

- Electronic Supplementary Information -  
for

***Brønsted acid-catalyzed annulation reaction of hydroxamic acids: synthesis of cyclopentane-fused isoxazolidines and their benzylic amide rearrangement***

Swati Lekha Mondal, Vinod Bhajammanavar, Isai Ramakrishna and  
Mahiuddin Baidya\*

Department of Chemistry, Indian Institute of Technology Madras,  
Chennai 600 036, Tamil Nadu, India  
E-mail: [mbaidya@iitm.ac.in](mailto:mbaidya@iitm.ac.in)

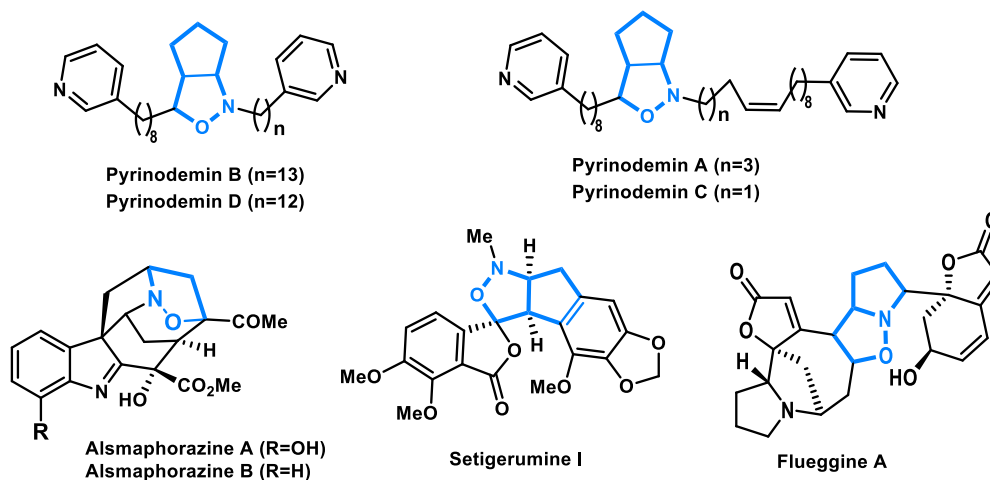
## *Contents*

	Page
1. General procedure .....	S3
2. General procedure for synthesis of cyclopentane fused isoxazolidines <b>3</b> .....	S4
3. Gram scale synthesis of compound <b>3a</b> .....	S13
4. Synthesis of compound <b>4a</b> .....	S13
5. Synthesis of compound <b>5a</b> .....	S14
6. General procedure for synthesis of compound <b>6</b> .....	S15
7. Crystallographic experimental data.....	S19
8. NMR spectra of synthesized compounds .....	S22

## General procedure:

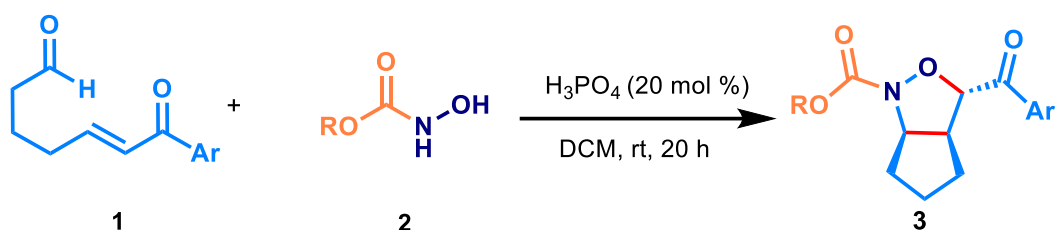
All non-aqueous reactions were carried out under an atmosphere of nitrogen in flame-dried glassware and were stirred using a magnetic stir plate. All reactions were carried out using commercial grade solvent unless otherwise noted. CH<sub>3</sub>CN, DCE, and CH<sub>2</sub>Cl<sub>2</sub> were dried over calcium hydride. Dry THF was prepared by distilling over sodium ketyl.

All reactions were monitored by thin layer chromatography (TLC) on WhatmanPartisil® K6F TLC plates (silica gel 60 Å, 0.25 mm thickness) and visualized using a UV lamp (366 or 254 nm) or by use of one of the following visualization reagents: PMA: 10 g phosphomolybdic acid/ 100 mL ethanol; KMnO<sub>4</sub>: 0.75 g potassium permanganate, 5 g K<sub>2</sub>CO<sub>3</sub>, / 100 mL water. Products were isolated by column chromatography (Merck silica gel 100-200µm). Yields refer to chromatographically and spectroscopically homogenous materials unless noted otherwise. <sup>13</sup>C and <sup>1</sup>H NMR spectra were recorded on a Bruker 400 or Bruker 500 MHz spectrometers. Chemical shift values (δ) are reported in ppm and calibrated to the residual solvent peak CDCl<sub>3</sub> δ = 7.2600 ppm for <sup>1</sup>H, δ = 77.16 for <sup>13</sup>C, DMSO-d<sub>6</sub> δ = 2.500 ppm for <sup>1</sup>H, δ = 39.500 ppm for <sup>13</sup>C; or calibrated to tetramethylsilane (δ = 0.00 ppm). All NMR spectra were recorded at ambient temperature (290 K) unless otherwise noted. <sup>1</sup>H NMR spectra are reported as follows: chemical shift (multiplicity, coupling constant, integration). The following abbreviations are used to indicate multiplicities: s, singlet; d, doublet; t, triplet; q, quartet; quint, quintet; sext, sextet; sept, septet; m, multiplet; dd, doublet of doublet; dt, doublet of triplet; dq, doublet of quartet; td, triplet of doublet; tt, triplet of triplet; dq, doublet of quartet; br, broad; app, apparent. Mass spectra were recorded by electrospray ionization (ESI) method on a Q-TOF Micro with lock spray source. The crystal data were collected and integrated using a BrukerAxs kappa apex2 CCD diffractometer, with graphite monochromated Mo-Kα radiation. The ω-formyl enone **1** was synthesized by following literature procedures (*Adv. Synth. Catal.* 2019, **361**, 208; *Org. Lett.* 2005, **7**, 18).

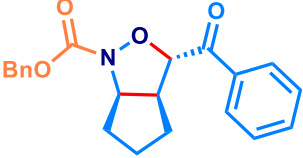
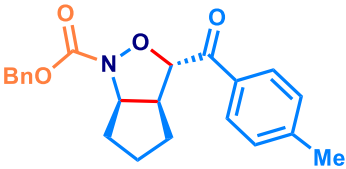
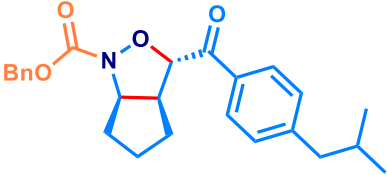
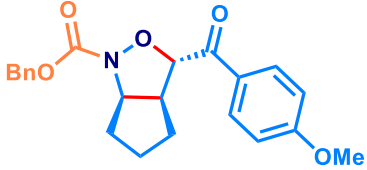


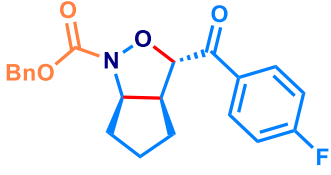
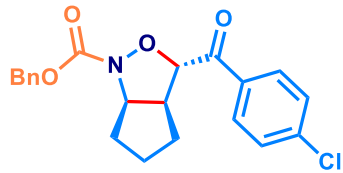
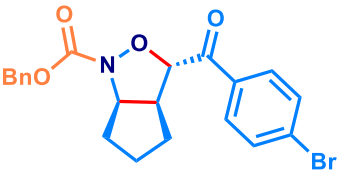
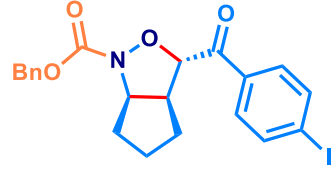
**Figure 1:** Examples of natural products with cyclopentane fused isoxazolidine framework.

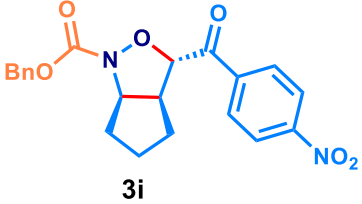
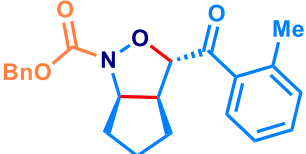
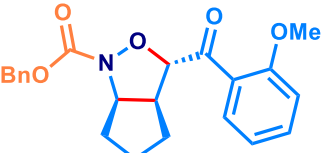
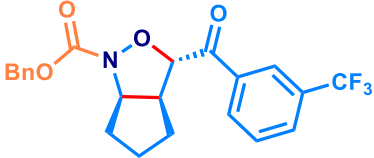
### General procedure for synthesis of cyclopentane fused isoxazolidines **3**

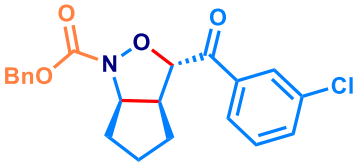
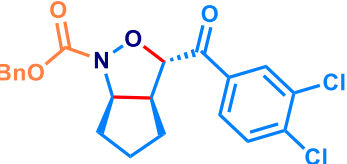
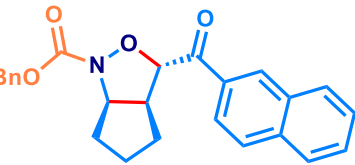
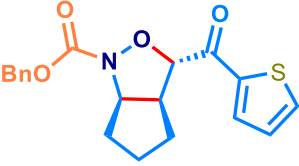


A 16x100 mm oven dried reaction tube equipped with a magnetic stir was charged with  $\omega$ -formyl enones **1** (0.20 mmol, 1.0 equiv), hydroxamic acids **2** (0.20 mmol, 1.0 equiv), and  $\text{H}_3\text{PO}_4$  (20 mol %). The reaction tube was capped with a rubber septum, evacuated, and backfilled with nitrogen gas. Then, anhydrous  $\text{CH}_2\text{Cl}_2$  (1.5 mL) was added via syringe. The reaction mixture was allowed to stir for 20 h at room temperature. After completion of the reaction (TLC monitored), volatiles were removed under reduced pressure and the crude product was purified by silica gel column chromatography to provide pure products **3**.

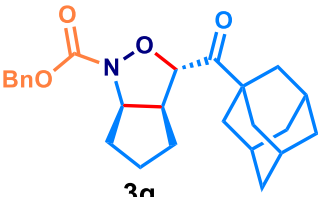
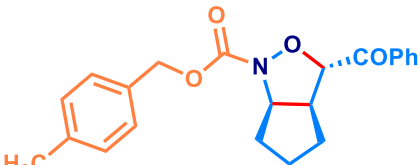
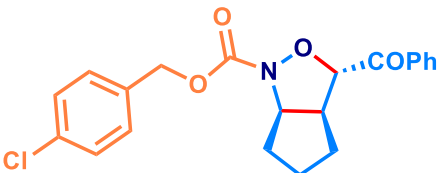
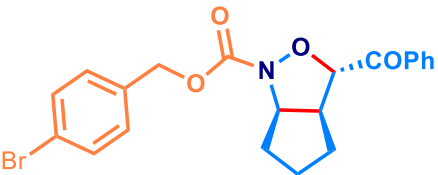
 <p style="text-align: center;"><b>3a</b></p>	<p>Compound <b>3a</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 86% (64 mg). <b><sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)</b> δ 7.97 – 7.94 (m, 2H), 7.57 – 7.54 (m, 1H), 7.44 – 7.41 (m, 2H), 7.23 – 7.19 (m, 3H), 7.10 – 7.09 (m, 2H), 5.11 (d, <i>J</i> = 1.6 Hz, 1H), 5.07 – 5.04 (m, 1H), 4.96 – 4.92 (m, 1H) 4.81 – 4.78 (m, 1H), 3.49 – 3.45 (m, 1H), 1.95 – 1.89 (m, 3H), 1.84 – 1.75 (m, 2H), 1.68 – 1.64 (m, 1H). <b><sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)</b> δ 195.2, 157.0, 135.6, 134.9, 133.6, 128.9, 128.7, 128.3, 128.0, 127.9, 86.3, 67.7, 65.1, 47.9, 34.1, 32.0, 25.6. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+Na]<sup>+</sup> Calculated for C<sub>21</sub>H<sub>21</sub>NO<sub>4</sub>Na<sup>+</sup> 374.1363; Found 374.1354.</p>
 <p style="text-align: center;"><b>3b</b></p>	<p>Compound <b>3b</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 83% (61 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 7.86 (d, <i>J</i> = 7.9 Hz, 2H), 7.25 – 7.19 (m, 5H), 7.11 – 7.09 (m, 2H), 5.09 – 4.92 (m, 3H), 4.81 – 4.77 (m, 1H), 3.50 – 3.45 (m, 1H), 2.40 (s, 3H), 1.98 – 1.87 (m, 3H), 1.84 – 1.75 (m, 2H), 1.69 – 1.62 (m, 1H). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 194.9, 157.1, 144.6, 135.8, 132.5, 129.5, 129.1, 128.3, 128.0, 127.9, 86.4, 67.8, 65.2, 48.0, 34.2, 32.1, 25.7, 21.8. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+Na]<sup>+</sup> Calculated for C<sub>22</sub>H<sub>23</sub>NO<sub>4</sub>Na<sup>+</sup> 388.1519; Found 388.1526.</p>
 <p style="text-align: center;"><b>3c</b></p>	<p>Compound <b>3c</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 80% (65 mg). <b><sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)</b> δ 7.88 (d, <i>J</i> = 8.3 Hz, 2H), 7.24 – 7.20 (m, 5H), 7.15 – 7.13 (m, 2H), 5.10 – 4.95 (m, 3H), 4.81 – 4.77 (m, 1H), 3.48 – 3.44 (m, 1H), 2.53 (d, <i>J</i> = 7.2 Hz, 2H), 1.96 – 1.87 (m, 4H), 1.85 – 1.76 (m, 2H), 1.68 – 1.64 (m, 1H, merged with water), 0.91 (d, <i>J</i> = 6.6 Hz, 6H). <b><sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)</b> δ 195.0, 157.1, 148.3, 135.9, 132.9, 129.5, 129.0, 128.4, 128.1, 128.0, 86.3, 67.8, 65.2, 48.2, 45.6, 34.2, 32.1, 30.2, 25.7, 22.5. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+H]<sup>+</sup> Calculated for C<sub>25</sub>H<sub>30</sub>NO<sub>4</sub><sup>+</sup> 408.2169; Found 408.2180.</p>
 <p style="text-align: center;"><b>3d</b></p>	<p>Compound <b>3d</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 79% (60 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 7.94 (d, <i>J</i> = 7.7 Hz, 2H), 7.23 – 7.22 (m, 3H), 7.11 (bs, 2H), 6.89 (d, <i>J</i> = 7.7 Hz, 2H), 5.07 – 4.93 (m, 3H), 4.80 – 4.76 (m, 1H), 3.86 (s, 3H), 3.51 – 3.46 (m, 1H), 1.99 – 1.89 (m, 3H), 1.84 – 1.75 (m, 2H), 1.70 – 1.67 (m, 1H, merged with water). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 193.8, 164.0, 157.1, 135.8, 131.4, 128.3, 128.05, 127.96, 125.8, 114.0, 86.3, 67.8, 65.2, 55.6, 48.0, 34.2, 32.1, 25.7. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+Na]<sup>+</sup> Calculated for C<sub>22</sub>H<sub>23</sub>NO<sub>5</sub>Na<sup>+</sup> 404.1468; Found 404.1473.</p>

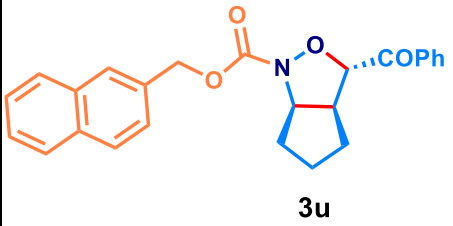
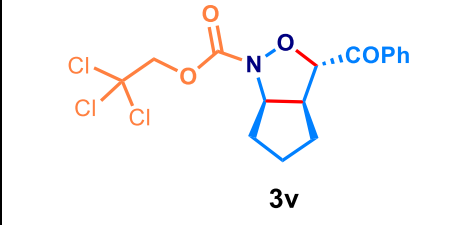
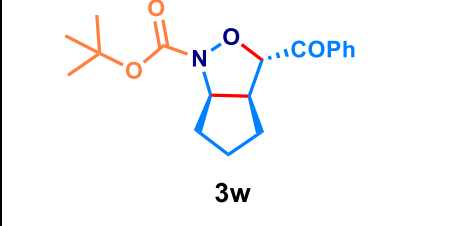
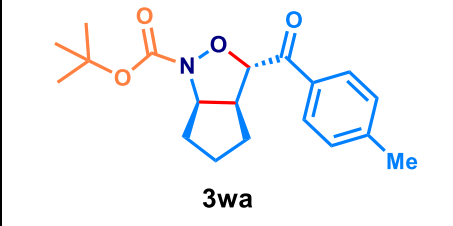
 <p style="text-align: center;"><b>3e</b></p>	<p>Compound <b>3e</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 85% (63 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 7.98 (dd, <i>J</i> = 8.5, 5.5 Hz, 2H), 7.26 – 7.23 (m, 3H), 7.09 – 7.04 (m, 4H), 5.03 – 4.91 (m, 3H), 4.81 – 4.77 (m, 1H), 3.56 – 3.51 (m, 1H), 2.00 – 1.90 (m, 3H), 1.83 – 1.74 (m, 2H), 1.69–1.65 (m, 1H). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 193.7, 166.0 (d, <sup>1</sup><i>J</i><sub>C-F</sub> = 255.8 Hz), 157.1, 135.5, 131.8 (d, <sup>3</sup><i>J</i><sub>C-F</sub> = 9.5 Hz) 131.5 (d, <sup>4</sup><i>J</i><sub>C-F</sub> = 3.1 Hz), 128.4, 128.1 (2×C), 115.8 (d, <sup>2</sup><i>J</i><sub>C-F</sub> = 21.9 Hz), 86.6, 67.9, 65.3, 47.5, 34.1, 32.0, 25.7. <b><sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)</b> δ -104.05. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+Na]<sup>+</sup> Calculated for C<sub>21</sub>H<sub>20</sub>FNO<sub>4</sub>Na<sup>+</sup> 392.1269; Found 392.1276.</p>
 <p style="text-align: center;"><b>3f</b></p>	<p>Compound <b>3f</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 72% (56 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 7.88 (d, <i>J</i> = 8.2 Hz, 2H), 7.35 (d, <i>J</i> = 8.1 Hz, 2H), 7.25 – 7.21 (m, 3H), 7.06 (d, <i>J</i> = 6.8 Hz, 2H), 5.02 – 4.89 (m, 3H), 4.79 – 4.77 (m, 1H), 3.56 – 3.51 (m, 1H), 1.97 – 1.86 (m, 3H), 1.84 – 1.74 (m, 2H), 1.70 – 1.64 (m, 1H, merged with water). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 194.1, 157.1, 140.2, 135.4, 133.4, 130.5, 129.1, 128.4, 128.2, 128.1, 86.7, 68.0, 65.4, 47.4, 34.2, 32.1, 25.8. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+Na]<sup>+</sup> Calculated for C<sub>21</sub>H<sub>20</sub>ClNO<sub>4</sub>Na<sup>+</sup> 408.0973; Found 408.0970.</p>
 <p style="text-align: center;"><b>3g</b></p>	<p>Compound <b>3g</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 76% (65 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 7.80 (d, <i>J</i> = 8.1 Hz, 2H), 7.52 (d, <i>J</i> = 8.1 Hz, 2H), 7.25 – 7.22 (m, 3H), 7.06 (d, <i>J</i> = 6.8 Hz, 2H), 5.01– 4.89 (m, 3H), 4.82 – 4.77 (m, 1H), 3.57 – 3.52 (m, 1H), 1.97 – 1.84 (m, 3H), 1.80–1.73 (m, 2H), 1.69–1.65 (m, 1H). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 194.3, 157.1, 135.4, 133.8, 132.1, 130.6, 129.0, 128.5, 128.19, 128.16, 86.7, 68.0, 65.4, 47.4, 34.2, 32.1, 25.8. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+Na]<sup>+</sup> Calculated for C<sub>21</sub>H<sub>20</sub>BrNO<sub>4</sub>Na<sup>+</sup> 452.0468; Found 452.0452.</p>
 <p style="text-align: center;"><b>3h</b></p>	<p>Compound <b>3h</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 71% (67 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 7.75 (d, <i>J</i> = 8.3 Hz, 2H), 7.64 (d, <i>J</i> = 8.3 Hz, 2H), 7.28 – 7.22 (m, 3H), 7.05 (d, <i>J</i> = 6.8 Hz, 2H), 5.00 – 4.88 (m, 3H), 4.81– 4.77 (m, 1H), 3.56 – 3.51 (m, 1H), 1.97– 1.88 (m, 3H), 1.84 – 1.75 (m, 2H), 1.73 – 1.67 (m, 1H). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 194.6, 157.1, 138.0, 135.4, 134.3, 130.4, 128.5, 128.2, 128.1, 102.0, 86.6, 68.0, 65.4, 47.3, 34.2, 32.0, 25.8. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+Na]<sup>+</sup> Calculated for C<sub>21</sub>H<sub>20</sub>I NO<sub>4</sub>Na<sup>+</sup> 500.0329; Found 500.0341.</p>

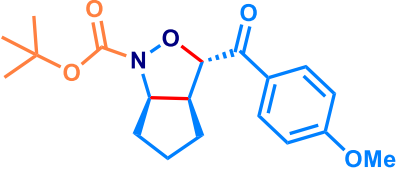
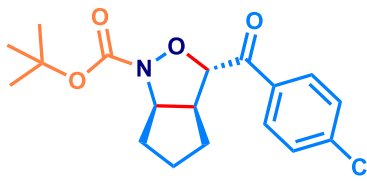
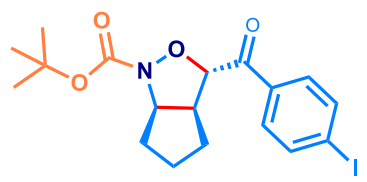
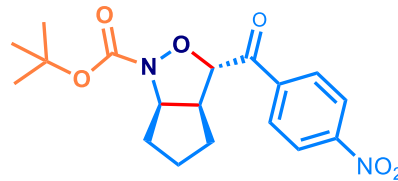
 <p style="text-align: center;"><b>3i</b></p>	<p>Compound <b>3i</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 63% (54 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 8.13 (d, <i>J</i> = 8.3 Hz, 2H), 8.06 (d, <i>J</i> = 8.5 Hz, 2H), 7.22 – 7.17 (m, 3H), 7.02 (d, <i>J</i> = 7.0 Hz, 2H), 5.02 (s, 1H), 4.94 – 4.79 (m, 3H), 3.66 – 3.61 (m, 1H), 2.00 – 1.90 (m, 3H), 1.81–1.76 (m, 2H), 1.71 – 1.66 (m, 1H). <b><sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)</b> δ 193.8, 157.1, 150.3, 139.5, 135.1, 130.1, 128.4, 128.3, 128.1, 123.7, 87.3, 68.1, 65.7, 46.8, 34.1, 32.0, 25.8. <b>HRMS (ESI/TOF-Q) m/z: [M+Na]<sup>+</sup></b> Calculated for C<sub>21</sub>H<sub>20</sub>N<sub>2</sub>O<sub>6</sub>Na<sup>+</sup> 419.1214; Found 419.1214.</p>
 <p style="text-align: center;"><b>3j</b></p>	<p>Compound <b>3j</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 80% (58 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 7.79 (d, <i>J</i> = 7.5 Hz, 1H), 7.40 (t, <i>J</i> = 7.3 Hz, 1H), 7.28 – 7.25 (m, 5H), 7.24 – 7.21 (m, 2H), 5.14 – 5.01 (m, 3H), 4.81 – 4.79 (m, 1H), 3.43– 4.39 (m, 1H), 2.45 (s, 3H), 1.94 – 1.92 (m, 3H), 1.86 – 1.76 (m, 2H), 1.71 – 1.67 (m, 1H). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 195.3, 157.0, 136.9, 135.1, 133.7, 133.6, 130.2, 129.1, 129.0, 128.7, 128.3, 125.9, 86.4, 66.1, 65.2, 48.0, 34.2, 32.1, 25.7, 18.8. <b>HRMS (ESI/TOF-Q) m/z: [M+Na]<sup>+</sup></b> Calculated for C<sub>22</sub>H<sub>23</sub>NO<sub>4</sub>Na<sup>+</sup> 388.1519; Found 388.1498.</p>
 <p style="text-align: center;"><b>3k</b></p>	<p>Compound <b>3k</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 62% (47 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 7.70 (d, <i>J</i> = 7.6 Hz, 1H), 7.50 – 7.46 (m, 1H), 7.37 – 7.28 (m, 5H), 7.01 – 6.94 (m, 2H), 5.25 – 5.13 (m, 3H), 4.65 – 4.61 (m, 1H), 3.91 (s, 3H), 3.28 – 3.23 (m, 1H), 2.01 – 1.92 (m, 2H), 1.84 – 1.78 (m, 3H), 1.70 – 1.64 (m, 1H, merged with water). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 198.4, 159.0, 136.4, 134.7 (2×C), 131.4, 128.5, 128.2, 128.0, 125.4, 121.2, 111.6, 89.5, 67.7, 63.6, 55.8, 49.8, 34.5, 32.7, 25.5. <b>HRMS (ESI/TOF-Q) m/z: [M+Na]<sup>+</sup></b> Calculated for C<sub>22</sub>H<sub>23</sub>NO<sub>5</sub>Na<sup>+</sup> 404.1468; Found 404.1484.</p>
 <p style="text-align: center;"><b>3l</b></p>	<p>Compound <b>3l</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 65% (55 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 8.21 (s, 1H), 8.13 (d, <i>J</i> = 8.0 Hz, 1H), 7.76 (d, <i>J</i> = 7.8 Hz, 1H), 7.51 (t, <i>J</i> = 7.8 Hz, 1H), 7.23 – 7.18 (m, 3H), 7.05 (d, <i>J</i> = 6.7 Hz, 2H), 5.05 (s, 1H), 5.00 (d, <i>J</i> = 12.2 Hz, 1H), 4.88 (d, <i>J</i> = 12.2 Hz, 1H), 4.84 – 4.79 (m, 1H), 3.60 – 3.55 (m, 1H), 1.98 – 1.89 (m, 3H), 1.84 – 1.76 (m, 2H), 1.70 – 1.66 (m, 1H). <b><sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)</b> δ 194.0, 157.1, 135.7, 135.4, 132.3, 131.3 (q, <sup>2</sup><i>J</i><sub>C-F</sub> = 33.0 Hz), 129.9 (q, <sup>3</sup><i>J</i><sub>C-F</sub> = 3.6 Hz), 129.4, 128.4, 128.2, 128.1, 125.9 (q, <sup>3</sup><i>J</i><sub>C-F</sub> = 3.7 Hz), 123.7 (q, <sup>1</sup><i>J</i><sub>C-F</sub> = 272 Hz) 86.8, 68.0, 65.5, 47.3, 34.2, 32.0, 25.8. <b><sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)</b> δ -62.8. <b>HRMS (ESI/TOF-Q) m/z: [M+H]<sup>+</sup></b> Calculated for C<sub>22</sub>H<sub>21</sub>F<sub>3</sub>NO<sub>4</sub><sup>+</sup> 420.1417; Found 420.1436.</p>

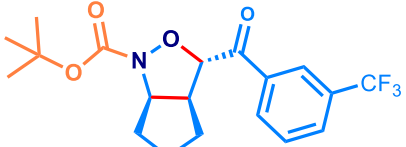
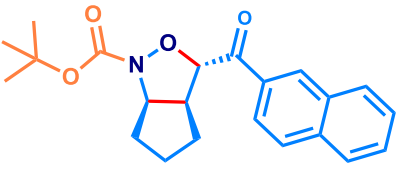
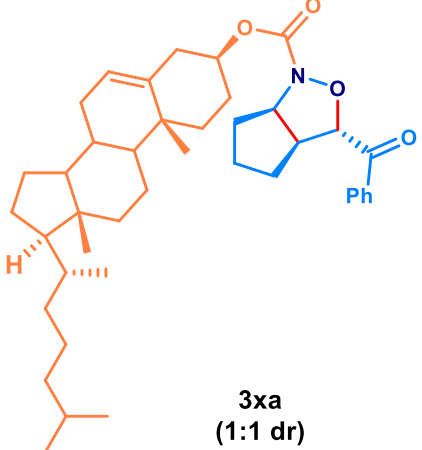
 <p style="text-align: center;"><b>3m</b></p>	<p>Compound <b>3m</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 78% (60 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 7.92 (s, 1H), 7.84 (d, <i>J</i> = 7.8 Hz, 1H), 7.50 (d, <i>J</i> = 8.0 Hz, 1H), 7.34 (t, <i>J</i> = 7.9 Hz, 1H), 7.28 – 7.22 (m, 3H), 7.11 – 7.09 (m, 2H), 5.04 – 4.92 (m, 3H), 4.84 – 4.79 (m, 1H), 3.55 – 3.50 (m, 1H), 1.98 – 1.89 (m, 3H), 1.84 – 1.75 (m, 2H), 1.70 – 1.65 (m, 1H). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 193.9, 157.1, 136.5, 135.4, 134.9, 133.5, 130.0, 128.9, 128.3, 128.1, 128.0, 127.1, 86.5, 67.9, 65.4, 47.4, 34.1, 32.0, 25.7. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+Na]<sup>+</sup> Calculated for C<sub>21</sub>H<sub>20</sub>ClNO<sub>4</sub>Na<sup>+</sup> 408.0973; Found 408.0981.</p>
 <p style="text-align: center;"><b>3n</b></p>	<p>Compound <b>3n</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 73% (61 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 7.99 (s, 1H), 7.75 (d, <i>J</i> = 8.3 Hz, 1H), 7.40 (d, <i>J</i> = 8.4 Hz, 1H), 7.26 – 7.20 (m, 3H), 7.05 (d, <i>J</i> = 7.1 Hz, 2H), 4.97 – 4.87 (m, 3H), 4.82 – 4.77 (m, 1H), 3.59 – 3.52 (m, 1H), 1.99 – 1.84 (m, 3H), 1.82 – 1.71 (m, 2H), 1.68 – 1.64 (m, 1H). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 193.0, 157.2, 138.2, 135.1, 134.6, 133.3, 131.0, 130.7, 128.4, 128.24, 128.16, 128.1, 86.9, 68.1, 65.6, 47.0, 34.2, 32.0, 25.8. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+Na]<sup>+</sup> Calculated for C<sub>21</sub>H<sub>19</sub>Cl<sub>2</sub>NO<sub>4</sub>Na<sup>+</sup> 442.0583; Found 442.0593.</p>
 <p style="text-align: center;"><b>3o</b></p>	<p>Compound <b>3o</b>: Sticky liquid; eluent (5% ethyl acetate in Hexane) <b>Yield</b>: 70% (56 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 8.54 (s, 1H), 7.98 – 7.93 (m, 2H), 7.87 – 7.83 (m, 2H), 7.64 – 7.53 (m, 2H), 7.13 – 6.93 (m, 5H), 5.27 (s, 1H), 5.02 – 4.87 (m, 3H), 3.60 – 3.55 (m, 1H), 2.00 – 1.94 (m, 3H), 1.87 – 1.81 (m, 2H), 1.71 – 1.67 (m, 1H). <b><sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)</b> δ 195.2, 157.2, 135.9, 135.5, 132.6, 132.4, 131.3, 130.0, 128.9, 128.7, 128.2, 127.95 (2×C), 127.91, 127.0, 124.3, 86.6, 67.9, 65.4, 47.9, 34.3, 32.1, 25.8. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+Na]<sup>+</sup> Calculated for C<sub>25</sub>H<sub>23</sub>NO<sub>4</sub>Na<sup>+</sup> 424.1519; Found 424.1535.</p>
 <p style="text-align: center;"><b>3p</b></p>	<p>Compound <b>3p</b>: Sticky liquid; eluent (5% Ethyl acetate in hexane) <b>Yield</b>: 74% (53 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 7.91 (d, <i>J</i> = 3.8 Hz, 1H), 7.65 (d, <i>J</i> = 5.0 Hz, 1H), 7.28 – 7.26 (m, 3H), 7.21 – 7.19 (m, 2H), 7.11 – 7.09 (m, 1H, merged with water), 5.13 – 5.02 (m, 2H), 4.89 (s, 1H), 4.76 – 4.71 (m, 1H), 3.52 – 3.47 (m, 1H), 1.96 – 1.85 (m, 3H), 1.84 – 1.75 (m, 2H), 1.70 – 1.63 (m, 1H). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 188.9, 156.7, 141.7, 137.4, 135.8, 135.0, 134.0, 128.6, 128.5, 128.1, 87.2, 67.9, 64.9, 48.6, 34.1, 32.0, 25.6. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+H]<sup>+</sup> Calculated for C<sub>19</sub>H<sub>20</sub>SNO<sub>4</sub><sup>+</sup> 358.1108; Found 358.1109.</p>

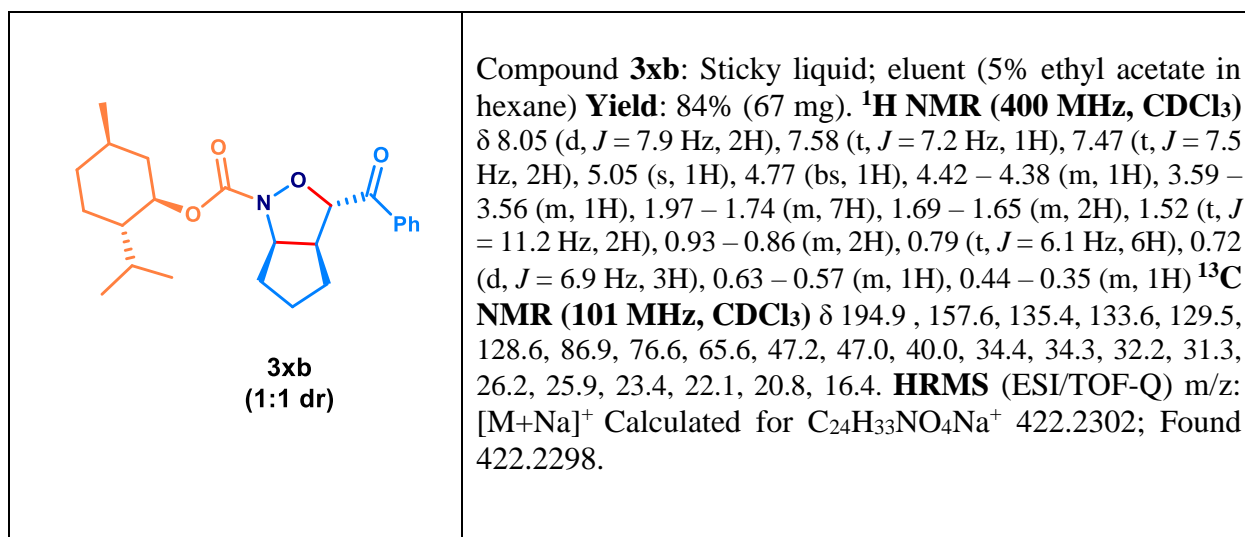


 <p style="text-align: center;"><b>3q</b></p>	<p>Compound <b>3q</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 77% (63 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 7.35 – 7.28 (m, 5H), 5.08 (d, <i>J</i> = 2.3 Hz, 2H), 4.79 – 4.76 (m, 1H), 4.62 (s, 1H), 3.05 – 3.00 (m, 1H), 1.95 (bs, 3H), 1.83 – 1.77 (m, 6H), 1.69 – 1.66 (m, 8H), 1.59 – 1.55 (m, 4H). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 209.7, 157.1, 135.9, 128.33, 128.27, 128.1, 82.7, 67.7, 65.3, 48.5, 45.8, 37.3, 36.3, 34.1, 31.9, 27.6, 25.6. <b>HRMS (ESI/TOF-Q) m/z</b>: [M+Na]<sup>+</sup> Calculated for C<sub>25</sub>H<sub>31</sub>NO<sub>4</sub>Na<sup>+</sup> 432.2145; Found 432.2154.</p>
 <p style="text-align: center;"><b>3r</b></p>	<p>Compound <b>3r</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 75% (55 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 7.96 (d, <i>J</i> = 7.9 Hz, 2H), 7.57 (t, <i>J</i> = 7.3 Hz, 1H), 7.44 (t, <i>J</i> = 7.4 Hz, 2H), 7.04 – 6.99 (m, 4H), 5.10 (s, 1H), 5.04 – 5.01 (m, 1H), 4.91– 4.88 (m, 1H), 4.80 – 4.76 (m, 1H), 3.50 – 3.45 (m, 1H), 2.31 (s, 3H), 1.99 – 1.86 (m, 3H), 1.83–1.76 (m, 2H), 1.68 – 1.62 (m, 1H, merged with water). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 195.3, 157.2, 137.8, 135.1, 133.6, 132.7, 129.1 (2×C), 128.8, 128.3, 86.4, 67.8, 65.2, 48.0, 34.2, 32.1, 25.7, 21.3. <b>HRMS (ESI/TOF-Q) m/z</b>: [M+Na]<sup>+</sup> Calculated for C<sub>12</sub>H<sub>23</sub>NO<sub>4</sub>Na<sup>+</sup> 388.1519; Found 388.1526.</p>
 <p style="text-align: center;"><b>3s</b></p>	<p>Compound <b>3s</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 72% (56 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 7.93 (d, <i>J</i> = 7.5 Hz, 2H), 7.58 (t, <i>J</i> = 7.4 Hz, 1H), 7.43 (t, <i>J</i> = 7.7 Hz, 2H), 7.16 (d, <i>J</i> = 8.3 Hz, 2H), 7.01 (d, <i>J</i> = 8.2 Hz, 2H), 5.12 (s, 1H), 4.95 (q, <i>J</i> = 12.4 Hz, 2H), 4.82 – 4.77 (m, 1H), 3.50 – 3.45 (m, 1H), 1.97 – 1.88 (m, 3H), 1.85 – 1.75 (m, 2H), 1.70 – 1.65 (m, 1H, merged with water). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 195.2, 157.0, 135.0, 134.2, 133.9, 133.7, 129.5, 129.0, 128.8, 128.6, 86.4, 67.0, 65.2, 48.0, 34.2, 32.1, 25.8. <b>HRMS (ESI/TOF-Q) m/z</b>: [M+Na]<sup>+</sup> Calculated for C<sub>21</sub>H<sub>20</sub>ClNO<sub>4</sub>Na<sup>+</sup> 408.0973; Found 408.0970.</p>
 <p style="text-align: center;"><b>3t</b></p>	<p>Compound <b>3t</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 70% (60 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 7.93 (d, <i>J</i> = 7.7 Hz, 2H), 7.59 – 7.56 (m, 1H), 7.45 – 7.41 (m, 2H), 7.32 (d, <i>J</i> = 8.2 Hz, 2H), 6.94 (d, <i>J</i> = 8.0 Hz, 2H), 5.12 (s, 1H), 4.97 – 4.89 (m, 2H), 4.80 – 4.77 (m, 1H), 3.50 – 3.44 (m, 1H), 1.97 – 1.89 (m, 3H), 1.85 – 1.75 (m, 2H), 1.70 – 1.65 (m, 1H, merged with water). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 195.2, 157.0, 135.0, 134.7, 133.7, 131.5, 129.8, 129.0, 128.8, 122.1, 86.4, 67.0, 65.2, 48.0, 34.2, 32.1, 25.8. <b>HRMS (ESI/TOF-Q) m/z</b>: [M+Na]<sup>+</sup> Calculated for C<sub>21</sub>H<sub>20</sub>BrNO<sub>4</sub>Na<sup>+</sup> 452.0468; Found 452.0450.</p>

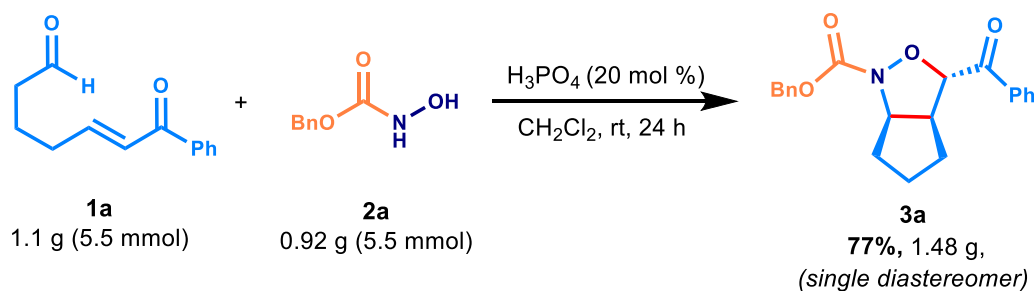
 <p style="text-align: center;"><b>3u</b></p>	<p>Compound <b>3u</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 72% (58 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 7.95 – 7.90 (m, 2H), 7.87 – 7.83 (m, 2H), 7.78 – 7.76 (m, 1H), 7.52 – 7.45 (m, 3H), 7.38 – 7.31 (m, 4H), 5.57 (d, <i>J</i> = 12.4 Hz, 1H), 5.37 (d, <i>J</i> = 12.4 Hz, 1H), 5.10 (s, 1H), 4.82 – 4.78 (m, 1H), 3.50 – 3.45 (m, 1H), 1.95 – 1.87 (m, 3H) 1.82 – 1.75 (m, 2H), 1.66 – 1.62 (m, 1H, merged with water). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 195.4, 157.1, 134.9, 133.6 (2×C), 131.6, 131.2, 129.1, 128.9, 128.7, 128.6, 127.2, 126.5, 125.9, 125.3, 123.7, 86.4, 66.0, 65.2, 48.0, 34.2, 32.1, 25.7. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+Na]<sup>+</sup> Calculated for C<sub>25</sub>H<sub>23</sub>NO<sub>4</sub>Na<sup>+</sup> 424.1519; Found 424.1519.</p>
 <p style="text-align: center;"><b>3v</b></p>	<p>Compound <b>3v</b>: Sticky liquid; eluent (5% ethyl acetate in Hexane) <b>Yield</b>: 76% (60mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 8.00 (d, <i>J</i> = 7.7 Hz, 2H), 7.58 (t, <i>J</i> = 7.3 Hz, 1H), 7.47 (t, <i>J</i> = 7.5 Hz, 2H), 5.18 (s, 1H), 4.80 – 4.76 (m, 1H), 4.71 (d, <i>J</i> = 11.8 Hz, 1H), 4.57 – 4.54 (m, 1H), 3.53 – 3.48 (m, 1H), 2.03 – 1.90 (m, 3H), 1.88 – 1.80 (m, 2H), 1.74 – 1.65 (m, 1H, merged with water). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 195.1, 154.4, 134.8, 133.9, 129.1, 128.9, 94.9, 86.3, 75.2, 65.1, 48.2, 34.1, 32.0, 25.7. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+Na]<sup>+</sup> Calculated for C<sub>16</sub>H<sub>16</sub>Cl<sub>3</sub>NO<sub>4</sub>Na<sup>+</sup> 414.0037; Found 414.0034.</p>
 <p style="text-align: center;"><b>3w</b></p>	<p>Compound <b>3w</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 73% (46 mg). <b><sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)</b> δ 8.07 – 8.05 (m, 2H), 7.59 – 7.56 (m, 1H), 7.49 – 7.46 (m, 2H), 5.04 (s, 1H), 4.81 – 4.77 (m, 1H), 3.57 – 3.53 (m, 1H), 1.96 – 1.88 (m, 2H), 1.85 – 1.71 (m, 3H), 1.67 – 1.59 (m, 1H), 1.13 (s, 9H). <b><sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)</b> δ 194.7, 156.8, 135.4, 133.6, 129.3, 128.7, 86.7, 81.8, 65.3, 46.8, 34.3, 32.1, 27.6, 25.9. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+Na]<sup>+</sup> Calculated for C<sub>18</sub>H<sub>23</sub>NO<sub>4</sub>Na<sup>+</sup> 340.1519; Found 340.1517.</p>
 <p style="text-align: center;"><b>3wa</b></p>	<p>Compound <b>3wa</b>: Sticky liquid; eluent (5% ethyl acetate in Hexane) <b>Yield</b>: 76% (50 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 7.96 – 7.95 (m, 2H), 7.28 – 7.26 (m, 2H), 5.03 (s, 1H), 4.79 – 4.77 (m, 1H), 3.53 – 3.51 (m, 1H), 2.41 (s, 3H), 2.00 – 1.90 (m, 2H), 1.83 – 1.70 (m, 3H), 1.63 – 1.15 (m, 1H), 1.15 (s, 9H). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 194.4, 156.8, 144.4, 132.9, 129.4, 129.3, 86.6, 81.7, 65.2, 46.9, 34.3, 32.1, 27.6, 25.9, 21.8. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+Na]<sup>+</sup> Calculated for C<sub>19</sub>H<sub>25</sub>NO<sub>4</sub>Na<sup>+</sup> 354.1676; Found 354.1688</p>

 <p style="text-align: center;"><b>3wb</b></p>	<p>Compound <b>3wb</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 75% (52 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 8.04 (d, <i>J</i> = 8.4 Hz, 2H), 6.95 (d, <i>J</i> = 8.4 Hz, 2H), 5.01 (s, 1H), 4.80 – 4.76 (m, 1H), 3.87 (s, 3H), 3.57 – 3.51 (m, 1H), 1.94 – 1.86 (m, 2H), 1.84 – 1.70 (m, 4H), 1.17 (s, 9H). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 193.3, 163.9, 156.9, 131.7, 128.6, 113.9, 86.6, 81.7, 65.3, 55.7, 46.9, 34.3, 32.1, 27.7, 25.9. <b>HRMS (ESI/TOF-Q)</b> m/z: [M+Na]<sup>+</sup> Calculated for C<sub>19</sub>H<sub>25</sub>NO<sub>5</sub>Na<sup>+</sup> 370.1625; Found 370.1632.</p>
 <p style="text-align: center;"><b>3wc</b></p>	<p>Compound <b>3wc</b>: Sticky liquid; eluent (5% Ethyl acetate/Hexane) <b>Yield</b>: 71% (50mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 8.02 (d, <i>J</i> = 8.6 Hz, 2H), 7.46 (d, <i>J</i> = 8.6 Hz, 2H), 4.97 (s, 1H), 4.80 – 4.75 (m, 1H), 3.59 – 3.54 (m, 1H), 1.96 – 1.78 (m, 4H), 1.71 – 1.60 (m, 2H), 1.16 (s, 9H). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 193.7, 156.7, 140.1, 133.9, 130.8, 129.0, 86.8, 82.0, 65.4, 46.7, 34.3, 32.1, 27.7, 25.9. <b>HRMS (ESI/TOF-Q)</b> m/z: [M+Na]<sup>+</sup> Calculated for C<sub>18</sub>H<sub>22</sub>ClNO<sub>4</sub>Na<sup>+</sup> 374.1130; Found 374.1132.</p>
 <p style="text-align: center;"><b>3wd</b></p>	<p>Compound <b>3wd</b>: Sticky liquid; eluent (5% Ethyl acetate/Hexane) <b>Yield</b>: 69% (61 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 7.85 (d, <i>J</i> = 8.4 Hz, 2H), 7.78 (d, <i>J</i> = 8.3 Hz, 2H), 4.96 (s, 1H), 4.79 – 4.75 (m, 1H), 3.58 – 3.53 (m, 1H), 1.97 – 1.89 (m, 2H), 1.84 – 1.69 (m, 3H), 1.52 – 1.47 (m, 1H), 1.16 (s, 9H). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 194.2, 156.7, 138.1, 134.8, 130.7, 101.7, 86.8, 82.0, 65.4, 46.7, 34.3, 32.1, 27.7, 25.9. <b>HRMS (ESI/TOF-Q)</b> m/z: [M+Na]<sup>+</sup> Calculated for C<sub>18</sub>H<sub>22</sub>INO<sub>4</sub>Na<sup>+</sup> 466.0486; Found 466.0496.</p>
 <p style="text-align: center;"><b>3we</b></p>	<p>Compound <b>3we</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 66% (51 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 8.33 (d, <i>J</i> = 8.0 Hz, 2H), 8.25 (d, <i>J</i> = 8.0 Hz, 2H), 5.00 (s, 1H), 4.80 – 4.76 (m, 1H), 3.64 – 3.59 (m, 1H), 2.01 – 1.91 (m, 2H), 1.86 – 1.72 (m, 3H), 1.68 – 1.64 (m, 1H), 1.16 (s, 9H). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 193.5, 156.6, 150.5, 140.1, 130.4, 123.9, 87.2, 82.3, 65.7, 46.5, 34.2, 32.1, 27.8, 25.9. <b>HRMS (ESI/TOF-Q)</b> m/z: [M+Na]<sup>+</sup> Calculated for C<sub>18</sub>H<sub>22</sub>N<sub>2</sub>O<sub>6</sub>Na<sup>+</sup> 385.1370; Found 385.1380.</p>

 <p style="text-align: center;"><b>3wf</b></p>	<p>Compound <b>3wf</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 62% (51 mg). <b><sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)</b> δ 8.34 (s, 1H), 8.27 (d, <i>J</i> = 7.9 Hz, 1H), 7.84 (d, <i>J</i> = 7.8 Hz, 1H), 7.64 (t, <i>J</i> = 7.8 Hz, 1H), 5.02 (s, 1H), 4.81– 4.78 (m, 1H), 3.63 – 3.59 (m, 1H), 1.99 – 1.91 (m, 2H), 1.85 – 1.78 (m, 2H), 1.77–1.72 (m, 1H), 1.66–1.62 (m, 1H), 1.12 (s, 9H). <b><sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)</b> δ 193.6, 156.7, 136.1, 132.6, 131.4 (q, <sup>2</sup><i>J</i><sub>C-F</sub> = 33.1 Hz), 129.9 (q, <sup>3</sup><i>J</i><sub>C-F</sub> = 3.6 Hz), 129.4, 126.3 (q, <sup>3</sup><i>J</i><sub>C-F</sub> = 3.7 Hz), 123.77 (q, <sup>1</sup><i>J</i><sub>C-F</sub> = 272.7 Hz), 87.0, 82.1, 65.5, 46.6, 34.3, 32.1, 27.6, 25.9. <b><sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)</b> δ -62.85. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+Na]<sup>+</sup> Calculated for C<sub>19</sub>H<sub>22</sub>F<sub>3</sub>NO<sub>4</sub>Na<sup>+</sup> 408.1393; Found 408.1404.</p>
 <p style="text-align: center;"><b>3wg</b></p>	<p>Compound <b>3wg</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 74% (57 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 8.65 (s, 1H), 8.08 (d, <i>J</i> = 8.6 Hz, 1H), 7.99 (d, <i>J</i> = 8.2 Hz, 1H), 7.89 (dd, <i>J</i> = 13.8, 8.4 Hz, 2H), 7.63 – 7.54 (m, 2H), 5.21 (s, 1H), 4.87 – 4.82 (m, 1H), 3.66 – 3.59 (m, 1H), 2.00 – 1.77 (m, 5H), 1.52 – 1.39 (m, 1H), 1.08 (s, 9H). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> δ 194.8, 156.9, 135.8, 132.7, 132.6, 131.5, 129.9, 128.9, 128.6, 127.9, 127.0, 124.5, 86.8, 81.8, 65.4, 47.0, 34.4, 32.2, 27.6, 26.0. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+Na]<sup>+</sup> Calculated for C<sub>22</sub>H<sub>25</sub>NO<sub>4</sub>Na<sup>+</sup> 390.1676; Found 390.1690.</p>
 <p style="text-align: center;"><b>3xa</b> (1:1 dr)</p>	<p>Compound <b>3xa</b>: Sticky liquid; eluent (5% ethyl acetate in hexane) <b>Yield</b>: 78% (94 mg). <b><sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)</b> δ 8.05 (d, <i>J</i> = 7.4 Hz, 2H), 7.58 (t, <i>J</i> = 7.0 Hz, 1H), 7.48 (t, <i>J</i> = 7.3 Hz, 2H), 5.26 (d, <i>J</i> = 18.6 Hz, 1H), 5.07 (s, 1H), 4.79 (s, 1H), 4.32 (s, 1H), 3.54 (s, 1H), 1.94 – 1.69 (m, 12H), 1.53 – 1.22 (m, 12H), 1.11 – 0.96 (m, 8H), 0.90 – 0.80 (m, 14H), 0.64 (s, 3H). <b><sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)</b> 195.1, 195.0 (C*), 157.1, 139.8, 139.7 (C*), 135.2, 133.7, 133.6 (C*), 129.3, 128.7, 122.55, 122.50 (C*), 86.90, 86.87 (C*), 76.2, 65.3, 56.7, 56.2, 50.0, 47.35, 47.30 (C*), 42.4, 39.8, 39.6 (C*), 37.9, 37.3, 36.9, 36.8 (C*), 36.6, 36.5 (C*), 36.3, 35.9, 34.3, 32.1, 32.0, 31.9 (C*), 28.3, 28.1, 27.6, 27.0, 25.9, 24.4, 23.9, 22.9, 22.7, 21.1, 19.3, 18.8, 11.9. [C* = signal from another diastereomer]; <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+Na]<sup>+</sup> Calculated for C<sub>41</sub>H<sub>59</sub>NO<sub>4</sub>Na<sup>+</sup> 652.4336; Found 652.4334.</p>

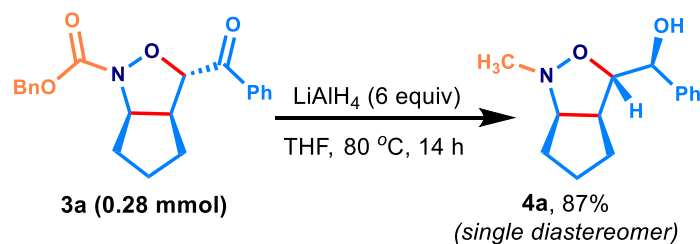


### Gram scale synthesis of compound 3a:



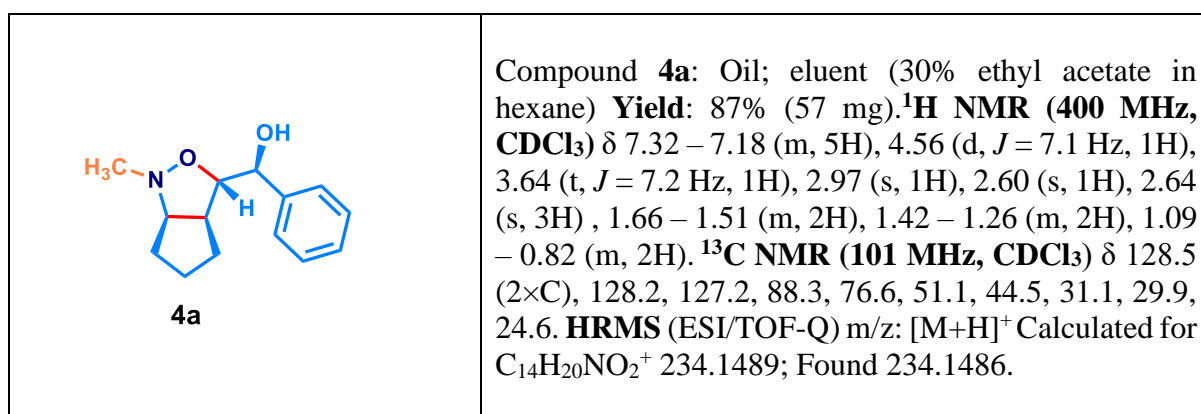
A 100 mL oven dried round bottom flask equipped with a magnetic stir was charged with formyl enone **1a** (1.1 g, 5.5 mmol, 1.0 equiv), benzylhydroxycarbamate **2a** (0.92 g, 5.5 mmol, 1.0 equiv), and H<sub>3</sub>PO<sub>4</sub> (0.57 mL, 20 mol %) under N<sub>2</sub> atmosphere. Then, anhydrous DCM (18 mL) was added via syringe. The reaction mixture was allowed to stir for 24 h at room temperature. After completion of the reaction (TLC monitored), volatiles were removed under reduced pressure and the crude product was purified by silica gel column chromatography to provide pure product **3a** (1.48 g, 77%).

### Synthesis of compound 4a:

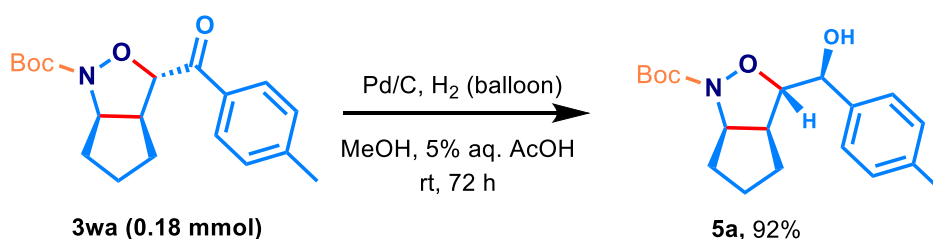


To an oven dried 16 mL sealed pressure tube equipped with a magnetic stir, was charged with the product **3a** (100 mg, 0.28 mmol, 1.0 equiv). The tube was capped with a rubber septum,

evacuated and backfilled with nitrogen gas. Then, 4 mL of anhydrous THF was added and the reaction temperature cooled down to 0 °C. Next, LiAlH<sub>4</sub> (60.5 mg, 1.68 mmol, 6 equiv) was added under N<sub>2</sub>. While addition, effervescence evolved and waited till it ceases at 0 °C and next the reaction mixture allowed to stir for 10 minutes at rt and the rubber septum was replaced with a cap under inert atmosphere. The reaction mixture was then heated to 80 °C for 14 h. After completion of the reaction, the reaction mixture was cooled down to rt and slowly quenched with ice-cold water and 15% NaOH (2 mL) was added. The resulting suspension was brought to room temperature and then filtered. The filtrate was washed with DCM (3 X 6 mL). Then organic layer was washed with Brine (6 mL) and dried under Na<sub>2</sub>SO<sub>4</sub>. The volatiles were evaporated under vacuum. Crude material was purified via column chromatography using 30% ethyl acetate in hexane to get pure **4a** as oil (57 mg, 87 % yield).

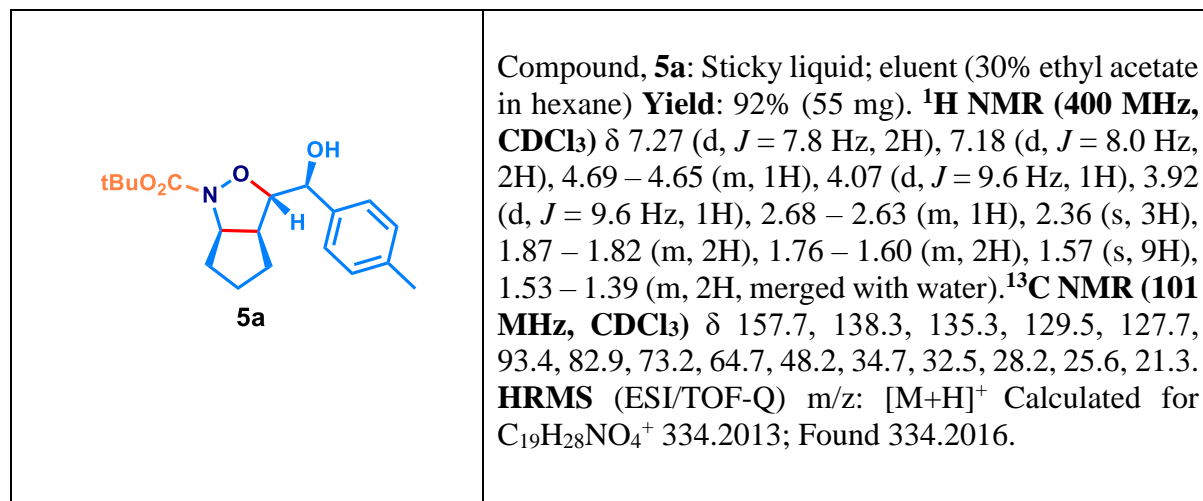


### Synthesis of compound 5a:

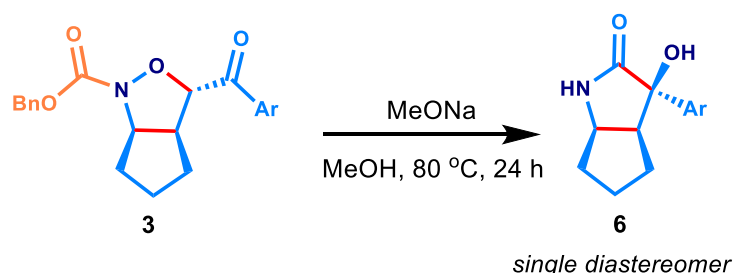


To an oven dried 25 mL round bottom flask, the product **3wa** (60 mg, 0.18 mmol, 1.0 equiv) was dissolved in 2 mL MeOH. Next, 15 mg 5% Pd/C in 1 mL of 5% aqueous AcOH was added. The round bottom flask was exposed to hydrogen gas by a hydrogen bladder and stirred at room temperature for 72 h. After completion, reaction mixture was filtered through a short celite pad and the solid was washed with dichloromethane. The filtrate was diluted with 2.5 mL of water, the aqueous layer was then extracted with dichloromethane (3 X 3 mL) and the

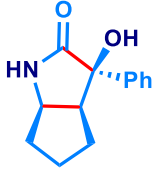
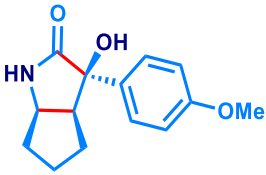
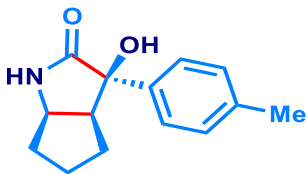
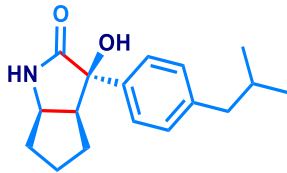
organic layer was dried over Na<sub>2</sub>SO<sub>4</sub>. After concentration under reduced pressure, the crude material was purified via column chromatography using 5% ethyl acetate in hexane to get **5a** as oil (55 mg, 92 % yield).



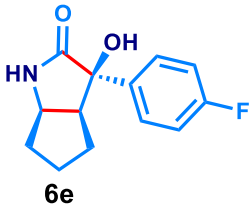
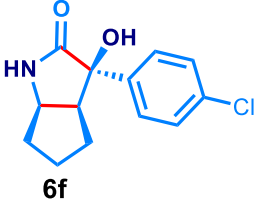
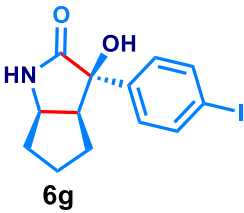
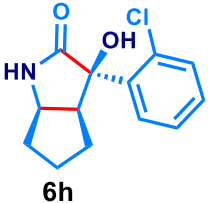
#### General procedure for synthesis of compound **6**:

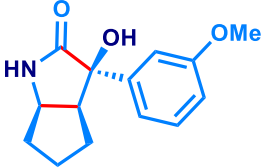
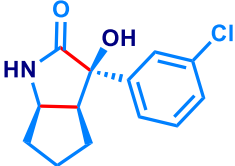
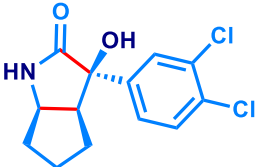


A 16 mL oven dried sealed pressure tube equipped with a magnetic stir was charged with product **3** (0.2 mmol, 1.0 equiv). The reaction tube was capped with a rubber septum, evacuated, and backfilled with nitrogen gas. Then, 5 mL of freshly prepared sodium methoxide (25% solution) in anhydrous methanol was added and the rubber septum was replaced with a reaction tube cap under inert atmosphere. Then the mixture was heated at 80 °C for 24 h. Next, the reaction mixture was cooled down to room temperature and slowly neutralized with aqueous HCl. The resulting suspension was washed with EtOAc (3 X 6 mL). Organic layer was washed with brine (6 mL) and dried under Na<sub>2</sub>SO<sub>4</sub>. The volatiles were evaporated in vacuum. The crude material was purified via silica gel column chromatography using (35-45% ethyl acetate: hexane) to get pure compound **6** as white solid.

 <p style="text-align: center;"><b>6a</b></p>	<p>Compound, <b>6a</b>: White solid; eluent (40% ethyl acetate in Hexane) <b>Yield</b>: 94% (57 mg). <b><sup>1</sup>H NMR (400 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 7.89 (s, 1H), 7.42 – 7.30 (m, 4H), 7.23 (t, <i>J</i> = 7.1 Hz, 1H), 5.74 (s, 1H), 3.96 (s, 1H), 2.72 – 2.69 (m, 1H), 2.10 – 2.06 (m, 1H), 1.65 – 1.44 (m, 5H). <b><sup>13</sup>C NMR (101 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 176.3, 146.1, 128.0, 126.9, 124.8, 78.2, 55.6, 51.6, 33.8, 26.0, 24.0. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+Na]<sup>+</sup> Calculated for C<sub>13</sub>H<sub>15</sub>NO<sub>2</sub>Na<sup>+</sup> 240.0995; Found 240.0998.</p>
 <p style="text-align: center;"><b>6b</b></p>	<p>Compound, <b>6b</b>: White solid; eluent (40% ethyl acetate in Hexane) <b>Yield</b>: 88% (41 mg). <b><sup>1</sup>H NMR (400 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 7.86 (s, 1H), 7.29 (d, <i>J</i> = 7.5 Hz, 2H), 7.12 (d, <i>J</i> = 7.6 Hz, 2H), 5.68 (s, 1H), 3.93 (s, 1H), 2.69 – 2.67 (m, 1H), 2.27 (s, 3H), 2.09 – 2.05 (m, 1H), 1.64 – 1.45 (m, 5H). <b><sup>13</sup>C NMR (101 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 176.4, 143.1, 135.8, 128.5, 124.8, 78.0, 55.5, 51.5, 33.8, 25.9, 23.9, 20.6. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+Na]<sup>+</sup> Calculated for C<sub>14</sub>H<sub>17</sub>NO<sub>2</sub>Na<sup>+</sup> 254.1151; Found 254.1171.</p>
 <p style="text-align: center;"><b>6c</b></p>	<p>Compound, <b>6c</b>: White solid; eluent (40% ethyl acetate in Hexane) <b>Yield</b>: 93% (51 mg). <b><sup>1</sup>H NMR (400 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 7.88 (s, 1H), 7.32 (d, <i>J</i> = 7.8 Hz, 2H), 7.10 (d, <i>J</i> = 7.7 Hz, 2H), 5.69 (s, 1H), 3.96 – 3.93 (m, 1H), 2.71 – 2.67 (m, 1H), 2.42 (d, <i>J</i> = 7.0 Hz, 2H), 2.10 – 2.06 (m, 1H), 1.85 – 1.75 (m, 1H), 1.65 – 1.46 (m, 5H), 0.85 (d, <i>J</i> = 6.5 Hz, 6H). <b><sup>13</sup>C NMR (101 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 176.4, 143.4, 139.6, 128.5, 124.6, 78.0, 55.5, 51.5, 44.2, 33.8, 29.7, 25.9, 23.9, 22.2 (2×C). <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+H]<sup>+</sup> Calculated for C<sub>17</sub>H<sub>24</sub>NO<sub>2</sub><sup>+</sup> 274.1802; Found 274.1800.</p>
 <p style="text-align: center;"><b>6d</b></p>	<p>Compound, <b>6d</b>: White solid; eluent (40% ethyl acetate in Hexane) <b>Yield</b>: 87% (43 mg). <b><sup>1</sup>H NMR (400 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 7.83 (s, 1H), 7.33 (d, <i>J</i> = 7.2 Hz, 2H), 6.88 (d, <i>J</i> = 7.5 Hz, 2H), 5.66 (s, 1H), 3.91 (bs, 1H), 3.73 (s, 3H), 2.71 – 2.68 (m, 1H), 2.07 – 2.04 (m, 1H), 1.64 – 1.46 (m, 5H). <b><sup>13</sup>C NMR (101 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 176.4, 158.2, 138.0, 126.1, 113.3, 77.8, 55.4, 55.1, 51.4, 33.7, 25.9, 23.9. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+H]<sup>+</sup> Calculated for C<sub>14</sub>H<sub>18</sub>NO<sub>3</sub><sup>+</sup> 248.1281; Found 248.1280.</p>

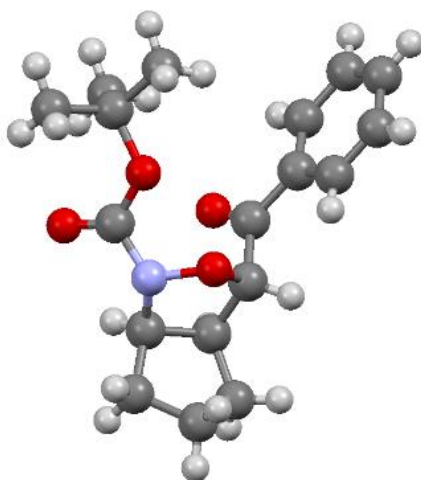


 <p style="text-align: center;"><b>6e</b></p>	<p>Compound, <b>6e</b>: White solid; eluent (40% ethyl acetate in Hexane) <b>Yield</b>: 95% (43 mg). <b><sup>1</sup>H NMR (400 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 7.93 (s, 1H), 7.46 – 7.42 (m, 2H), 7.14 (t, <i>J</i> = 8.7 Hz, 2H), 5.87 (s, 1H), 3.96 (s, 1H), 2.71 – 2.68 (m, 1H), 2.09 – 2.05 (m, 1H), 1.64 – 1.46 (m, 5H). <b><sup>13</sup>C NMR (101 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 176.1, 161.2 (d, <sup>1</sup><i>J</i><sub>C-F</sub> = 242.7 Hz), 142.2 (d, <sup>4</sup><i>J</i><sub>C-F</sub> = 2.8 Hz), 126.9 (d, <sup>3</sup><i>J</i><sub>C-F</sub> = 8.1 Hz), 114.7 (d, <sup>2</sup><i>J</i><sub>C-F</sub> = 21.1 Hz), 77.8, 55.5, 51.4, 33.8, 25.9, 23.9. <b><sup>19</sup>F NMR (471 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ -115.8. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+H]<sup>+</sup> Calculated for C<sub>13</sub>H<sub>15</sub>FNO<sub>2</sub><sup>+</sup> 236.1081; Found 236.1080.</p>
 <p style="text-align: center;"><b>6f</b></p>	<p>Compound, <b>6f</b>: White solid; eluent (40% ethyl acetate in Hexane) <b>Yield</b>: 89% (45 mg). <b><sup>1</sup>H NMR (400 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 7.96 (s, 1H), 7.40 (q, <i>J</i> = 8.2 Hz, 4H), 5.93 (s, 1H), 3.97 (bs, 1H), 2.70 – 2.67 (m, 1H), 2.09 – 2.05 (m, 1H), 1.66 – 1.46 (m, 5H). <b><sup>13</sup>C NMR (101 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 176.3, 145.5, 131.9, 128.4, 127.3, 78.3, 55.9, 51.7, 34.2, 26.4, 24.3. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+H]<sup>+</sup> Calculated for C<sub>13</sub>H<sub>15</sub>ClNO<sub>2</sub><sup>+</sup> 252.0786; Found 252.0785.</p>
 <p style="text-align: center;"><b>6g</b></p>	<p>Compound, <b>6g</b>: White solid; eluent (40% ethyl acetate in Hexane) <b>Yield</b>: 87% (57 mg). <b><sup>1</sup>H NMR (400 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 7.96 (s, 1H), 7.68 (d, <i>J</i> = 8.1 Hz, 2H), 7.22 (d, <i>J</i> = 8.1 Hz, 2H), 5.90 (s, 1H), 3.95 (bs, 1H), 2.68 – 2.63 (m, 1H), 2.08 – 2.04 (m, 1H), 1.63 – 1.43 (m, 5H). <b><sup>13</sup>C NMR (101 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 175.8, 146.0, 136.7, 127.3, 92.8, 77.9, 55.5, 51.2, 33.8, 25.9, 23.9. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+H]<sup>+</sup> Calculated for C<sub>13</sub>H<sub>15</sub>INO<sub>2</sub><sup>+</sup> 344.0142; Found 344.0141.</p>
 <p style="text-align: center;"><b>6h</b></p>	<p>Compound, <b>6h</b>: White solid; eluent (40% ethyl acetate in Hexane) <b>Yield</b>: 91% (47 mg). <b><sup>1</sup>H NMR (400 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 7.89 (s, 1H), 7.42 (s, 1H), 7.34 – 7.30 (m, 2H), 7.25 – 7.21 (m, 1H), 5.75 (s, 1H), 3.97 – 3.95 (m, 1H), 2.72 – 2.67 (m, 1H), 2.11 – 2.05 (m, 1H), 1.66 – 1.44 (m, 5H). <b><sup>13</sup>C NMR (101 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 176.4, 146.1, 128.1(2C), 127.0, 124.9(2C), 78.3, 55.7, 51.6, 33.9, 26.0, 24.0. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+H]<sup>+</sup> Calculated for C<sub>13</sub>H<sub>15</sub>ClNO<sub>2</sub><sup>+</sup> 252.0786; Found 252.0784.</p>

 <p style="text-align: center;"><b>6i</b></p>	<p>Compound, <b>6i</b>: White solid; eluent (40% ethyl acetate in Hexane) <b>Yield</b>: 89% (44 mg). <b><sup>1</sup>H NMR (400 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 7.91 (s, 1H), 7.24 (t, <i>J</i> = 7.9 Hz, 1H), 6.99 (s, 1H), 6.94 (d, <i>J</i> = 7.7 Hz, 1H), 6.81 (d, <i>J</i> = 8.1 Hz, 1H), 5.76 (s, 1H), 3.96 (s, 1H), 3.74 (s, 3H), 2.70 – 2.68 (m, 1H), 2.09 – 2.05 (m, 1H), 1.64 – 1.45 (m, 5H). <b><sup>13</sup>C NMR (101 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 176.2, 159.1, 147.8, 129.1, 116.9, 112.0, 110.8, 78.1, 55.6, 55.0, 51.5, 33.8, 26.0, 23.9. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+Na]<sup>+</sup> Calculated for C<sub>14</sub>H<sub>18</sub>NO<sub>3</sub><sup>+</sup> 248.1281; Found 248.1280.</p>
 <p style="text-align: center;"><b>6j</b></p>	<p>Compound, <b>6j</b>: White solid; eluent (40% ethyl acetate in Hexane) <b>Yield</b>: 87% (44 mg). <b><sup>1</sup>H NMR (400 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 7.98 (s, 1H), 7.45 (s, 1H), 7.39 – 7.29 (m, 3H), 5.98 (s, 1H), 4.01 – 3.98 (m, 1H), 2.73 – 2.68 (m, 1H), 2.10 – 2.04 (m, 1H), 1.66 – 1.62 (m, 2H), 1.57 – 1.48 (m, 3H). <b><sup>13</sup>C NMR (101 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 175.6, 148.6, 132.9, 130.0, 126.8, 124.9, 123.4, 77.9, 55.6, 51.1, 33.8, 25.9, 23.9. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+H]<sup>+</sup> Calculated for C<sub>13</sub>H<sub>15</sub>ClNO<sub>2</sub><sup>+</sup> 252.0786; Found 252.0782.</p>
 <p style="text-align: center;"><b>6k</b></p>	<p>Compound, <b>6k</b>: White solid; eluent (40% ethyl acetate in Hexane) <b>Yield</b>: 94% (54 mg). <b><sup>1</sup>H NMR (400 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 8.03 (s, 1H), 7.60 – 7.58 (m, 2H), 7.35 (d, <i>J</i> = 8.4 Hz, 1H), 6.11 (s, 1H), 4.00 (bs, 1H), 2.73 – 2.71 (m, 1H), 2.07 – 2.05 (m, 1H), 1.64 – 1.63 (m, 2H), 1.54 – 1.46 (m, 3H). <b><sup>13</sup>C NMR (101 MHz, DMSO-<i>d</i><sub>6</sub>)</b> δ 175.3, 147.2, 130.8, 130.4, 129.5, 127.1, 125.3, 77.7, 55.6, 50.9, 33.8, 26.0, 23.9. <b>HRMS (ESI/TOF-Q)</b> <i>m/z</i>: [M+H]<sup>+</sup> Calculated for C<sub>13</sub>H<sub>14</sub>Cl<sub>2</sub>NO<sub>2</sub><sup>+</sup> 286.0396; Found 286.0395.</p>

## Crystallographic experimental data

### Crystal Data for **3w**:



**Crystallization:** Crystals of compound **3w** were obtained through slow evaporation technique at room temperature from the solution in CDCl<sub>3</sub>. Crystal structure of compound **3w** has the **CCDC number: 2243707** (Ellipsoid Probability 50%).

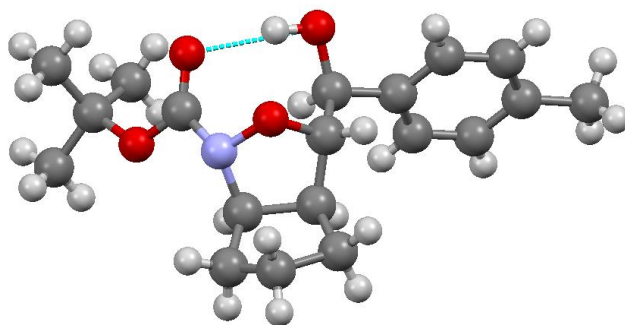
### Crystal data and structure refinement for **3w**.

Identification code	3w
Empirical formula	C <sub>13</sub> H <sub>27</sub> N O <sub>4</sub>
Formula weight	317.388
Temperature	296.15 K
Wavelength	0.71073 Å
Crystal system	Orthorhombic
Space group	Pna2 <sub>1</sub>
Unit cell dimensions	a = 12.2934 (5) Å    α = 90°. b = 14.3475 (5) Å    β = 90°. c = 9.7444(4) Å    γ = 90°.
Volume	1718.71(12) Å <sup>3</sup>

Z	4
Density (calculated)	1.227 g/m <sup>3</sup>
Absorption coefficient	0.086 mm <sup>-1</sup>
F(000)	680.5
Crystal size	0.25 x 0.22 x 0.1 mm <sup>3</sup>
Theta range for data collection/°	4.36 to 49.98.
Index ranges	-14 ≤ h ≤ 14, -17 ≤ k ≤ 15, -11 ≤ l ≤ 11
Reflections collected	11670
Independent reflections	3028 [R <sub>int</sub> = 0.0231, R <sub>sigma</sub> = 0.0230]
Data / restraints / parameters	3028 / 1 / 94
Goodness-of-fit on F <sup>2</sup>	1.697
Final R indices [I > 2σ(I)]	R1 = 0.1131, wR2 = 0.3572
R indices (all data)	R1 = 0.1239, wR2 = 0.3789
Extinction coefficient	n/a
Largest diff. peak and hole	0.62/-0.42 e.Å <sup>-3</sup>

### Crystal Data for 5a:

Crystals of compound **5a** were obtained through slow evaporation technique at room temperature from the solution in CDCl<sub>3</sub>. Crystal structure of compound **5a** has the **CCDC number: 2245191** (Ellipsoid Probability 50%).

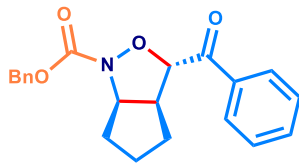


Crystal data and structure refinement for **5a**

Identification code	5a
Empirical formula	C <sub>19</sub> H <sub>27</sub> N O <sub>4</sub>
Formula weight	333.41
Temperature	298(2) K
Wavelength	1.54178 Å
Crystal system, space group	Monoclinic, C 2/c
Unit cell dimensions	a = 26.239(5) Å      α = 90 deg. b = 14.599(3) Å      β = 112.952(8) deg. c = 10.4681(18) Å    γ = 90 deg.
Volume	3692.3(11) Å <sup>3</sup>
Z	8
Calculated density	1.200 Mg/m <sup>3</sup>
Absorption coefficient	0.675 mm <sup>-1</sup>
F (000)	1440
Crystal size	0.206 x 0.198 x 0.112 mm
Theta range for data collection	3.537 to 68.245 deg.
Limiting indices	-31 ≤ h ≤ 31, -17 ≤ k ≤ 17, -12 ≤ l ≤ 12
Reflections collected / unique	50618 / 3387 [R(int) = 0.0495]
Completeness to theta = 67.679	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7531 and 0.6279
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	3387 / 52 / 235
Goodness-of-fit on F <sup>2</sup>	1.078

Final R indices [I>2sigma(I)]	R1 = 0.0453, wR2 = 0.1286
R indices (all data)	R1 = 0.0557, wR2 = 0.1419
Extinction coefficient	0.00084(12)
Largest diff. peak and hole	0.243 and -0.241 e.A <sup>-3</sup>

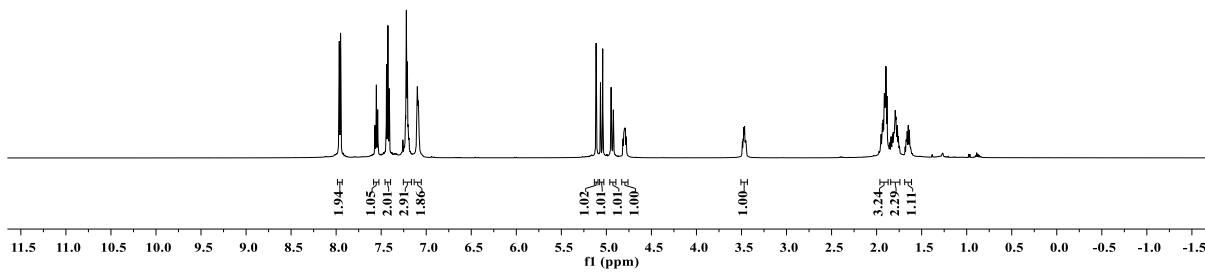
## **NMR SPECTRA OF SYNTHESISED COMPOUNDS**



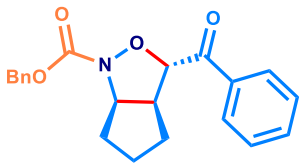
**3a**

500 MHz, CDCl<sub>3</sub>

7.97, 7.95, 7.94, 7.57, 7.55, 7.54, 7.44, 7.43, 7.41, 7.23, 7.22, 7.21, 7.19, 7.10, 7.09, 5.12, 5.11, 5.07, 5.04, 4.96, 4.92, 4.81, 4.80, 4.79, 4.78, 3.49, 3.48, 3.47, 3.46, 3.45, 1.95, 1.94, 1.93, 1.91, 1.90, 1.89, 1.84, 1.82, 1.81, 1.79, 1.78, 1.77, 1.75, 1.68, 1.67, 1.66, 1.65, 1.64

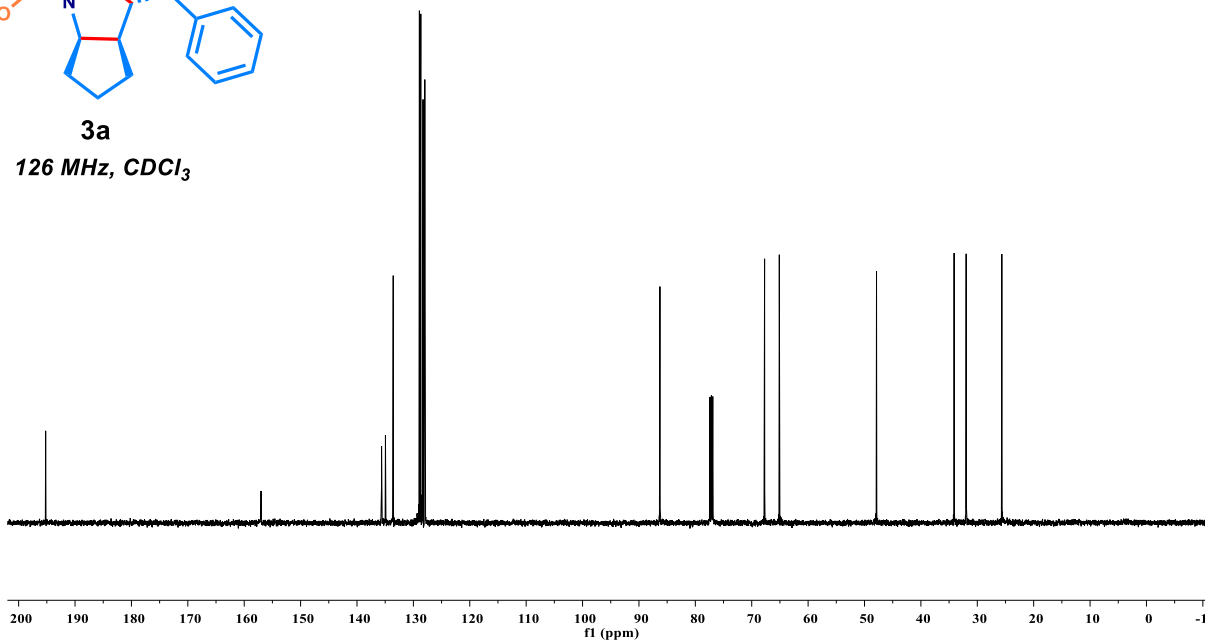


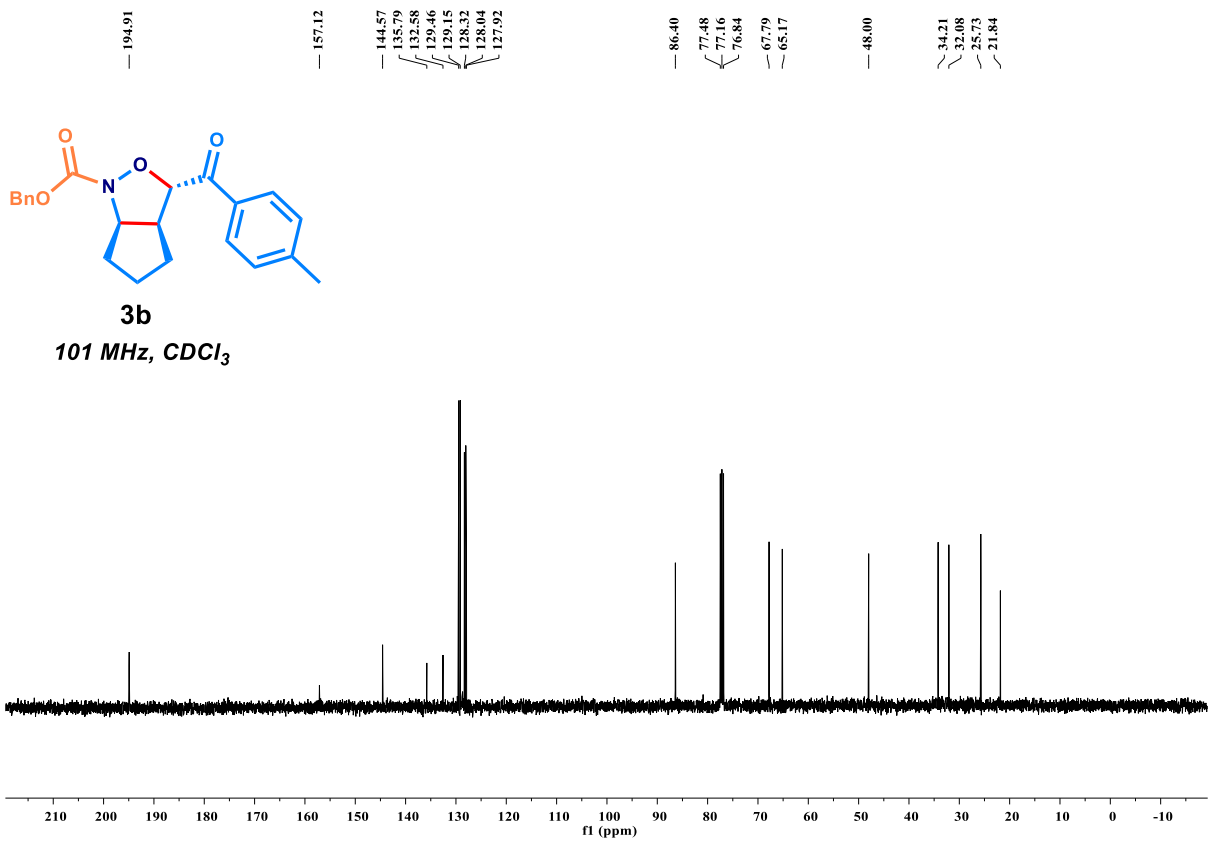
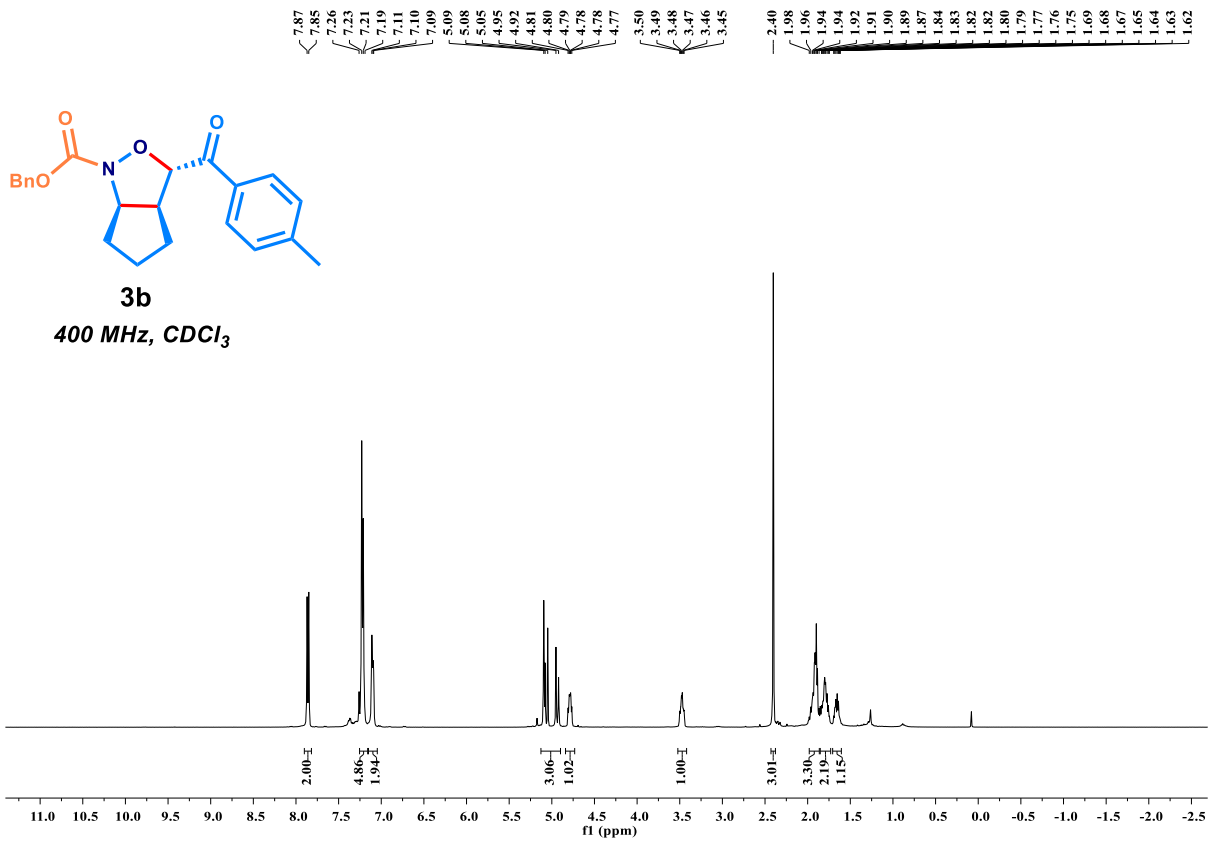
195.21, 157.02, 135.64, 134.95, 133.58, 128.93, 128.68, 128.29, 127.97, 127.92, 86.28, 77.41, 77.16, 76.90, 67.72, 65.11, 47.87, 34.11, 31.97, 25.65



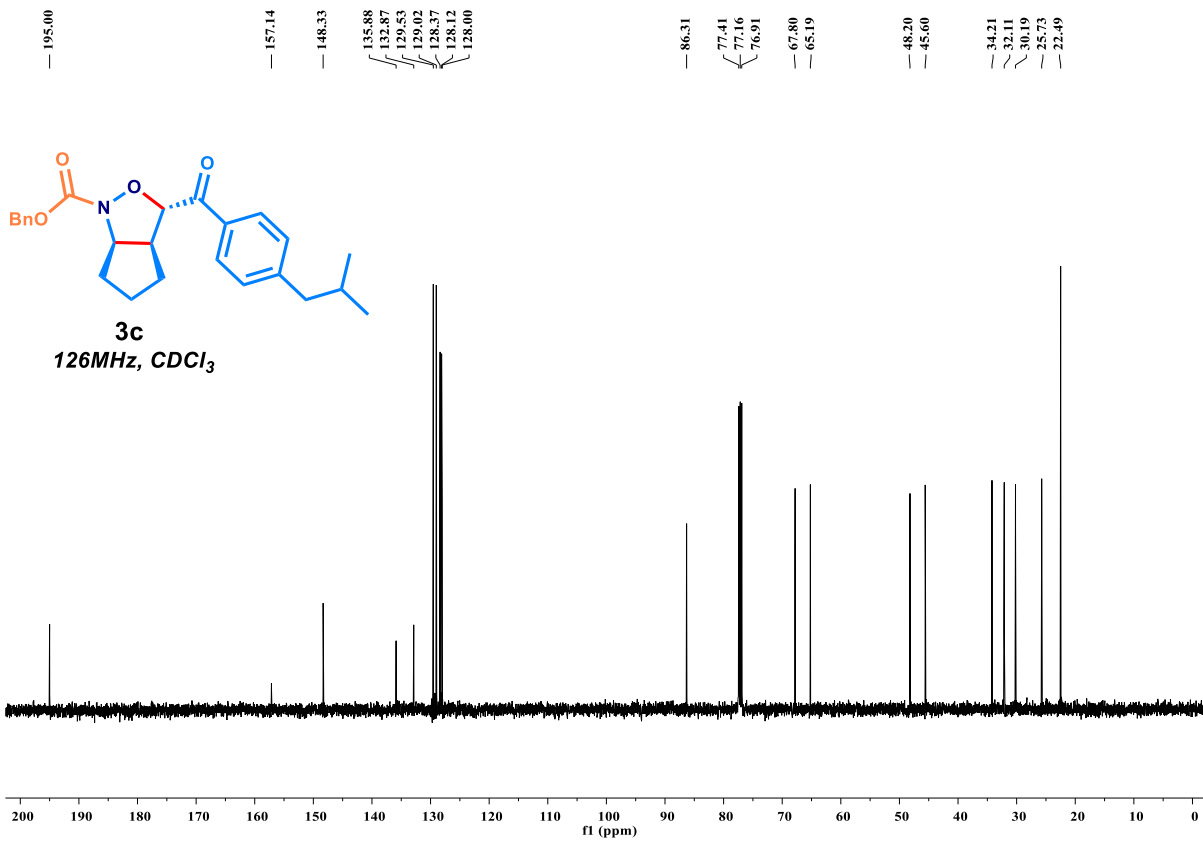
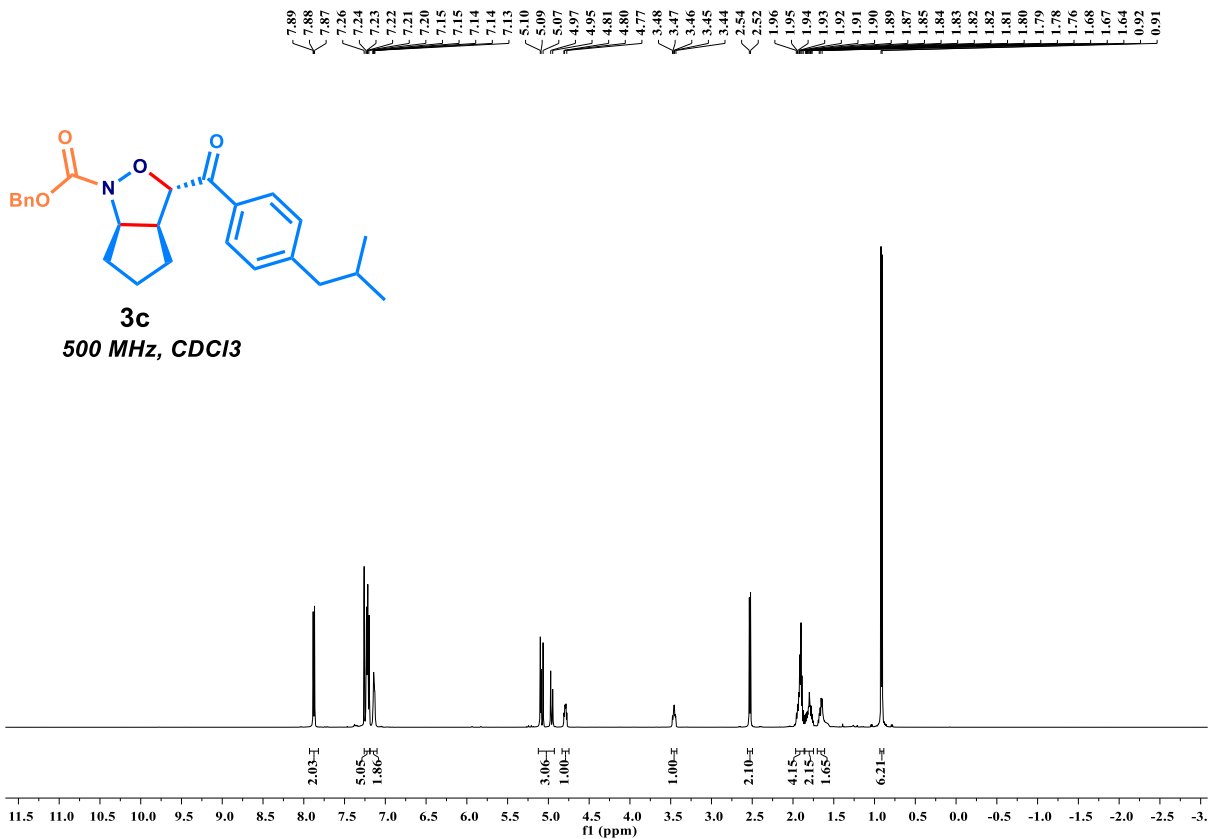
**3a**

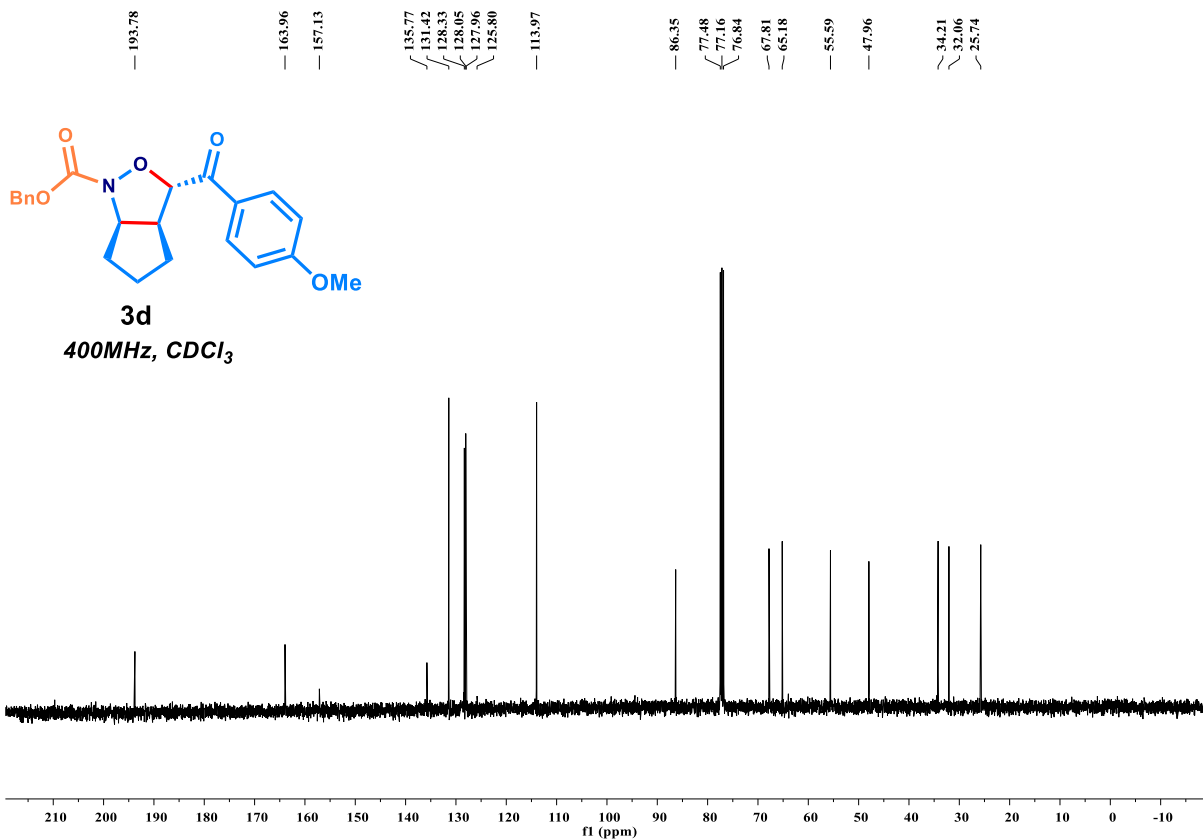
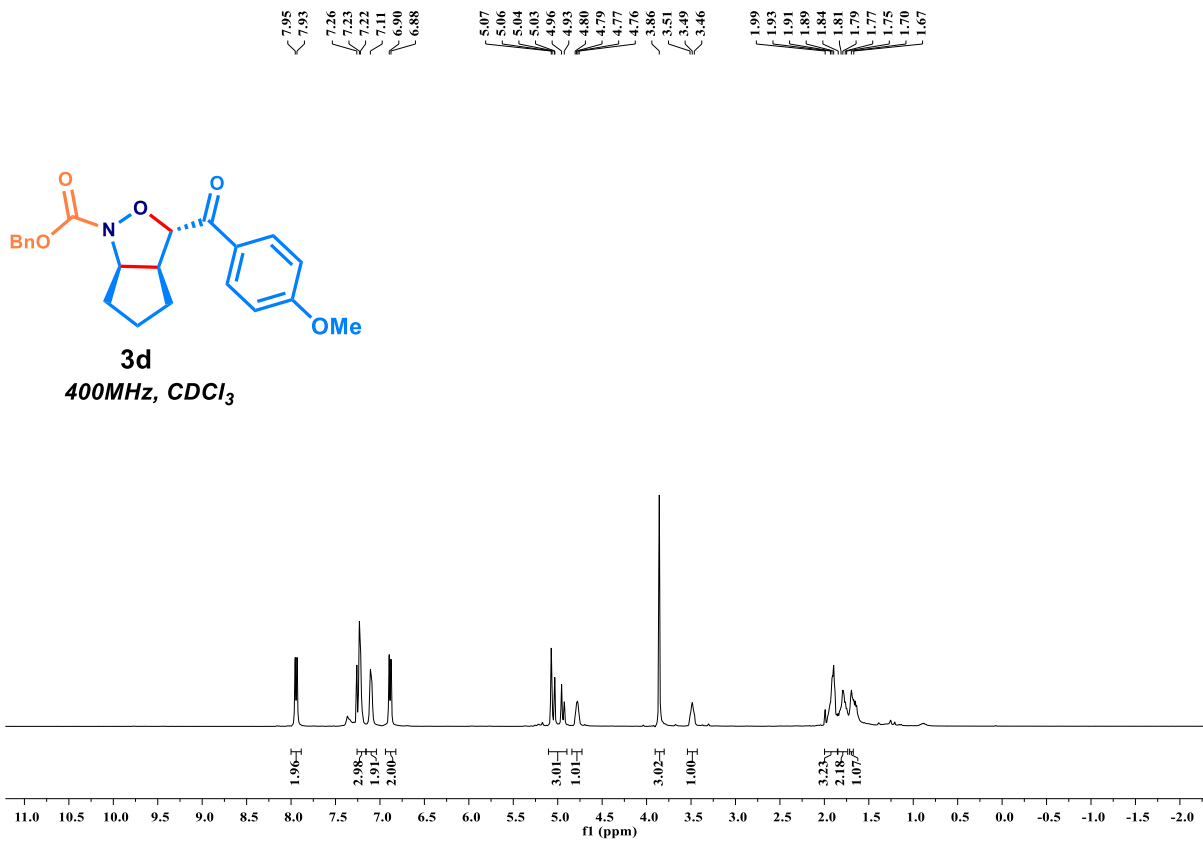
126 MHz, CDCl<sub>3</sub>

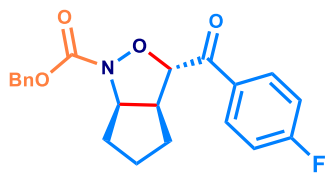








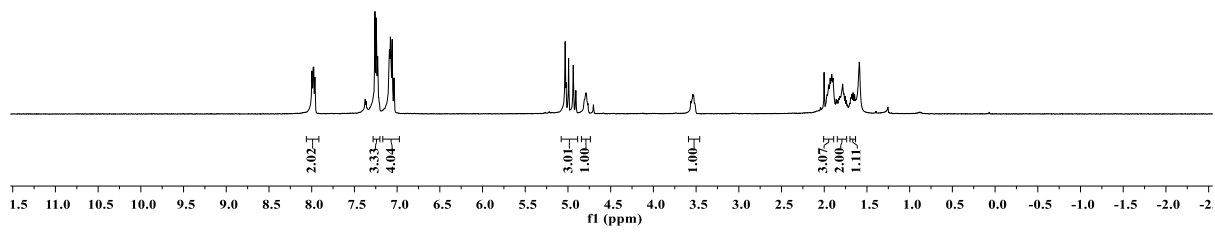




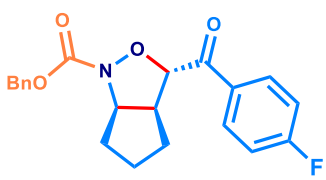
**3e**

400 MHz, CDCl<sub>3</sub>

- 8.00
- 7.98
- 7.98
- 7.96
- 7.26
- 7.24
- 7.23
- 7.09
- 7.08
- 7.06
- 7.04
- 5.03
- 5.02
- 4.99
- 4.94
- 4.91
- 4.81
- 4.79
- 4.78
- 4.77
- 3.56
- 3.54
- 3.51
- 3.51
- 2.00
- 1.97
- 1.96
- 1.93
- 1.91
- 1.90
- 1.83
- 1.80
- 1.78
- 1.74
- 1.69
- 1.68
- 1.66

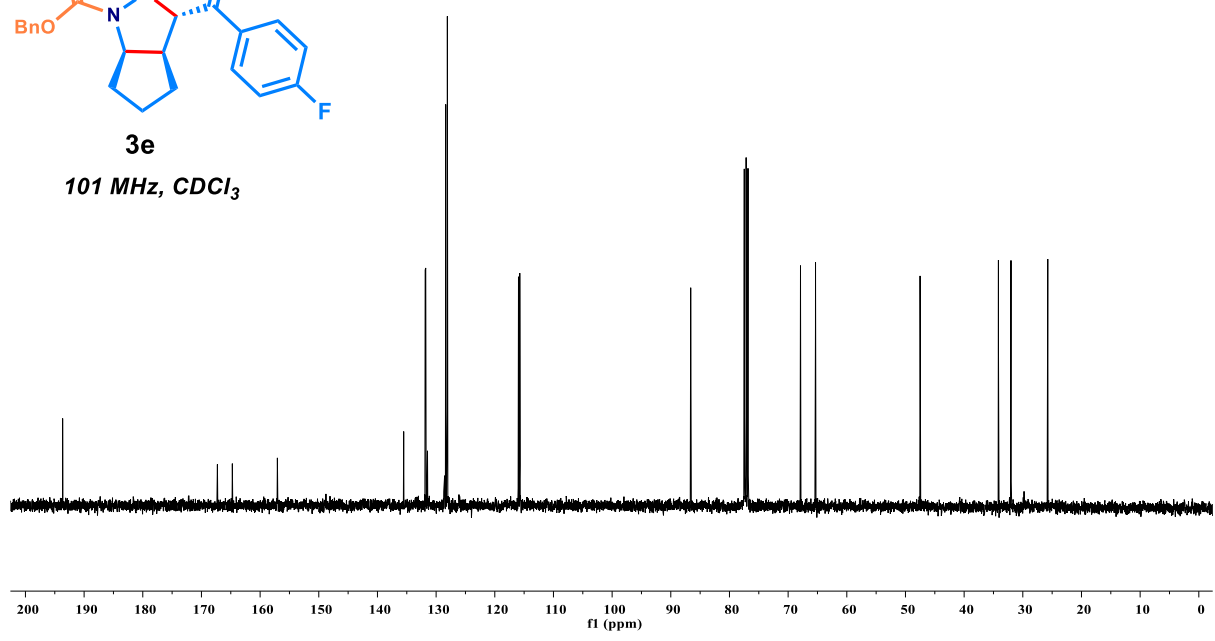


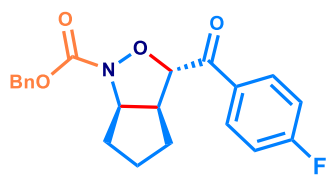
- 193.66
- 167.28
- 164.74
- 157.06
- 135.53
- 131.87
- 131.77
- 131.50
- 131.47
- 128.37
- 128.09
- 115.96
- 115.74
- 86.61
- 77.48
- 77.16
- 76.84
- 67.89
- 65.33
- 47.51
- 34.15
- 32.02
- 25.74



**3e**

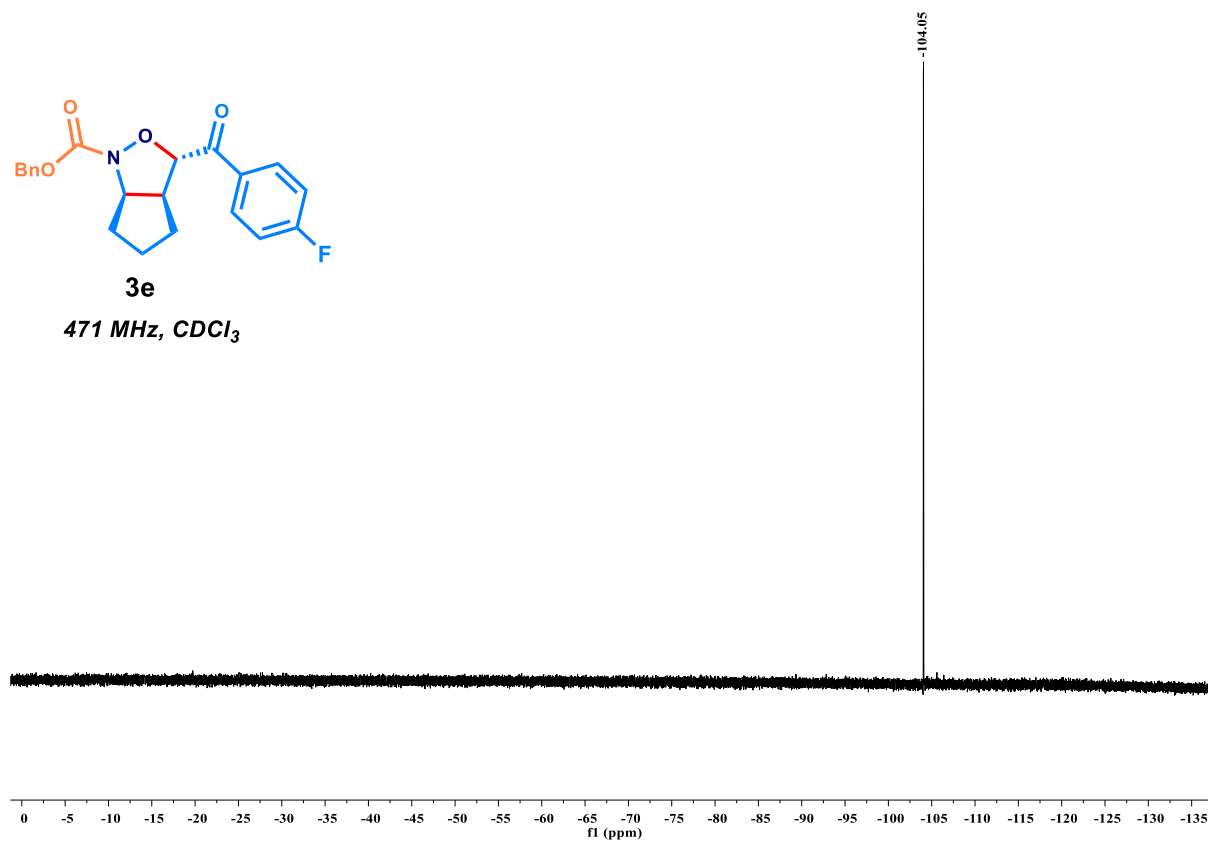
101 MHz, CDCl<sub>3</sub>

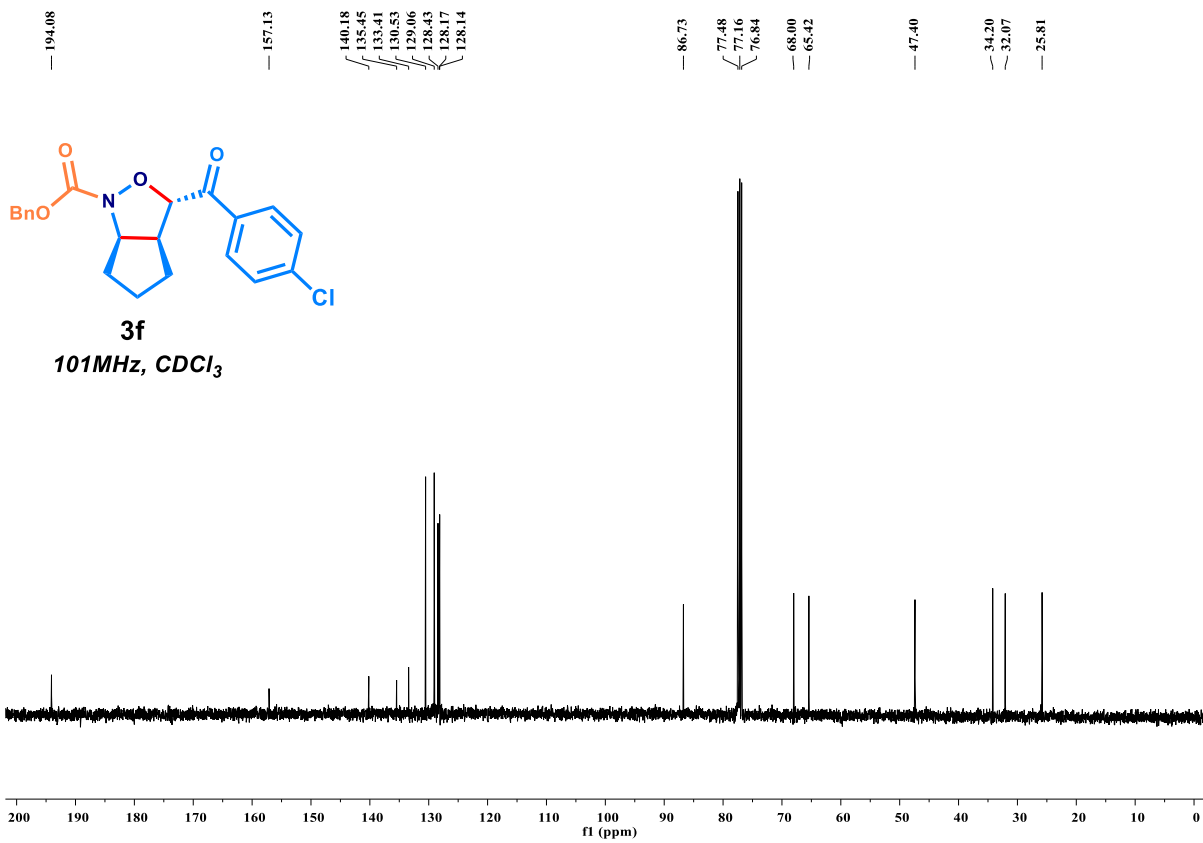
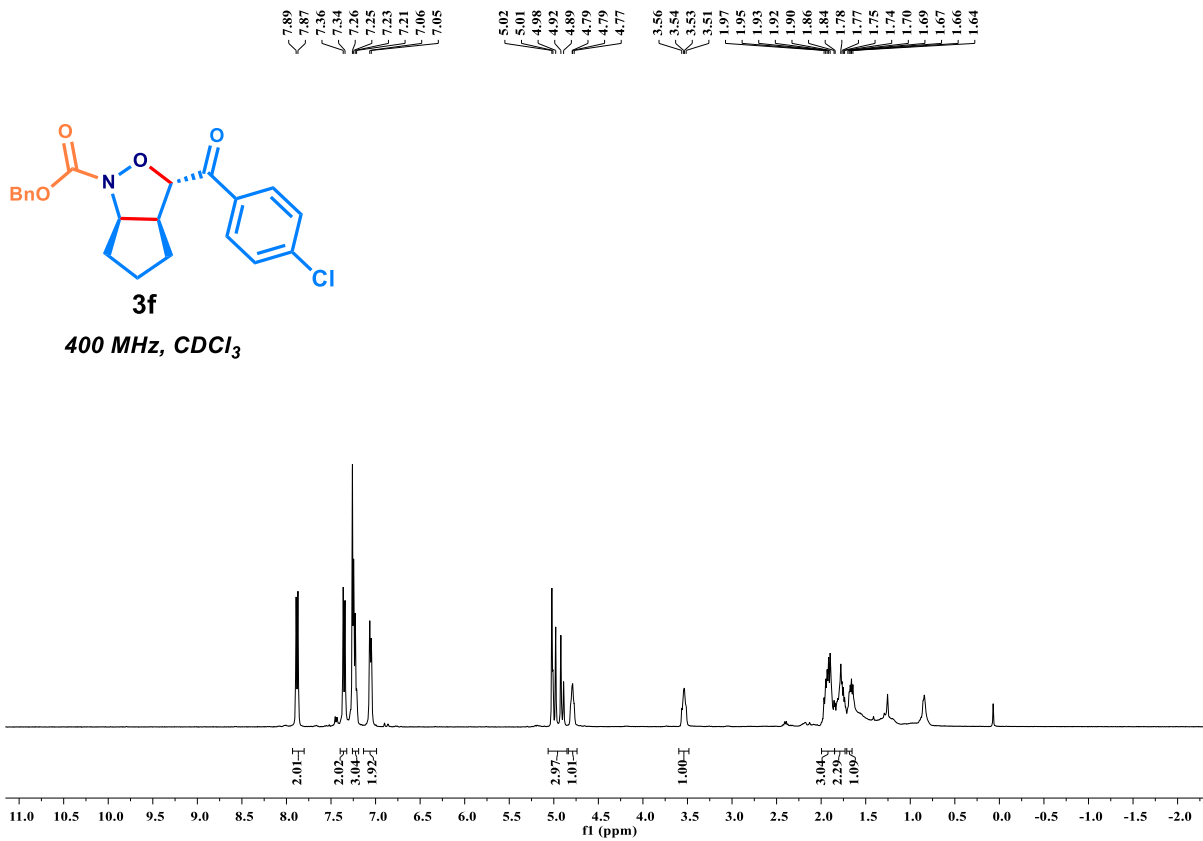


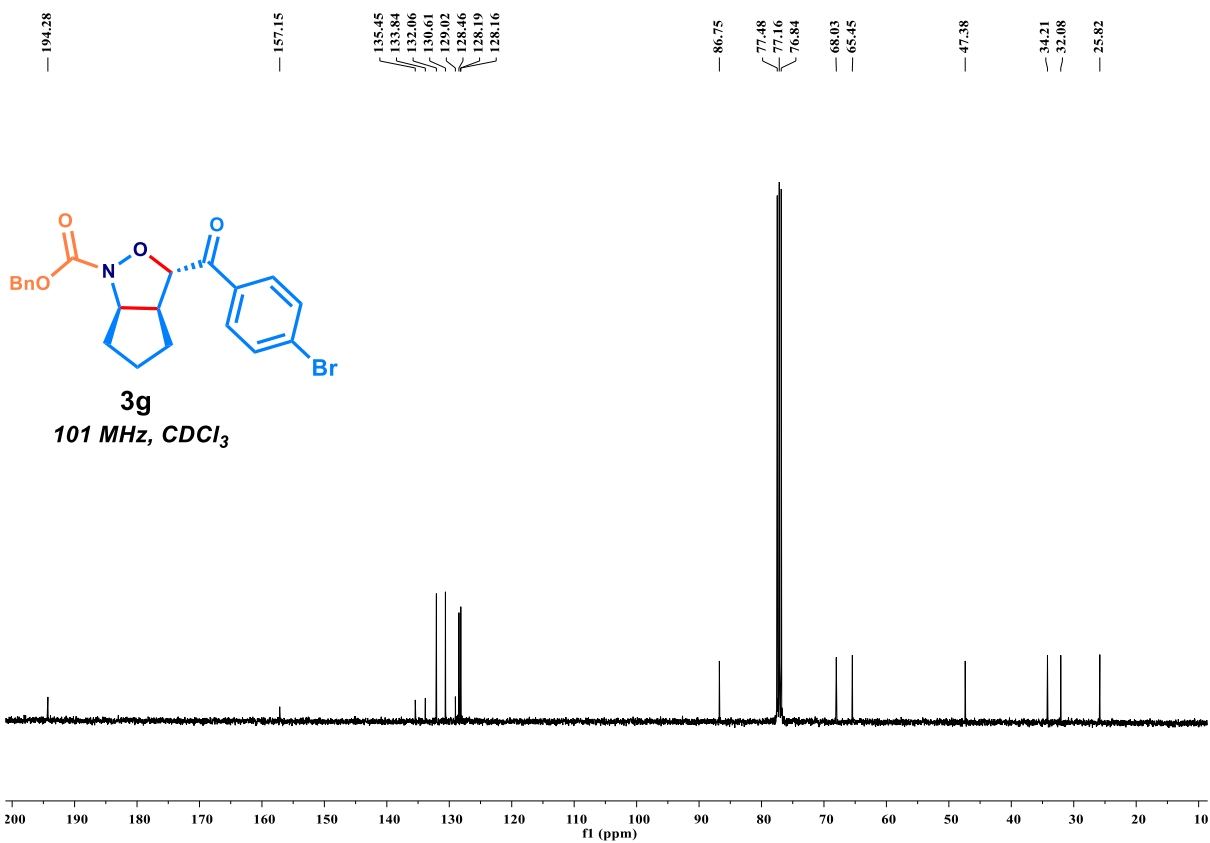
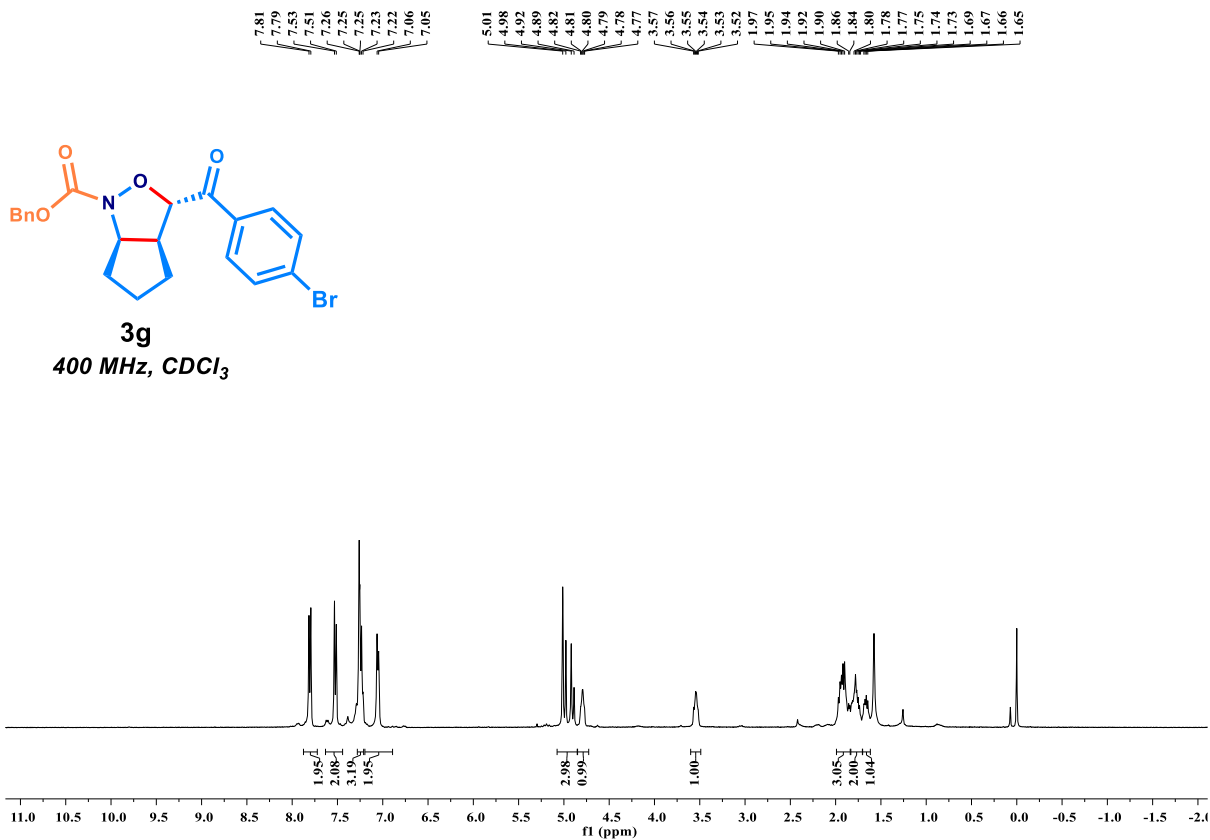


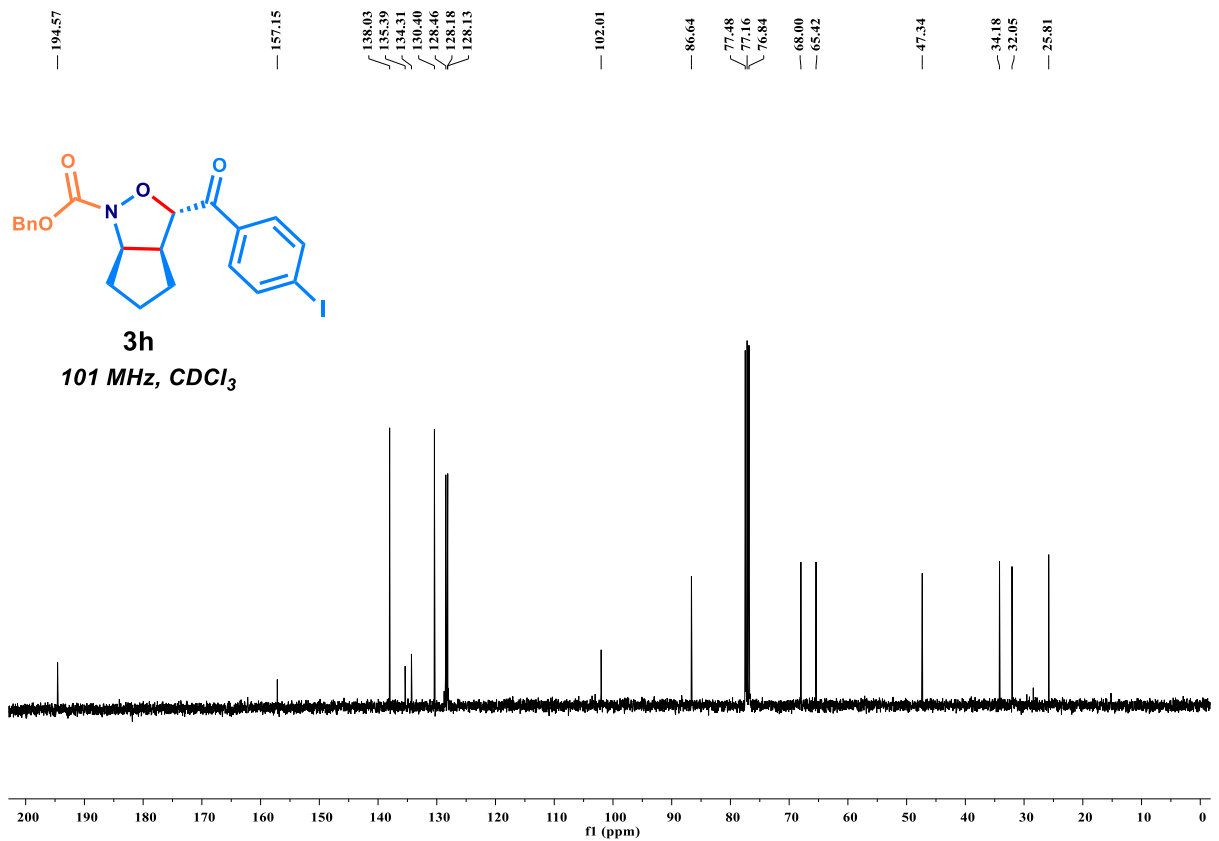
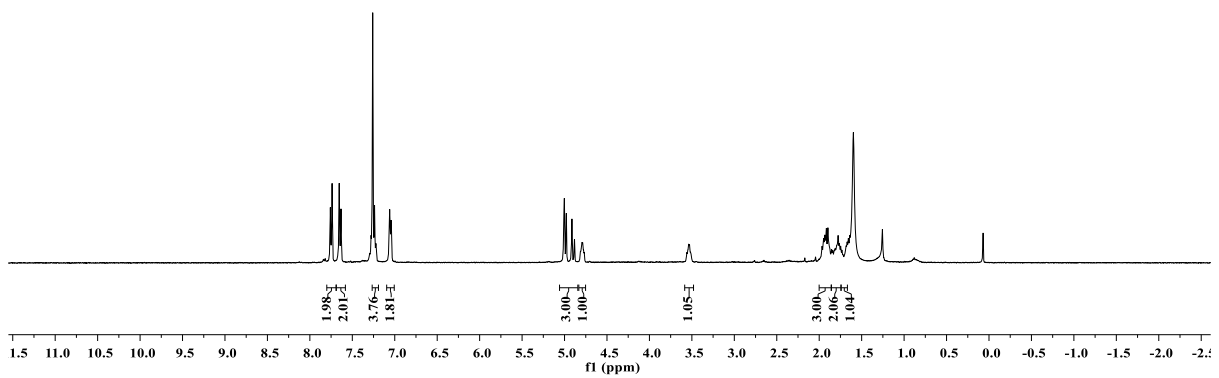
**3e**

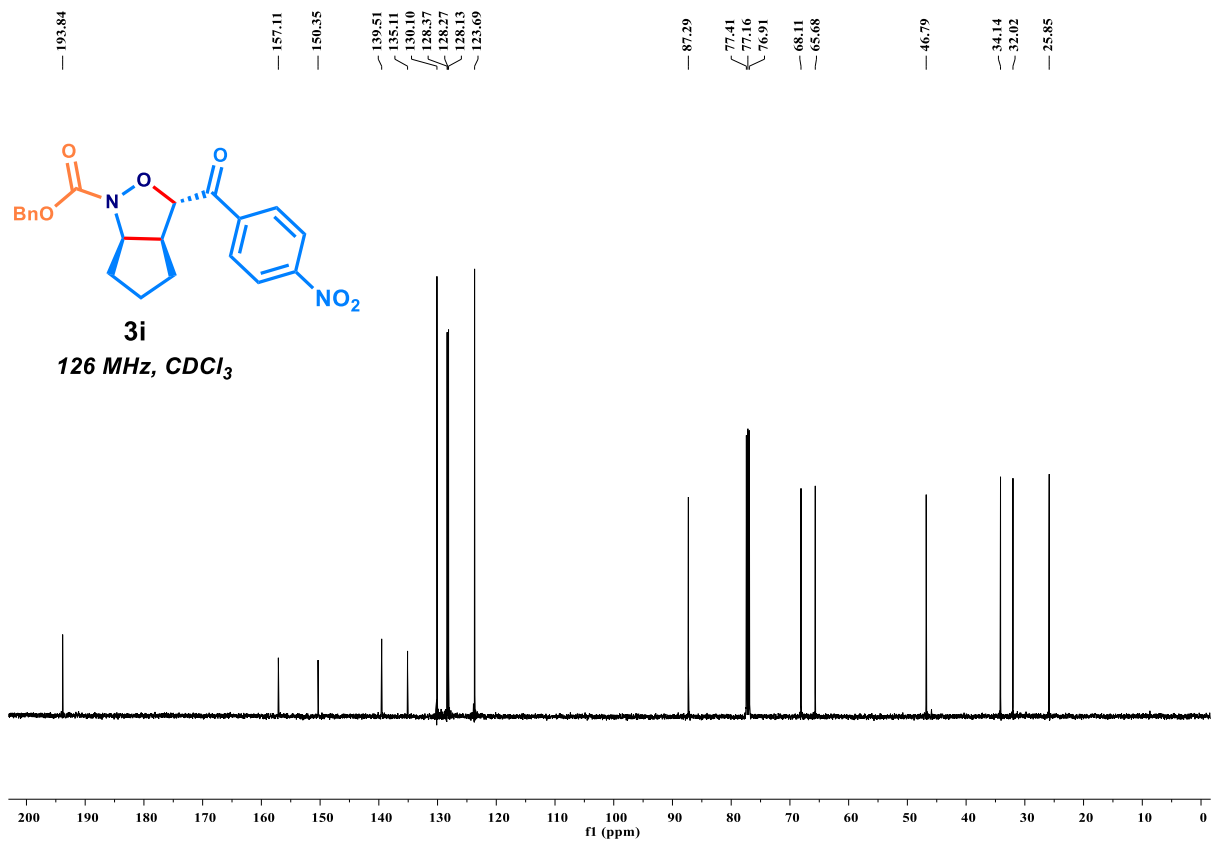
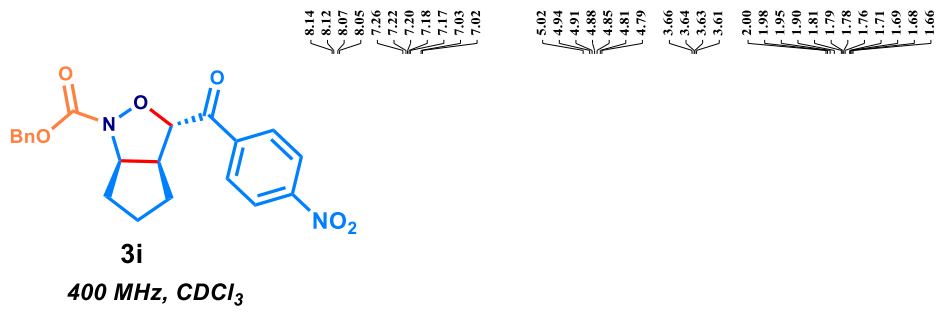
471 MHz, CDCl<sub>3</sub>



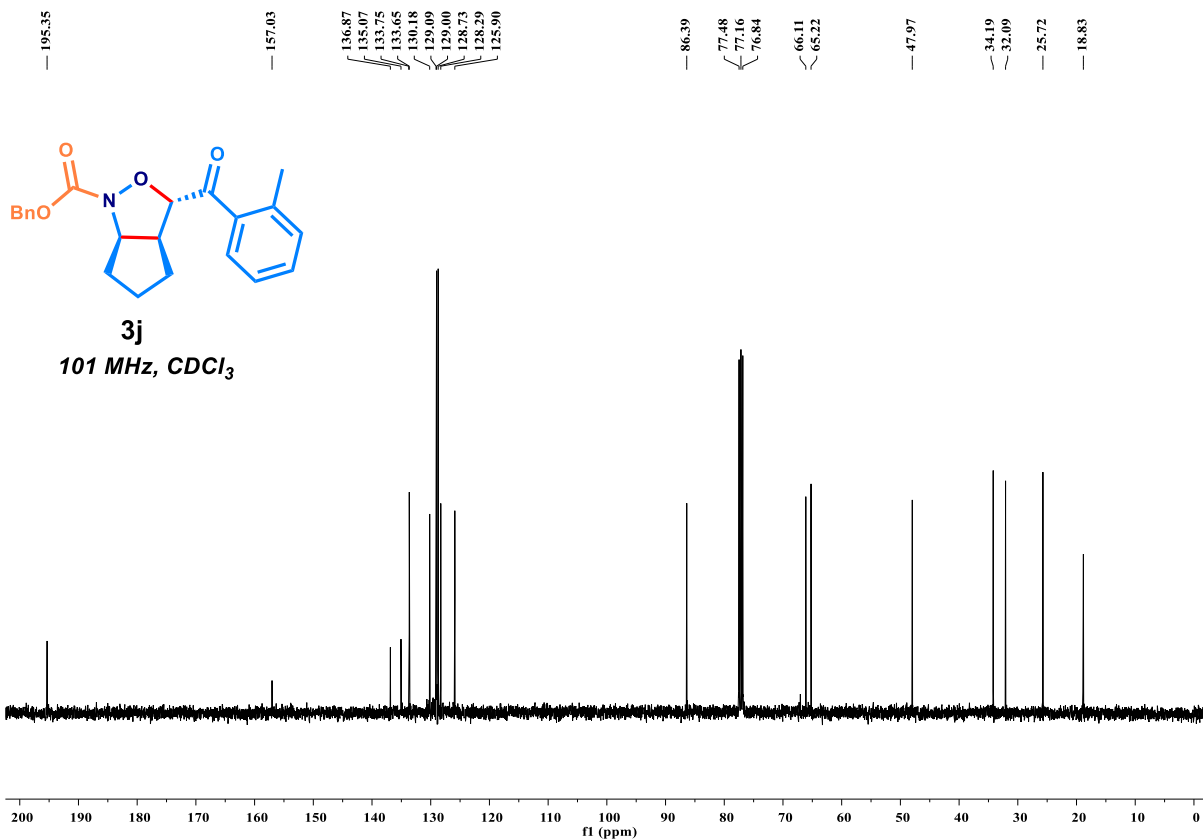
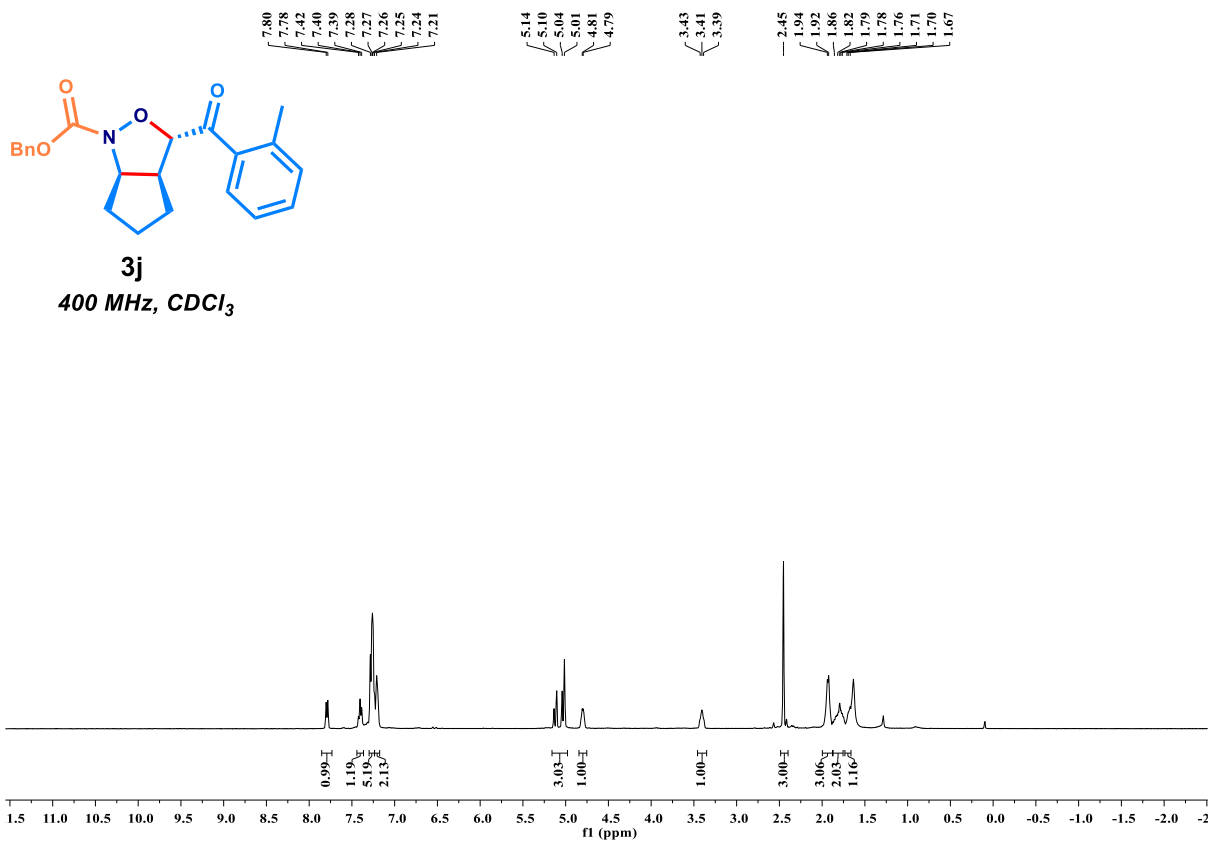


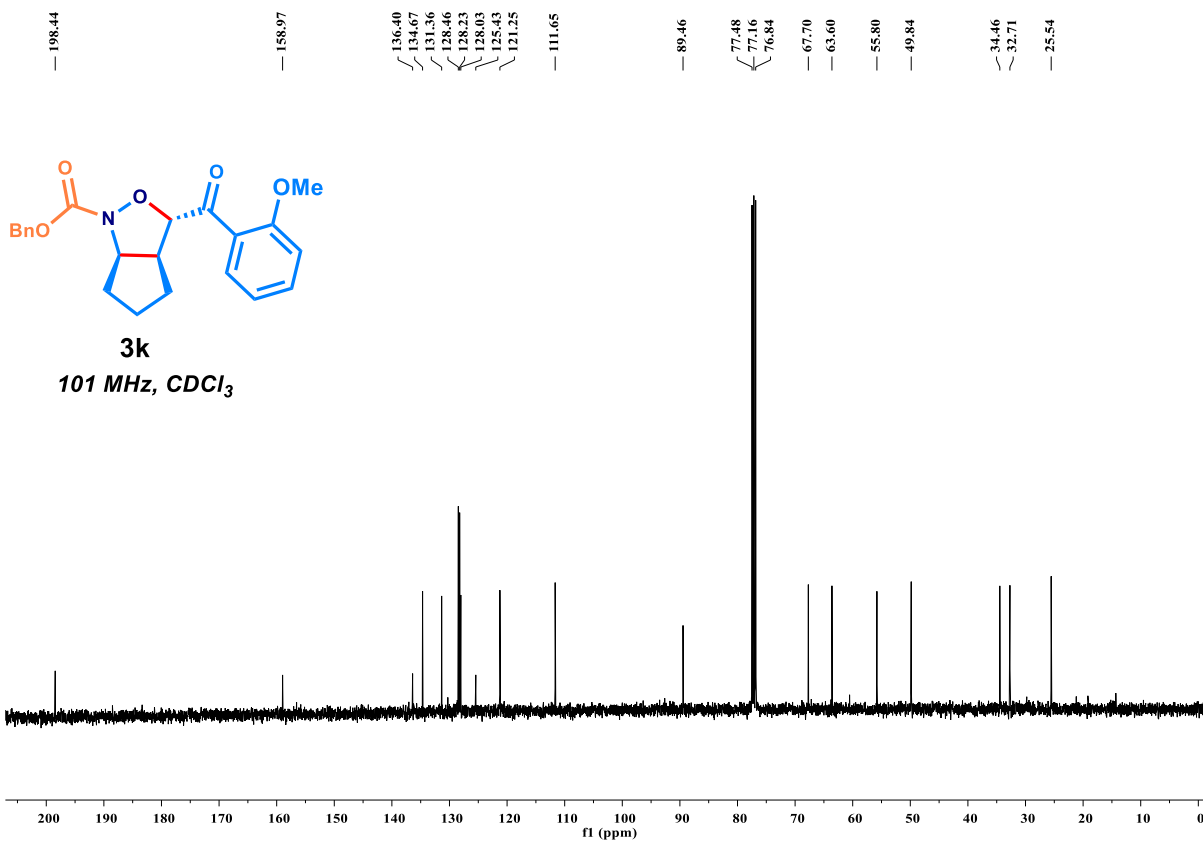
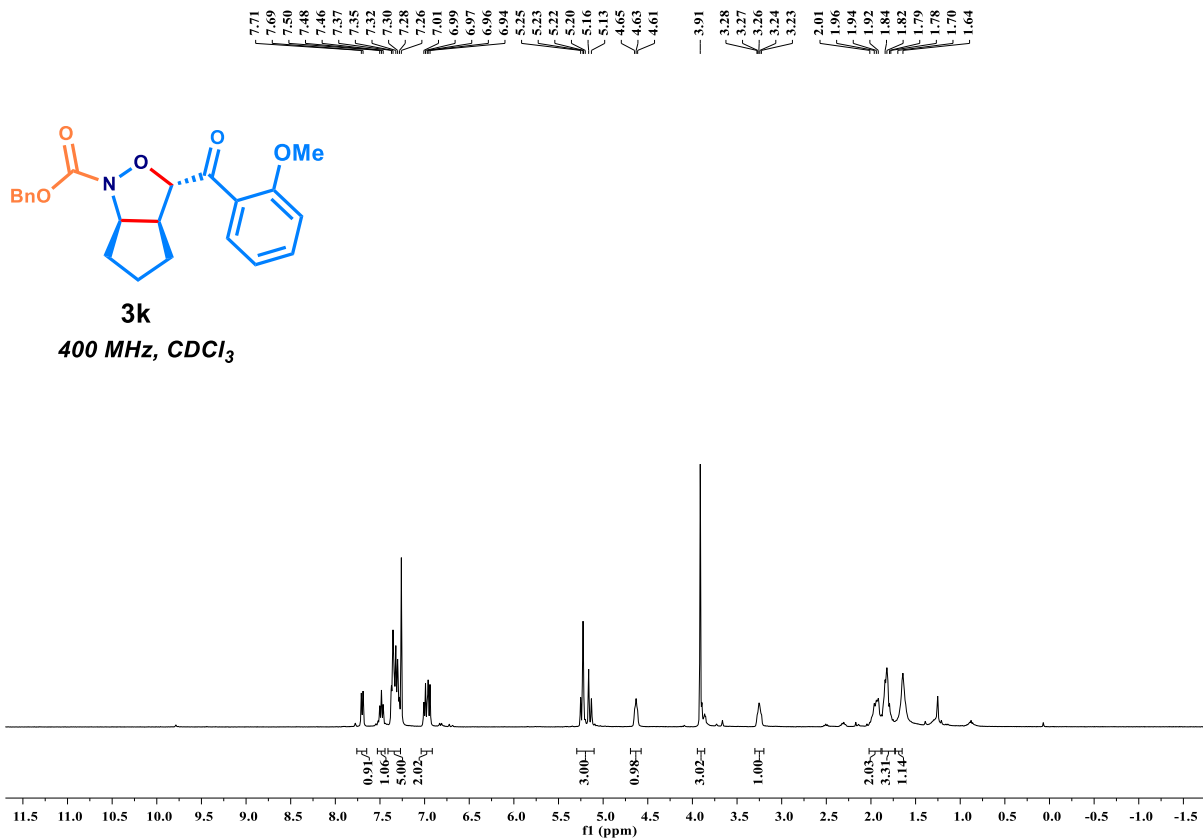


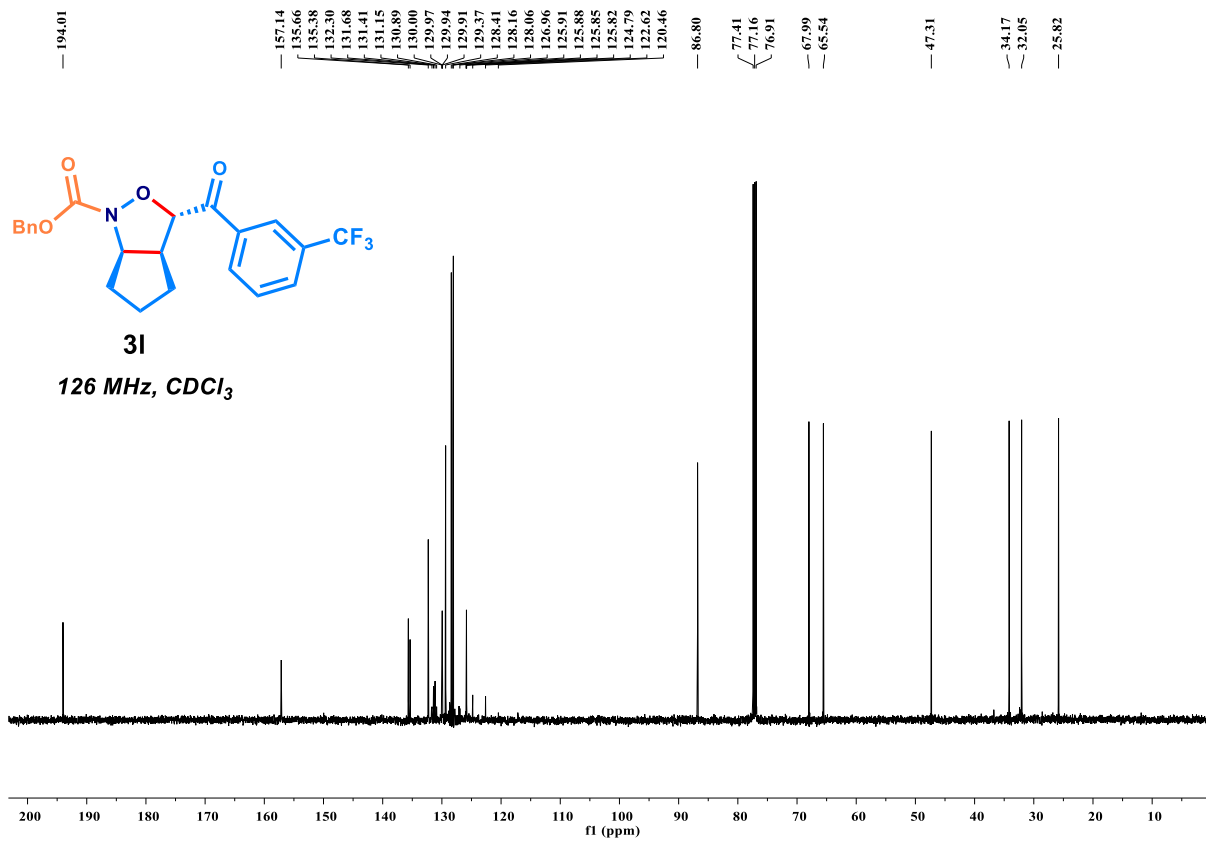
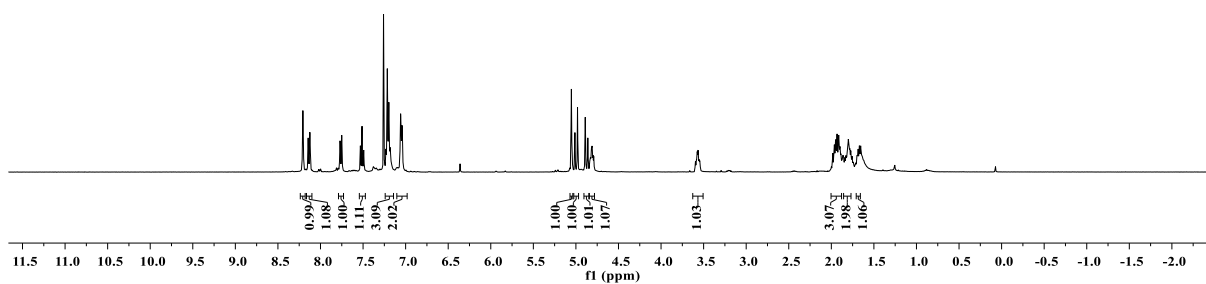
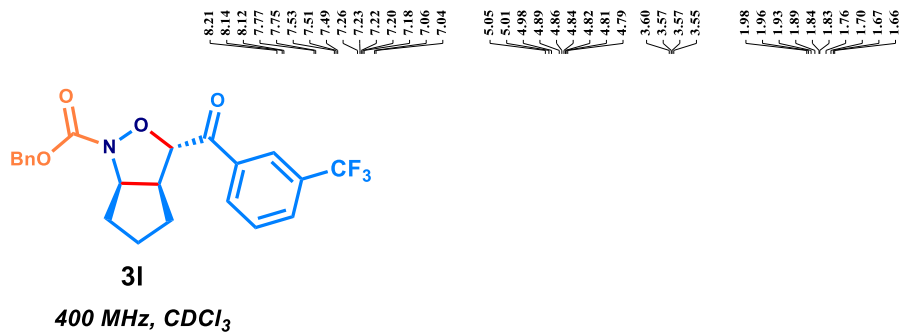


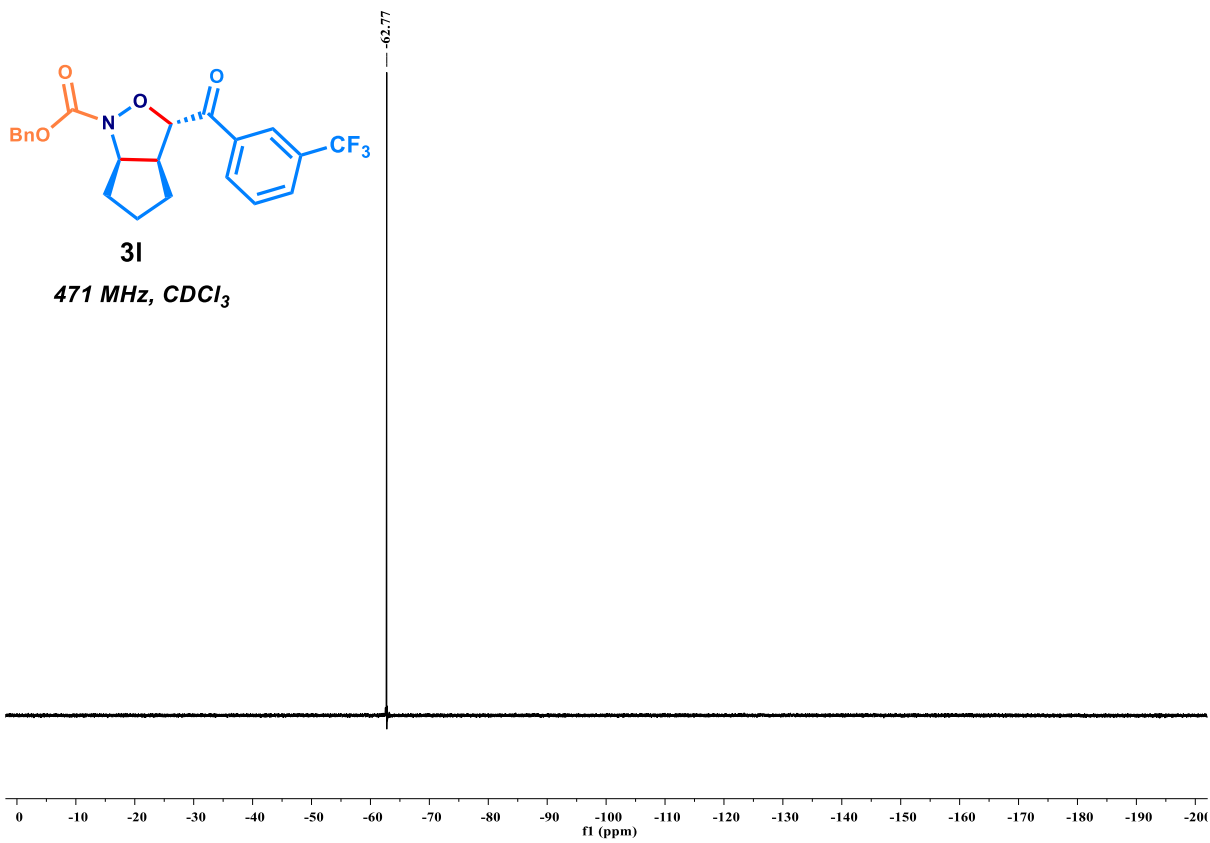


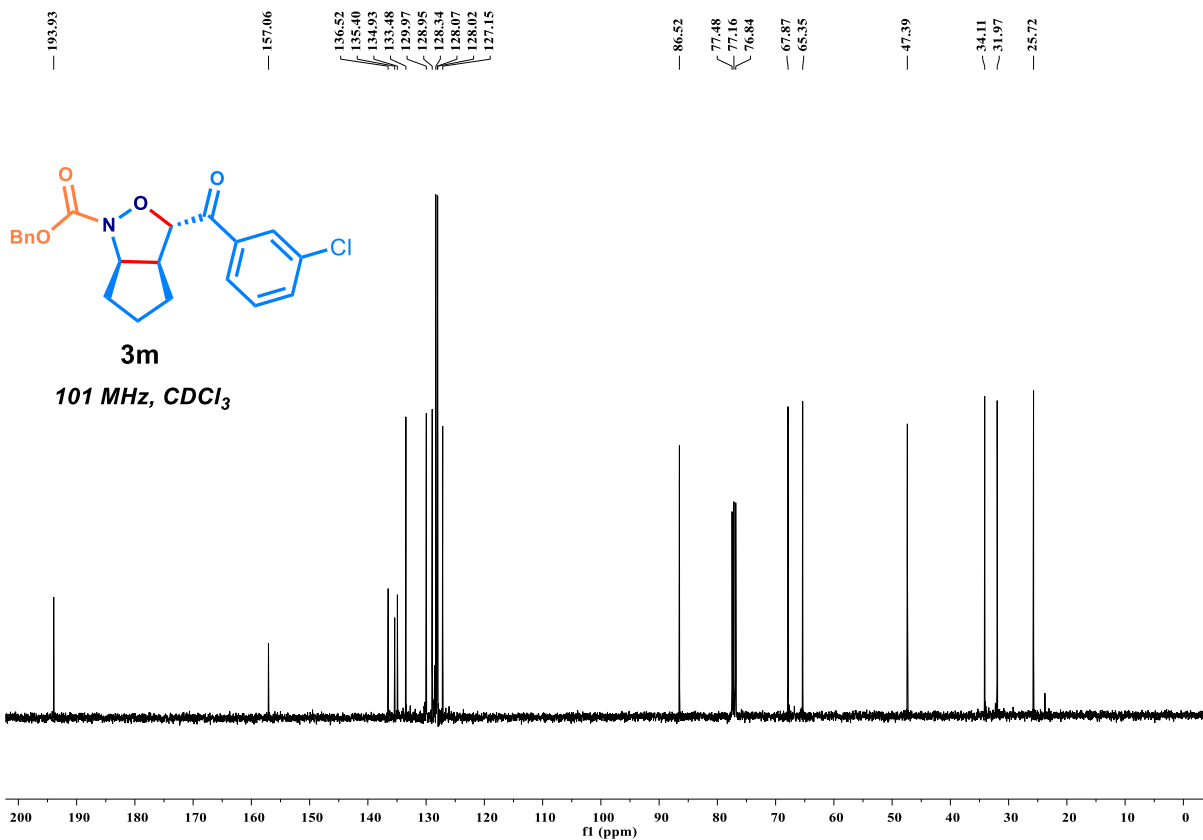
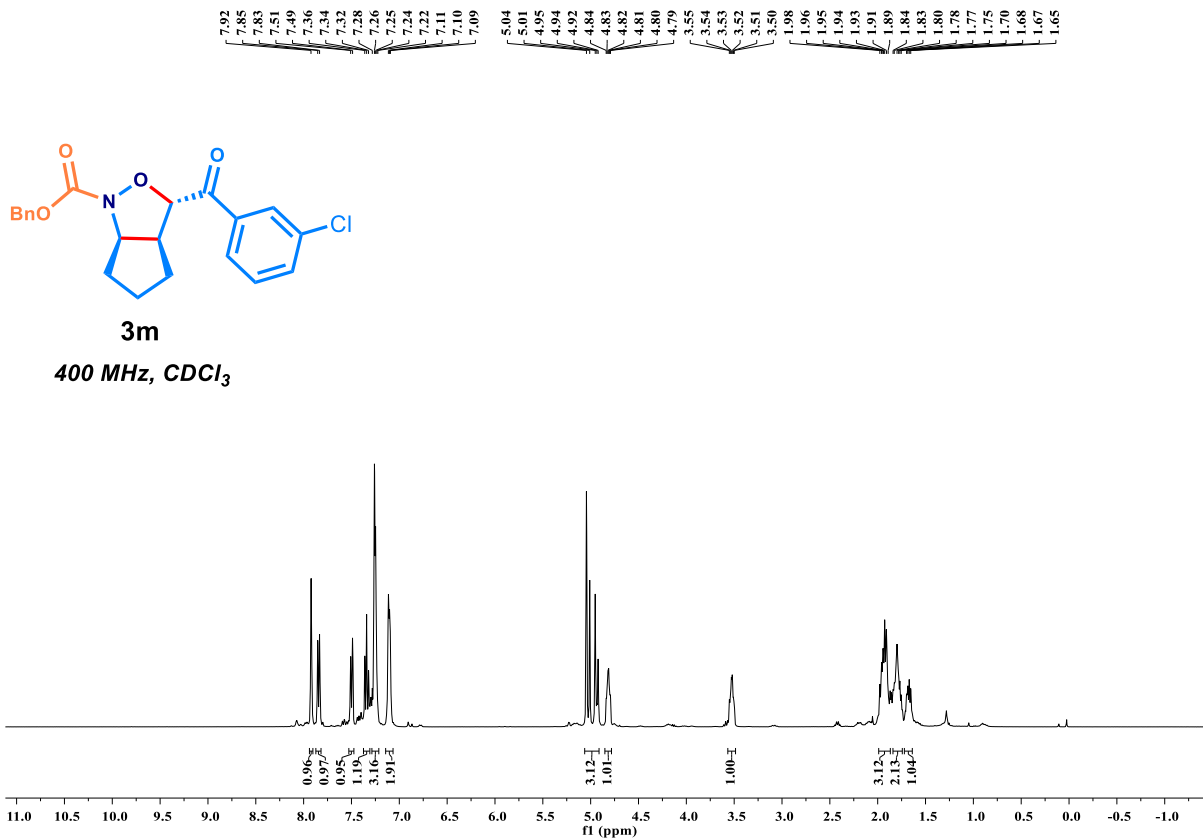


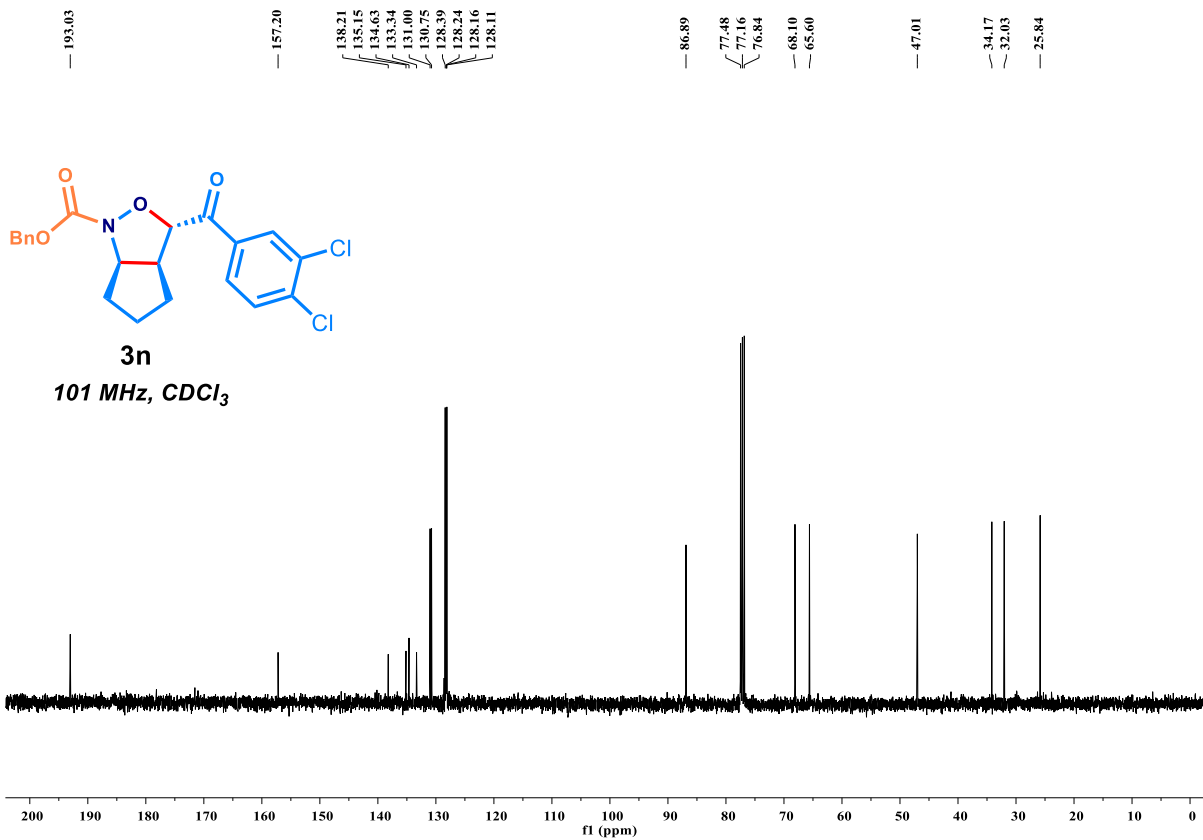
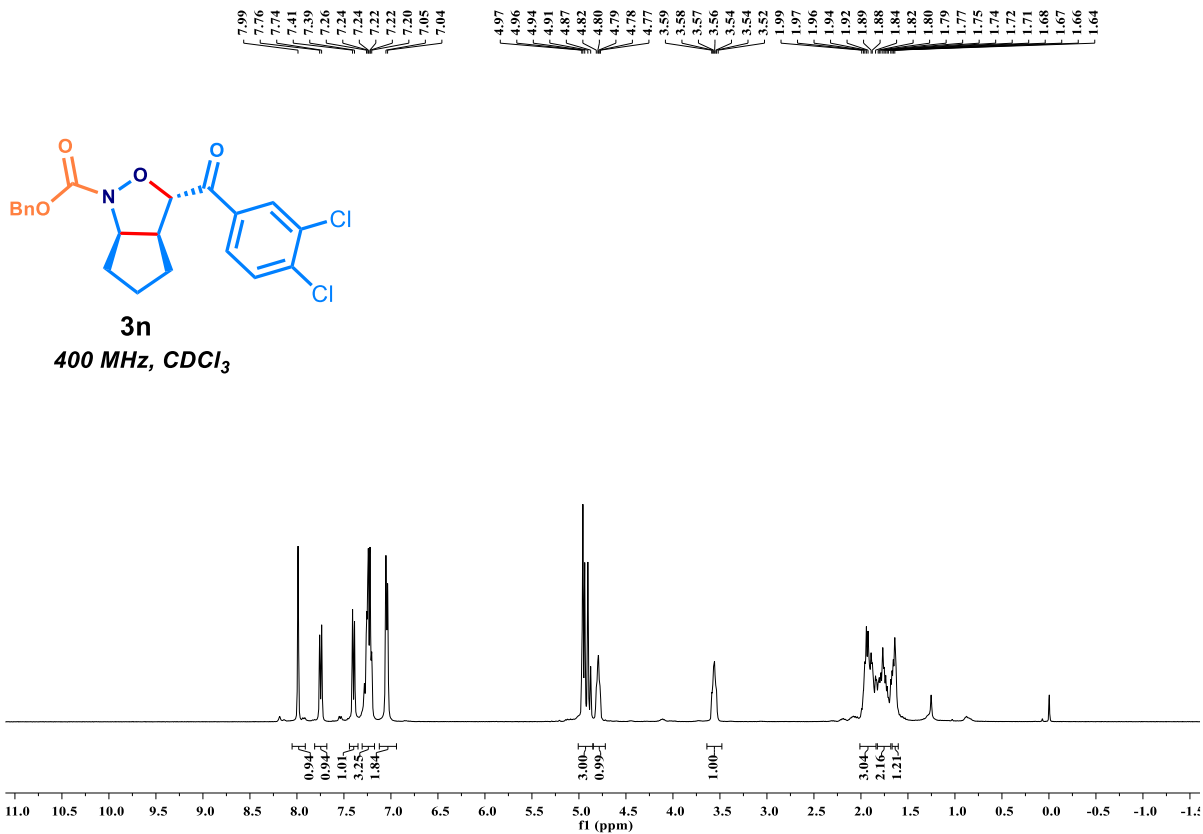


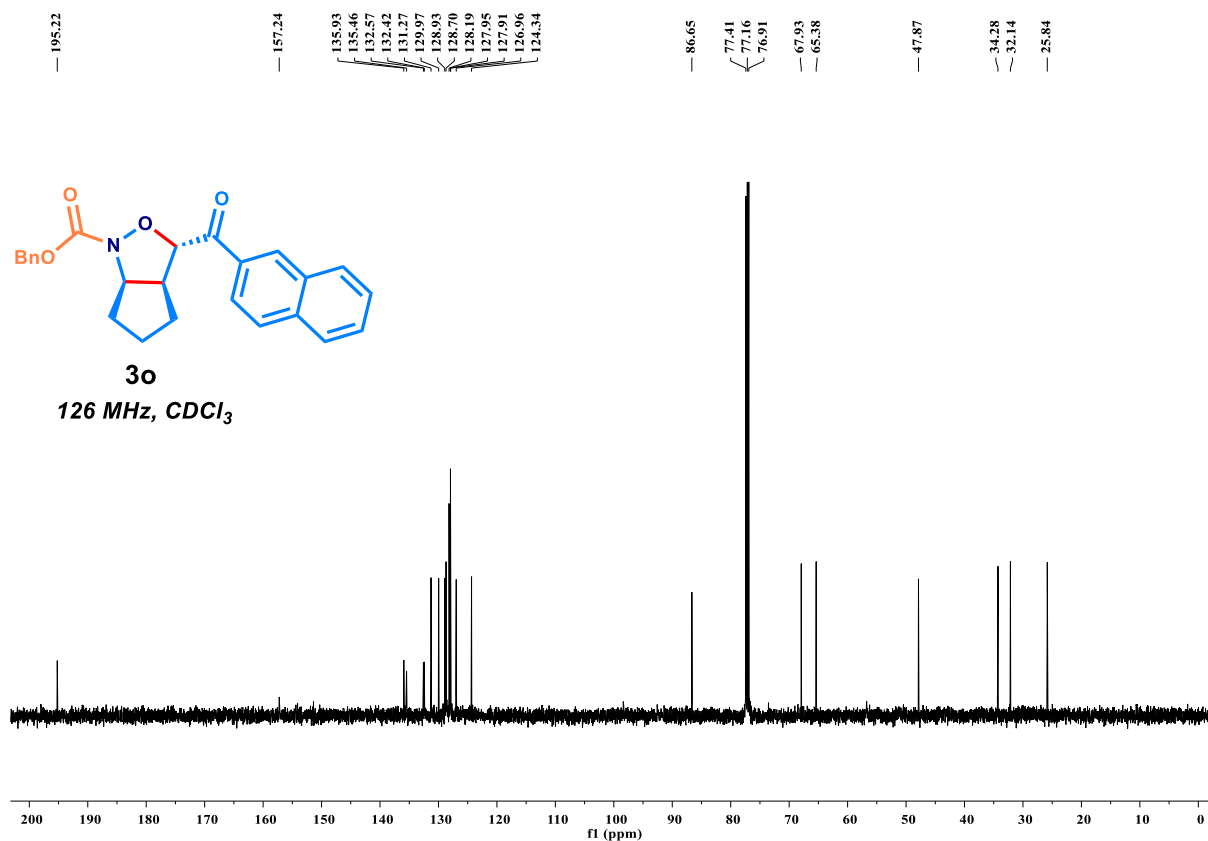
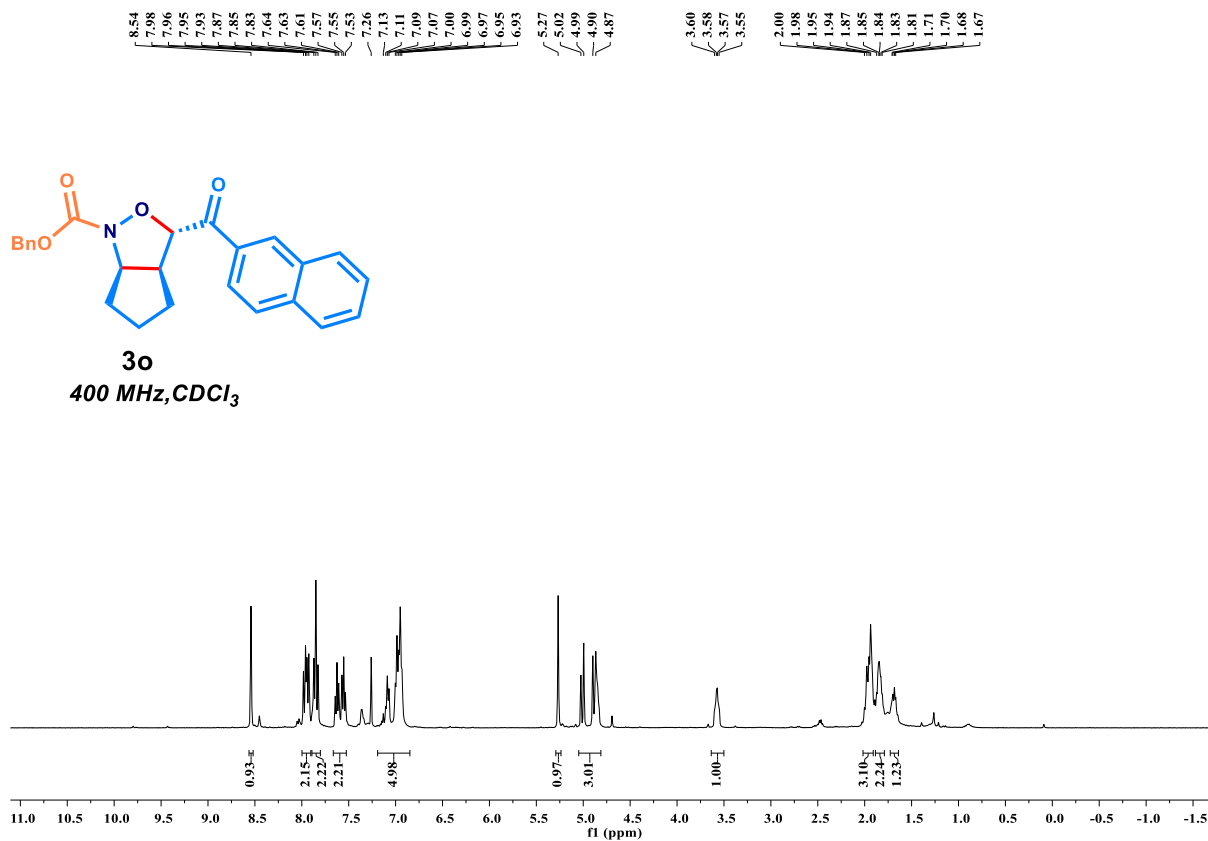


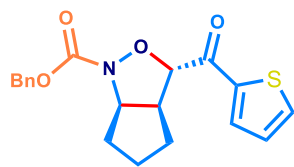




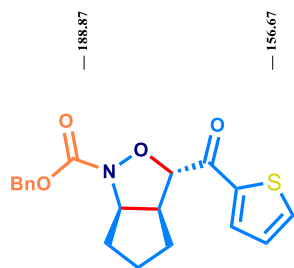
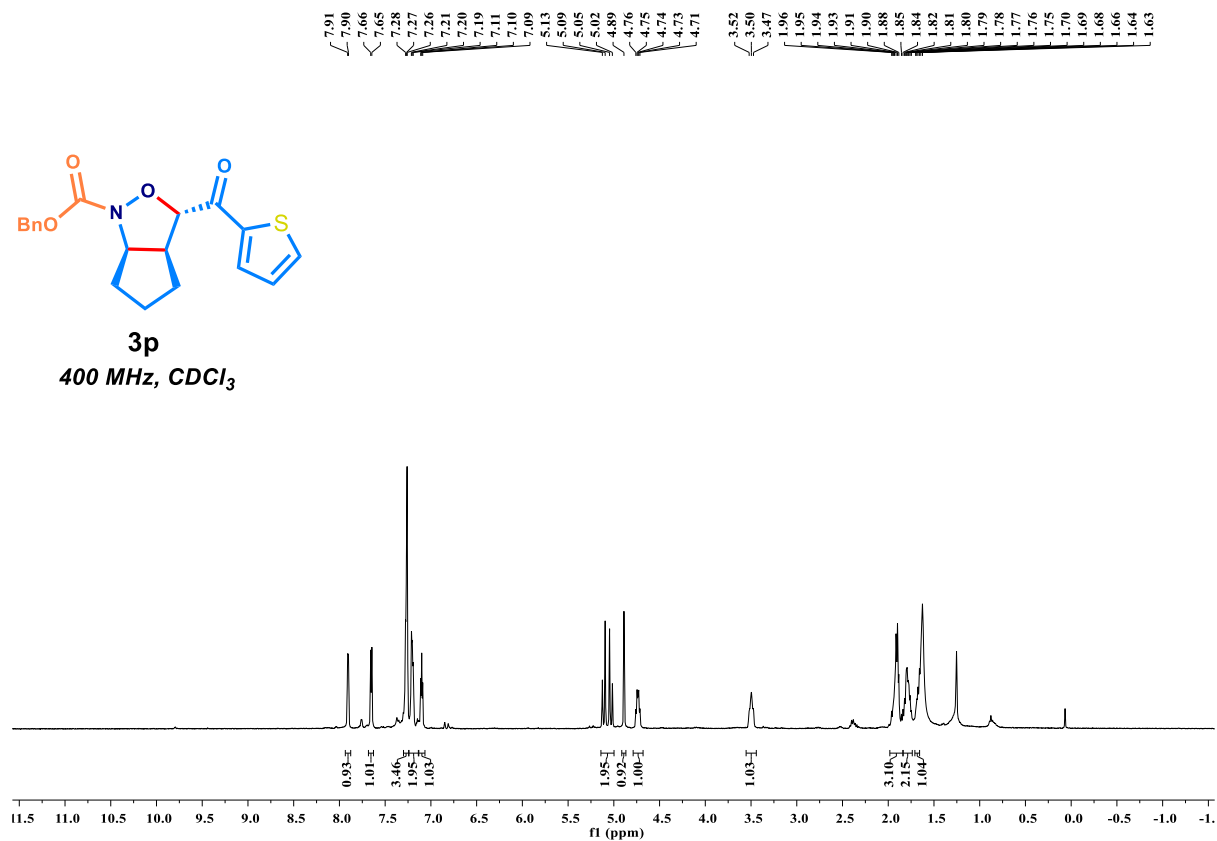




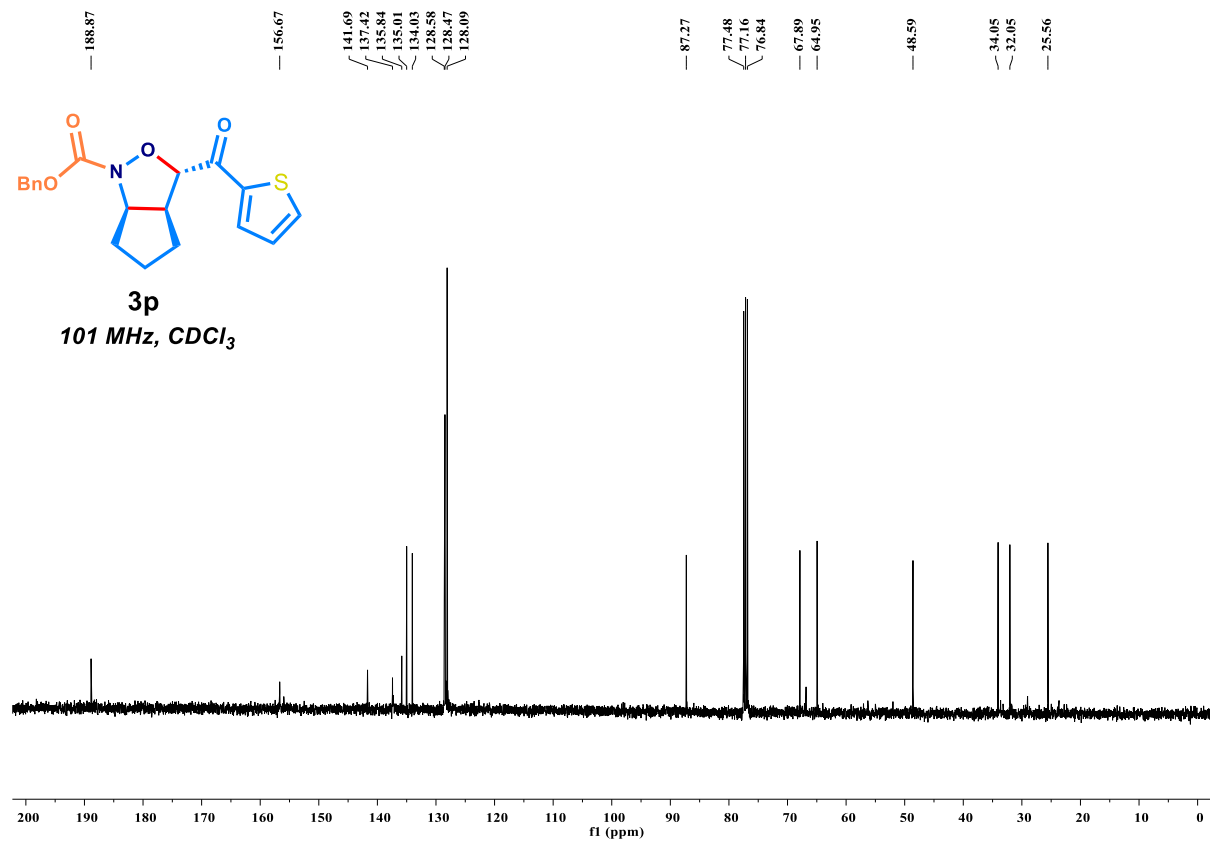




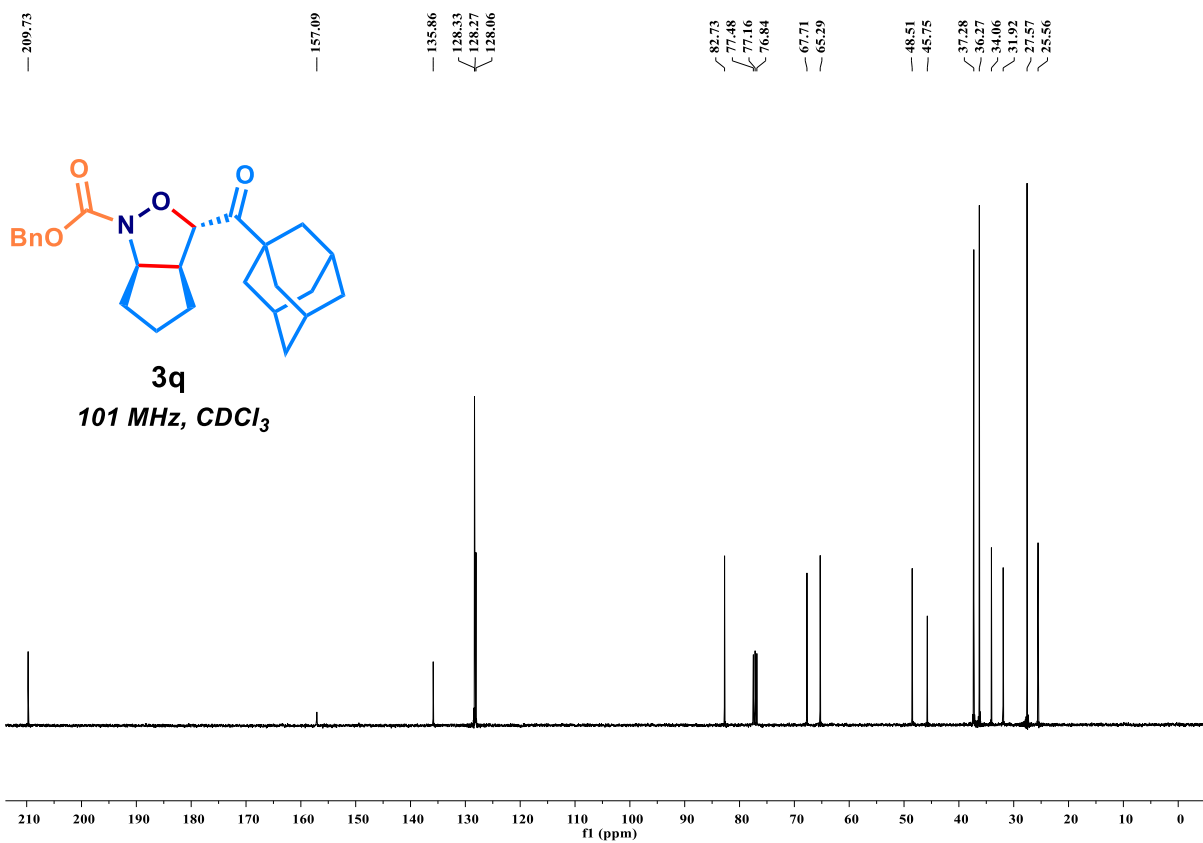
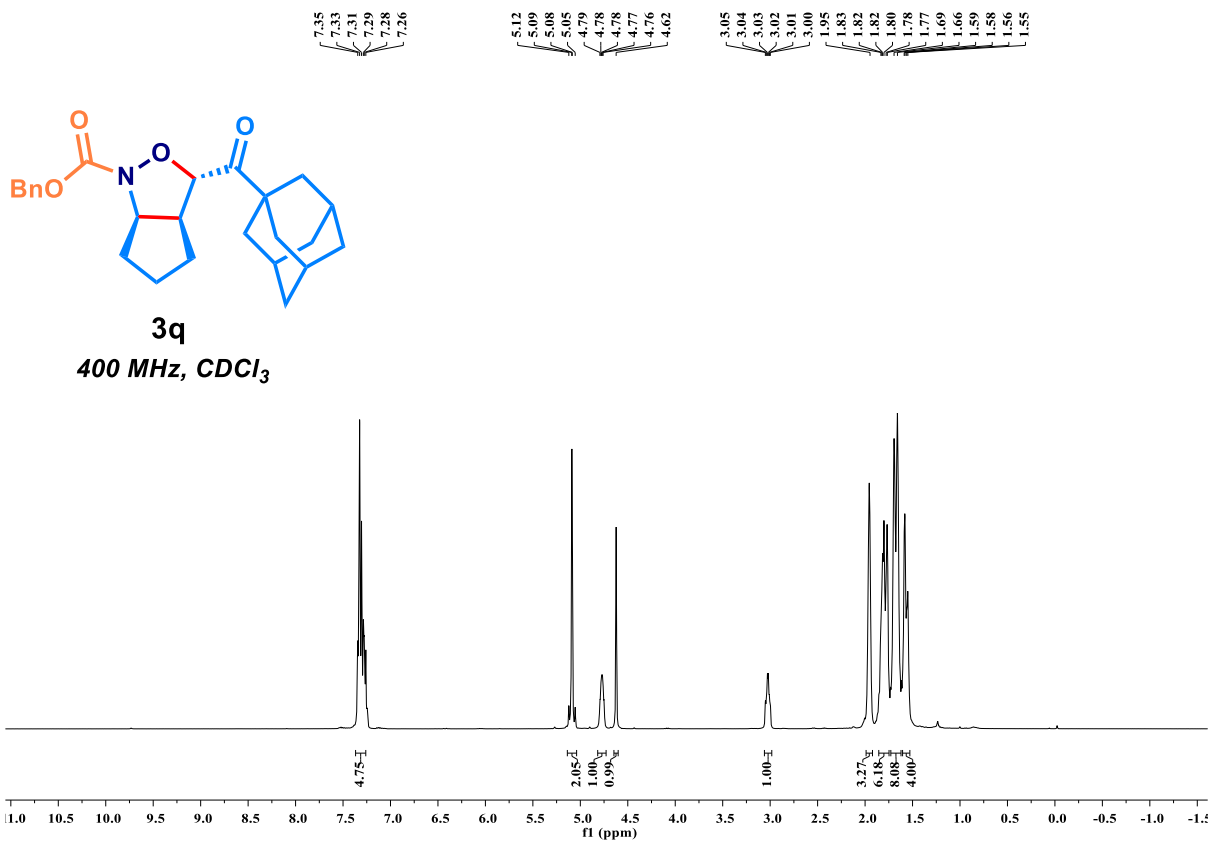
**3p**  
400 MHz, CDCl<sub>3</sub>

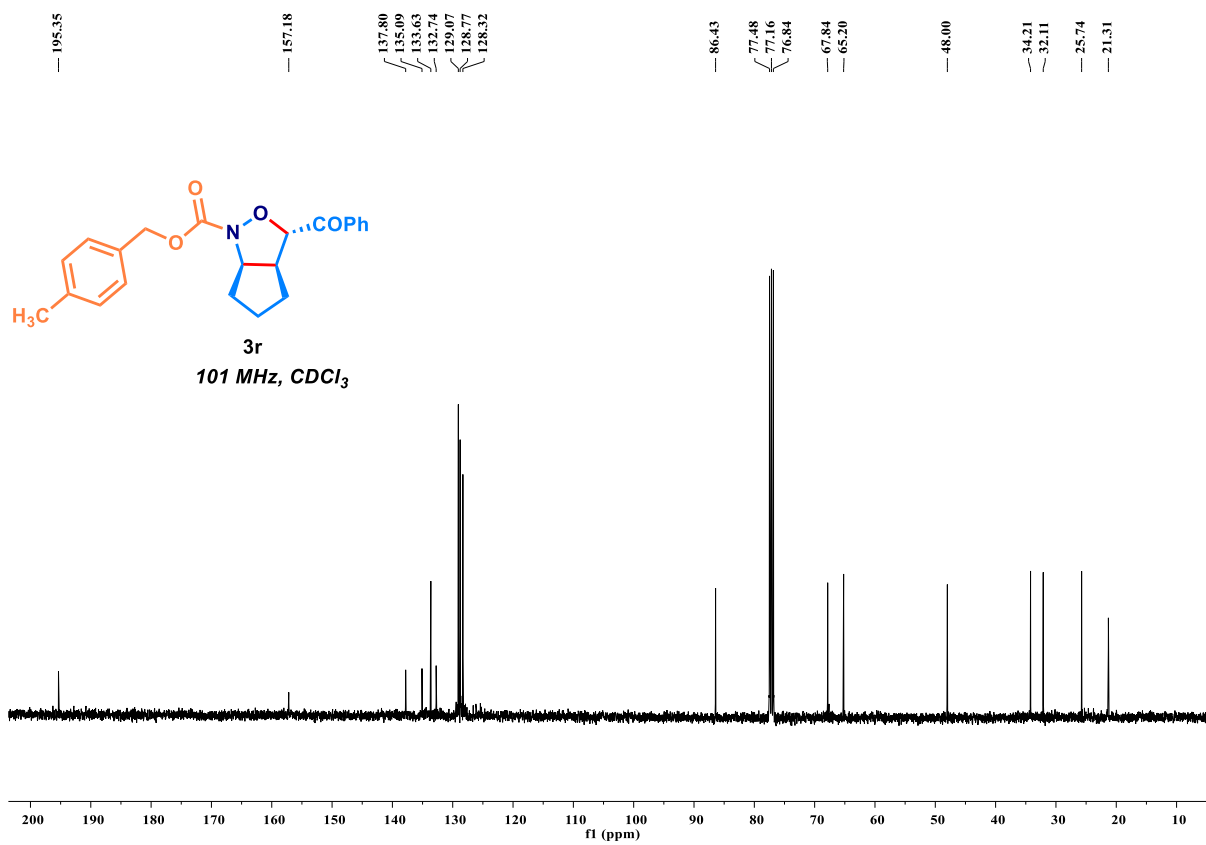
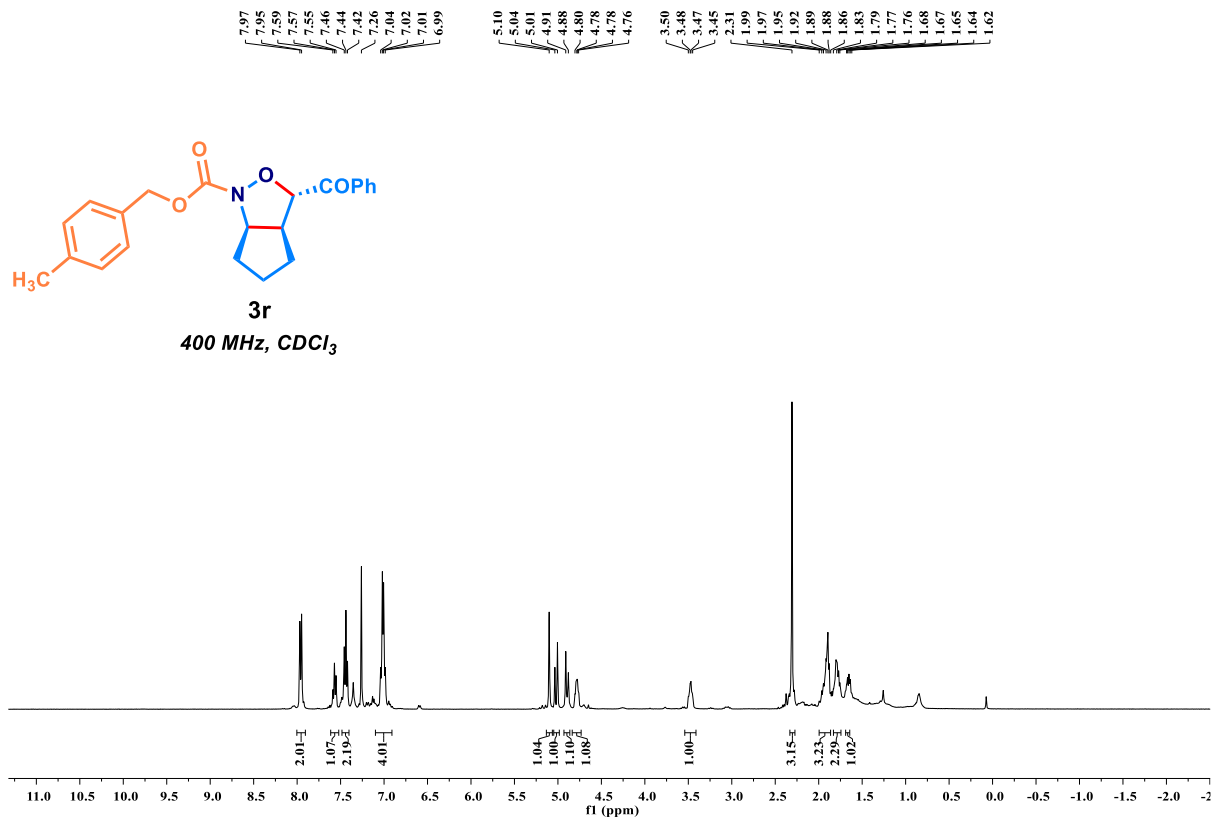


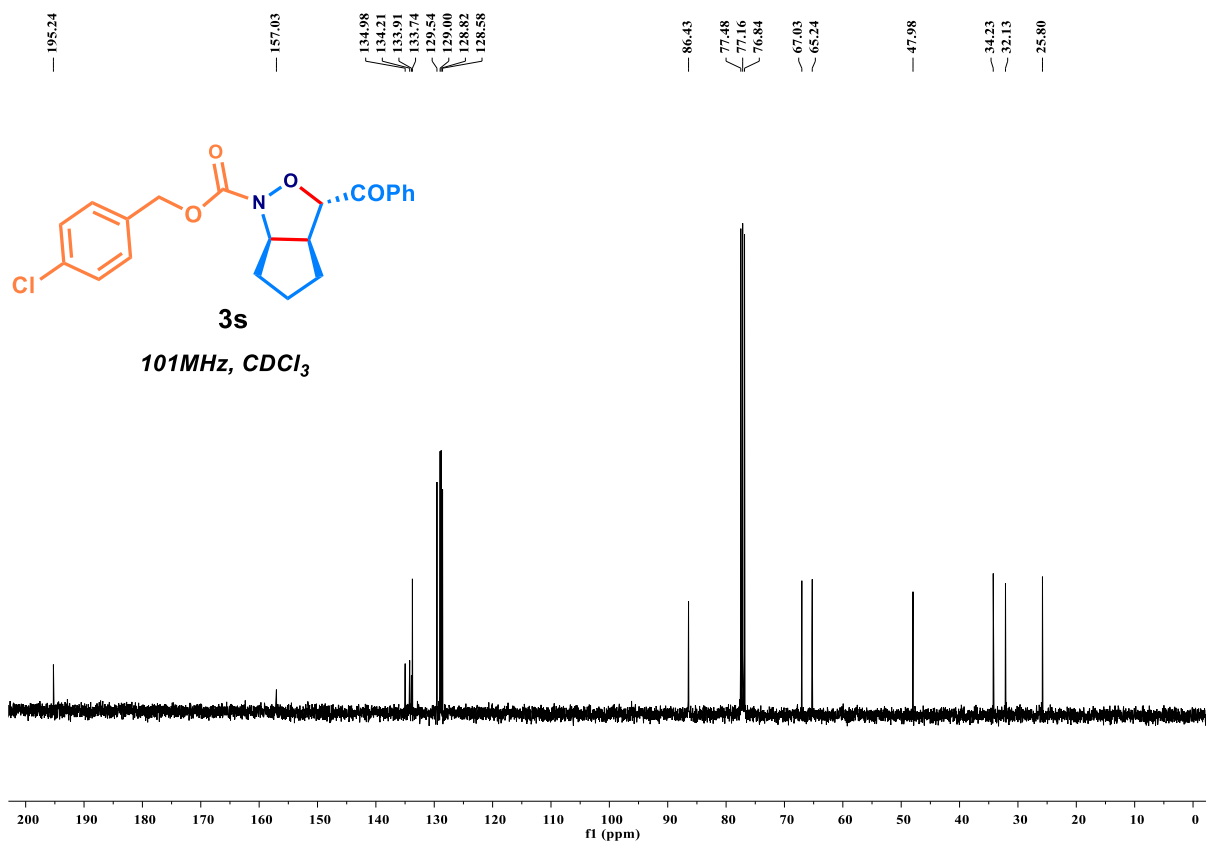
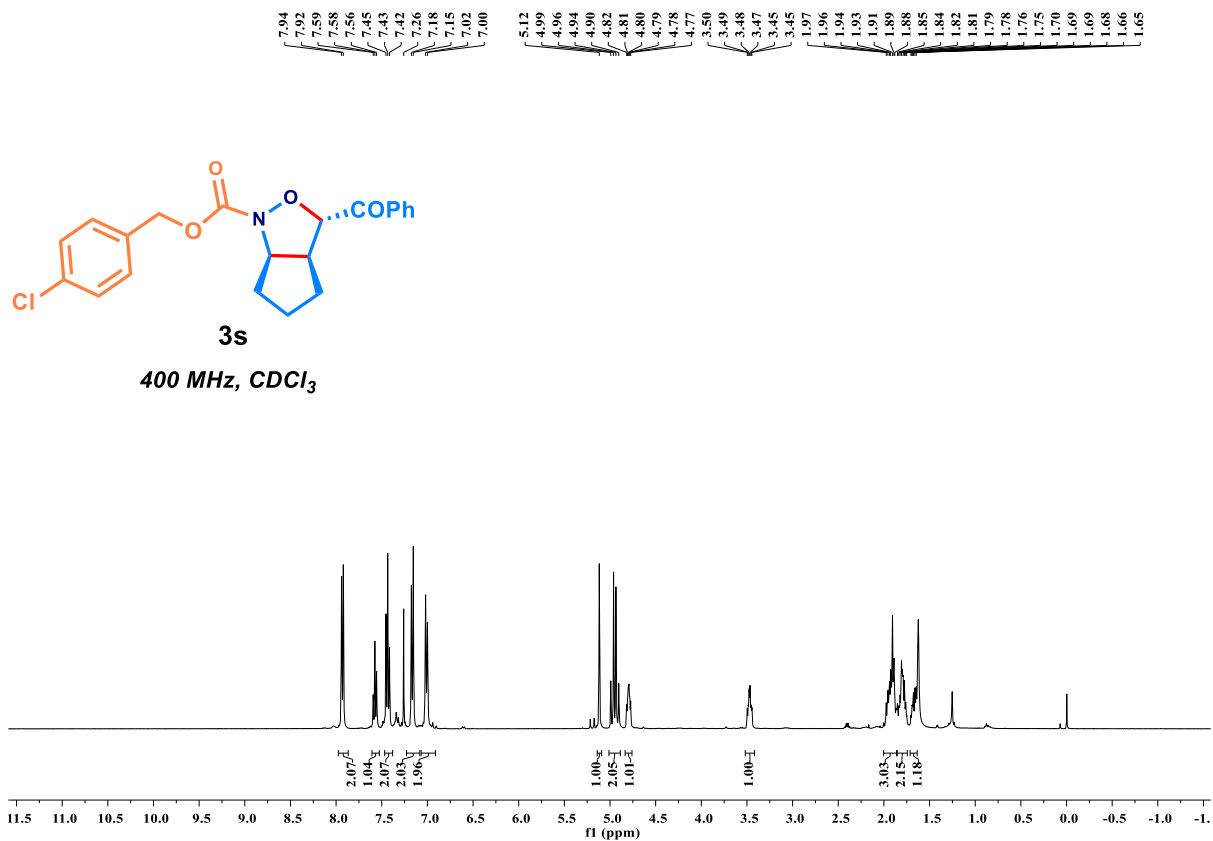
**3p**  
101 MHz, CDCl<sub>3</sub>

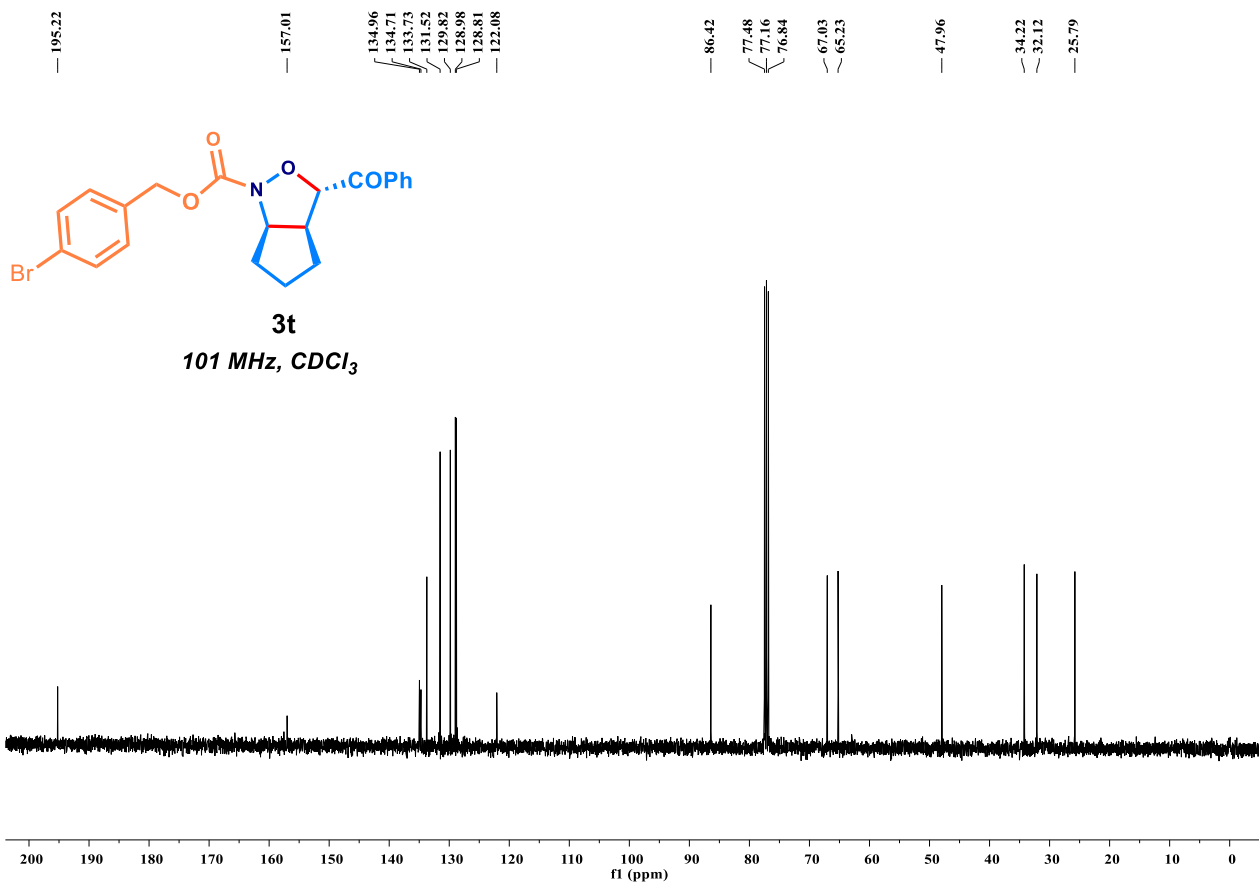
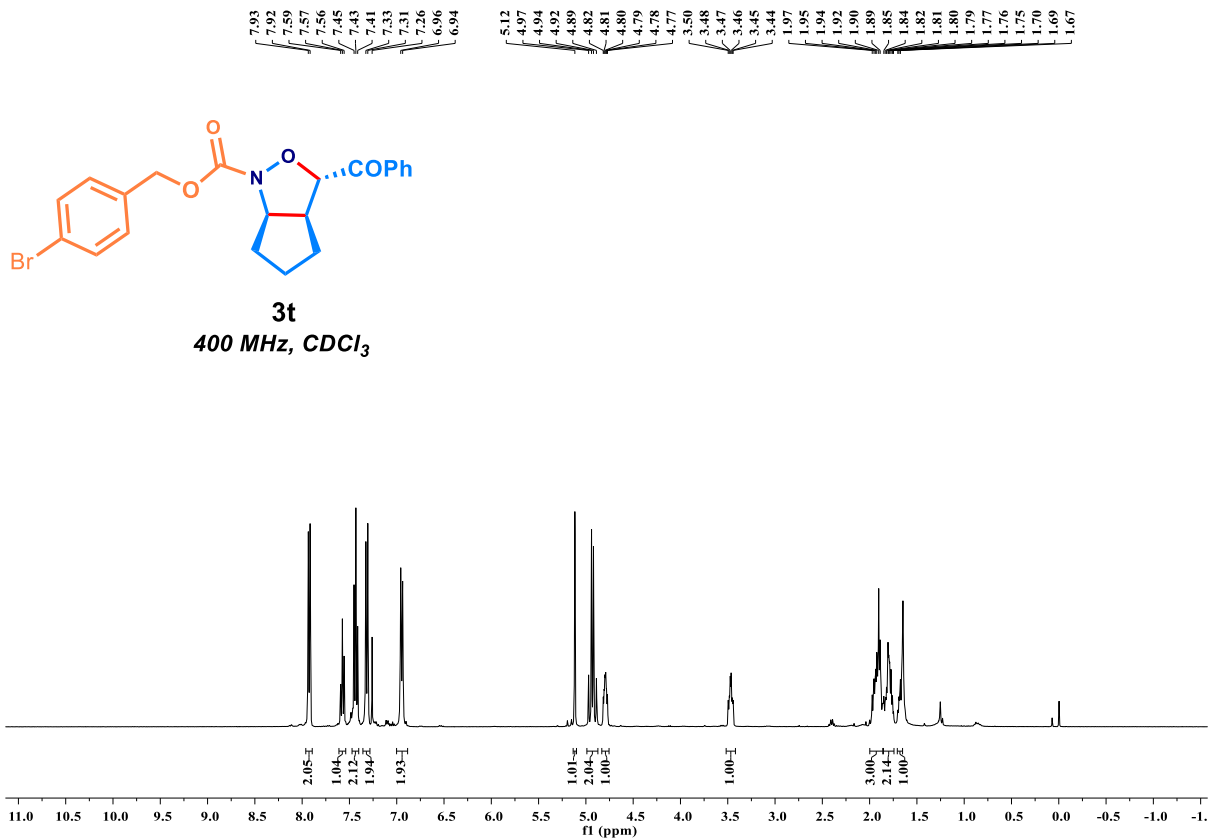


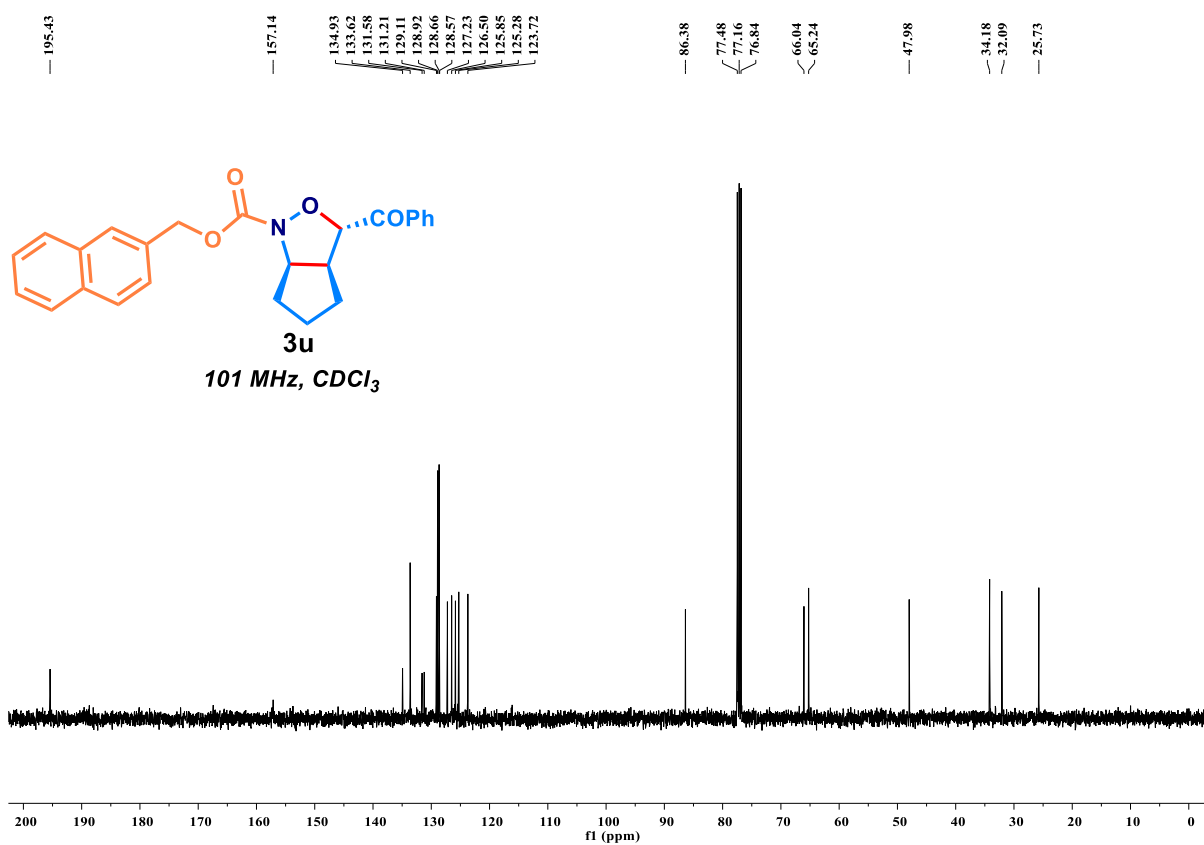
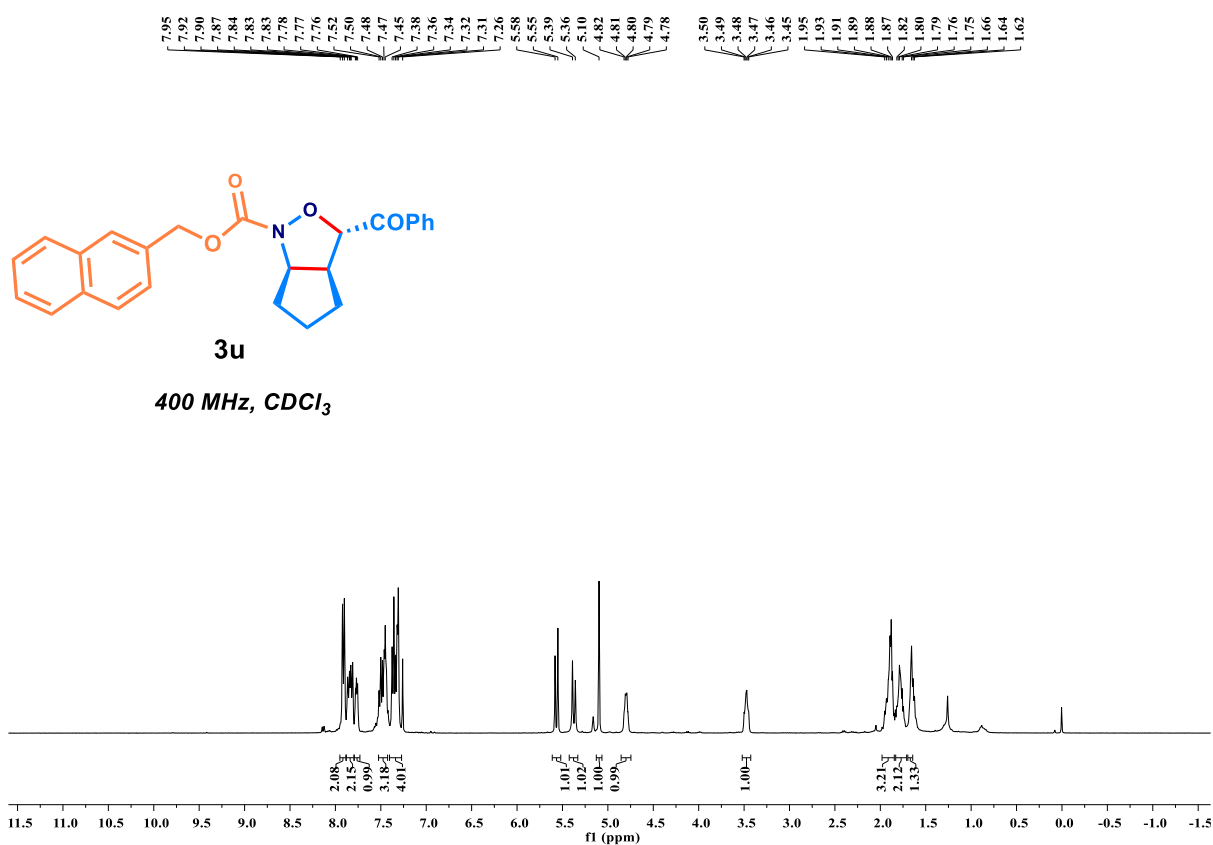


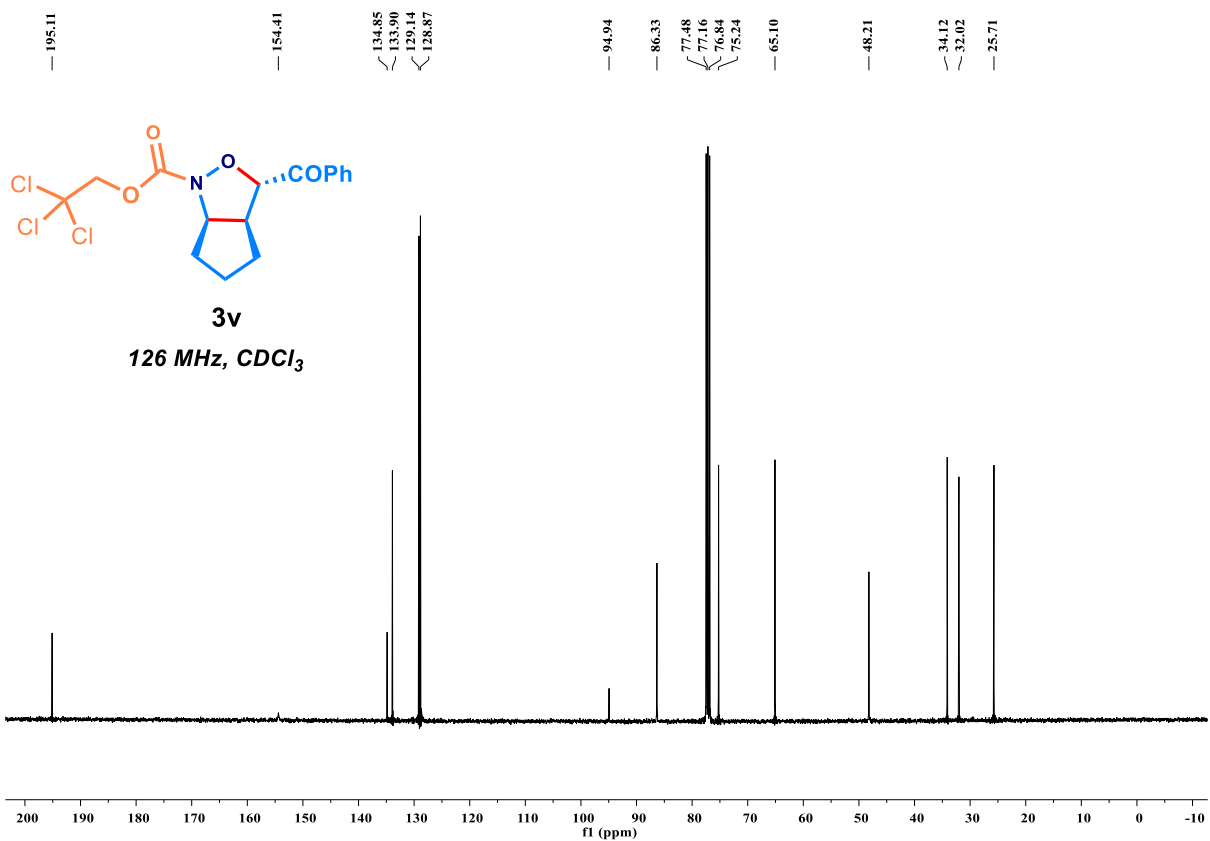
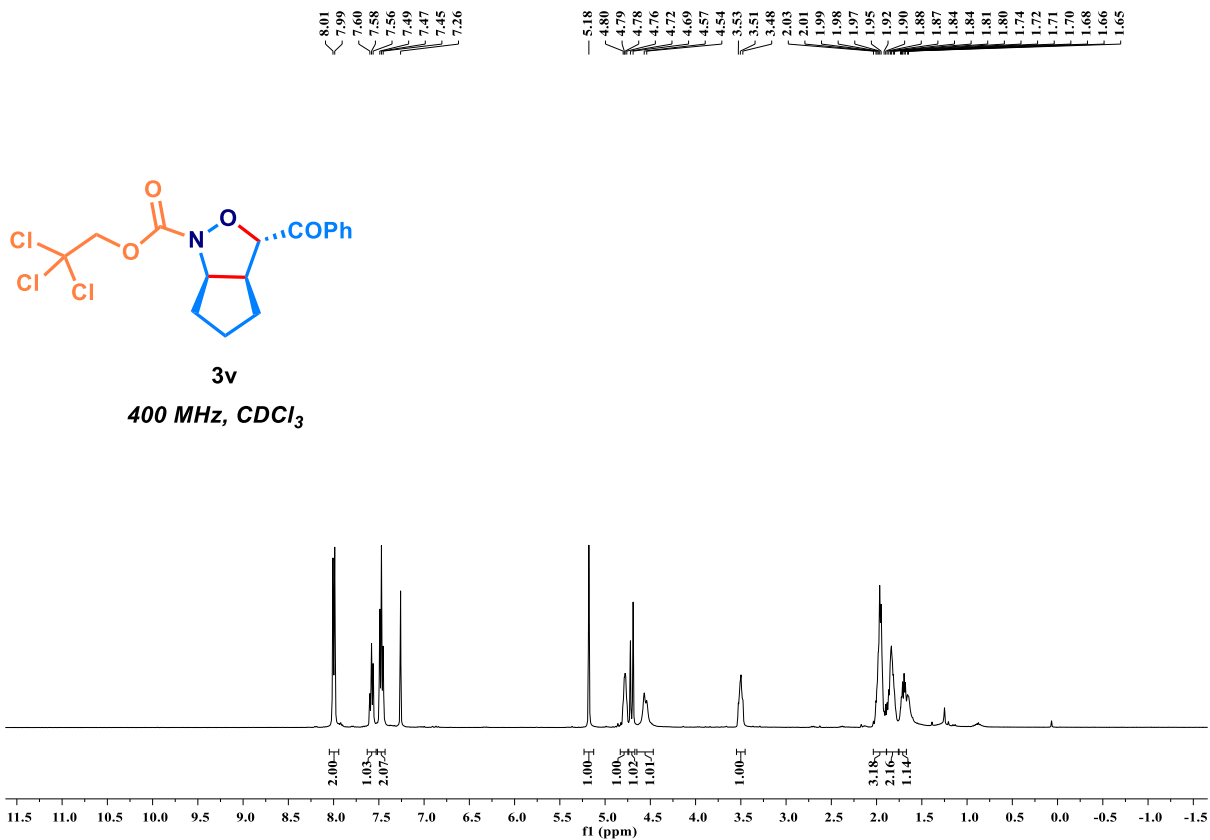


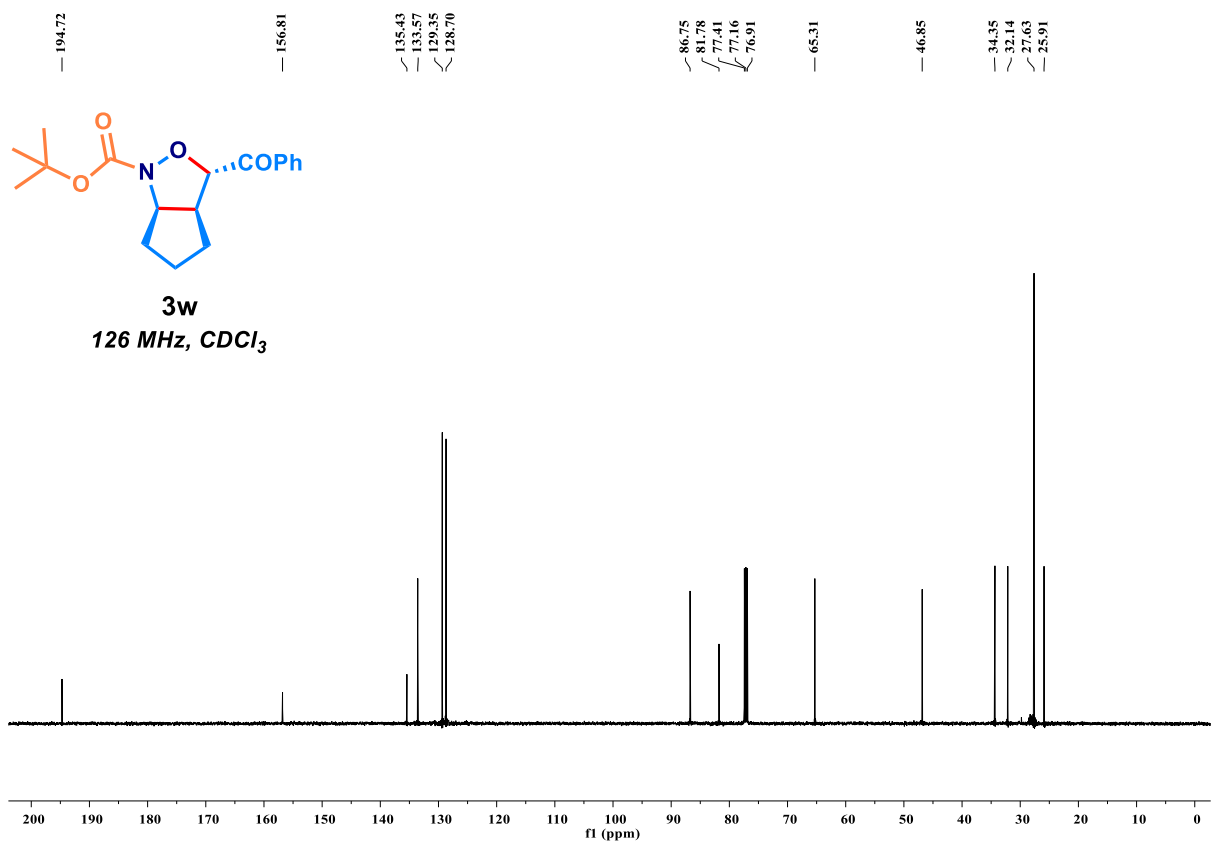
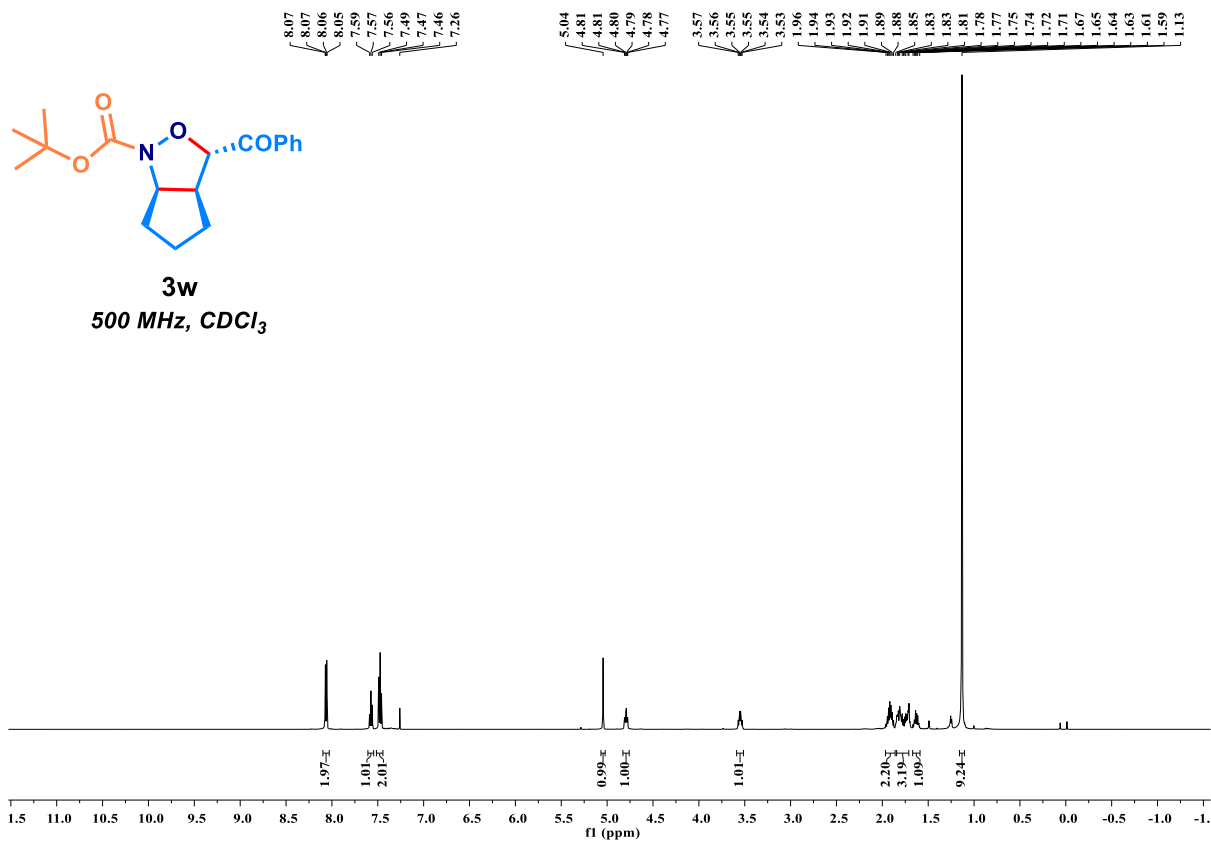


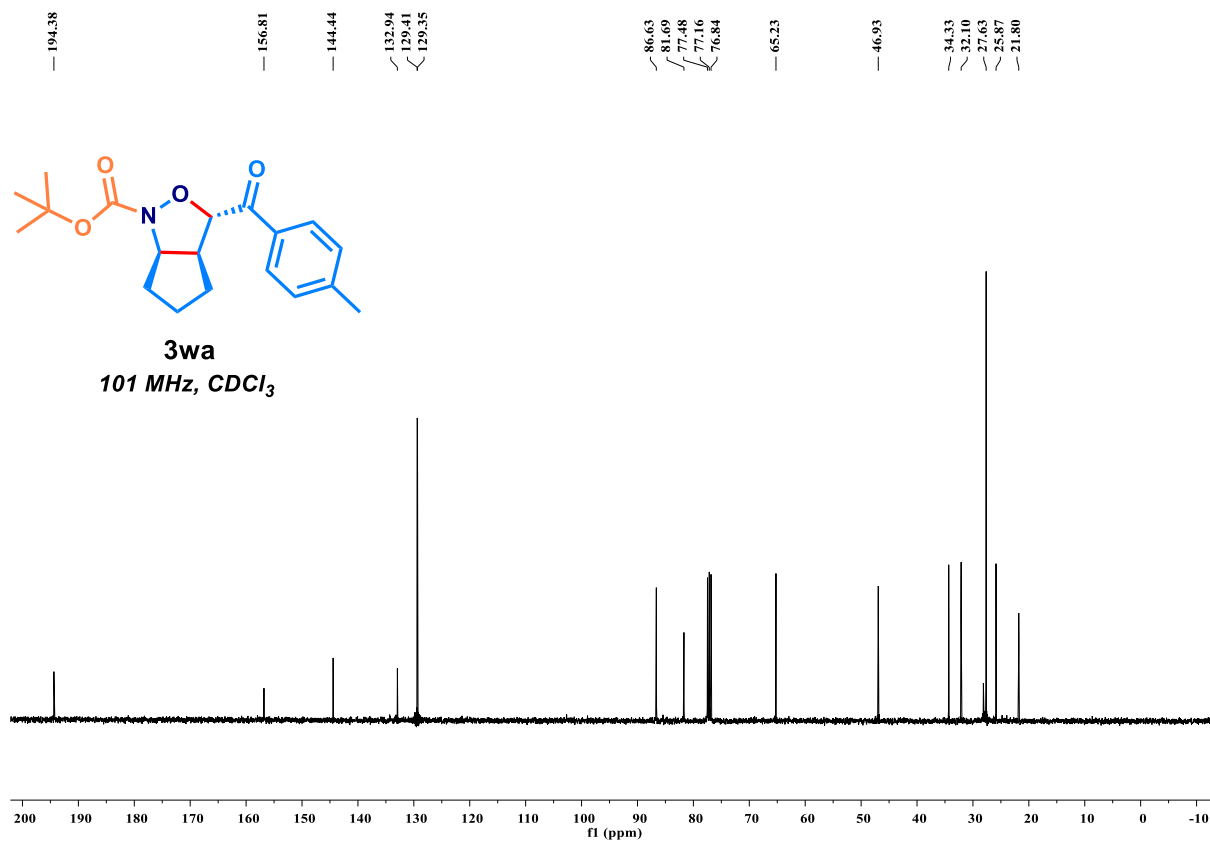
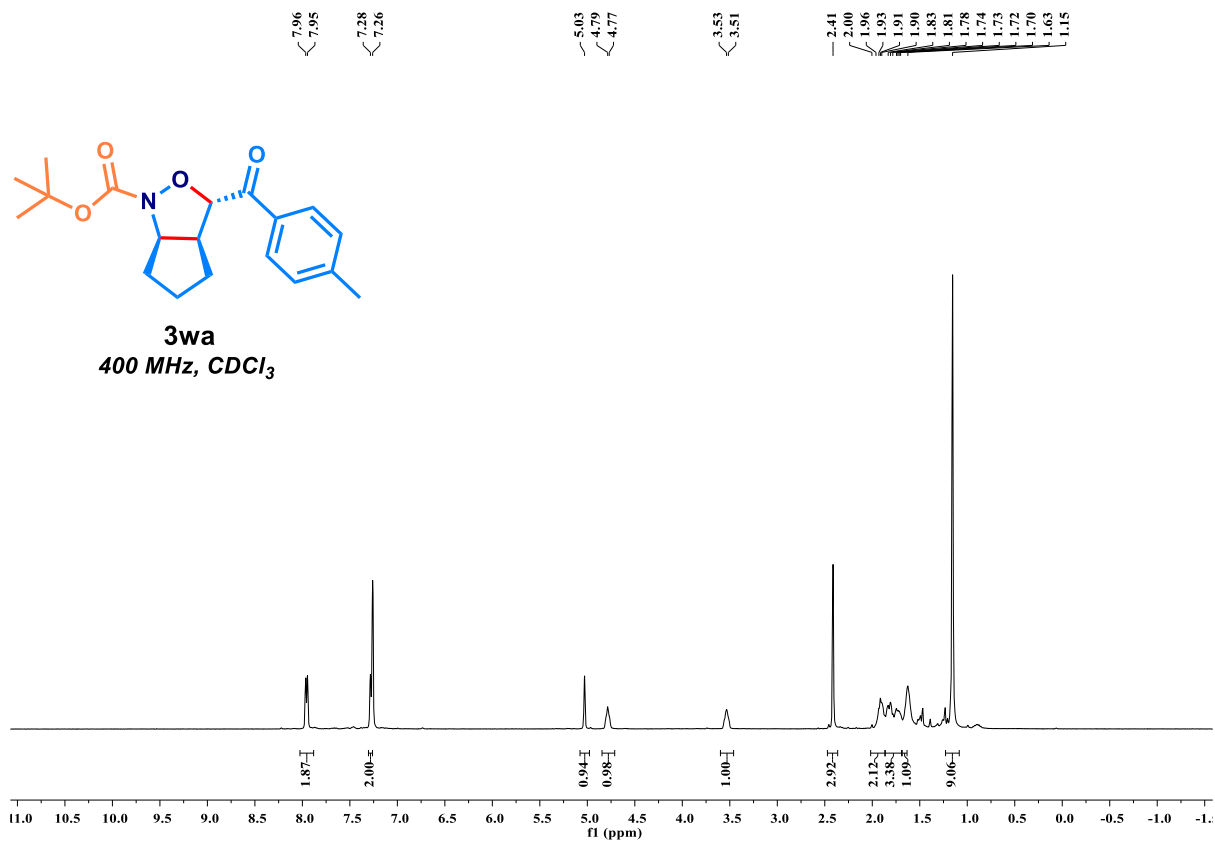




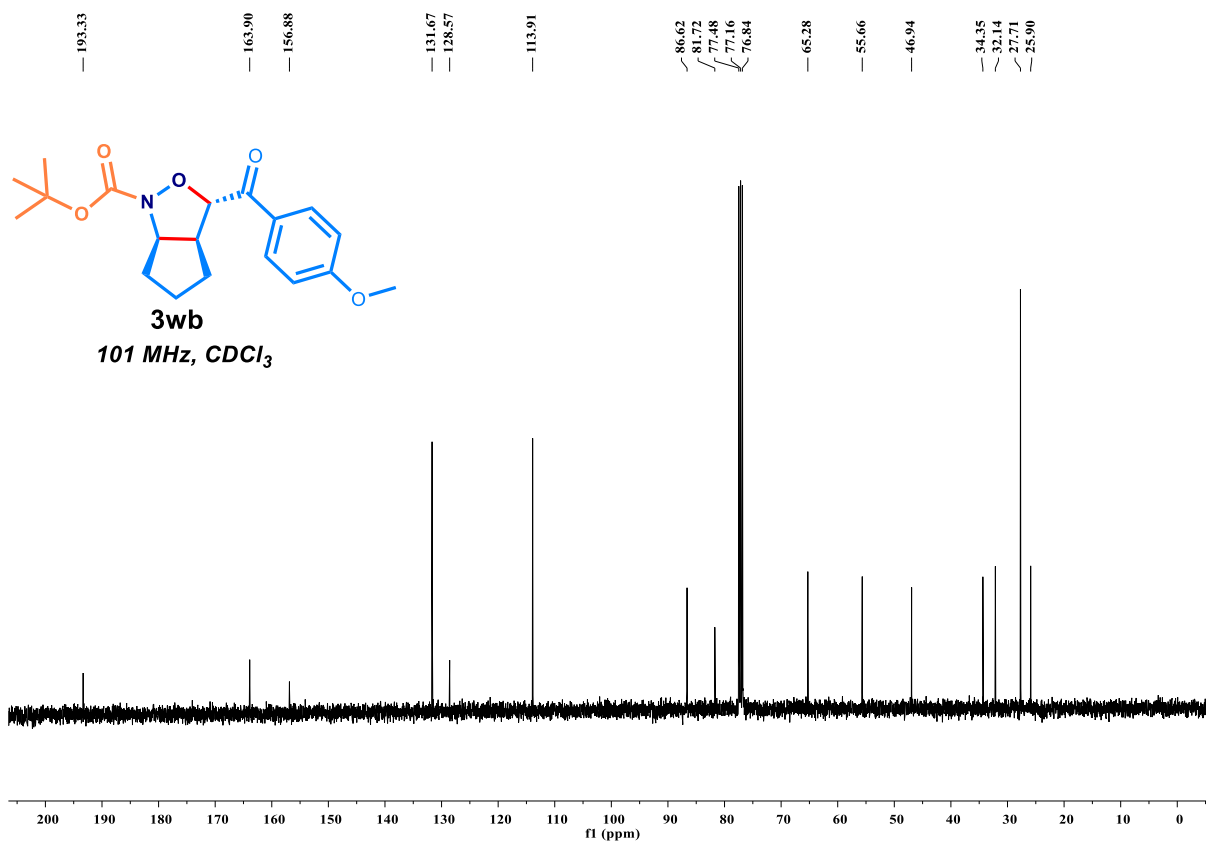
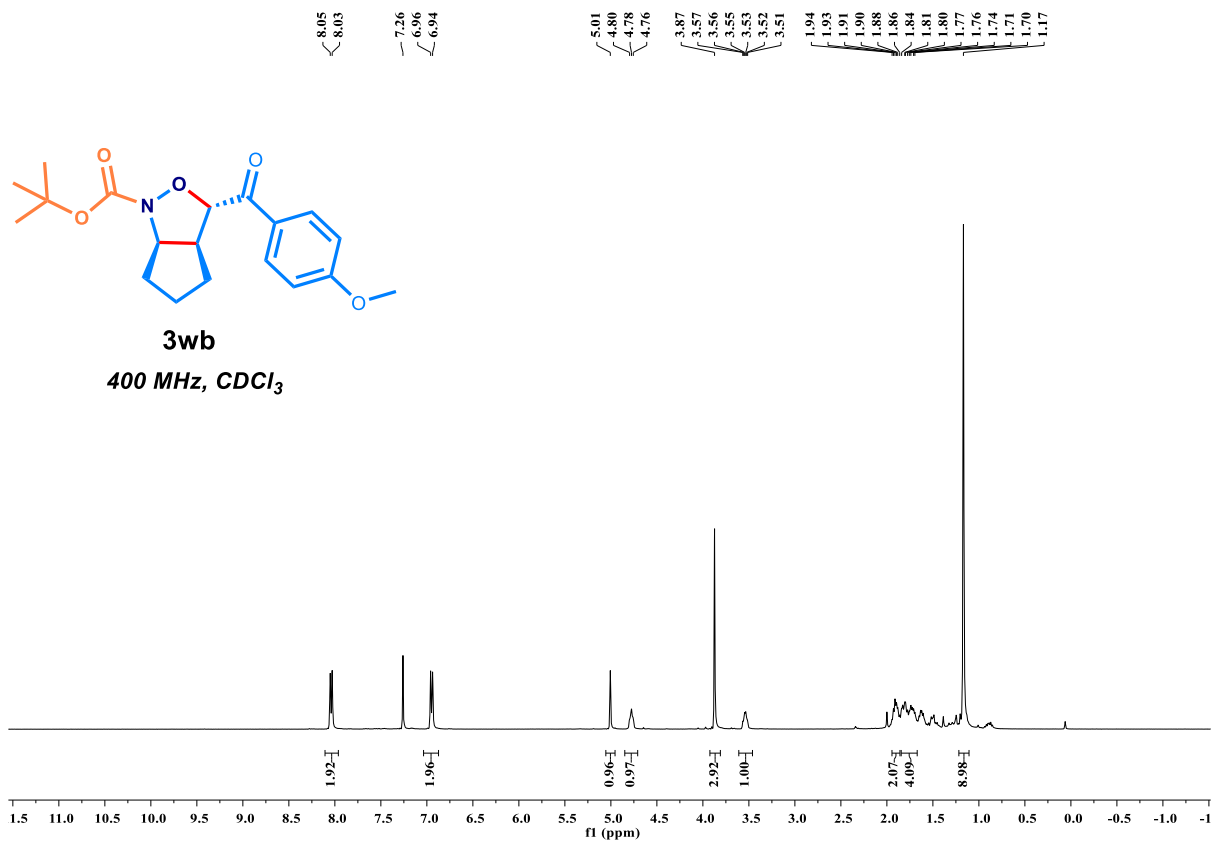


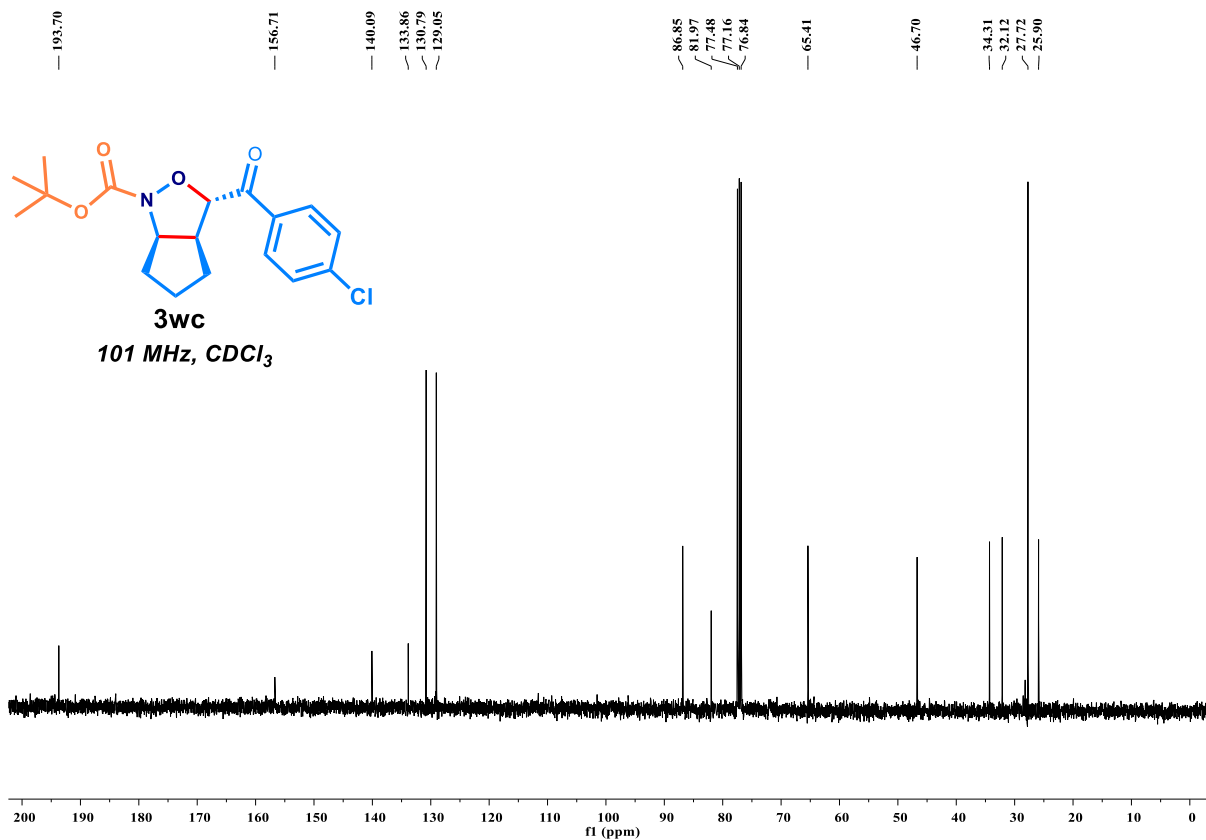
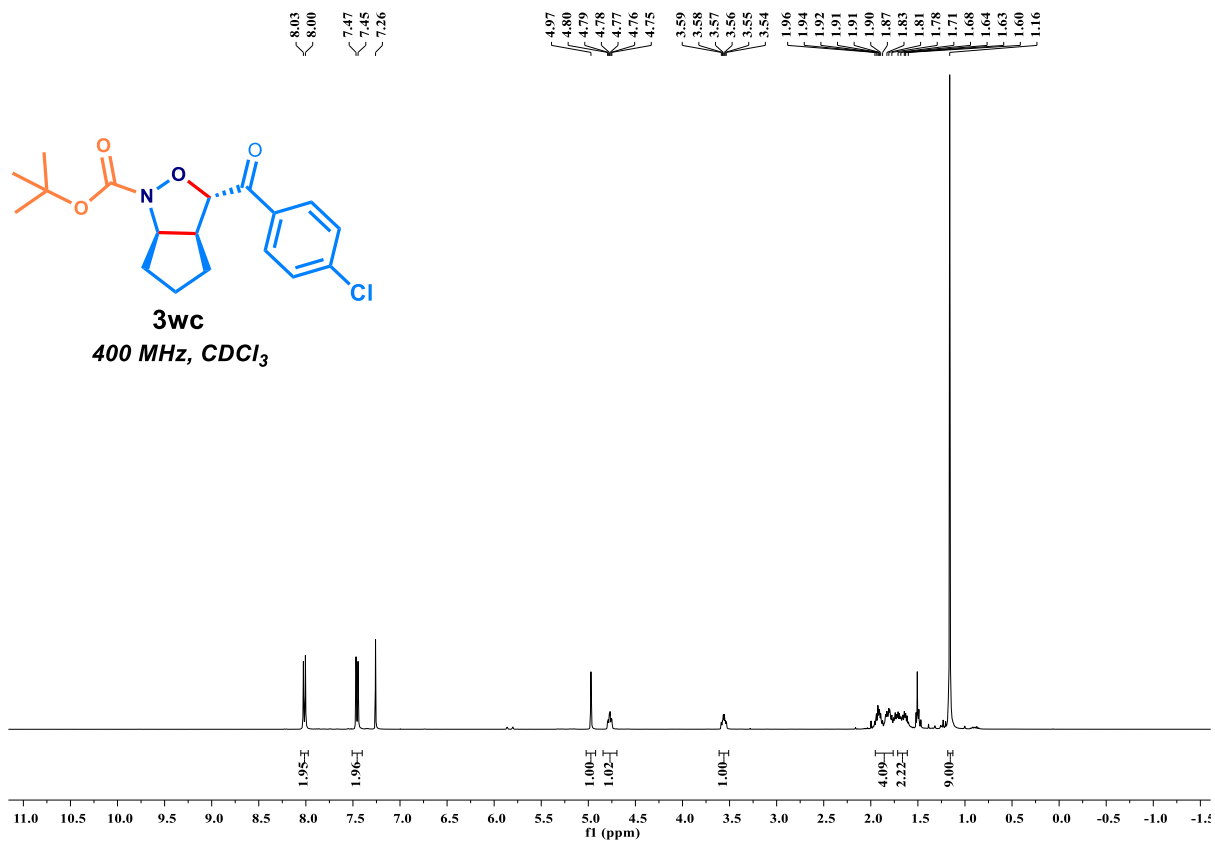


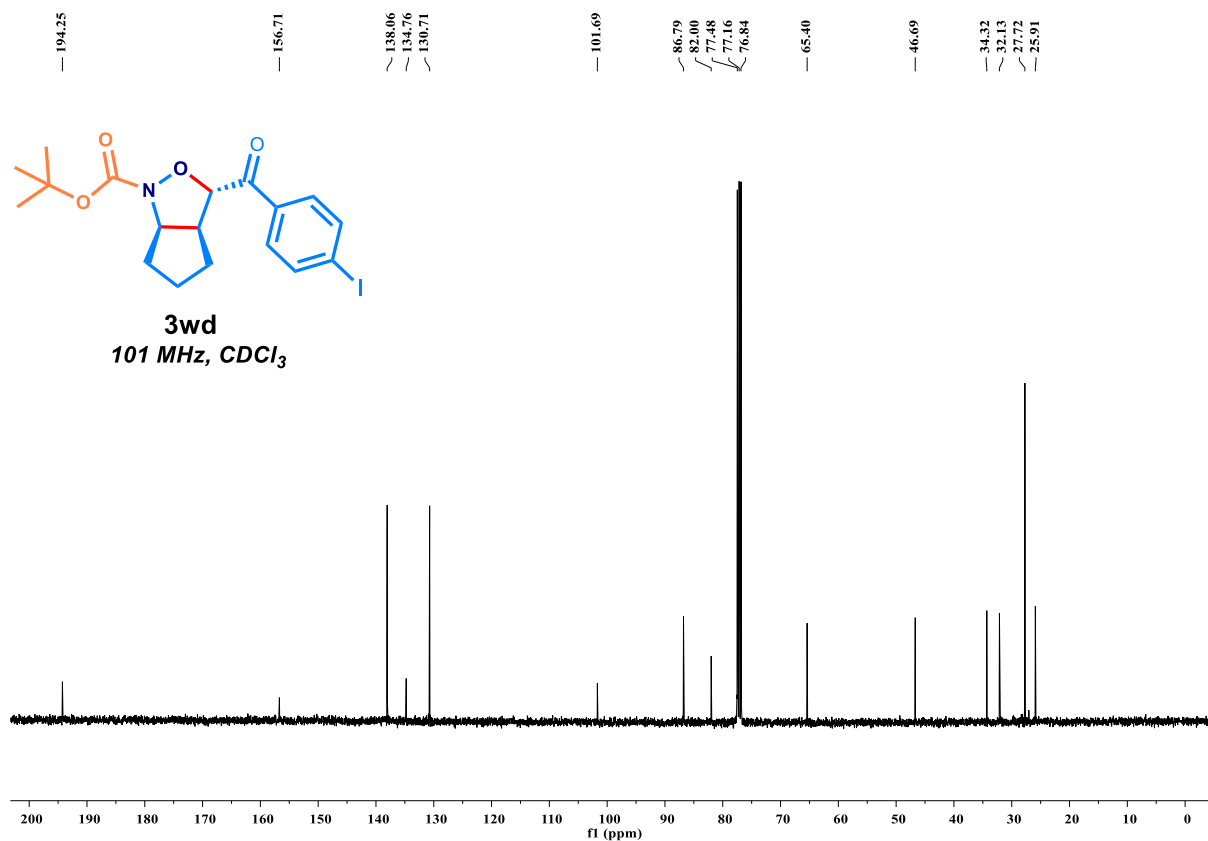
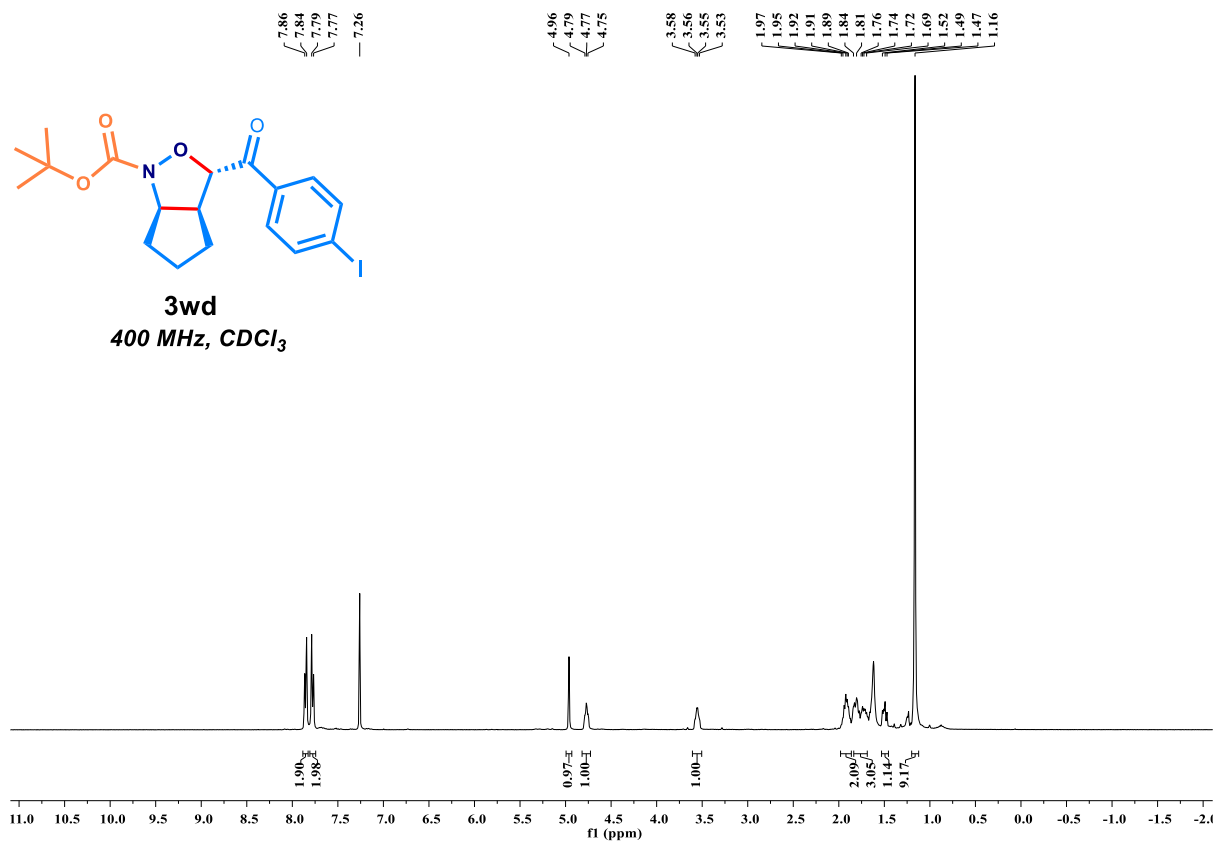


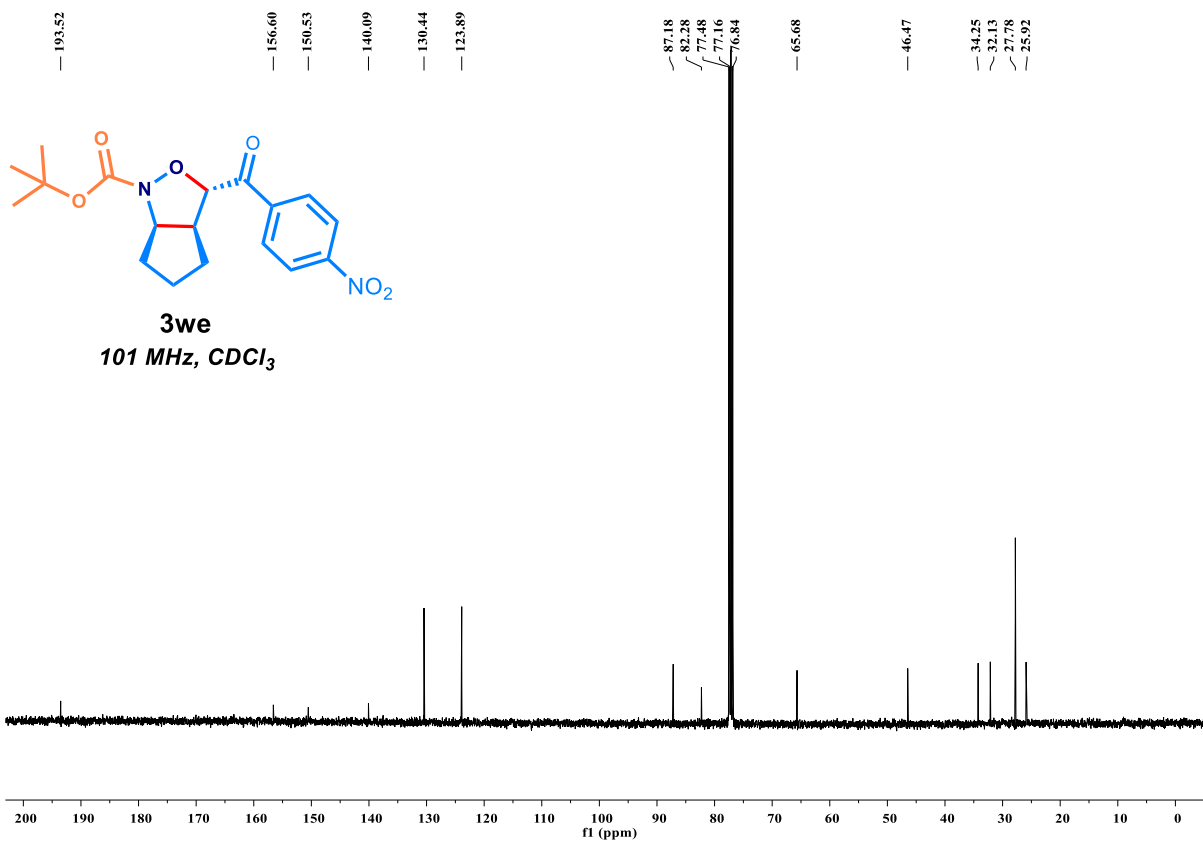
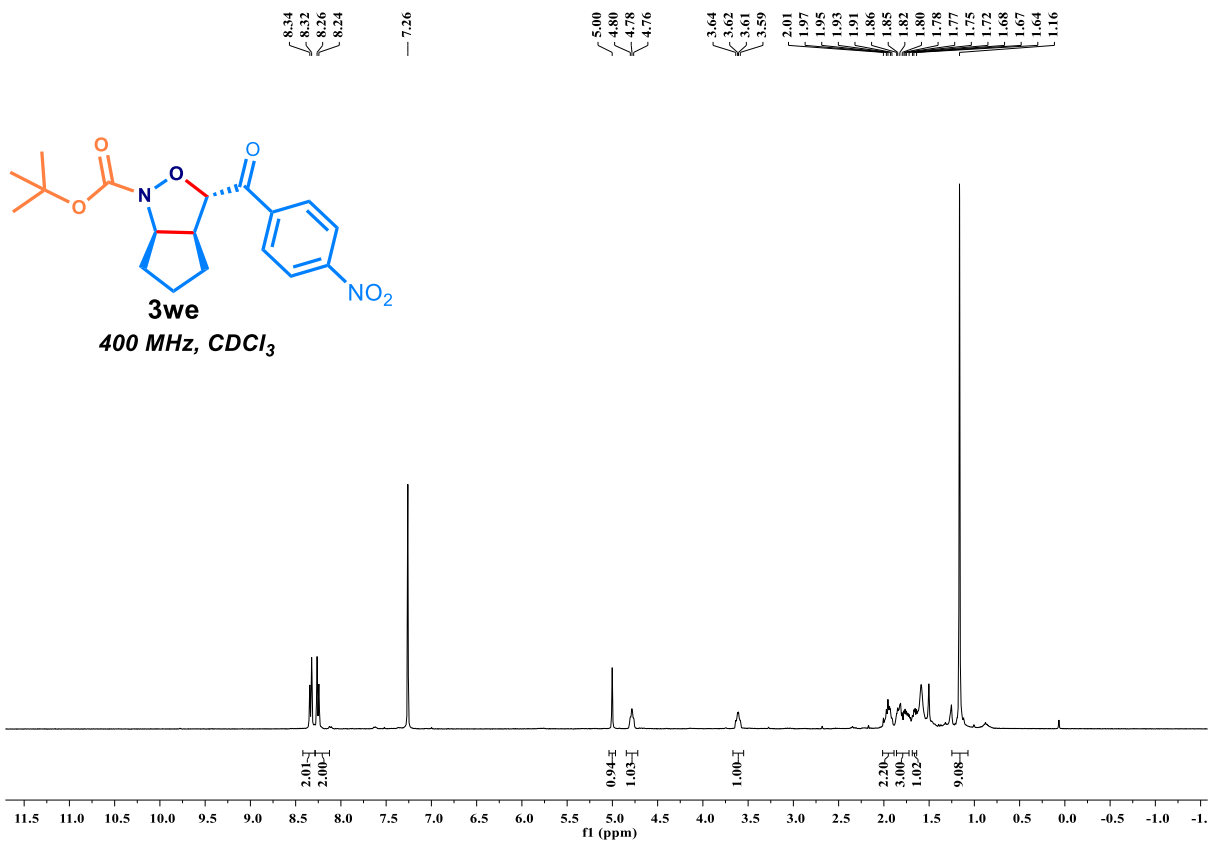


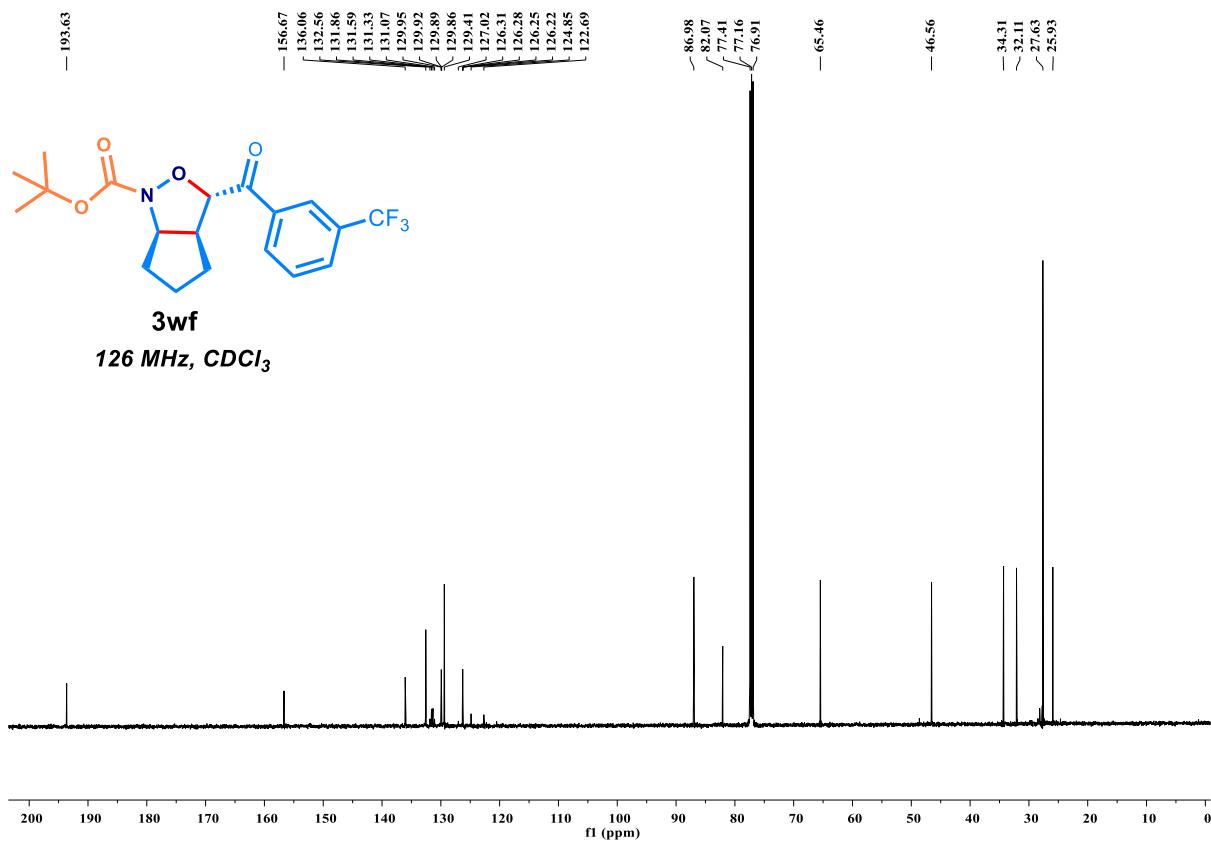
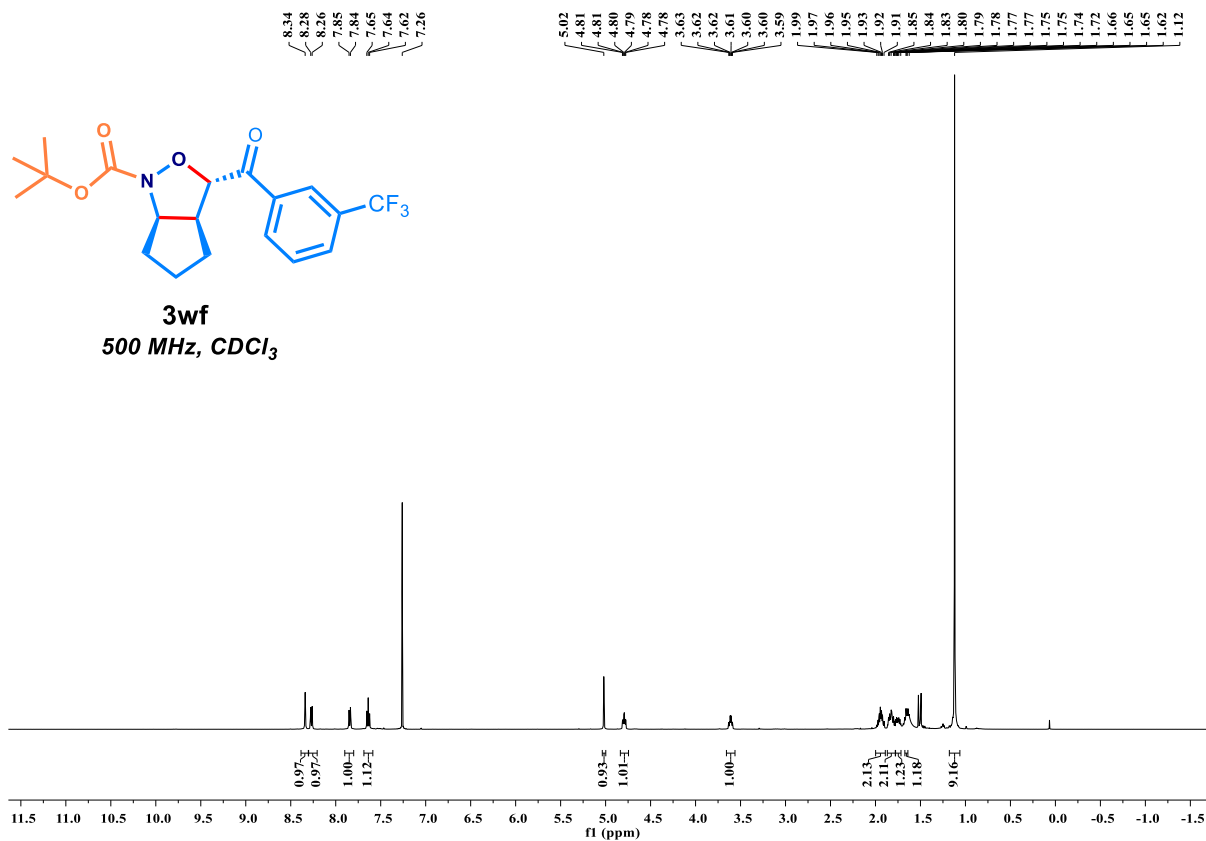


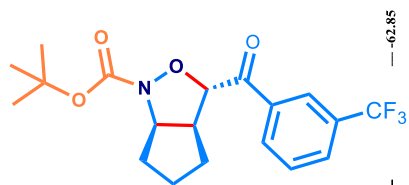






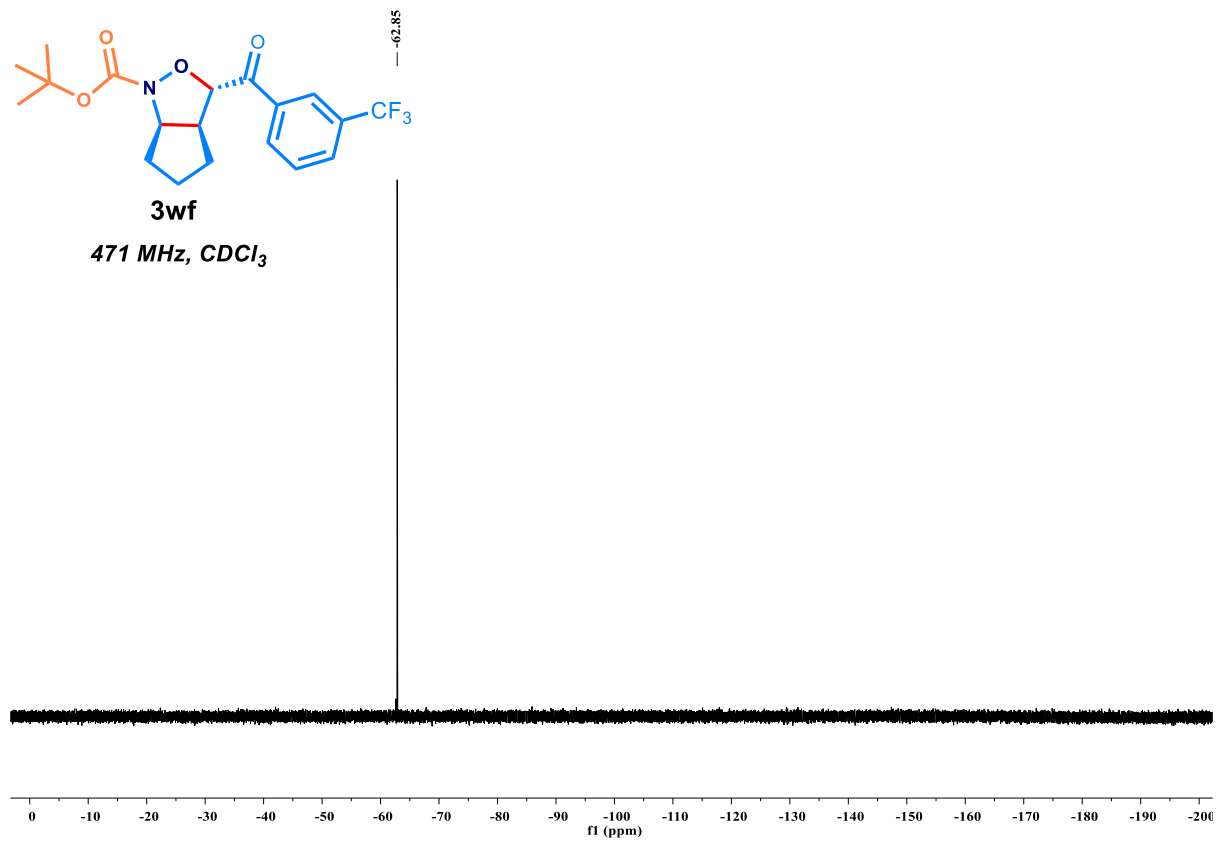


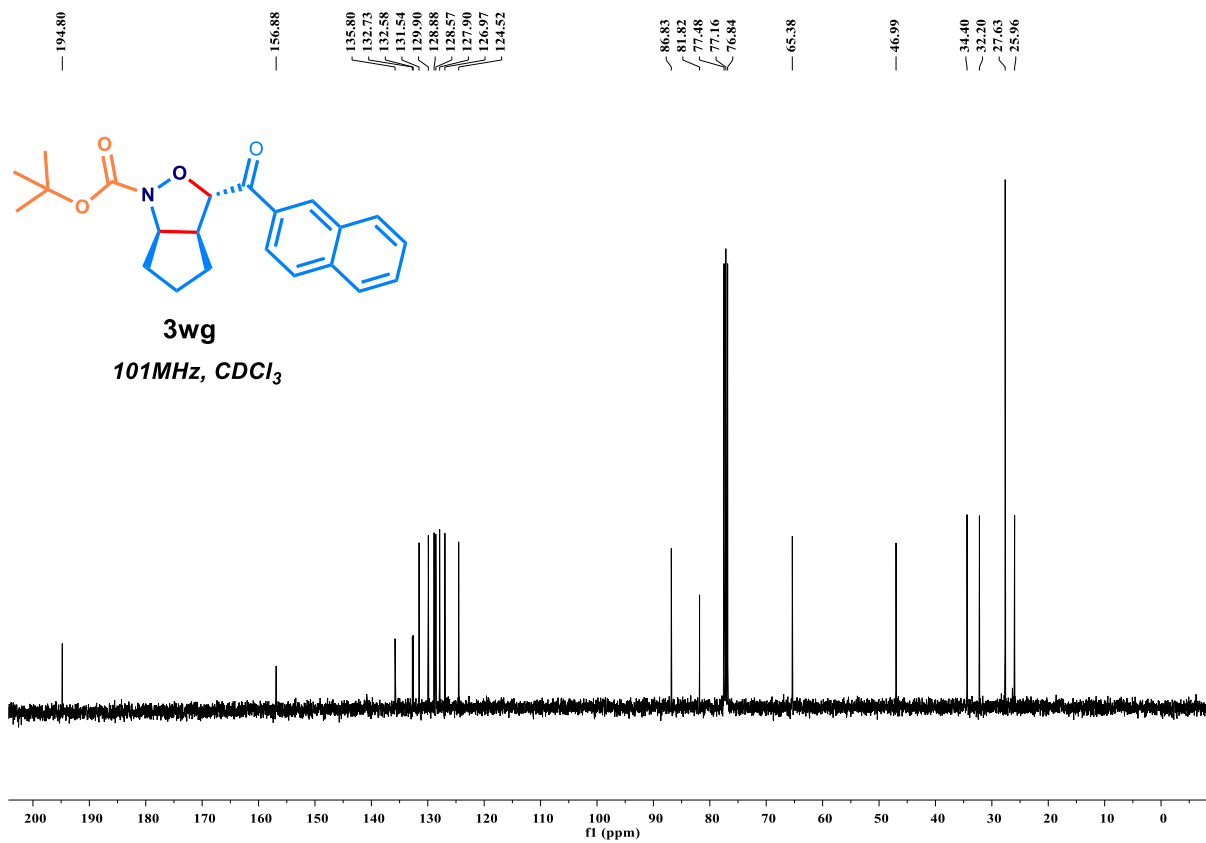
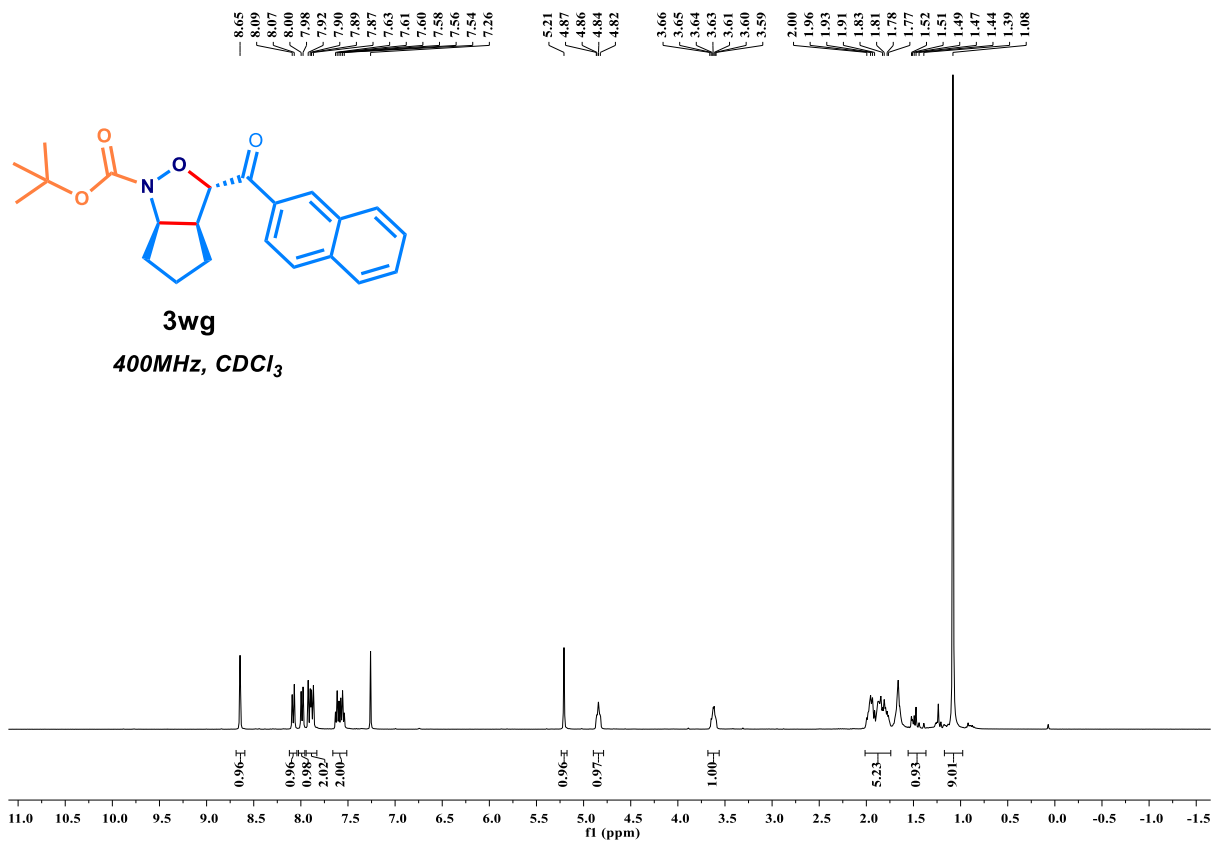


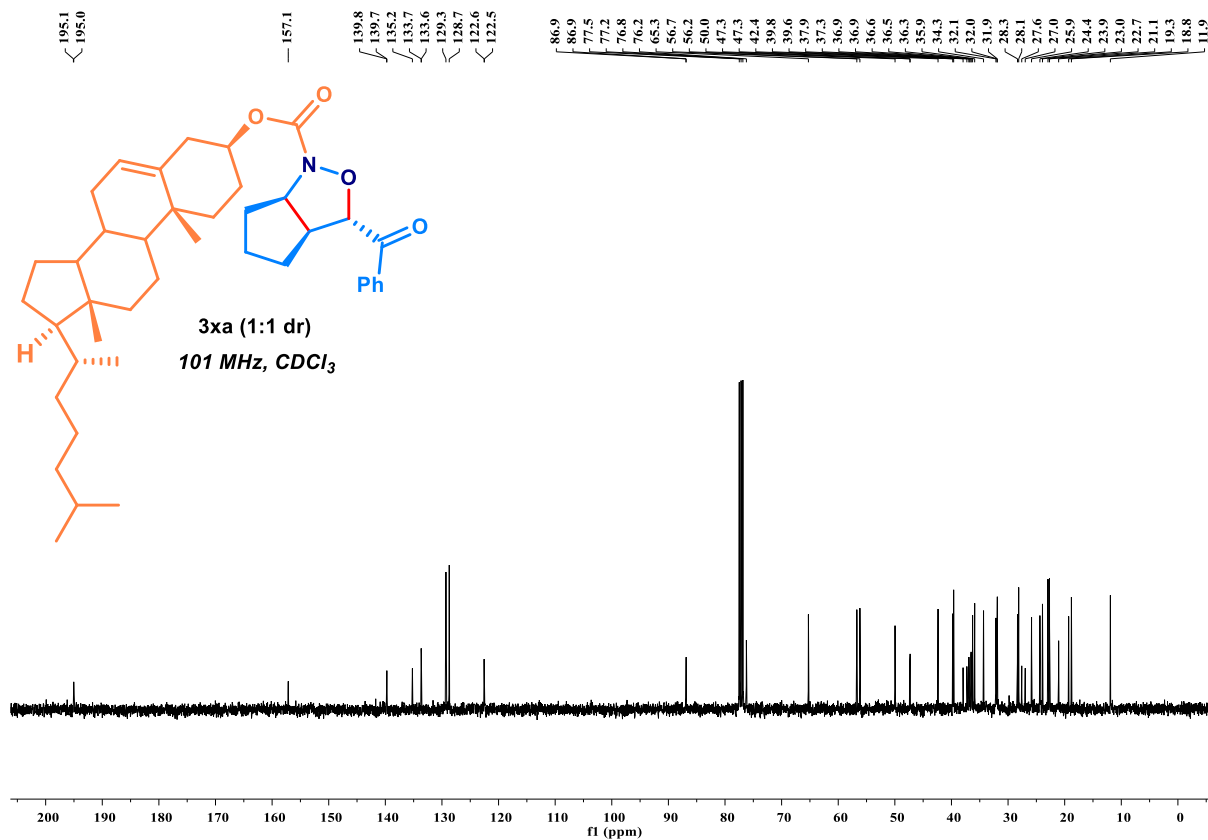
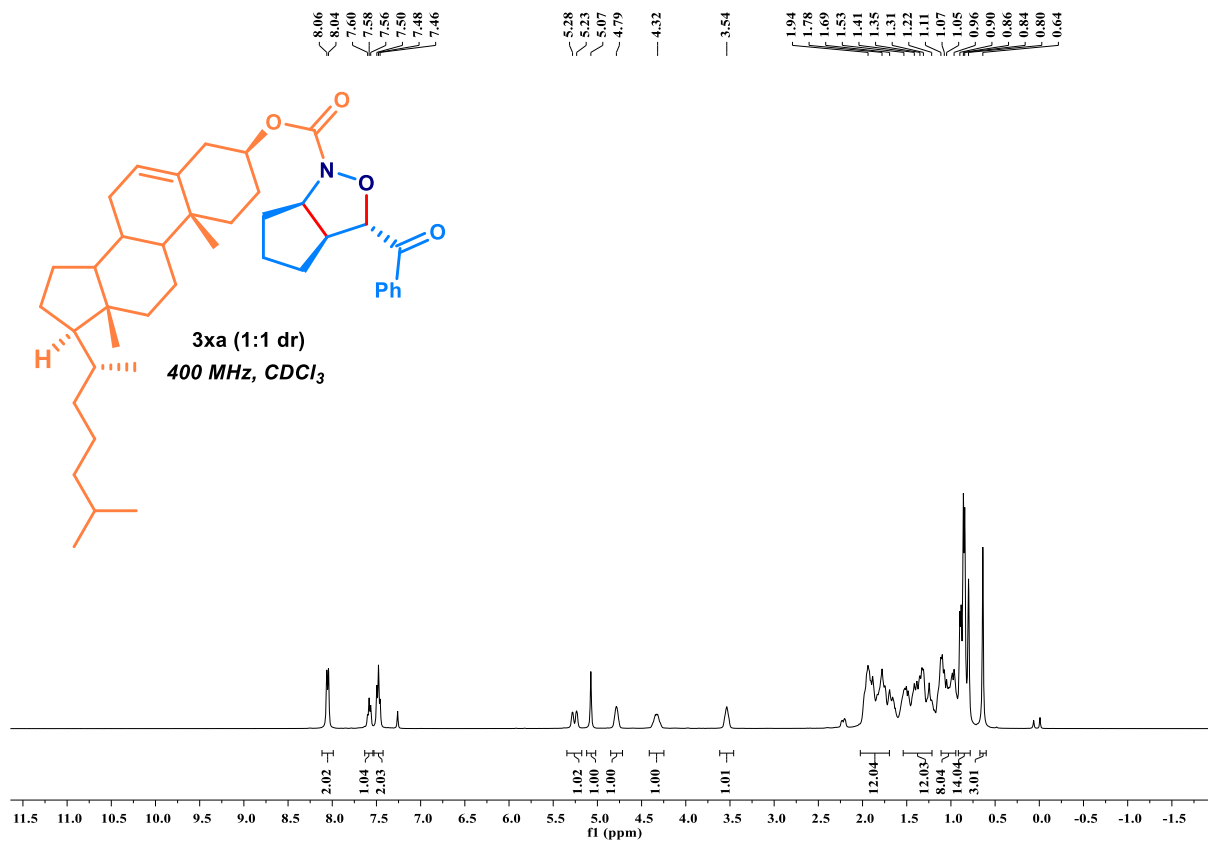


**3wf**

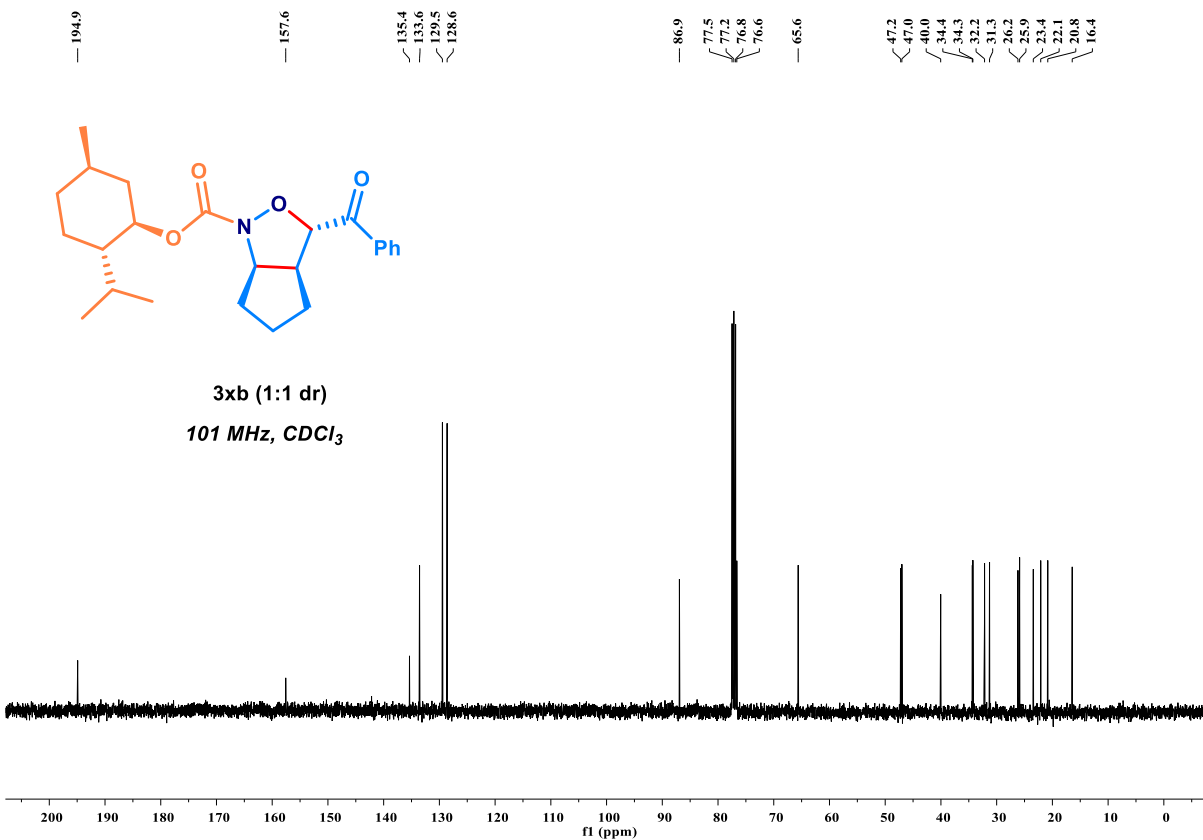
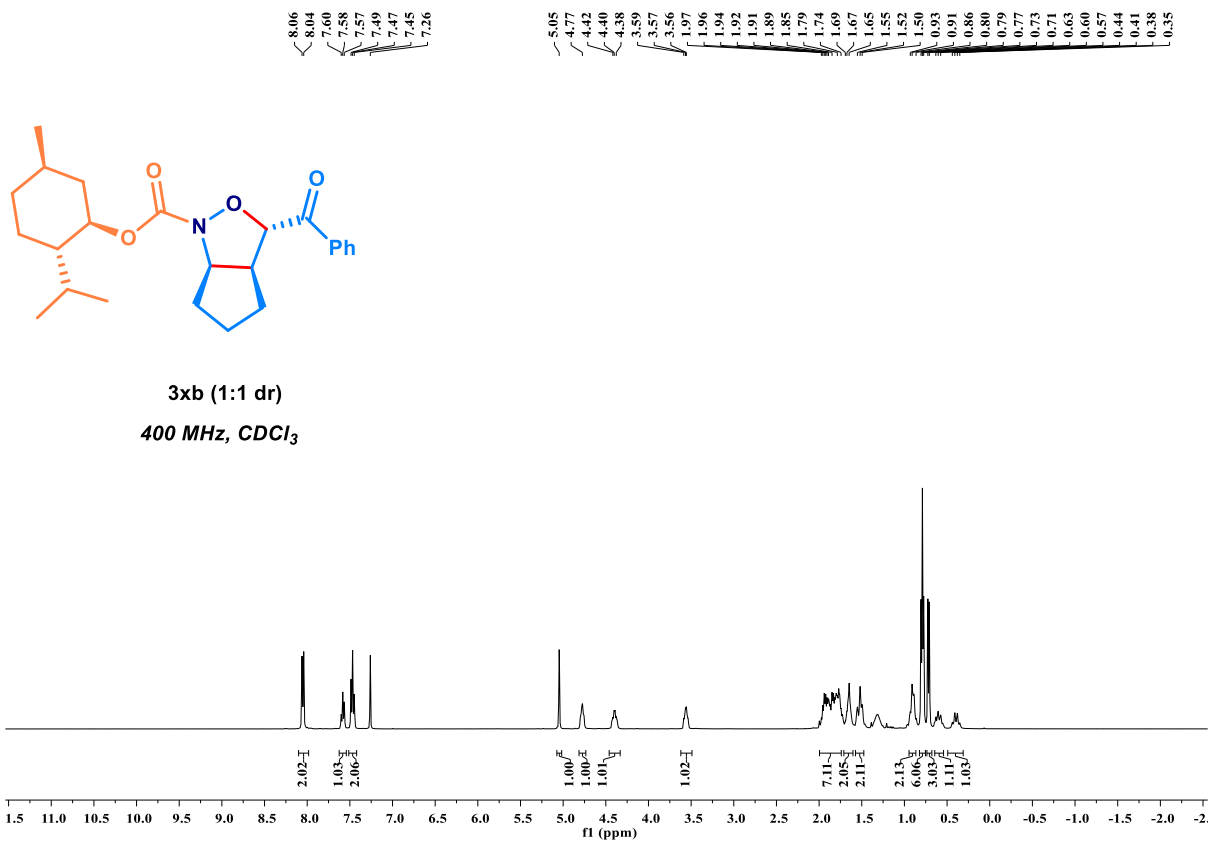
471 MHz, CDCl<sub>3</sub>

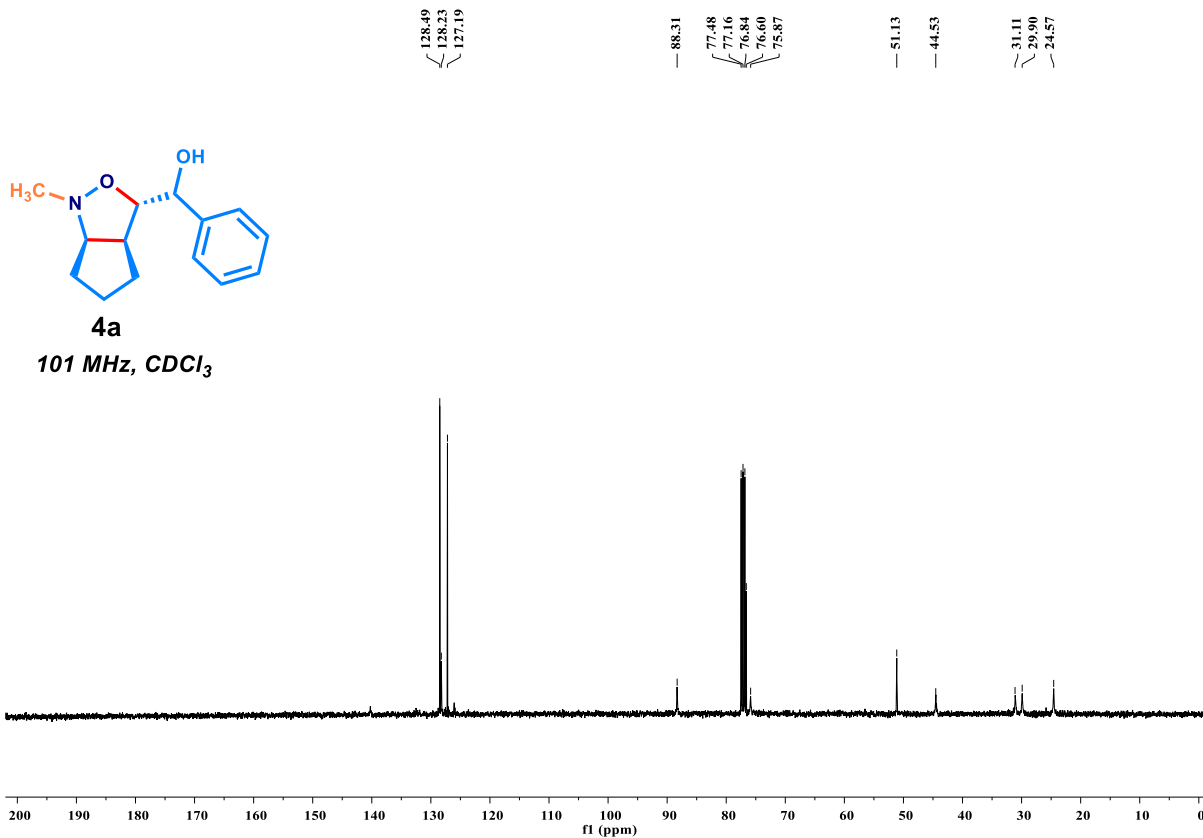
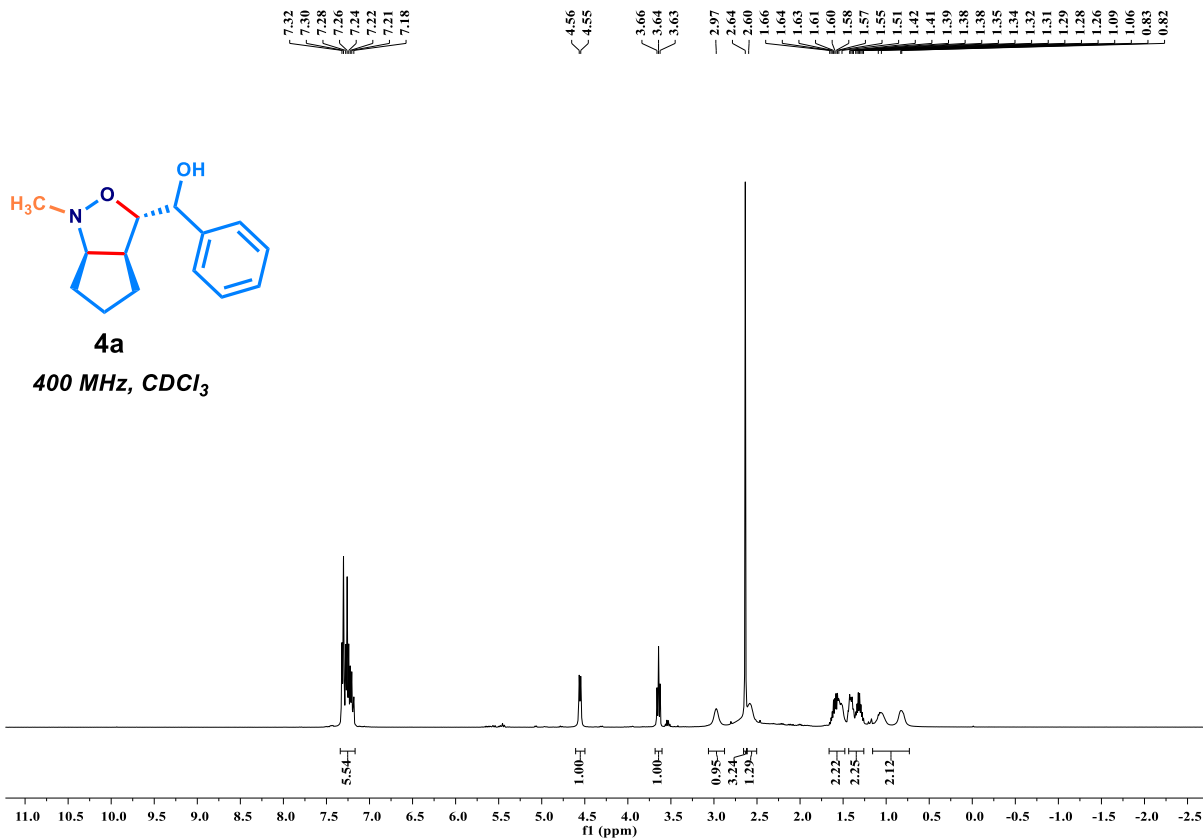


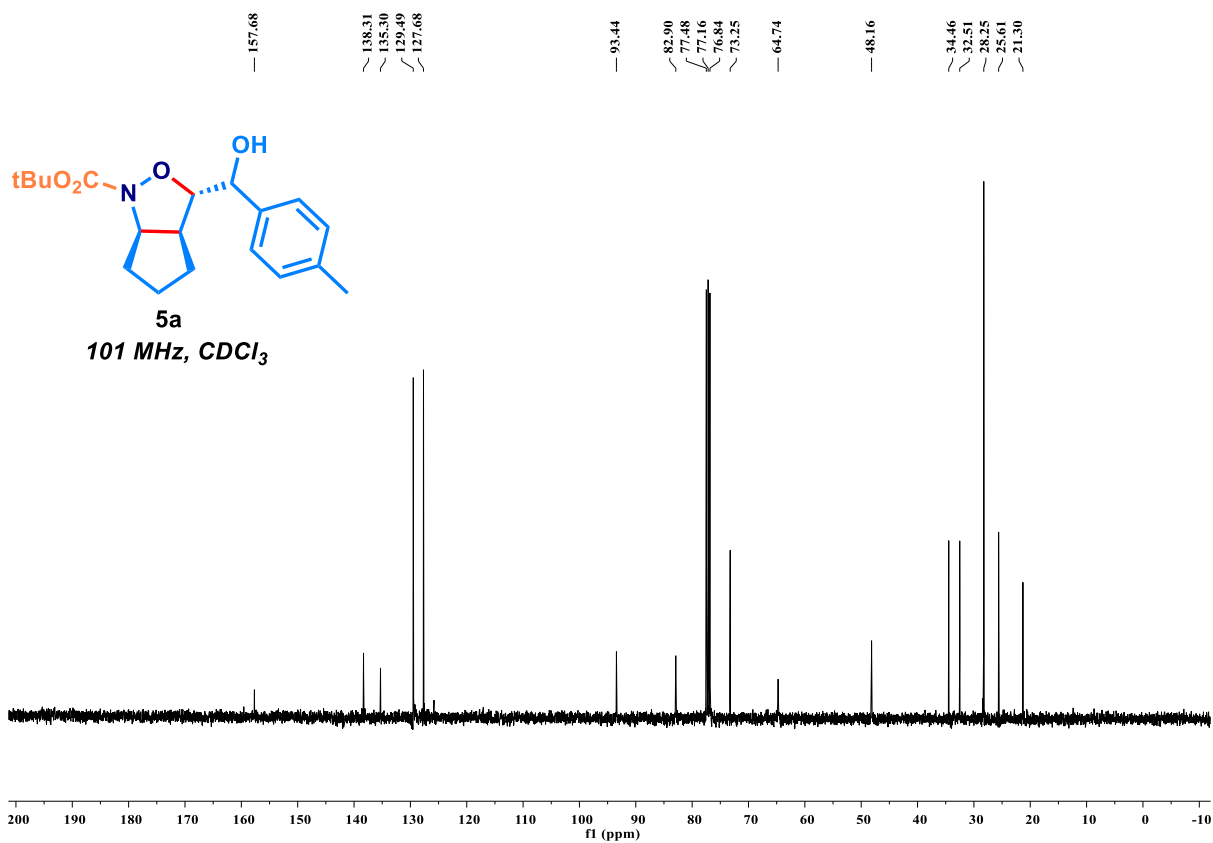
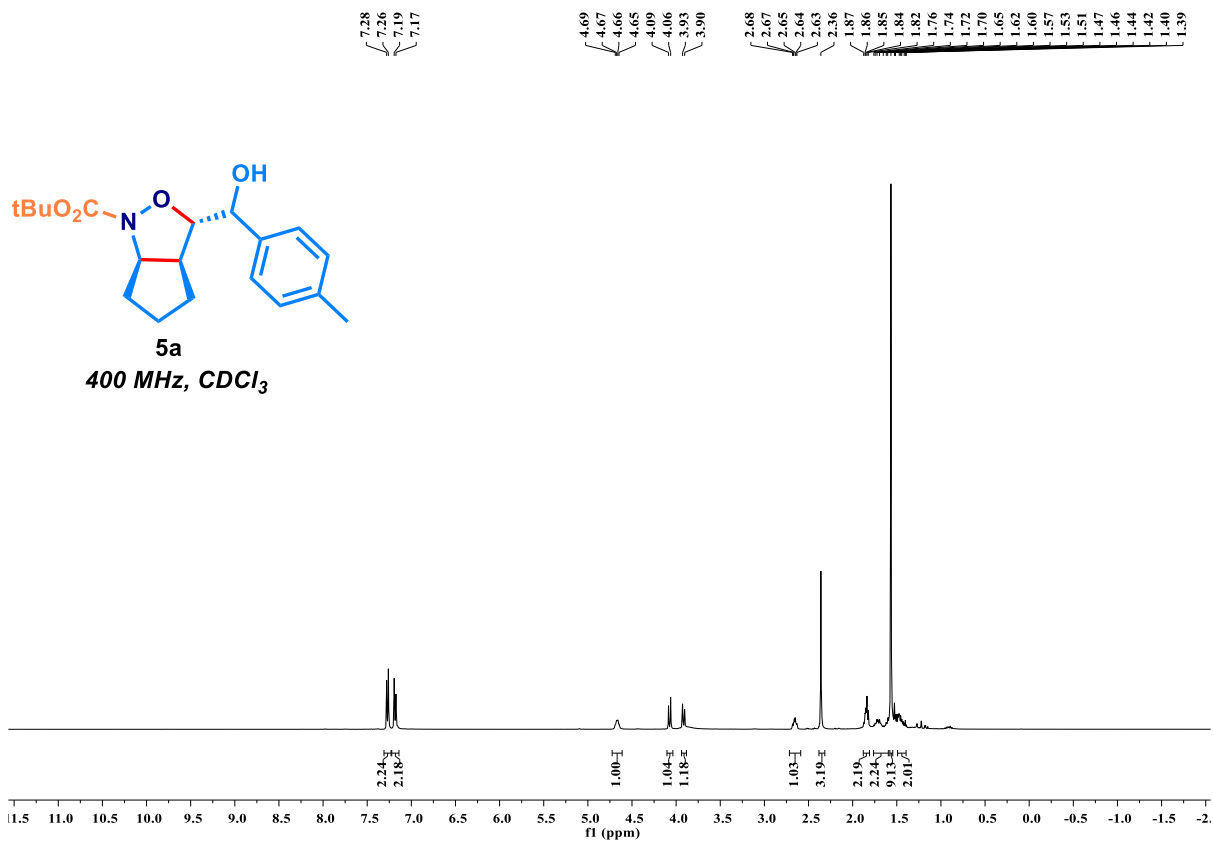


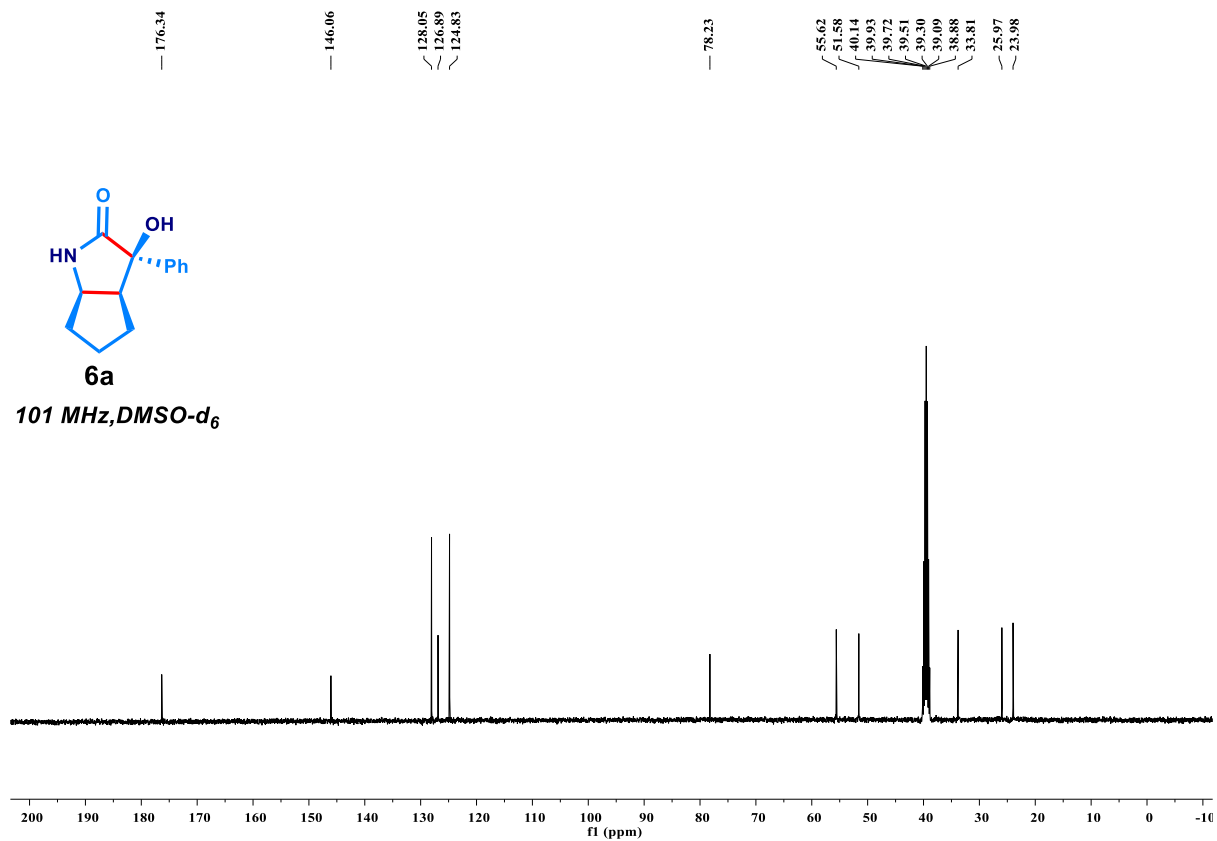
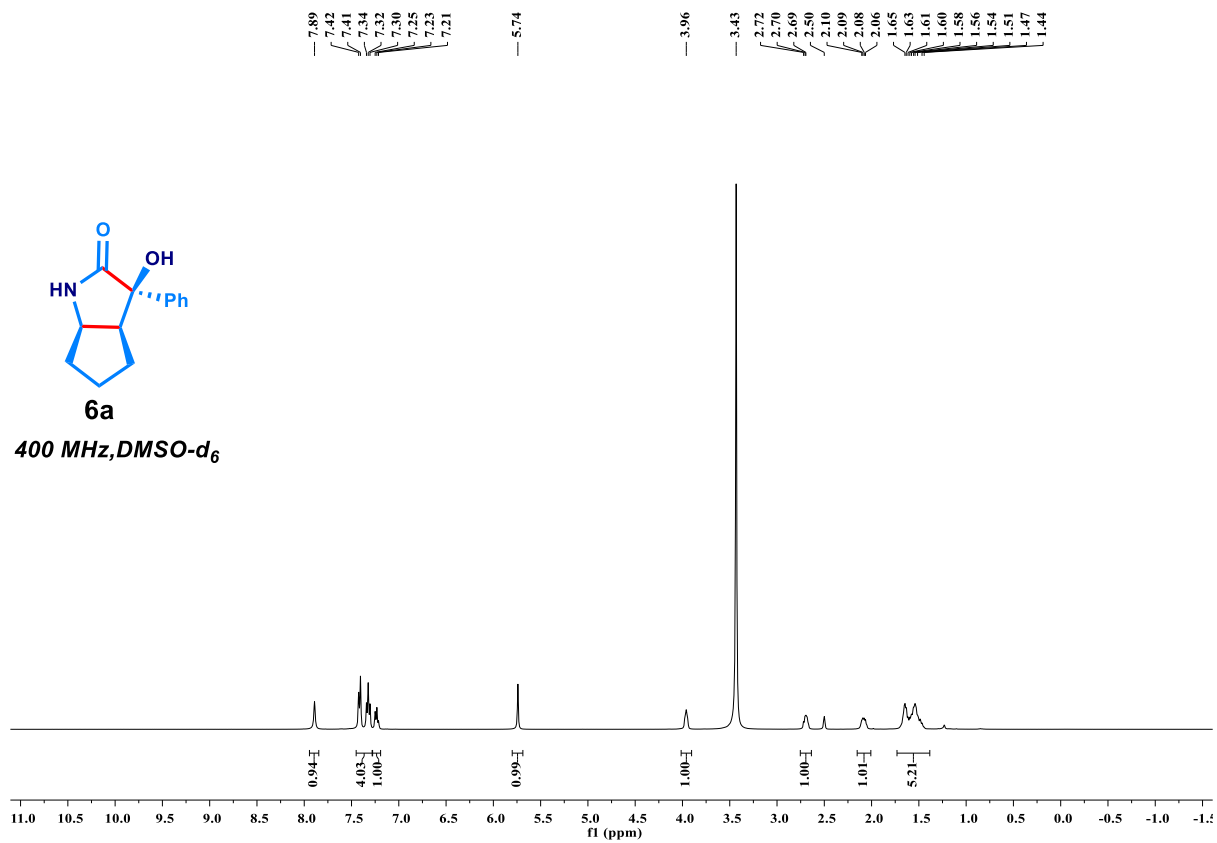


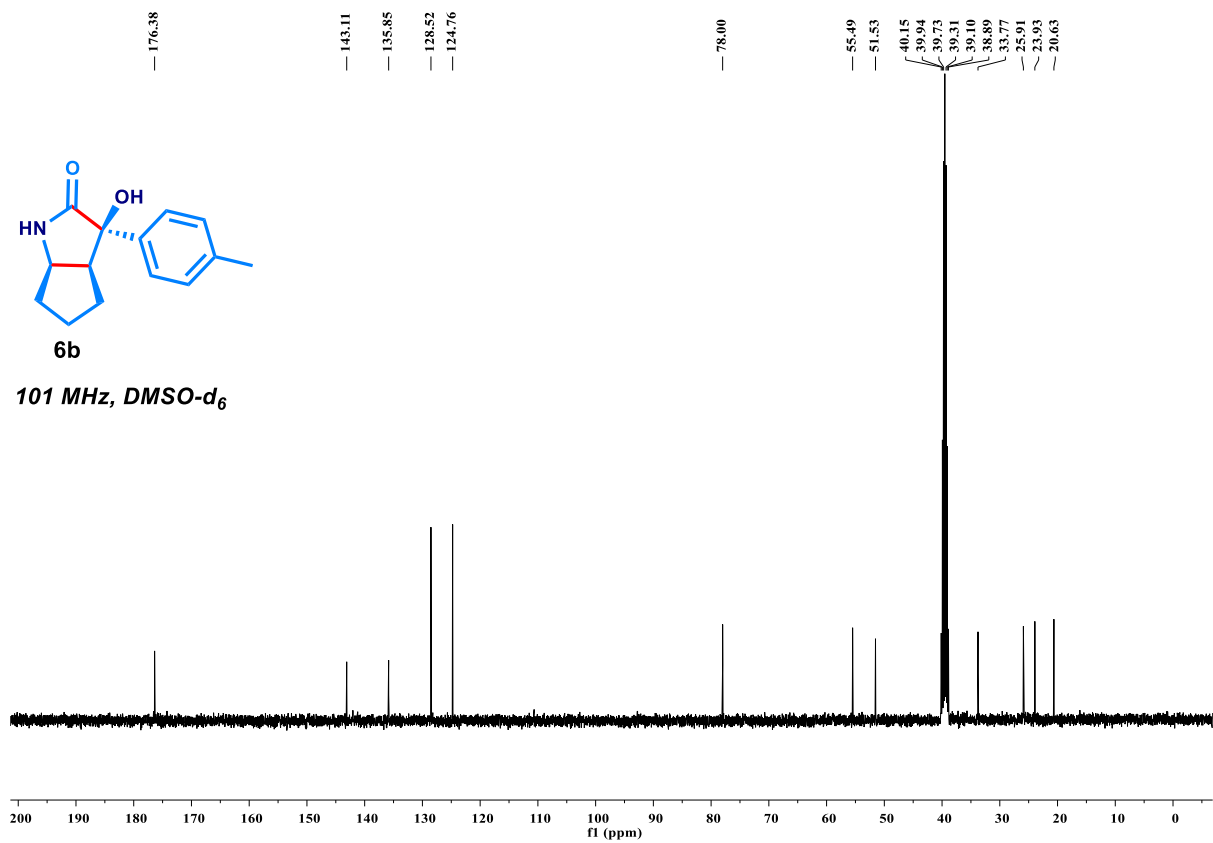
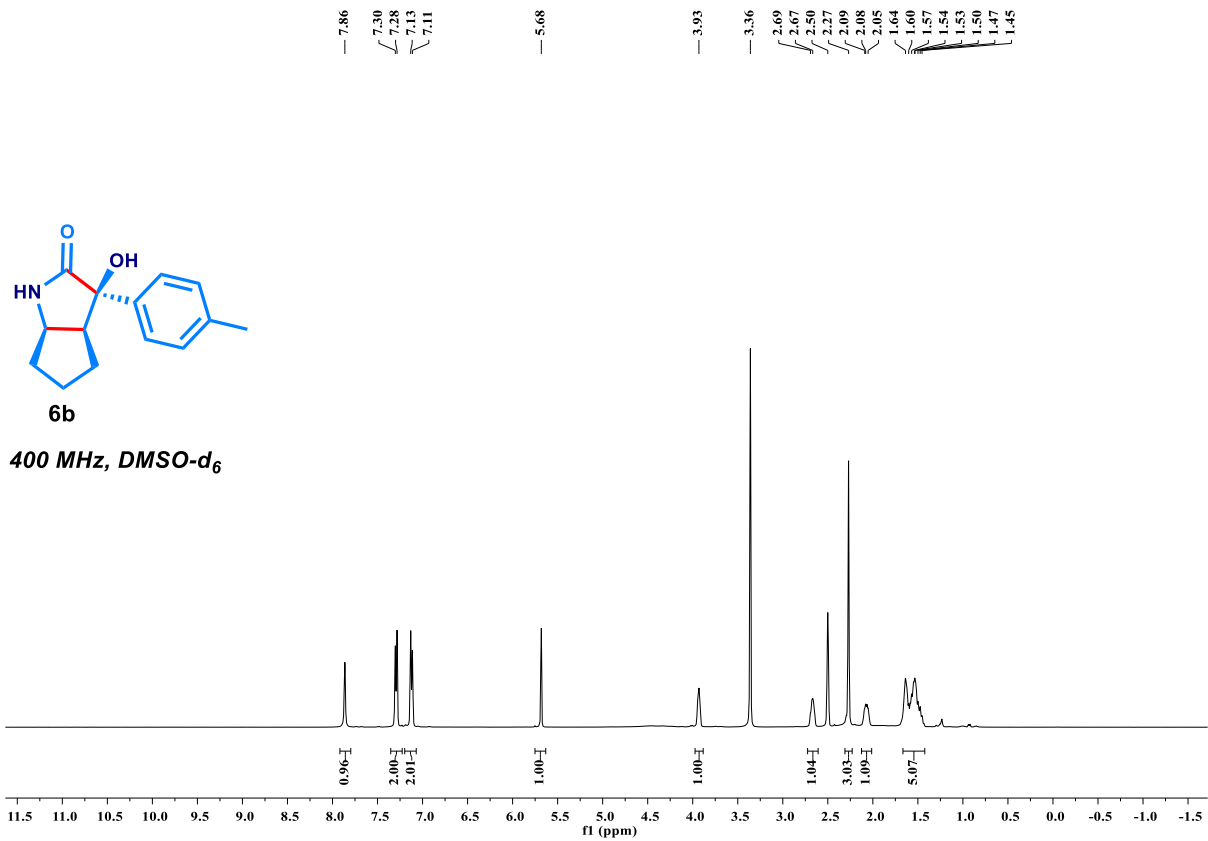


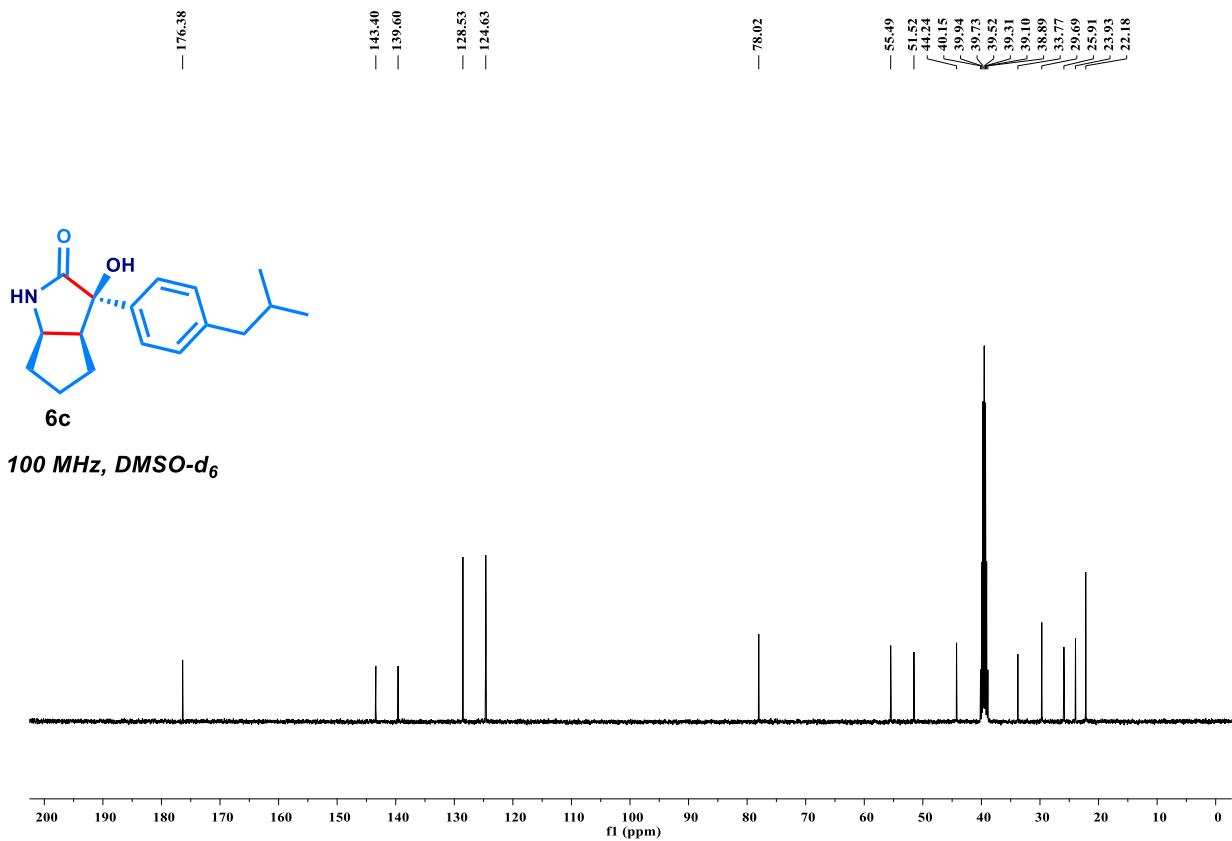
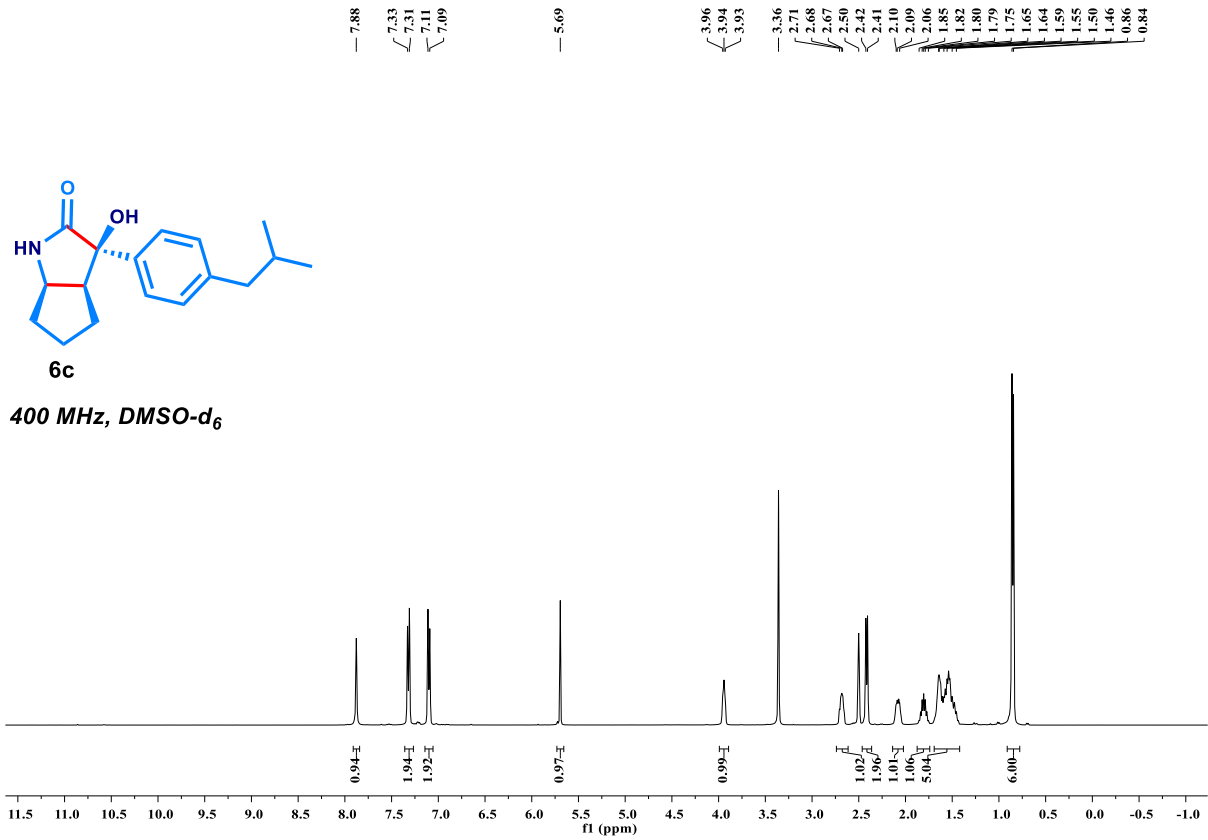


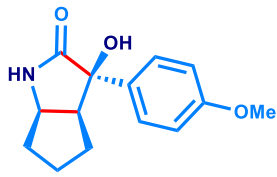






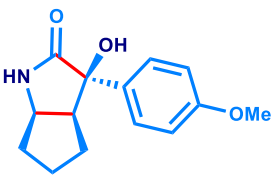
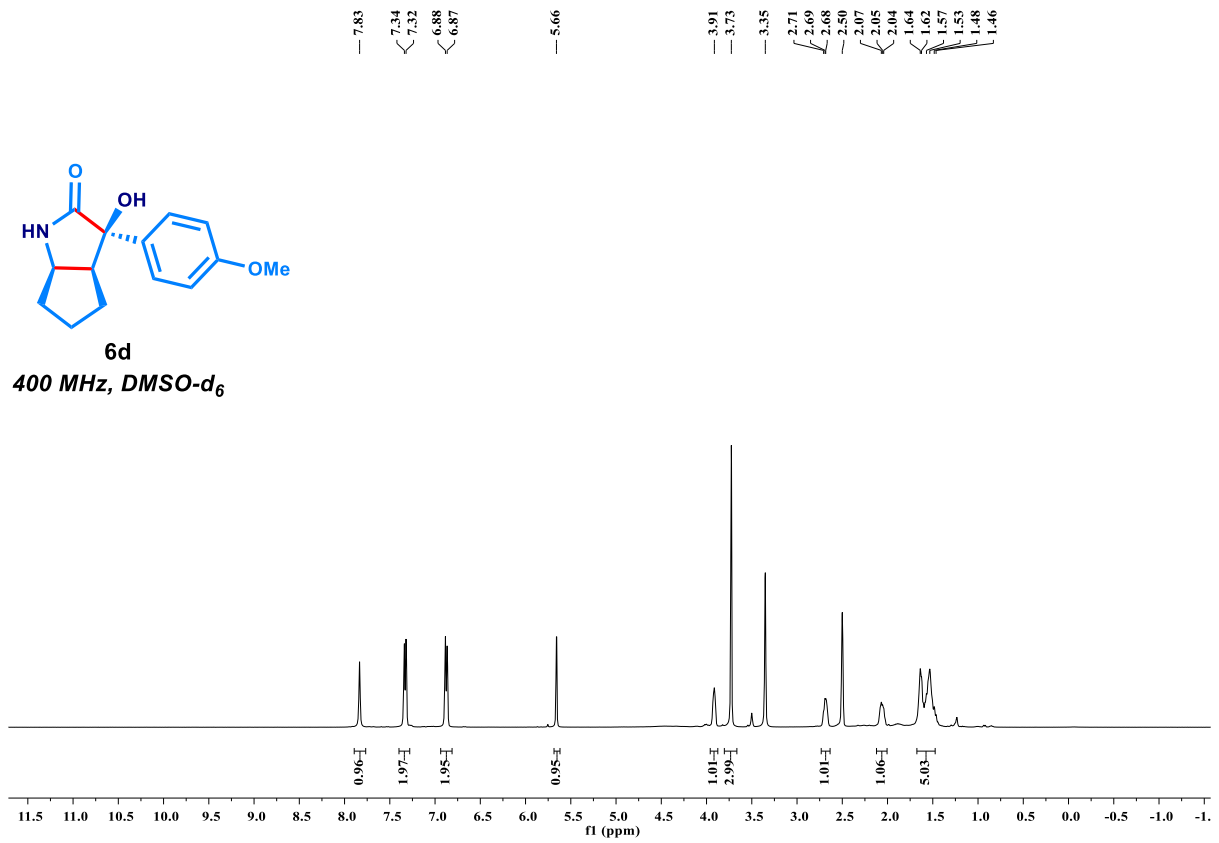






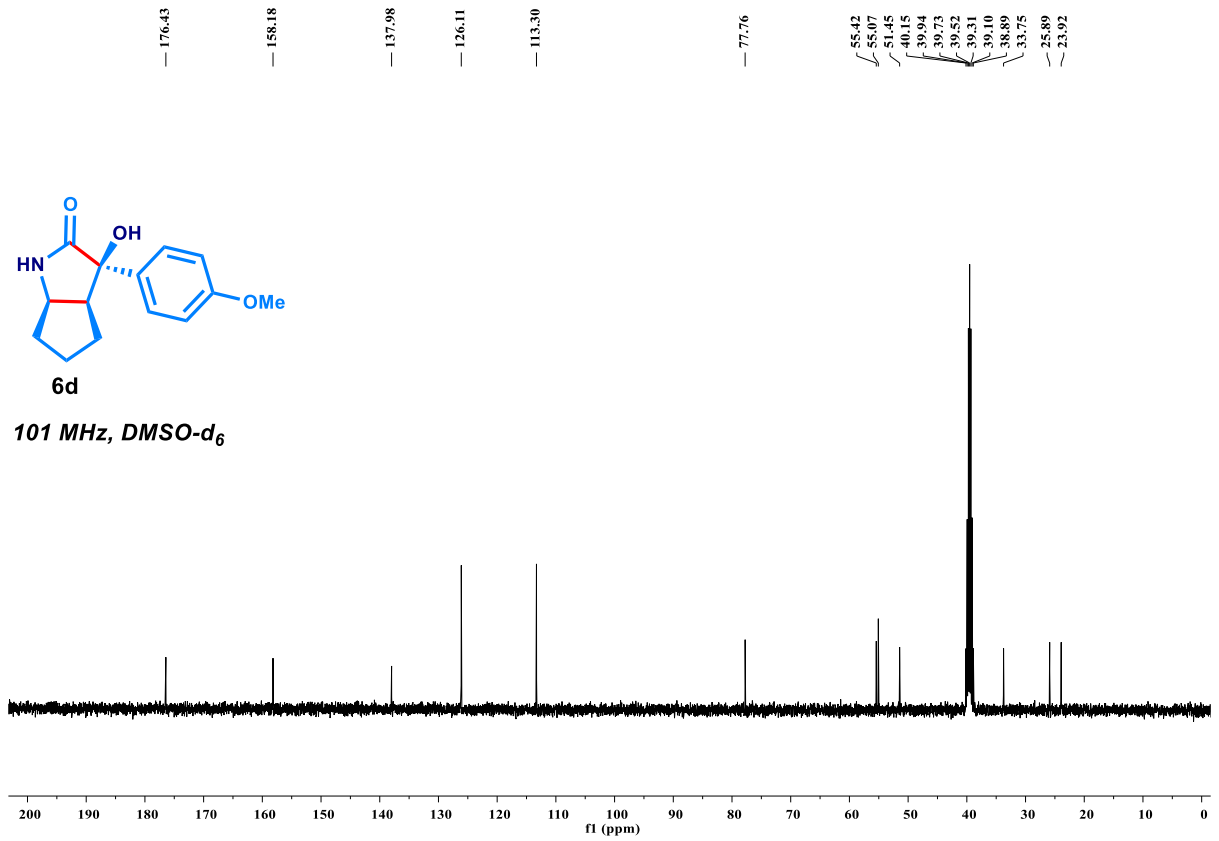
6d

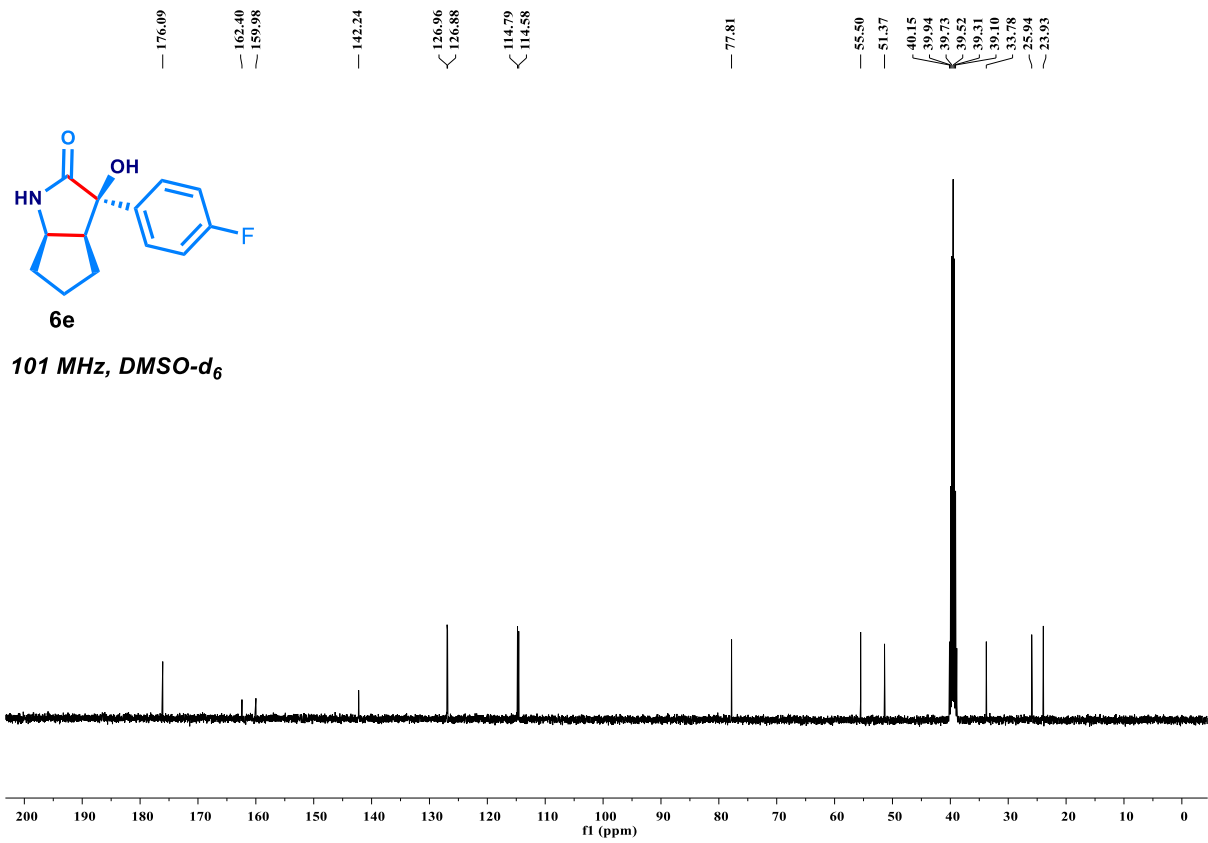
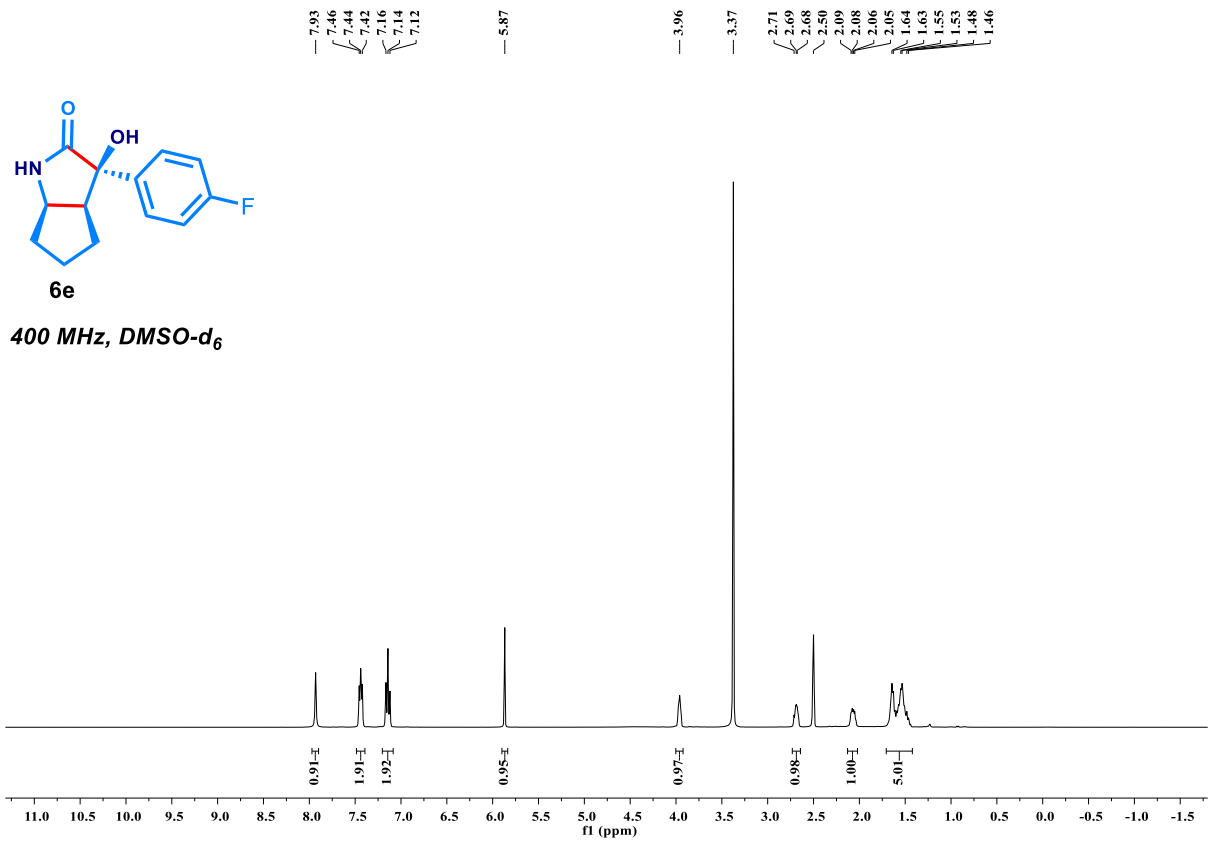
400 MHz, DMSO- $d_6$



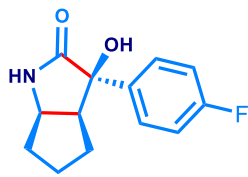
6d

101 MHz, DMSO- $d_6$



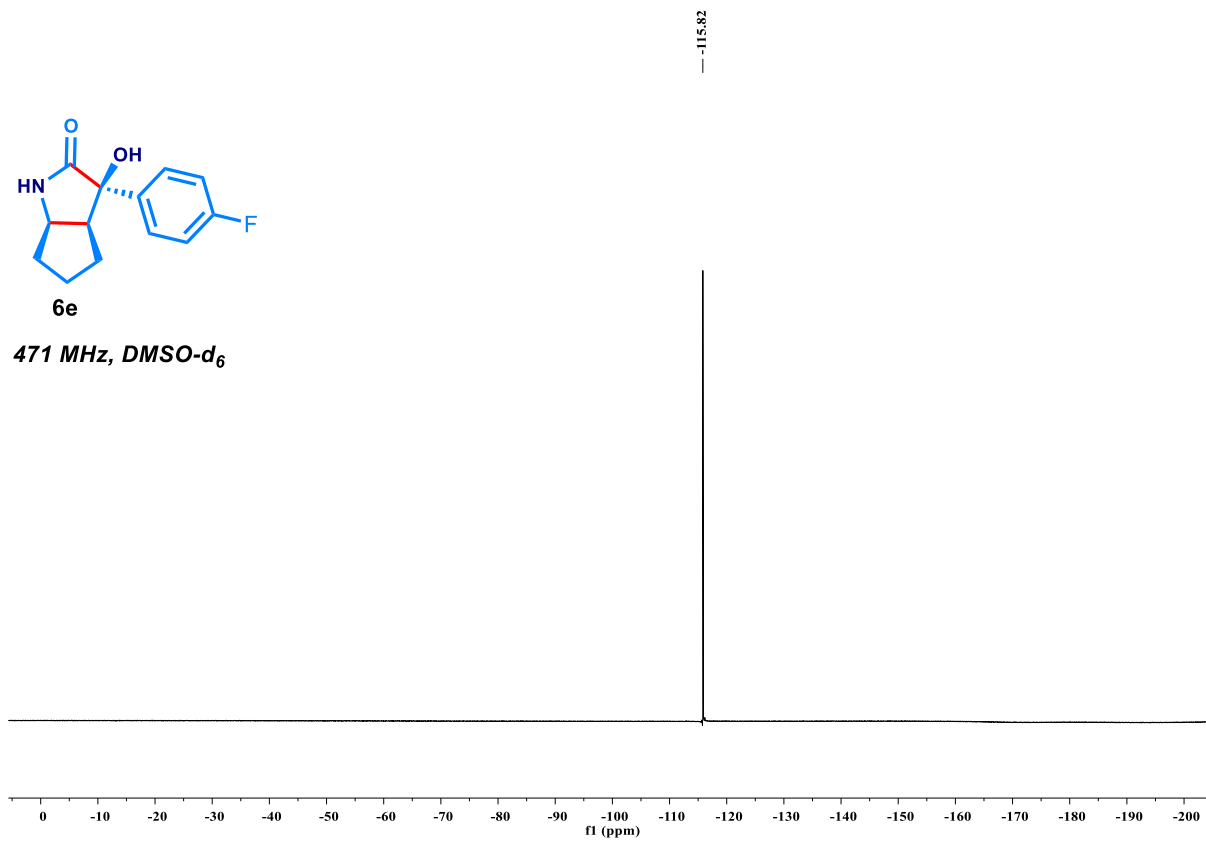


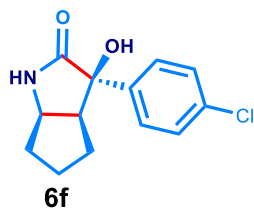




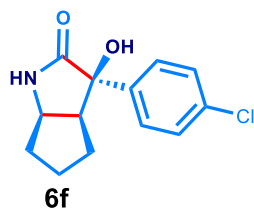
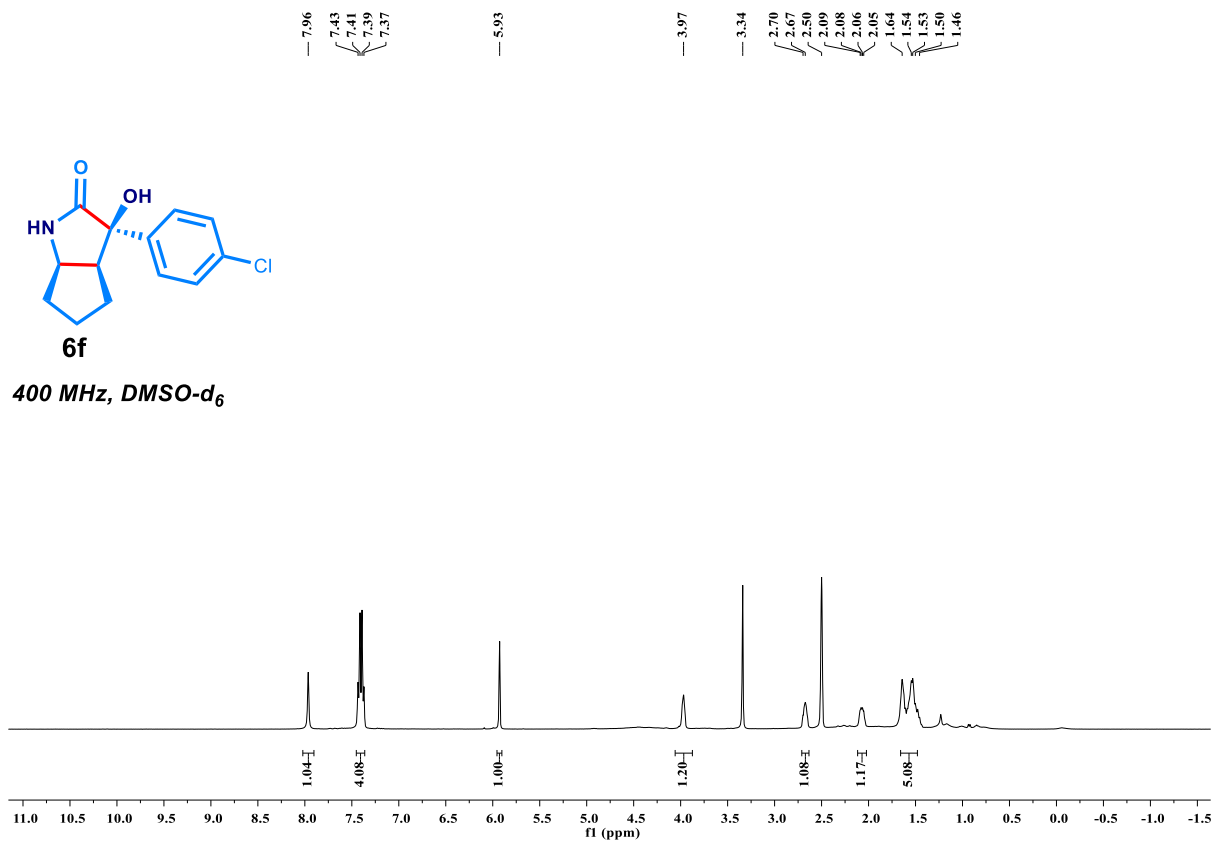
6e

471 MHz, DMSO- $d_6$

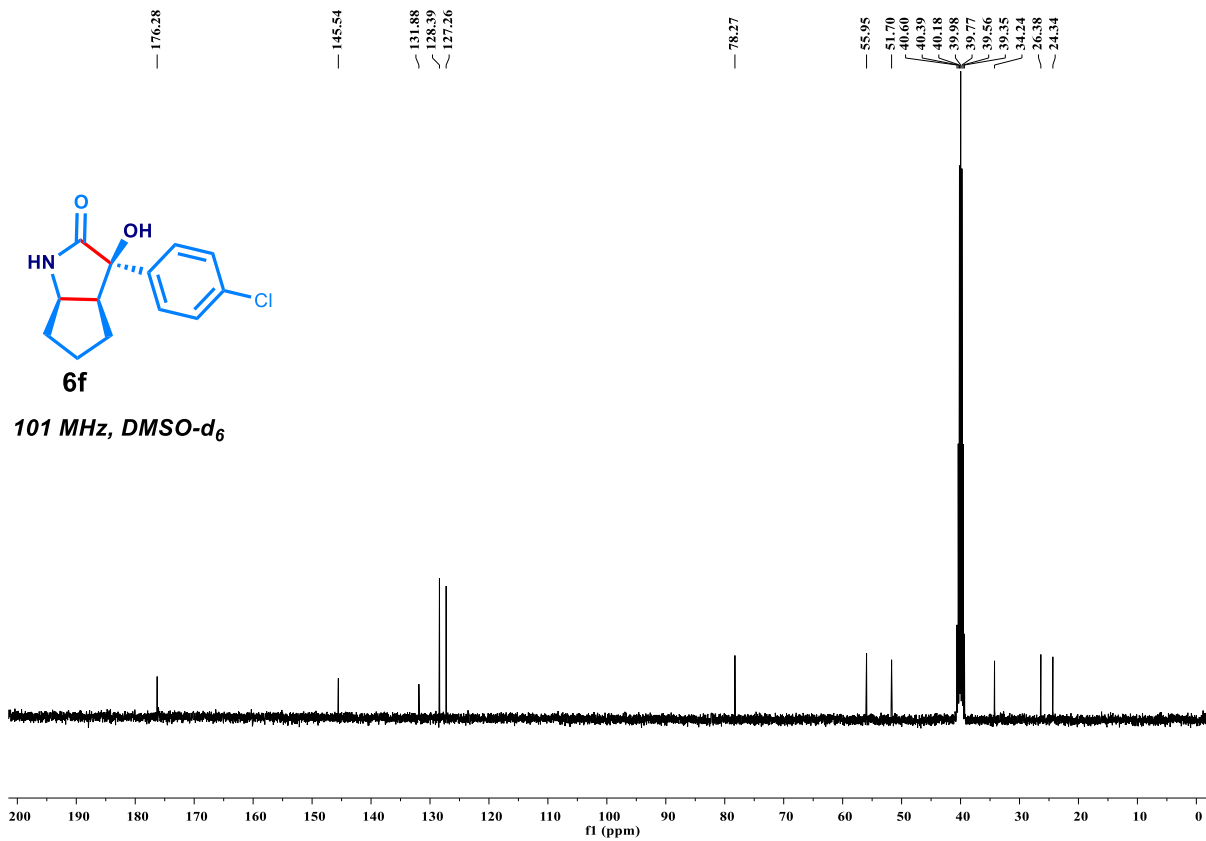


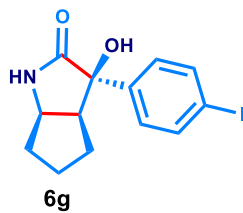


400 MHz, DMSO-d<sub>6</sub>

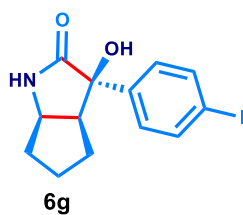
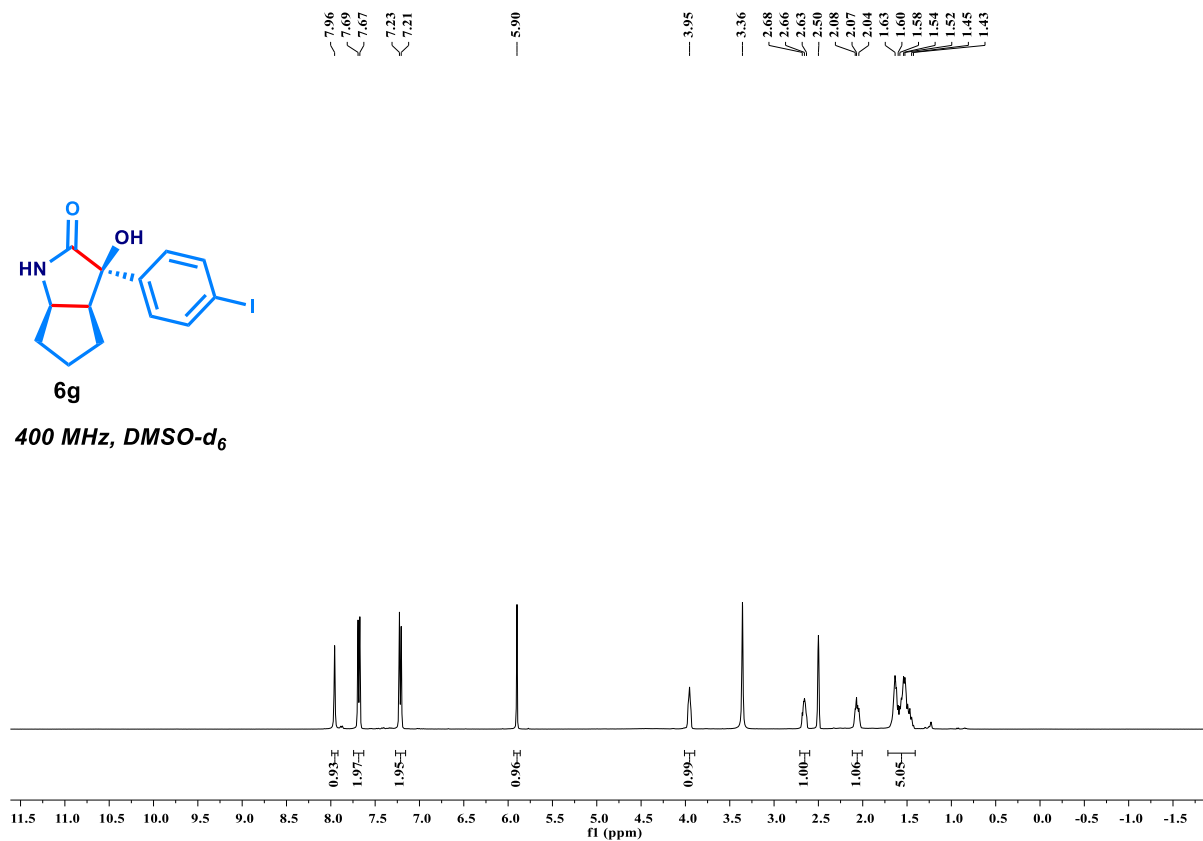


101 MHz, DMSO-d<sub>6</sub>

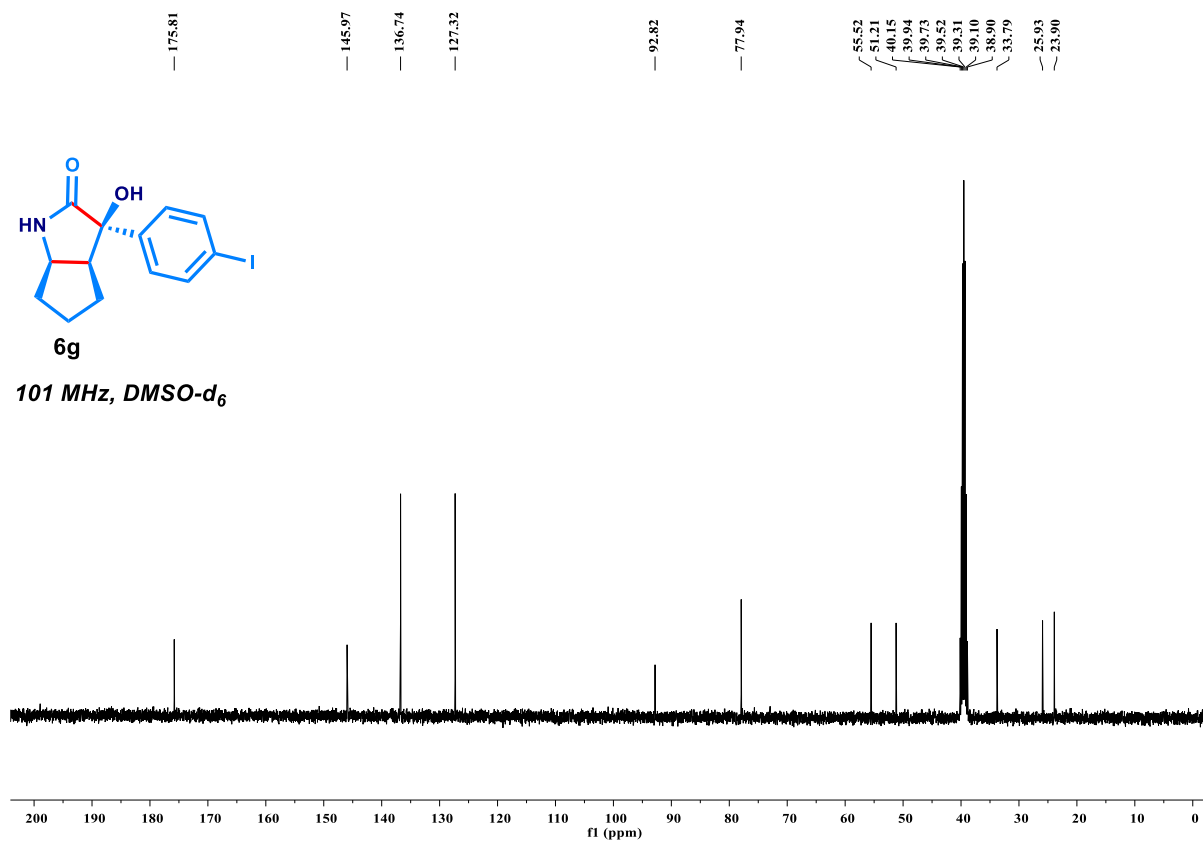


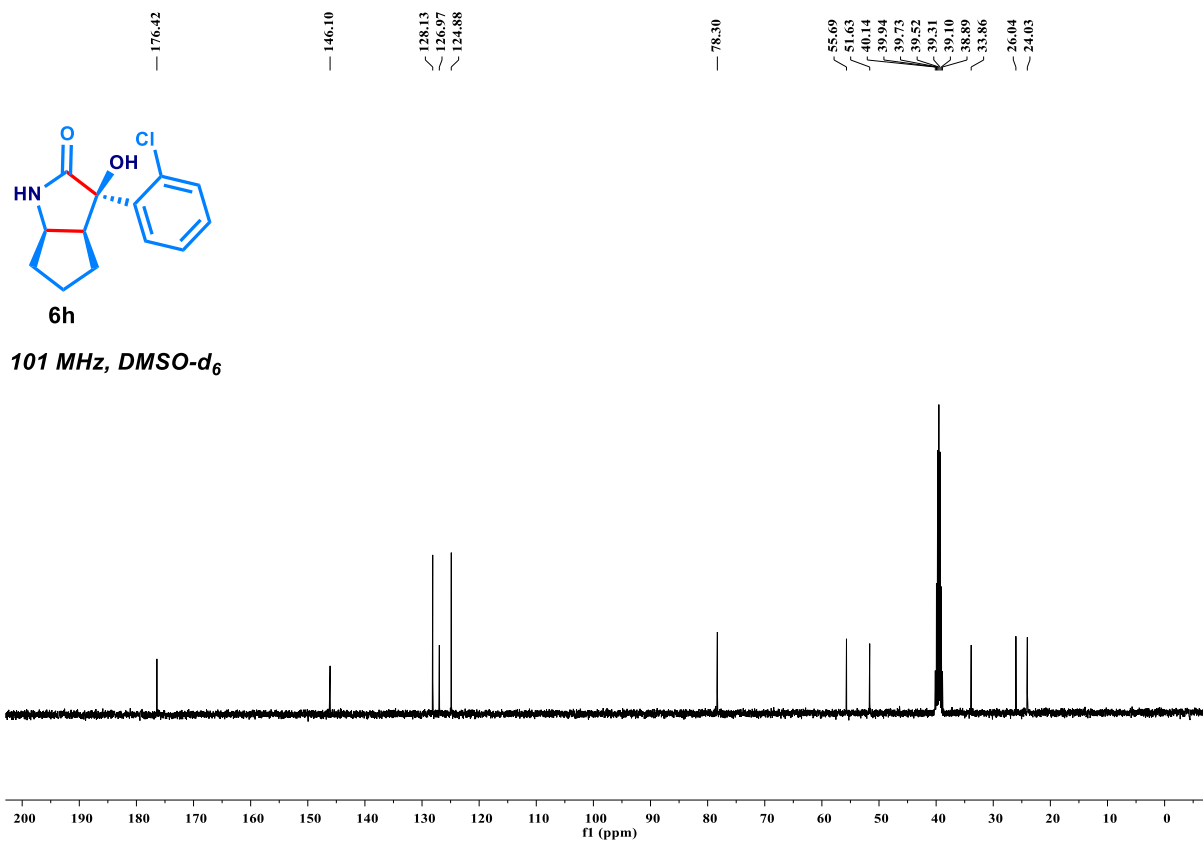
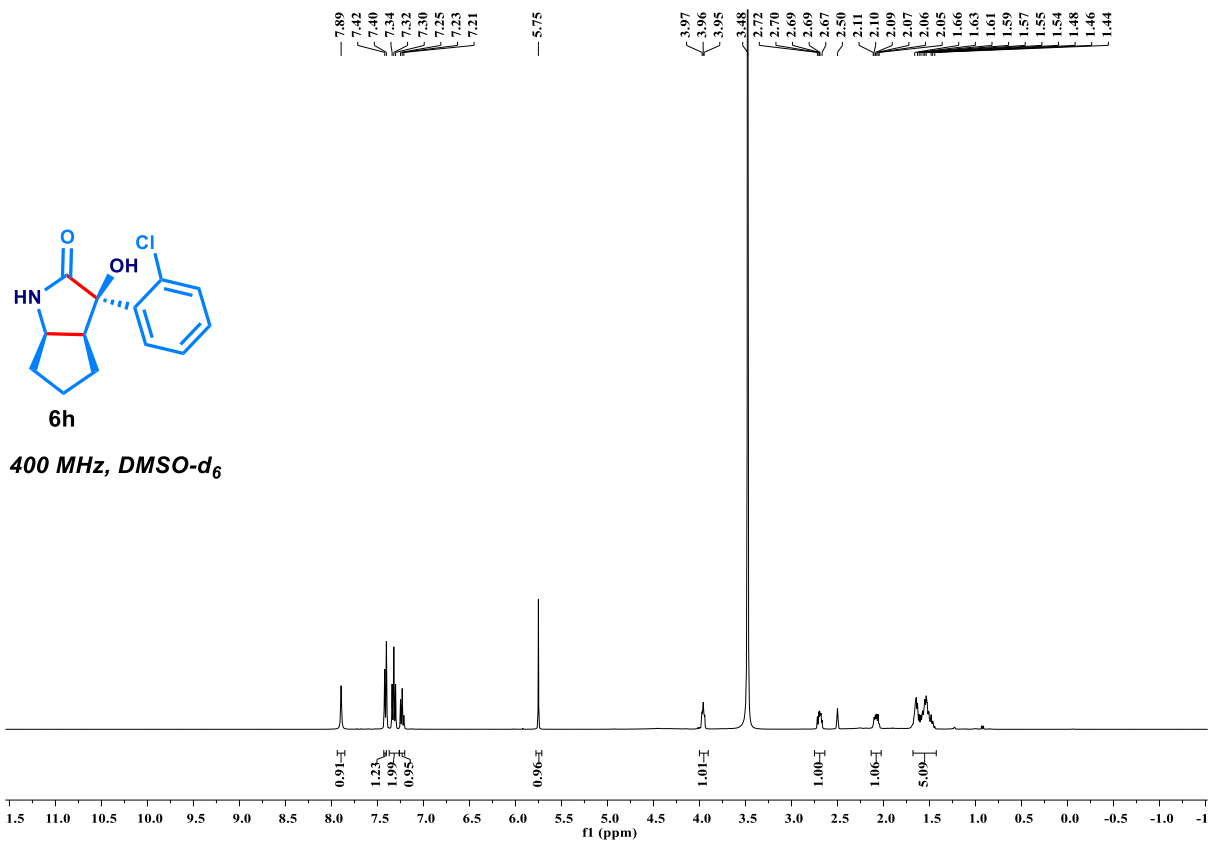


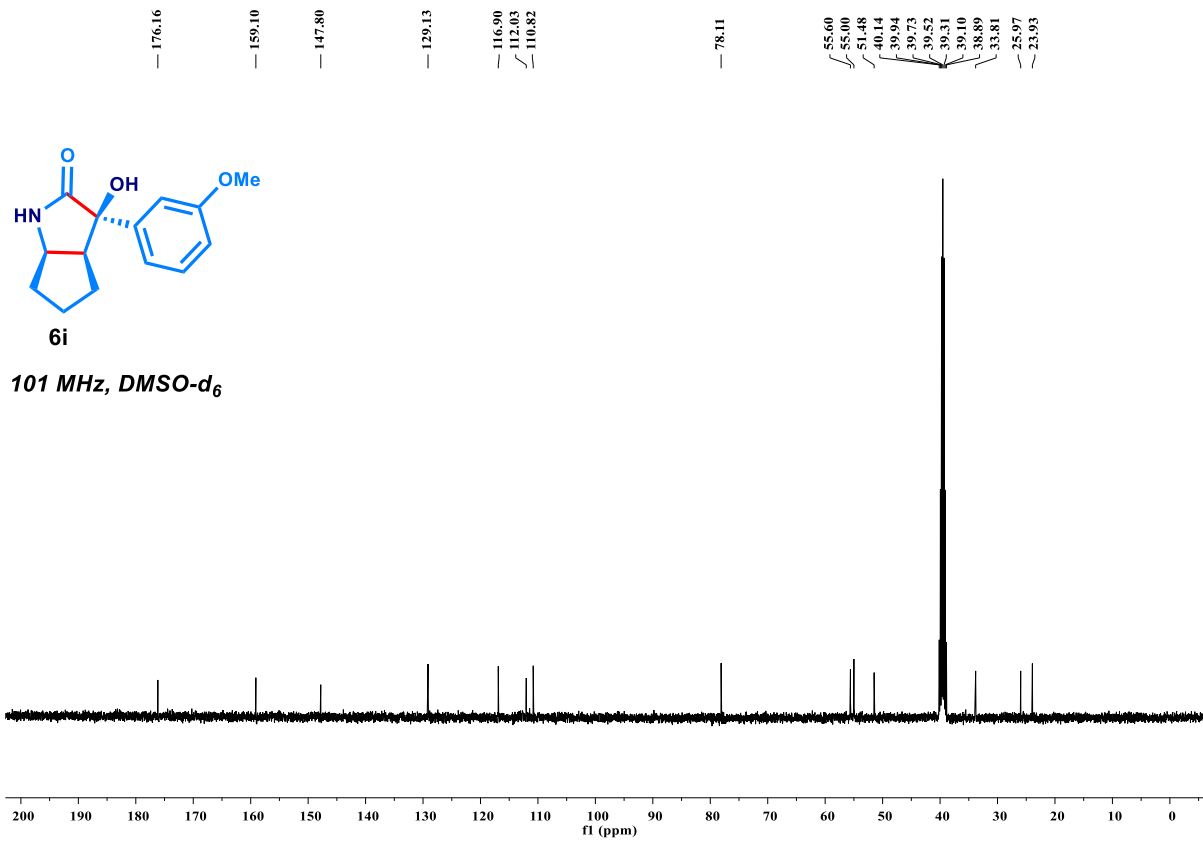
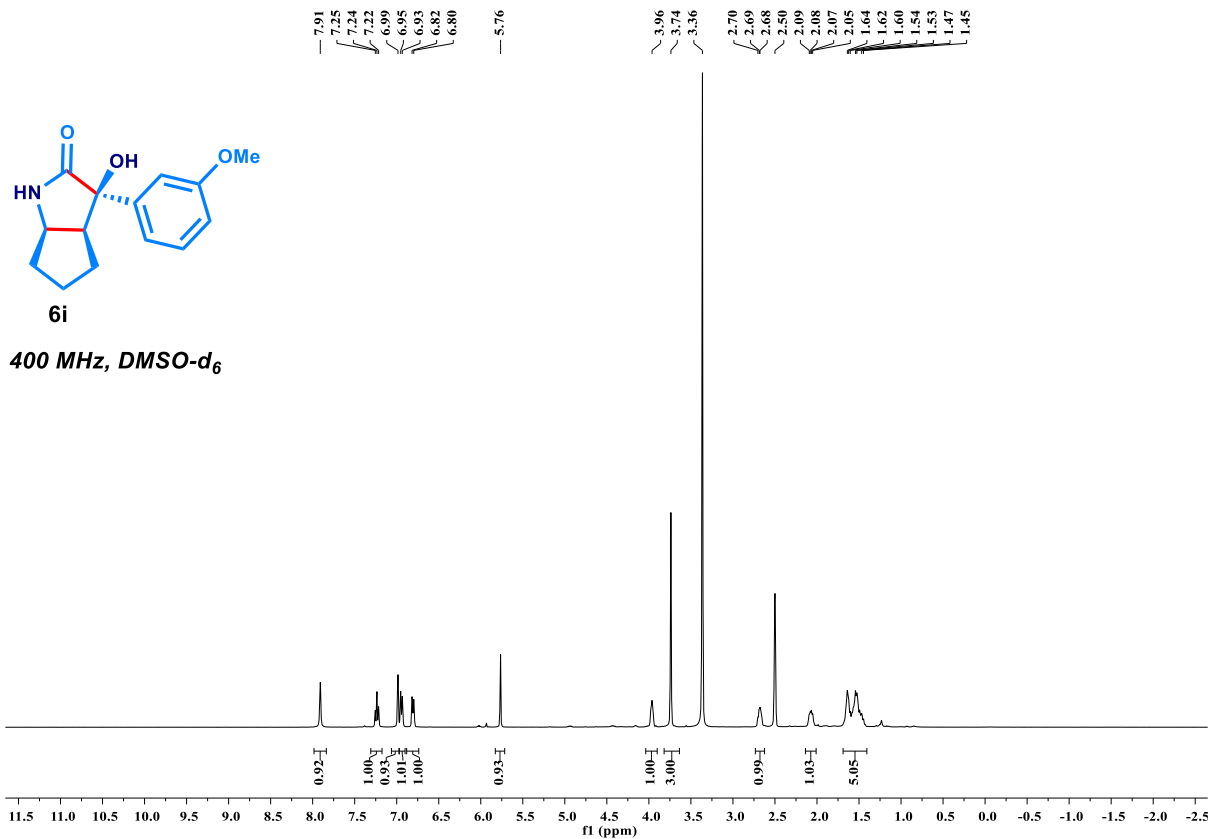
400 MHz, DMSO- $d_6$

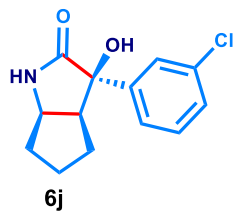


101 MHz, DMSO- $d_6$

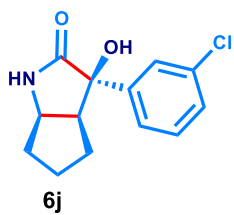
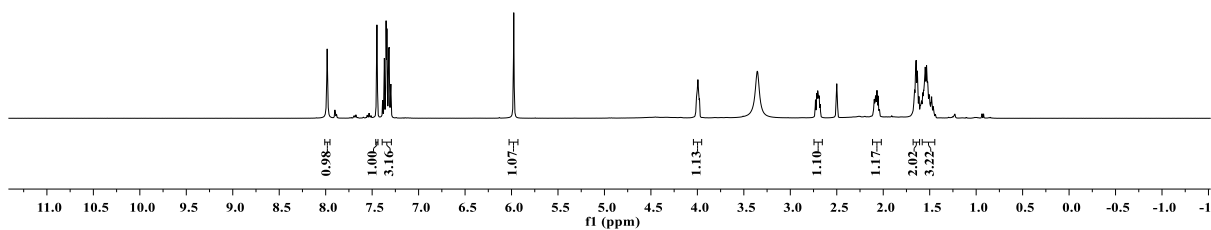




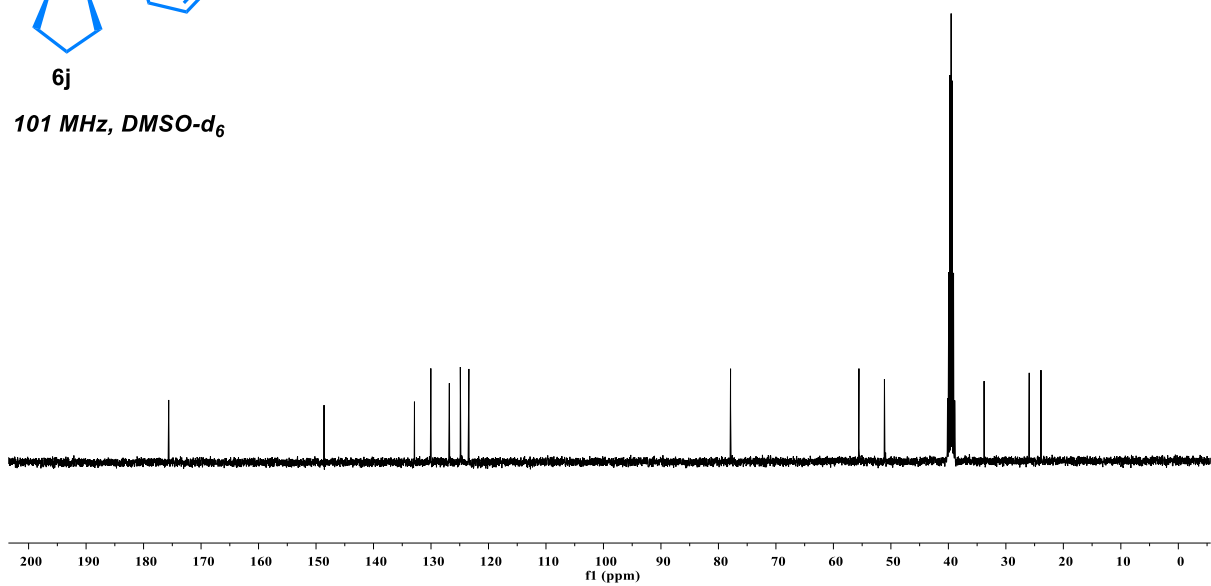


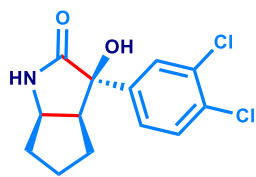


400 MHz, DMSO- $d_6$



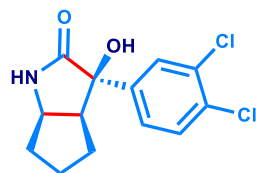
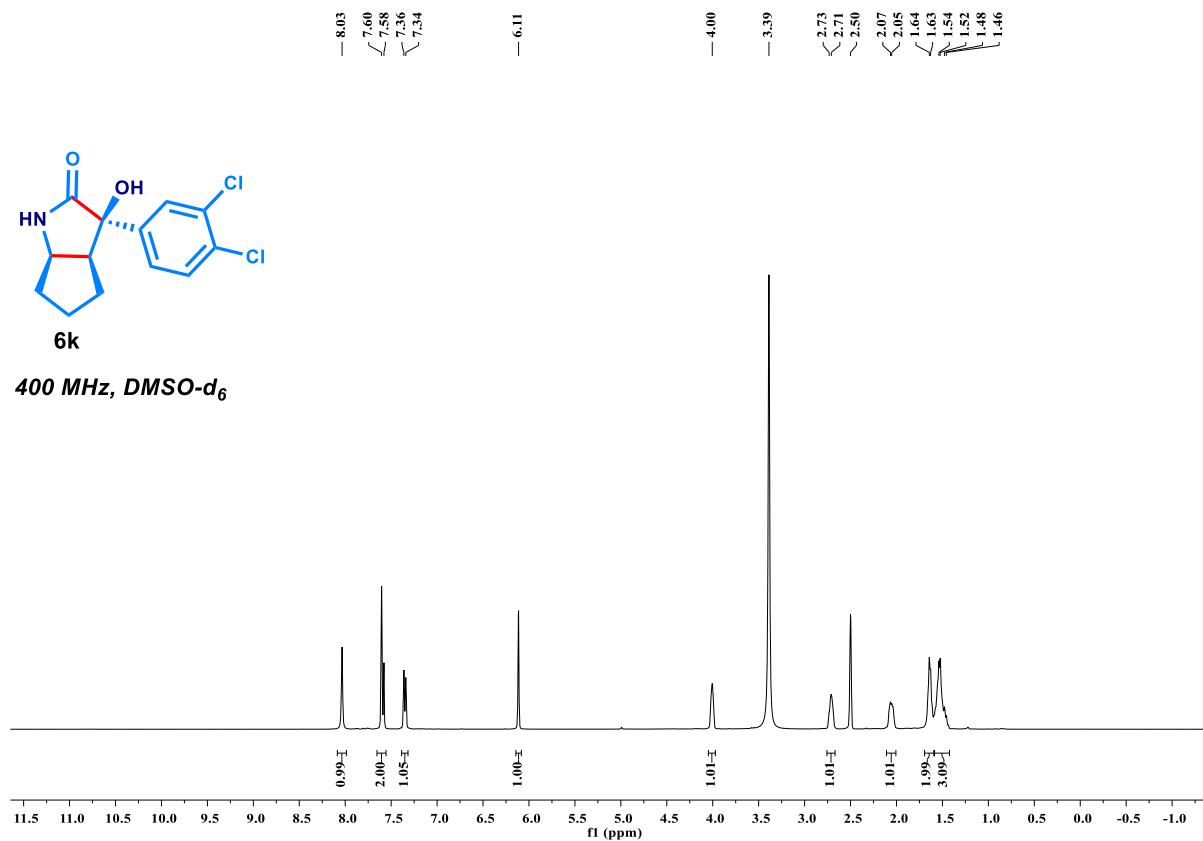
101 MHz, DMSO- $d_6$





6k

400 MHz, DMSO-d<sub>6</sub>



6k

101 MHz, DMSO-d<sub>6</sub>

