

## Supporting Information for

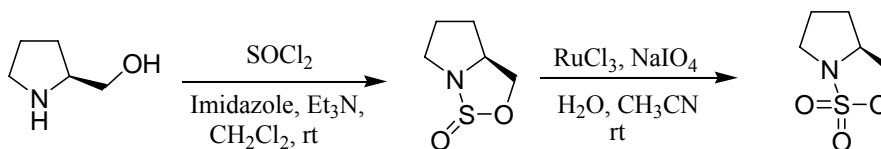
### Combinatorial Synthesis of Functionalized Chiral and Double Chiral Ionic Liquids and Their Applications as Asymmetric Non-Covalent Bifunctional Organocatalysts

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**Synthesis of chiral cyclic sulfate and cyclic sulfamidates followed previous publications with minor modifications (*Tetrahedron*, 2003, 59, 2581-2616; *J. Org. Chem.* 2002, 67, 5164-5169):**

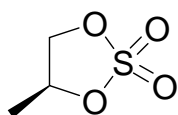


To a solution of imidazole (10.88 g, 0.16 mol) in 400 ml CH<sub>2</sub>Cl<sub>2</sub> was added triethylamine (12.24 mL, 0.088 mol) and prolinol (4.046 g, 0.04 mol) with stirring, the reaction was cooled to 0°C, followed by the dropwise addition of thionyl chloride (3.28 mL, 0.042 mol), the mixture was allowed to warm to rt and stirred overnight, then 200 ml

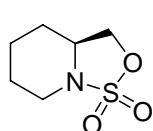
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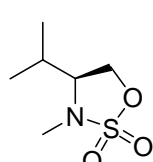
water was added, the organic phase was separated and the aqueous phase was washed with  $\text{CH}_2\text{Cl}_2$  (40 mL  $\times$  2) twice, the combined organic phase was washed with water (40 mL), dried with  $\text{Na}_2\text{SO}_4$  and concentrated in *vacuo* after filtration to give a yellow oil, which was used without any purification in the next step.

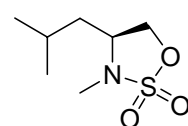
The yellow oil was dissolved in  $\text{CH}_3\text{CN}$  (250 mL) under ice-water bath,  $\text{RuCl}_3$  (80 mg, 0.38 mmol) and  $\text{NaIO}_4$  (12.8 g, 0.06 mol) was added in portion, followed by the addition of 200 ml water, the solution was stirred overnight under rt, quenched with 200 ml  $\text{Et}_2\text{O}$ , the organic phase was collected, the aqueous phase was filtered and then transferred to a separatory funnel, washed with  $\text{Et}_2\text{O}$  (40 mL  $\times$  2), the combined organic phase was washed with 50 ml saturated aqueous  $\text{NaHCO}_3$ , 50 ml brine, dried with  $\text{Na}_2\text{SO}_4$ , filtered, concentrated in *vacuo*, the residue was purified by flash chromatography on silica gel to afford the sulfamidate as a white solid (5.62 g, total yield of 86%).  $[\alpha]_{\text{D}}^{20\text{C}} = +40.1^\circ$  (C=1.0,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.74-1.83 (1H, m), 1.86-1.95 (2H, m), 2.08-2.20 (1H, m), 3.17-3.26 (1H, m), 3.57-3.65 (1H, m), 3.98-4.04 (1H, m), 4.19-4.27 (1H, m), 4.48-4.54 (1H, m);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  25.13, 31.30, 51.06, 62.44, 71.73; HRMS (EI) for  $\text{C}_5\text{H}_9\text{NO}_3\text{S}$  (M): calcd. 163.0303, found 163.0305.

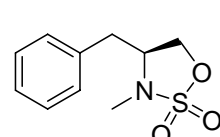


$[\alpha]_{\text{D}}^{20\text{C}} = +0.6^\circ$  (C=1.0,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  1.55 (3H, d,  $J=6.22\text{Hz}$ ), 4.29 (1H, t,  $J=8.67\text{Hz}$ ), 4.72 (1H, dd,  $J=5.65\text{ Hz}$ , 5.65Hz, 8.67 Hz), 5.07-5.14 (1H, m);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  17.60, 74.33, 80.15. HRMS (EI) for  $\text{C}_3\text{H}_5\text{O}_4\text{S}$  (M-1): calcd. 136.9909, found 136.9907.

  $[\alpha]_D^{20} = +48.4^\circ$  (C=1.0, CHCl<sub>3</sub>); <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 1.26-1.45 (2H, m), 1.51-1.64 (1H, m), 1.74-1.88 (3H, m), 2.70 (1H, td, *J* = 11.87 Hz, 2.83 Hz), 3.35-3.49 (2H, m), 4.13 (1H, dd, *J* = 7.91 Hz, 8.1 Hz, 9.79 Hz), 4.54 (1H, dd, *J* = 5.27 Hz, 5.84 Hz, 7.91 Hz); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ 21.79, 23.32, 27.63, 43.58, 57.16, 73.75; HRMS (EI) for C<sub>6</sub>H<sub>11</sub>NO<sub>3</sub>S (M): calcd. 177.0460, found 177.0459.

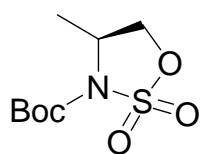
  $[\alpha]_D^{20} = +33.6^\circ$  (C=1.0, CHCl<sub>3</sub>); <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 0.96 (6H, dd, *J* = 4.90 Hz, 4.90 Hz, 6.97 Hz), 1.96-2.08 (1H, m), 2.77 (3H, s), 3.30-3.36 (1H, m), 4.23 (1H, dd, *J* = 7.35 Hz, 7.91 Hz, 8.67 Hz), 4.44 (1H, dd, *J* = 7.35 Hz, 7.35 Hz, 8.48 Hz); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ 21.79, 23.32, 27.63, 43.58, 57.16, 73.75; HRMS (EI) for C<sub>6</sub>H<sub>13</sub>NO<sub>3</sub>S (M): calcd. 179.0616, found 179.0615.

  $[\alpha]_D^{20} = +67.7^\circ$  (C=1.0, CHCl<sub>3</sub>); <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 0.92 (6H, t, *J* = 6.4 Hz), 1.38-1.46 (1H, m), 1.54-1.67 (2H, m), 2.72 (3H, s), 3.46-3.55 (1H, m), 4.10 (1H, t, *J* = 8.28 Hz), 4.55 (1H, dd, *J* = 6.60 Hz, 6.78 Hz, 8.29 Hz); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ 22.04, 23.33, 24.38, 32.22, 40.36, 59.21, 71.92; HRMS (EI) for C<sub>7</sub>H<sub>15</sub>NO<sub>3</sub>S (M): calcd. 193.0773, found 193.0772.

  $[\alpha]_D^{20} = +25.1^\circ$  (C=1.0, CHCl<sub>3</sub>); <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 2.72-2.84 (4H, m), 3.16 (1H, dd, *J* = 5.66 Hz, 5.84 Hz, 13.57 Hz), 3.65-3.76 (1H, m), 4.19 (1H, dd, *J* = 7.5 Hz, 7.72 Hz, 8.67 Hz), 4.35 (1H, dd, *J* = 6.6 Hz, 6.6 Hz, 8.66 Hz), 7.15-7.21 (2H, m), 7.27-7.37 (3H, m); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ

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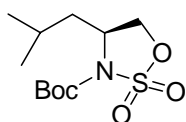
32.69, 37.93, 61.72, 71.81, 127.53, 129.04, 129.07, 134.65; HRMS (EI) for C<sub>10</sub>H<sub>13</sub>NO<sub>3</sub>S  
(M): calcd. 227.0616, found 227.0613.



$[\alpha]_D^{20} = +3.7^\circ$  (C=1.0, CHCl<sub>3</sub>); <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$

1.50 (3H, d,  $J = 6.41$  Hz), 1.55 (9H, s), 4.19 (1H, dd,  $J = 3.02$  Hz, 9.04 Hz), 4.36-4.48 (1H, m), 4.66 (1H, dd,  $J = 6.02$  Hz, 9.05 Hz); <sup>13</sup>C

NMR (CDCl<sub>3</sub>, 75 MHz):  $\delta$  18.31, 27.94, 53.80, 71.31, 85.36, 148.47; HRMS (EI) for C<sub>8</sub>H<sub>15</sub>NO<sub>5</sub>S (M): calcd. 237.0671, found 237.0673.

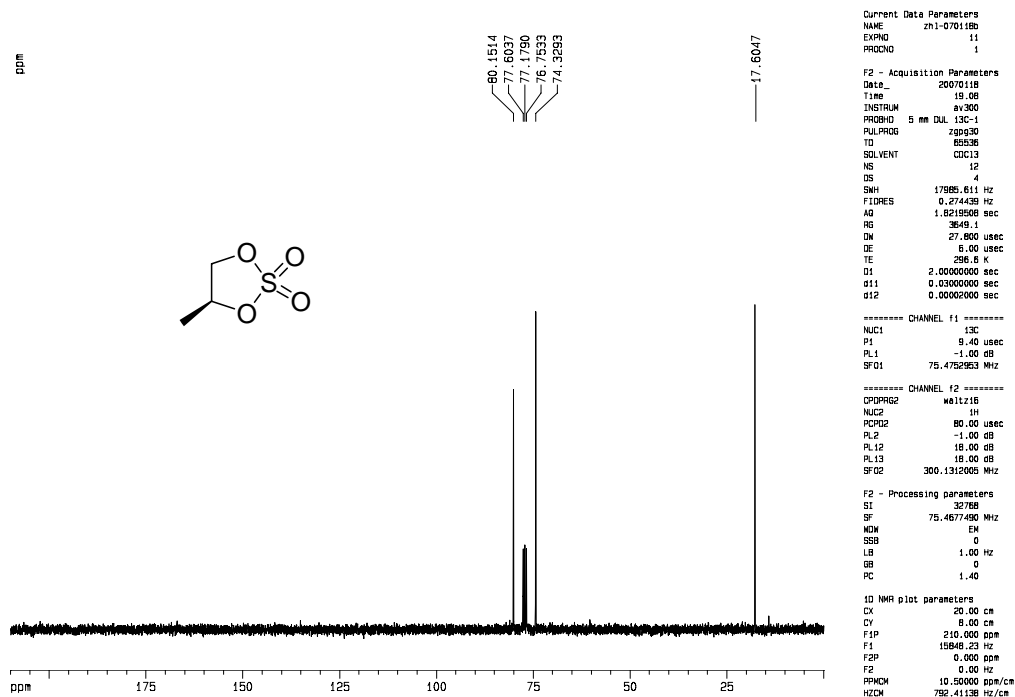
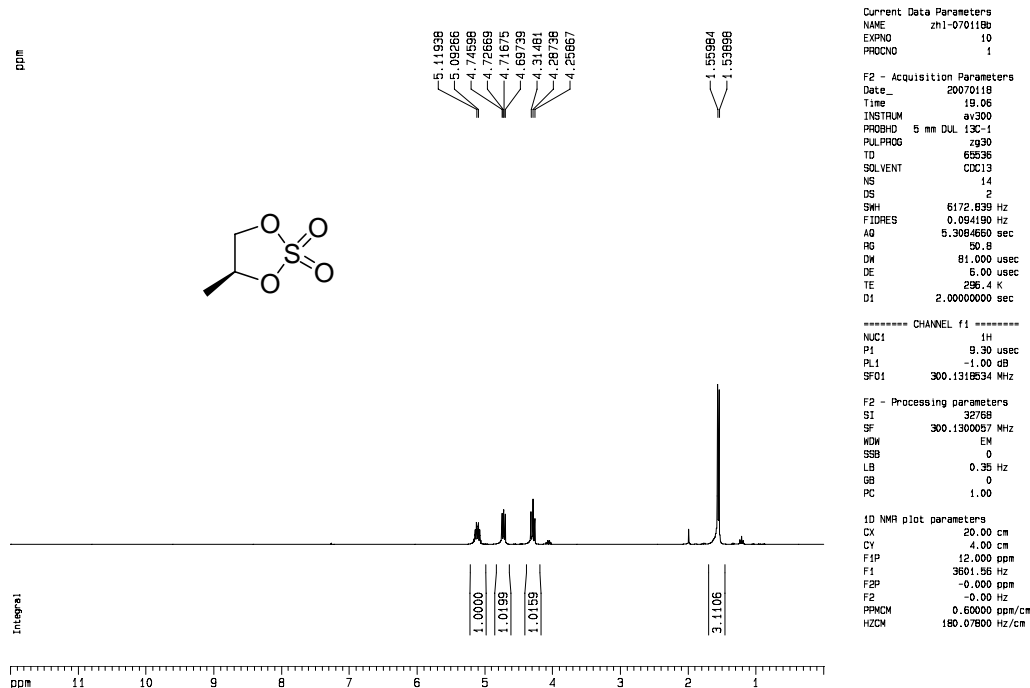


$[\alpha]_D^{20} = +14.5^\circ$  (C=1.0, CHCl<sub>3</sub>); <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  0.96

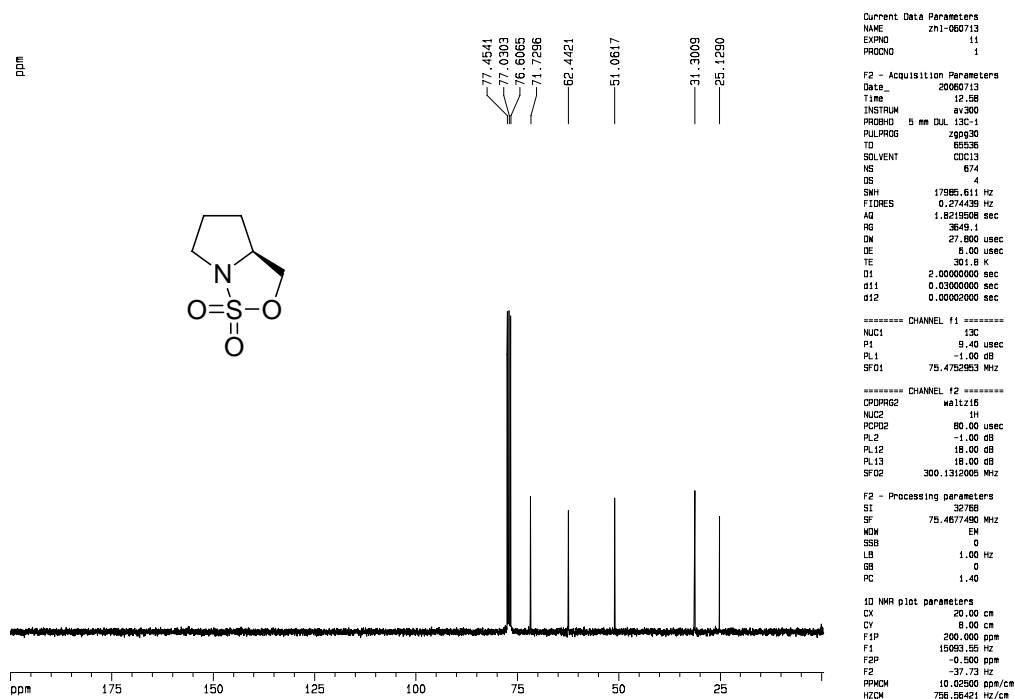
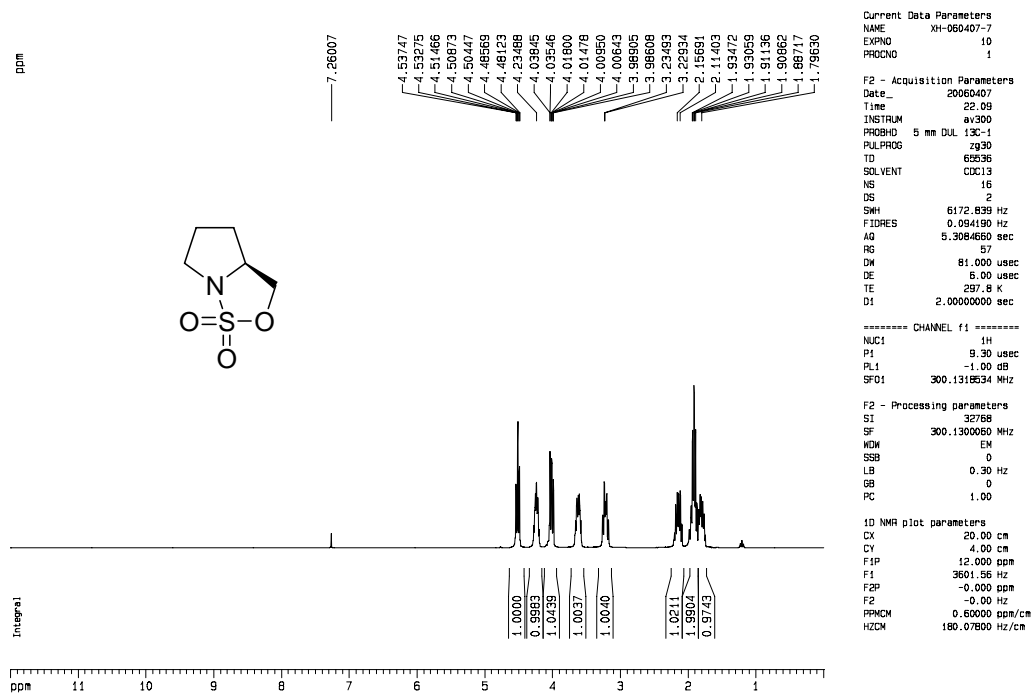
(6H, dd,  $J = 6.59$  Hz, 6.59 Hz, 9.23 Hz), 1.54 (9H, s), 1.57-1.84 (3H, m),

4.27-4.36 (2H, m), 4.63 (1H, dd,  $J = 6.03$  Hz, 9.23 Hz); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz):  $\delta$  21.66, 23.23, 24.68, 27.92, 40.96, 56.30, 70.03, 85.32, 148.61; HRMS (EI) for C<sub>11</sub>H<sub>21</sub>NO<sub>5</sub>S (M): calcd. 279.1140, found 279.1143.

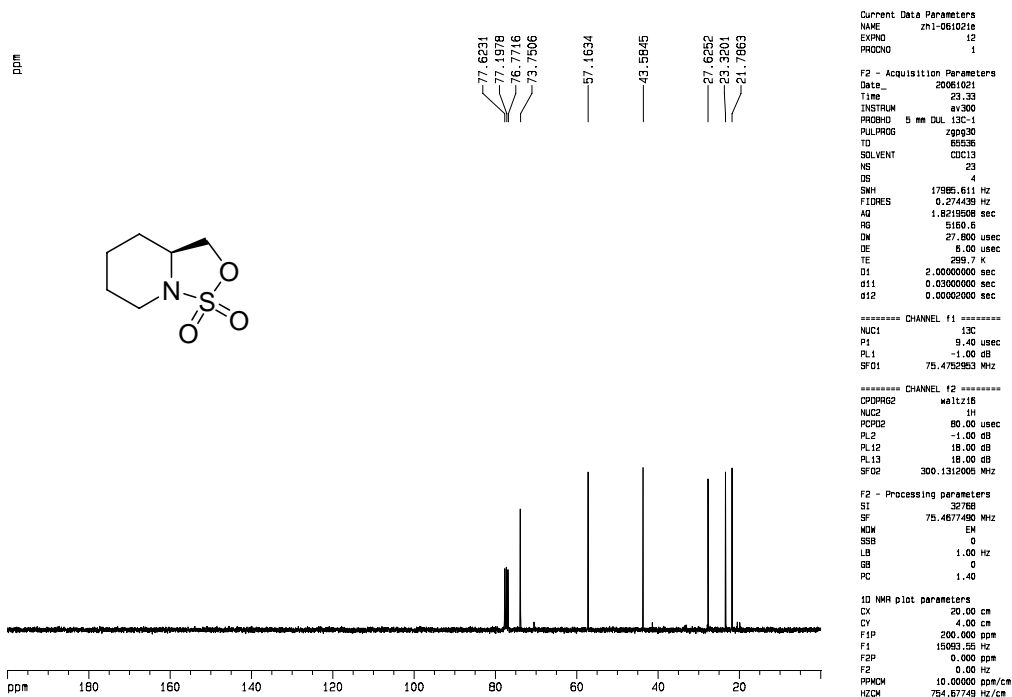
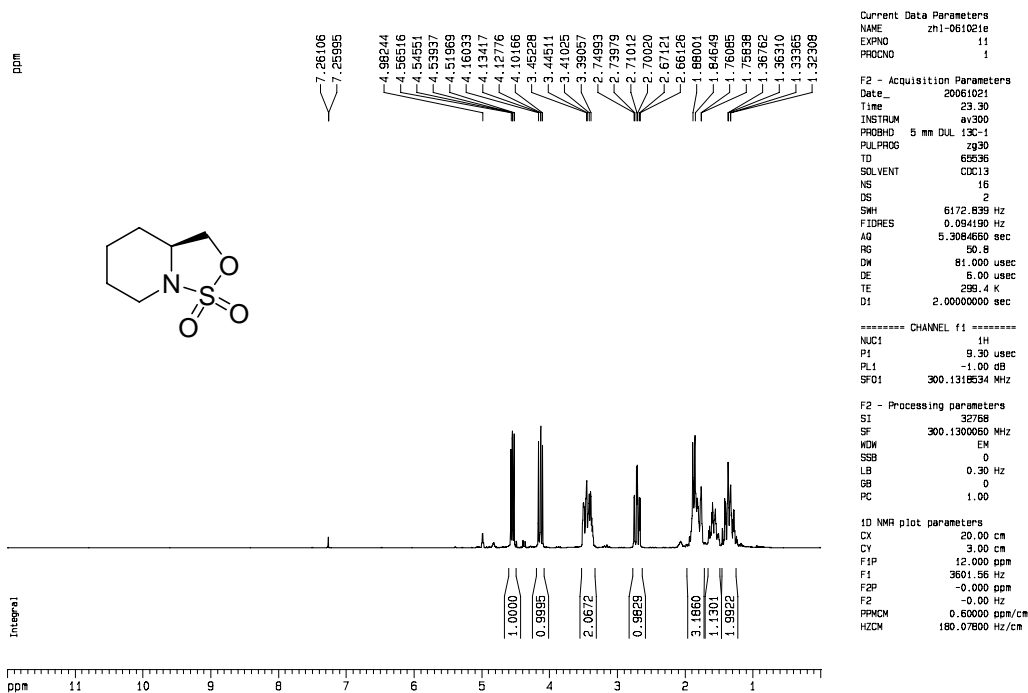
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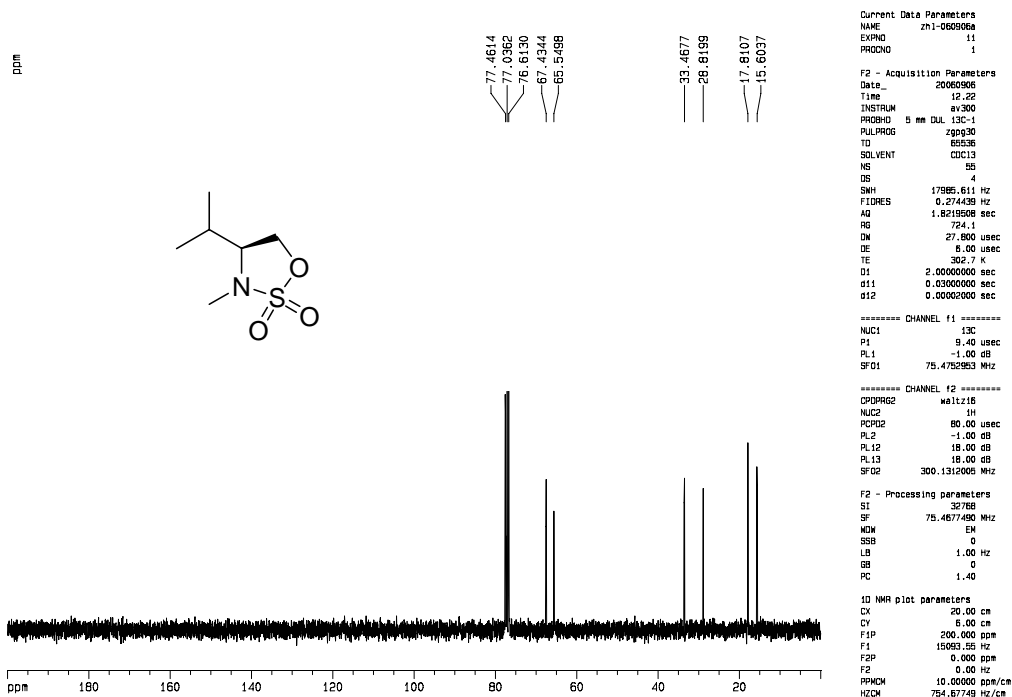
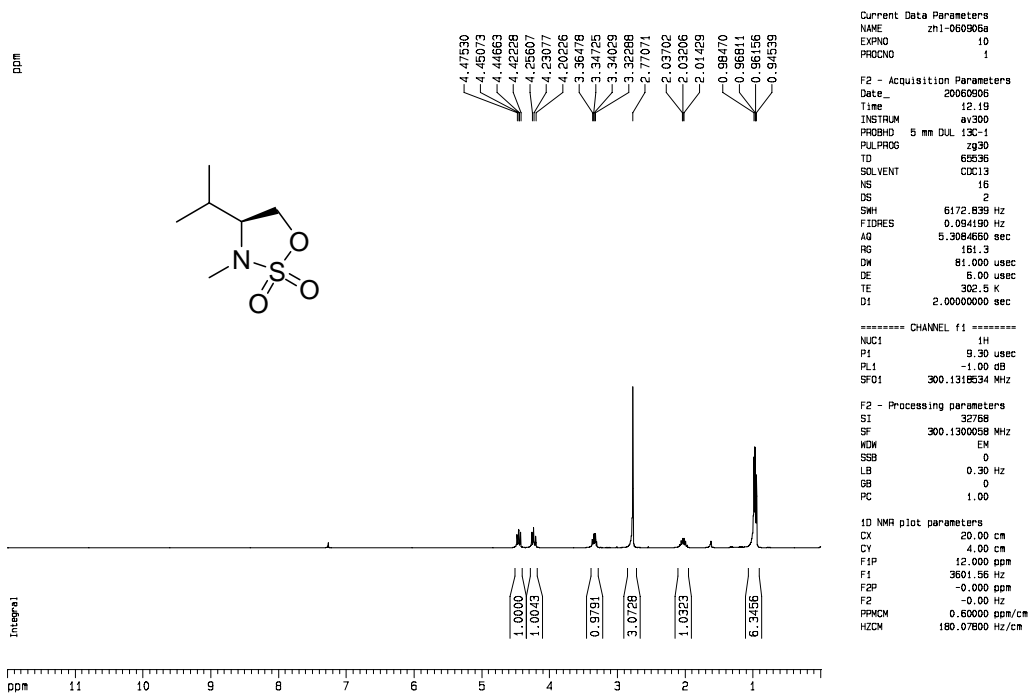
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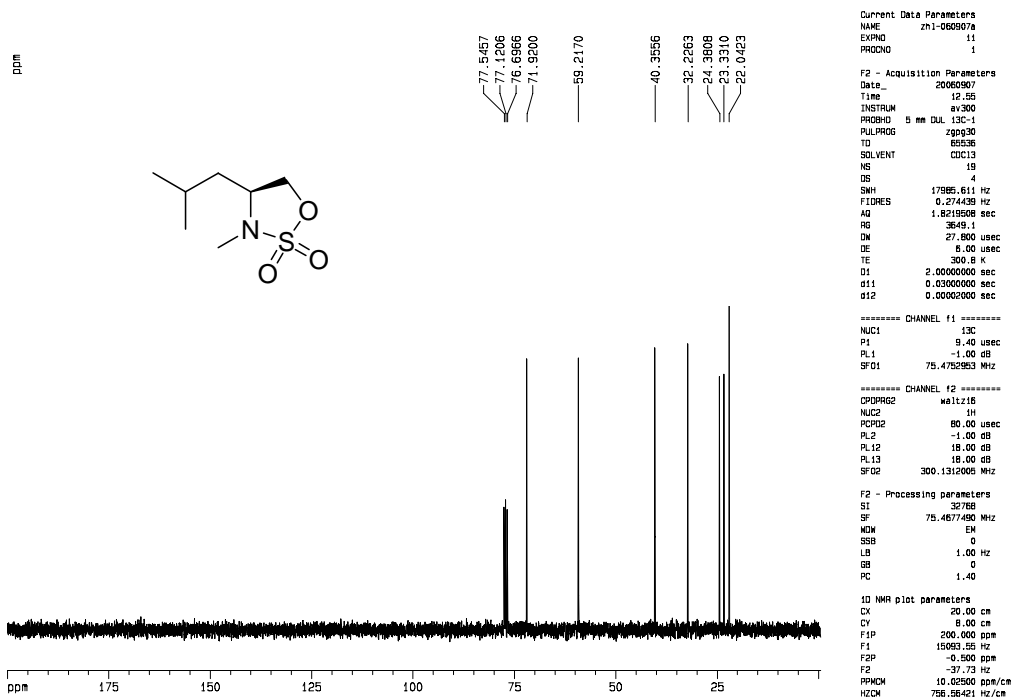
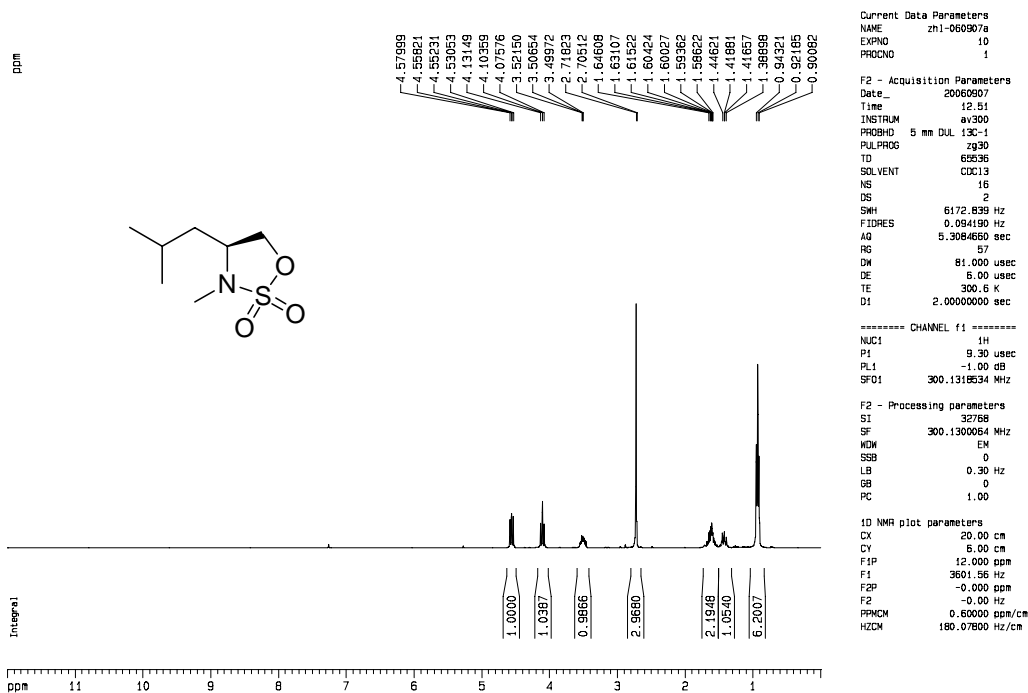


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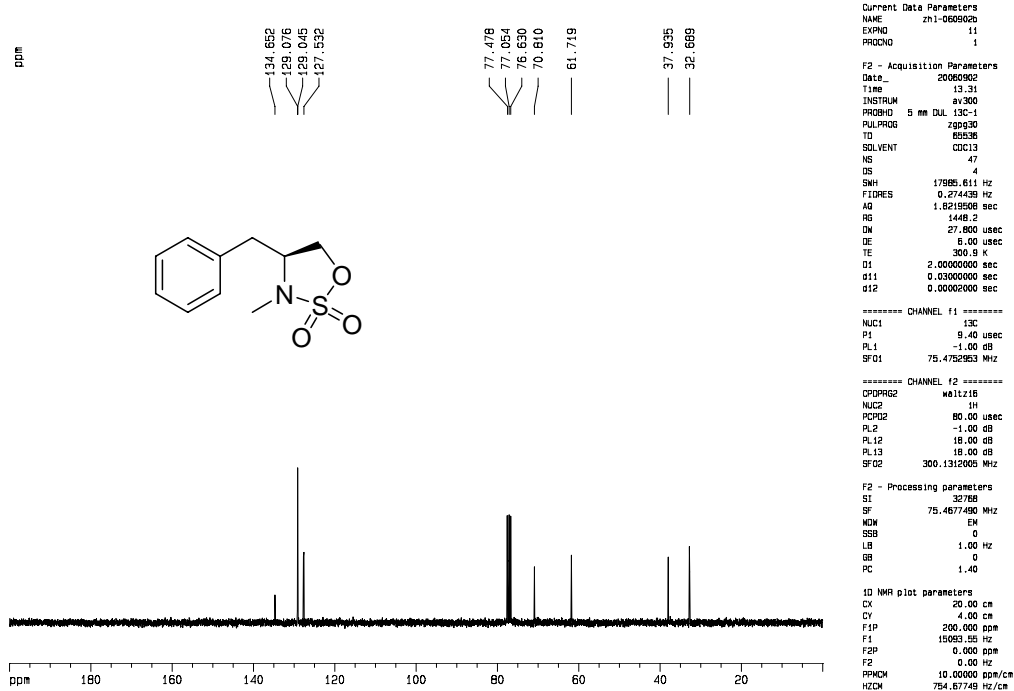
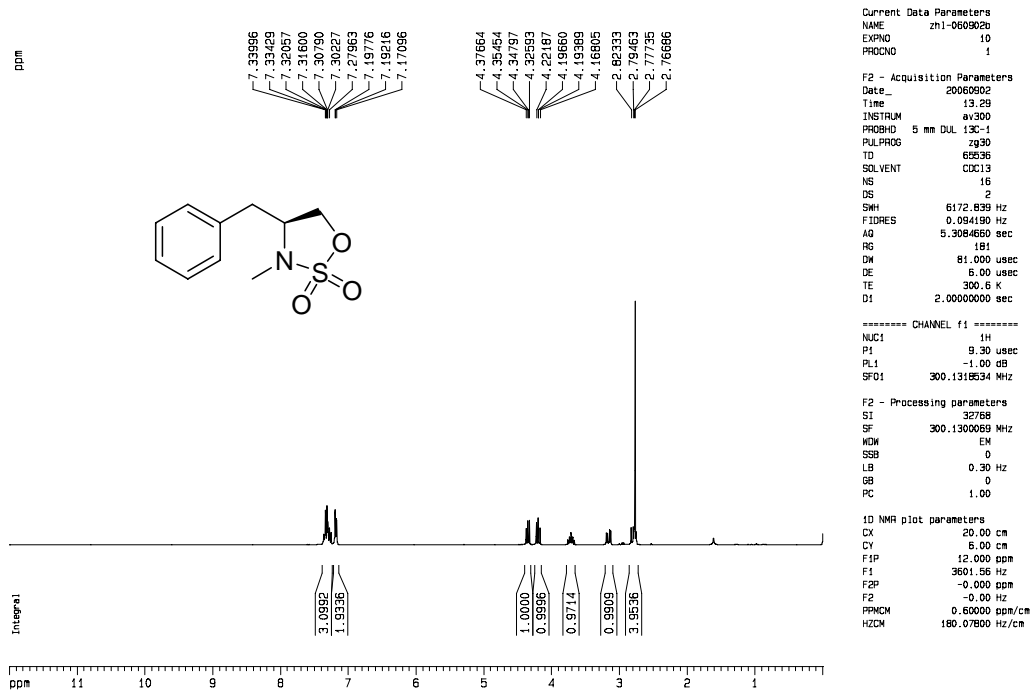




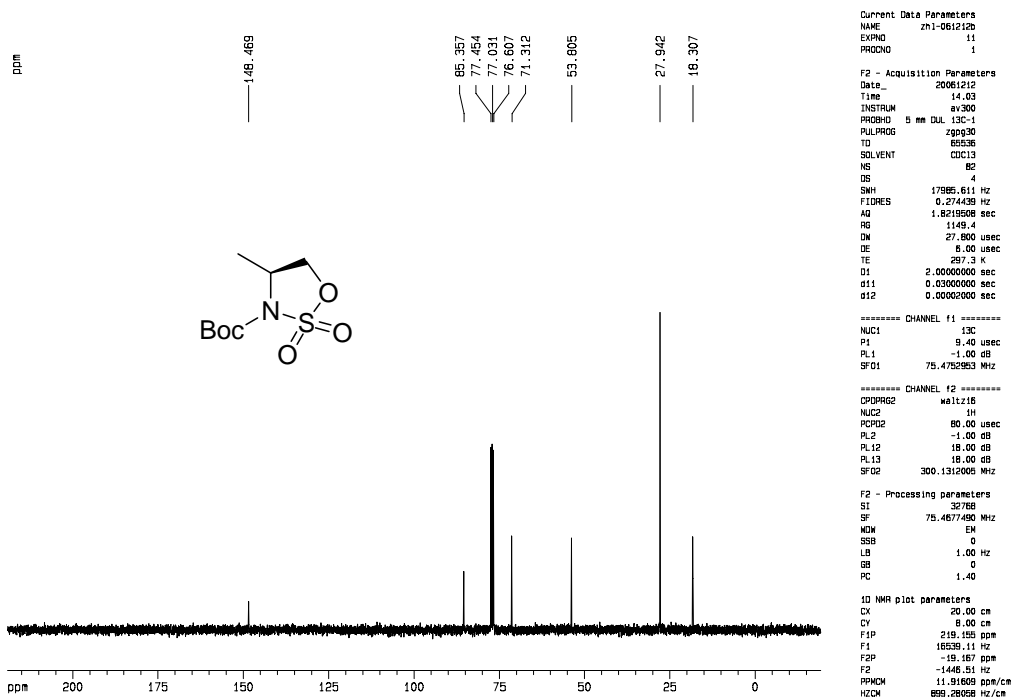
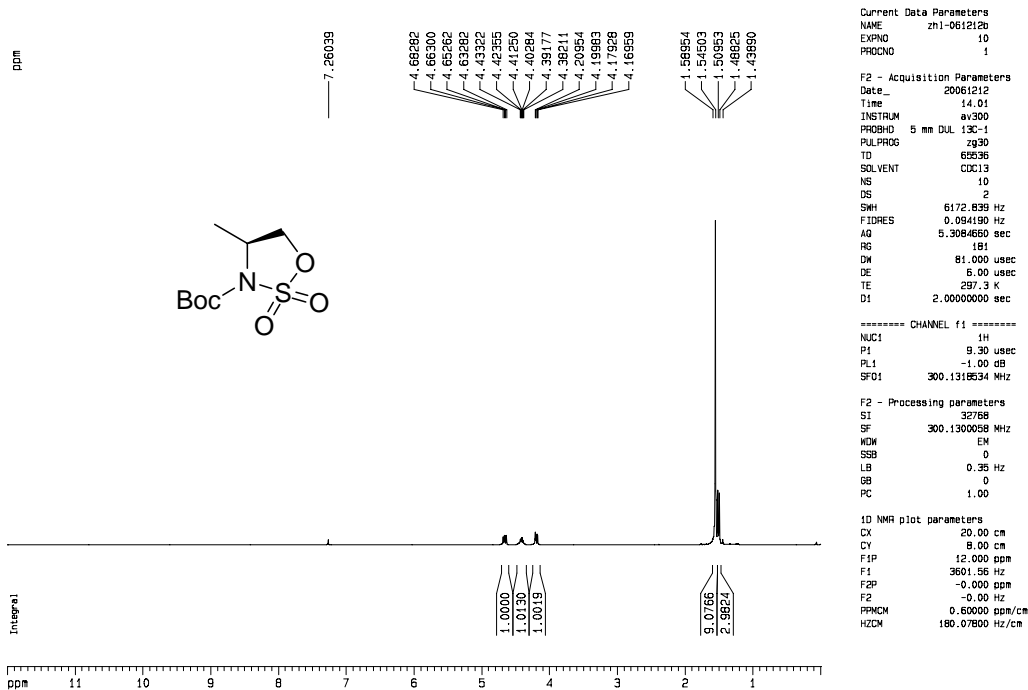
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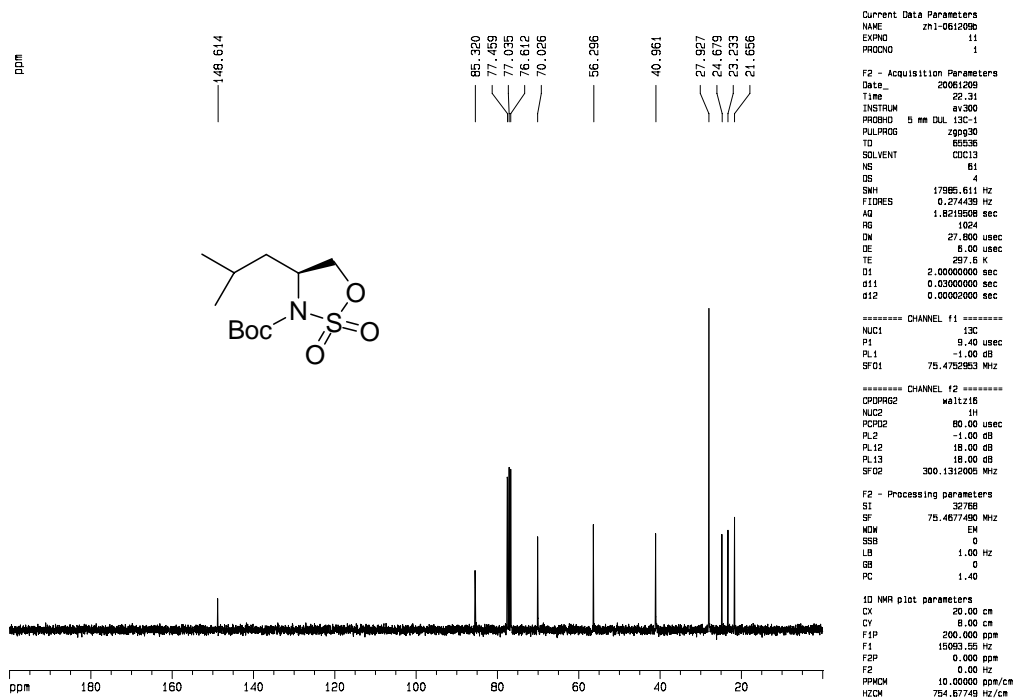
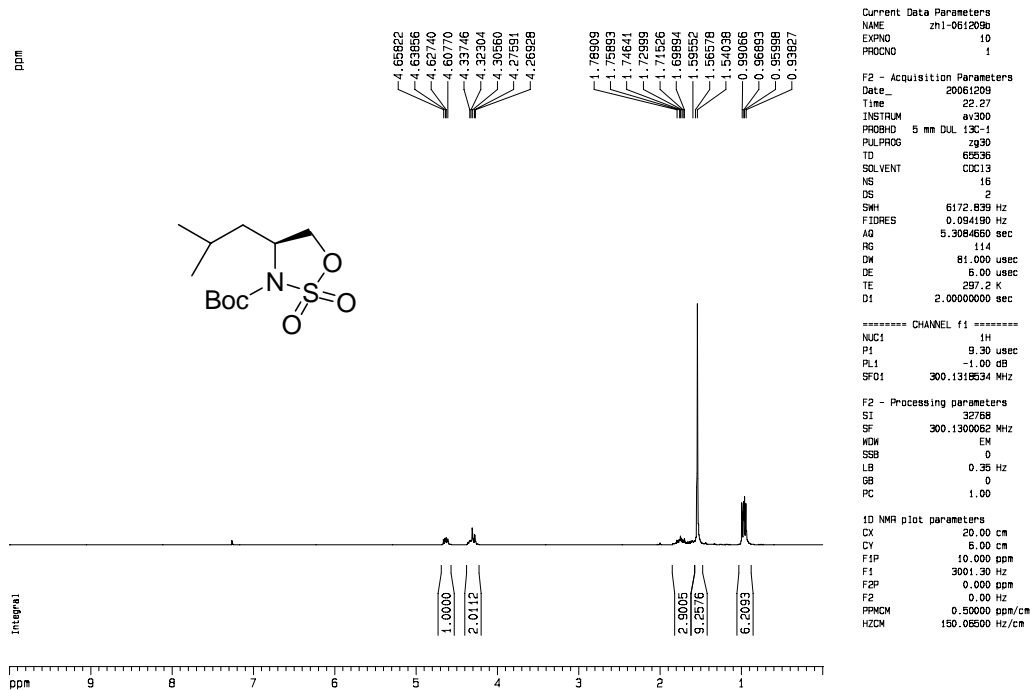
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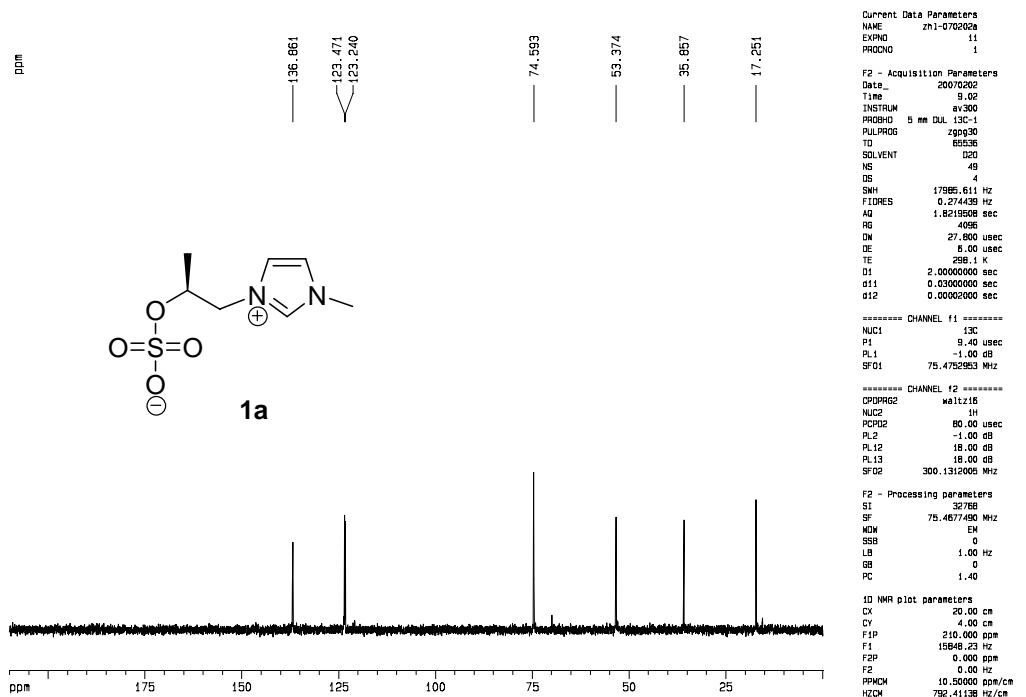
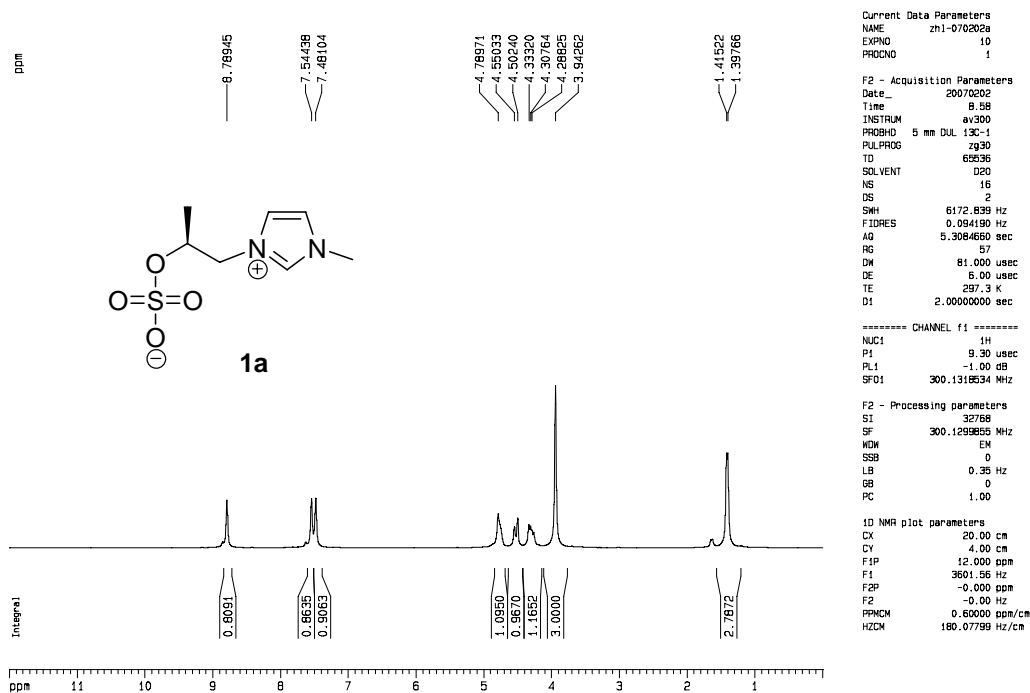
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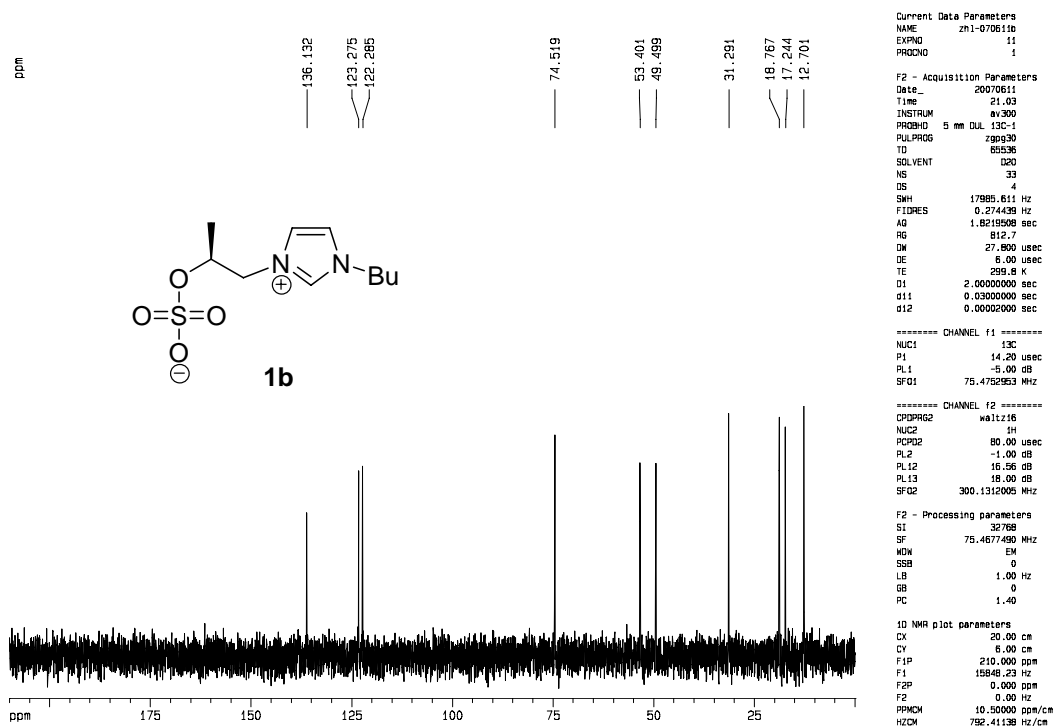
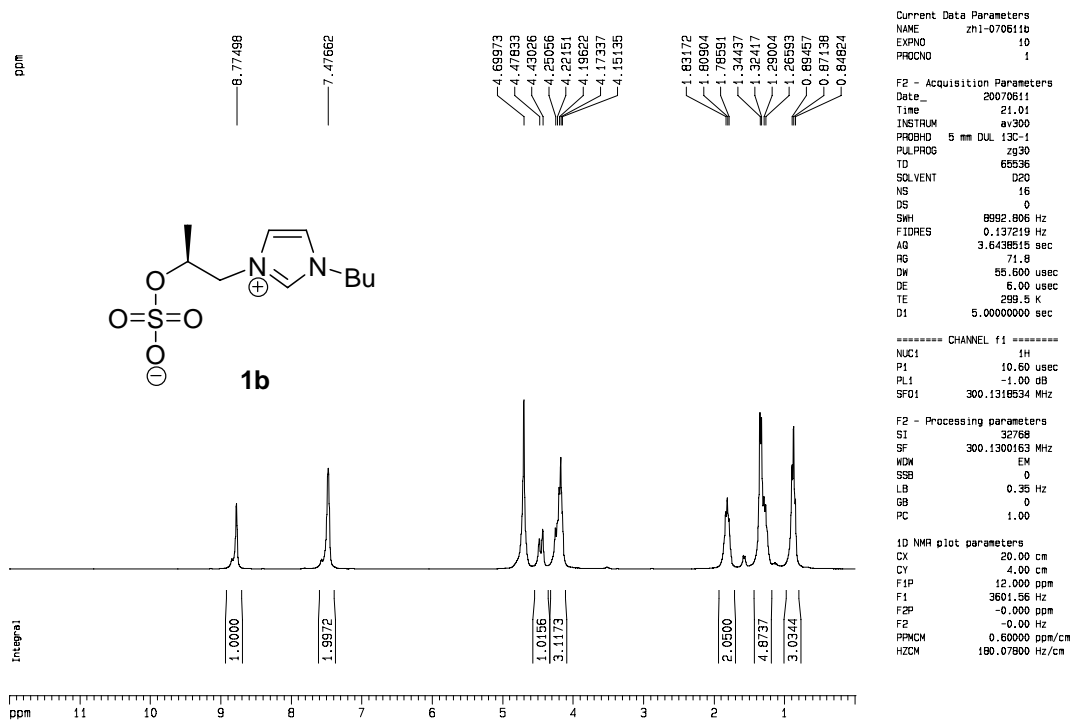
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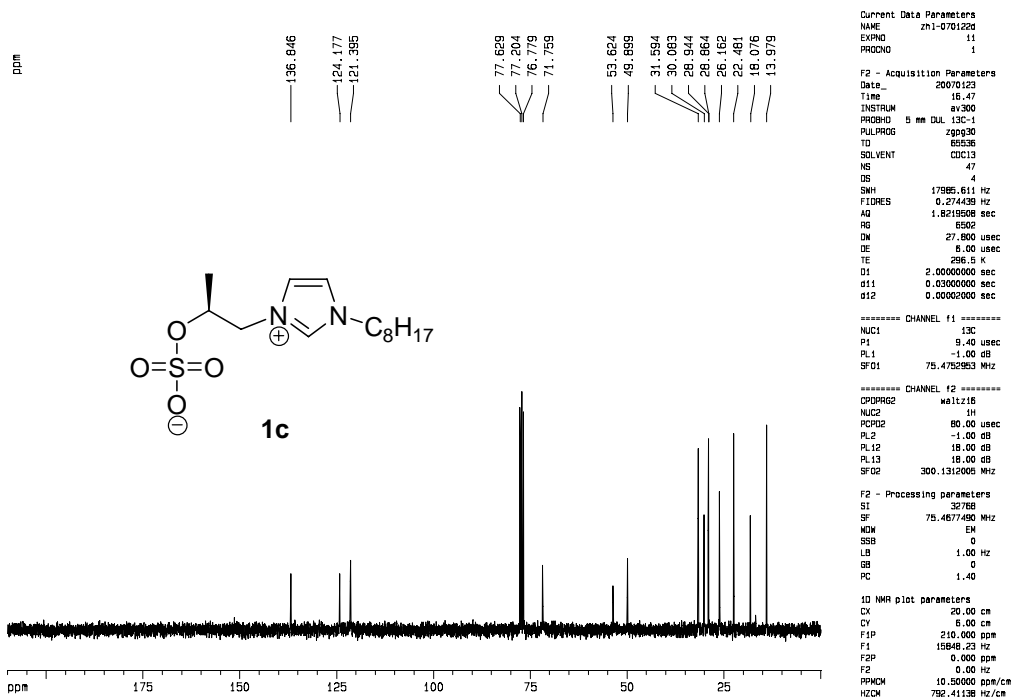
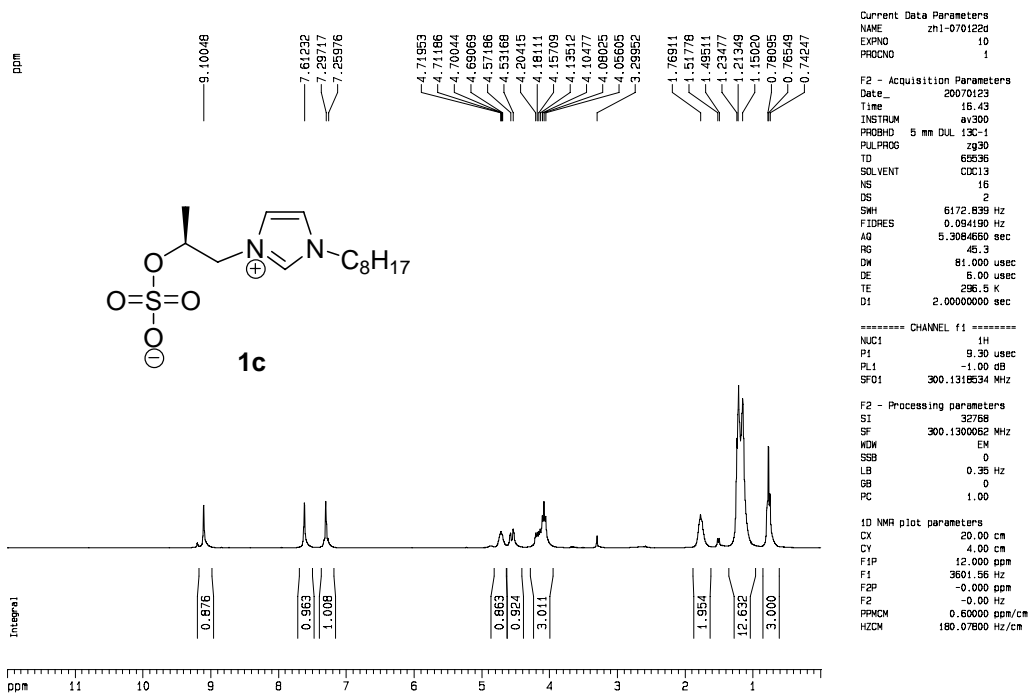
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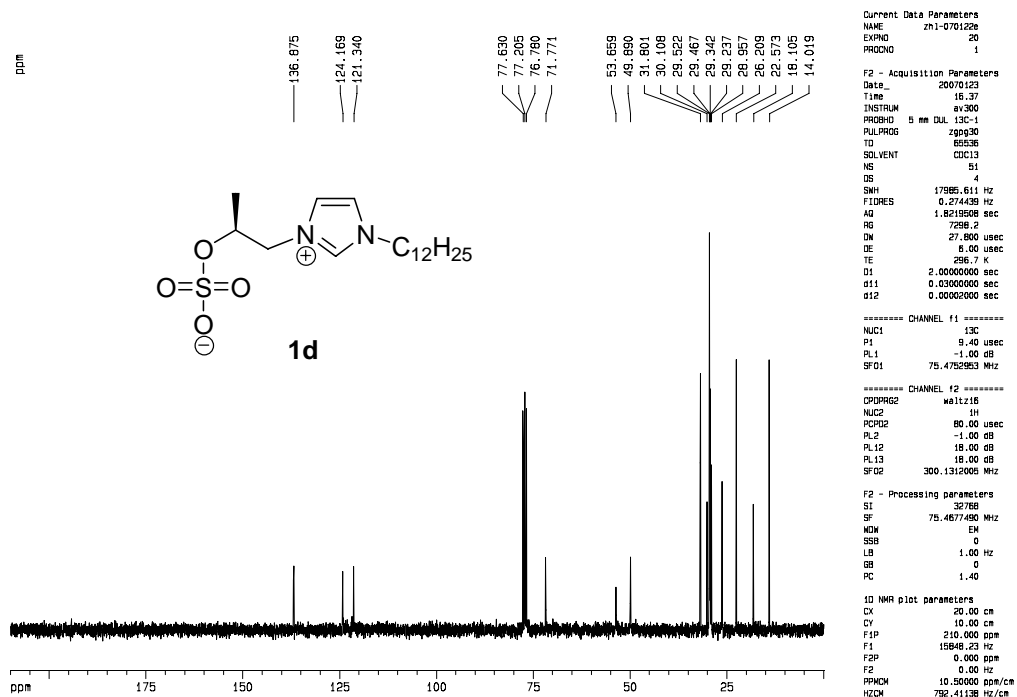
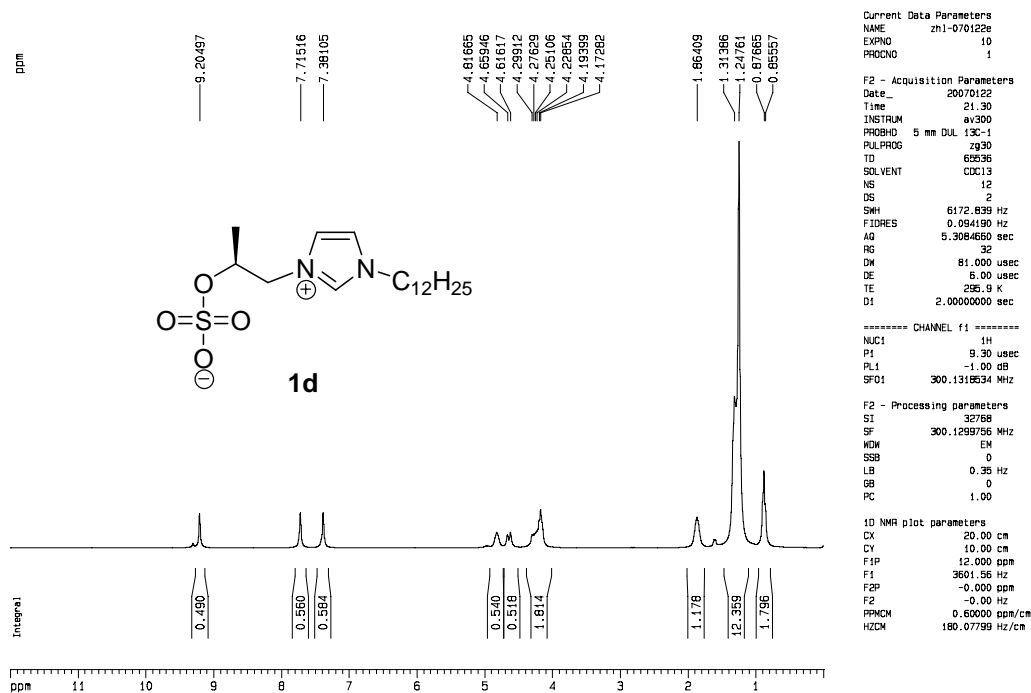
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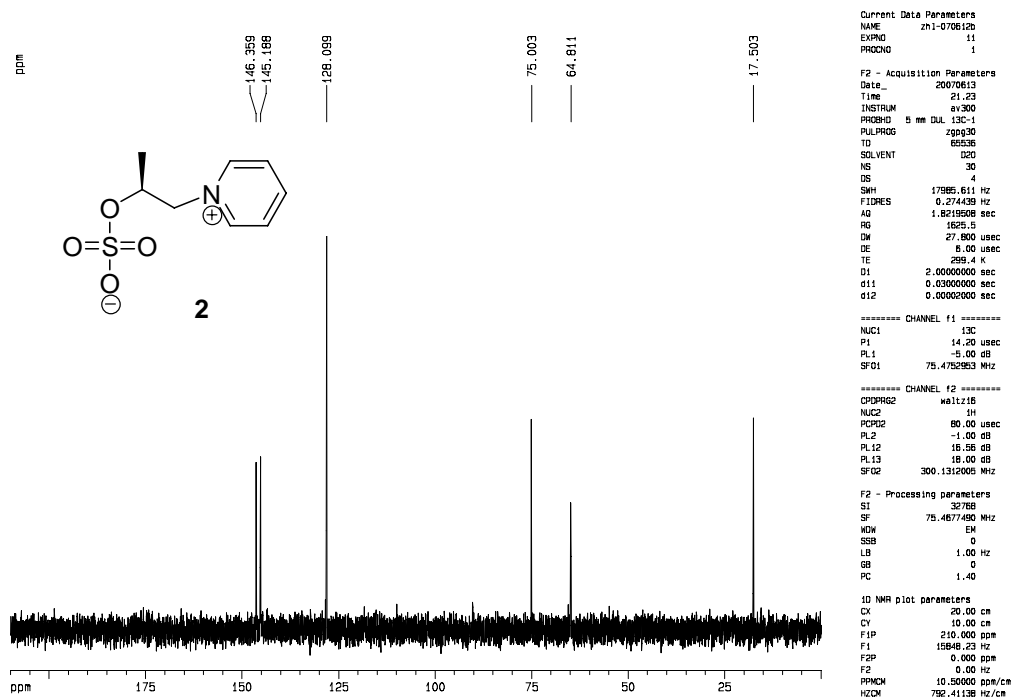
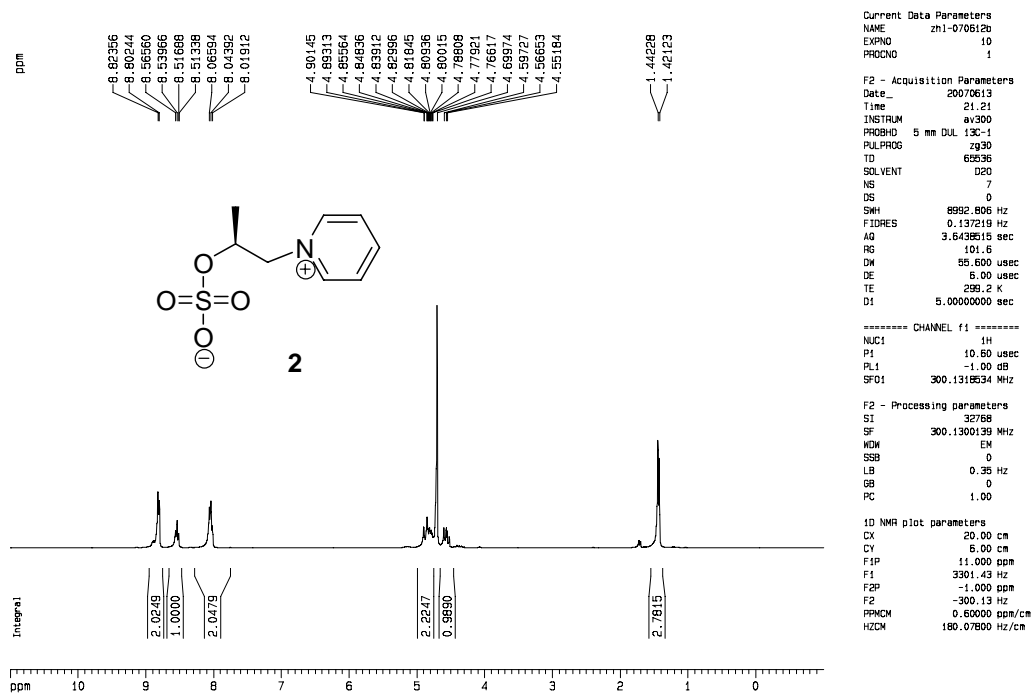


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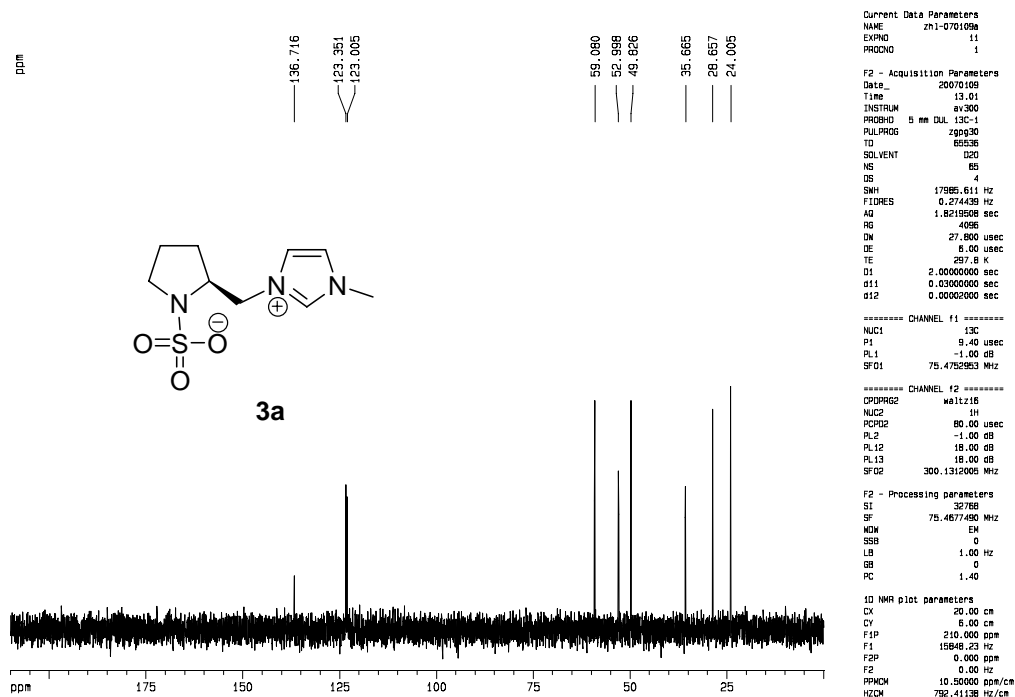
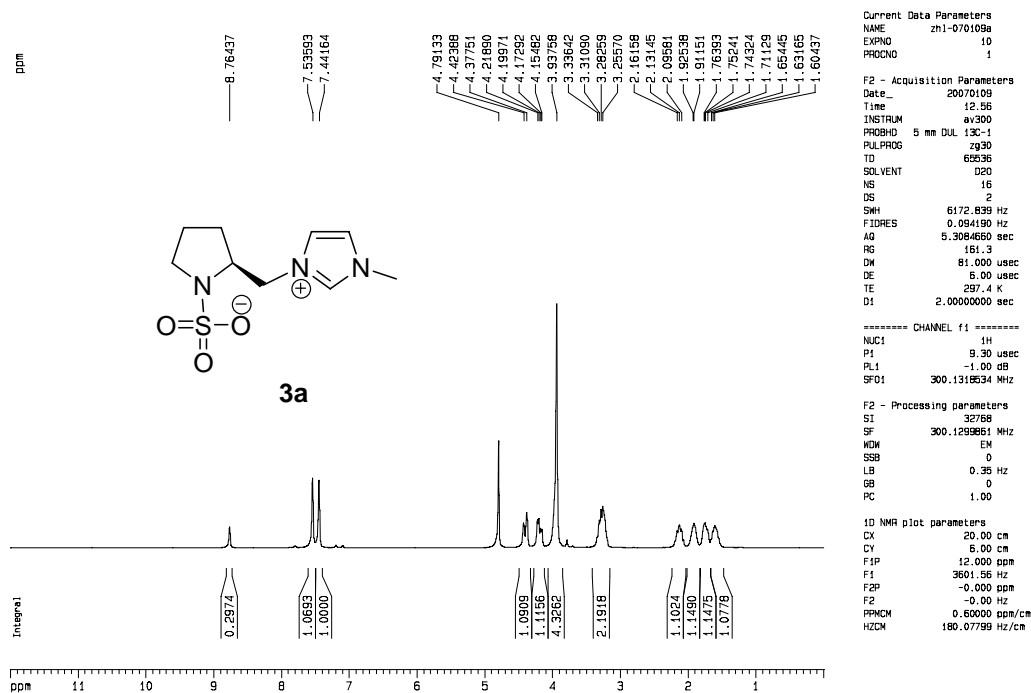




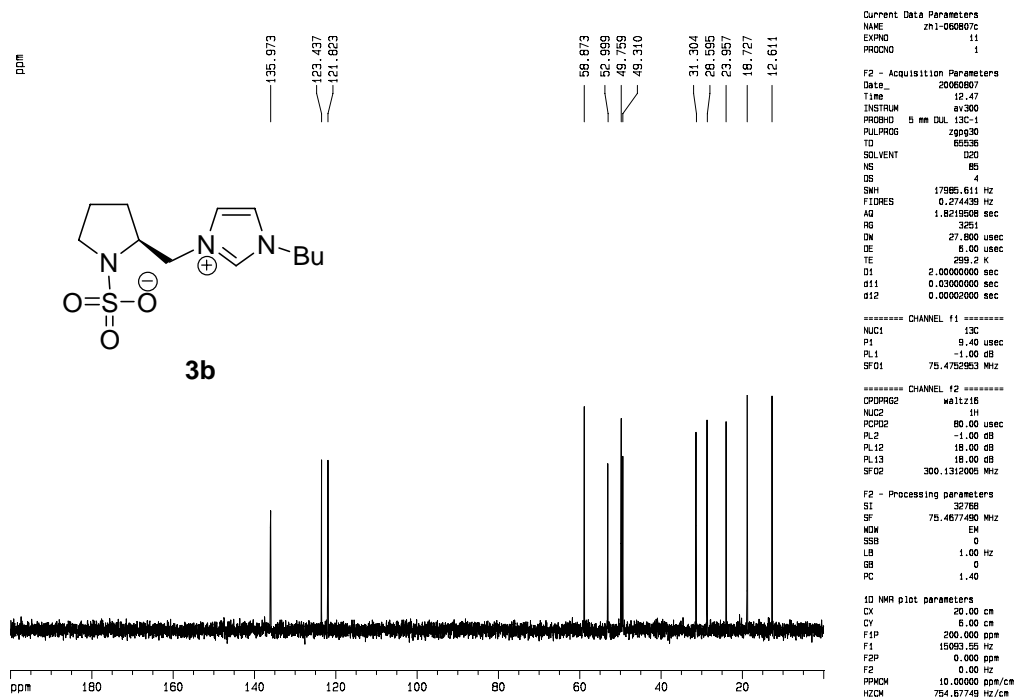
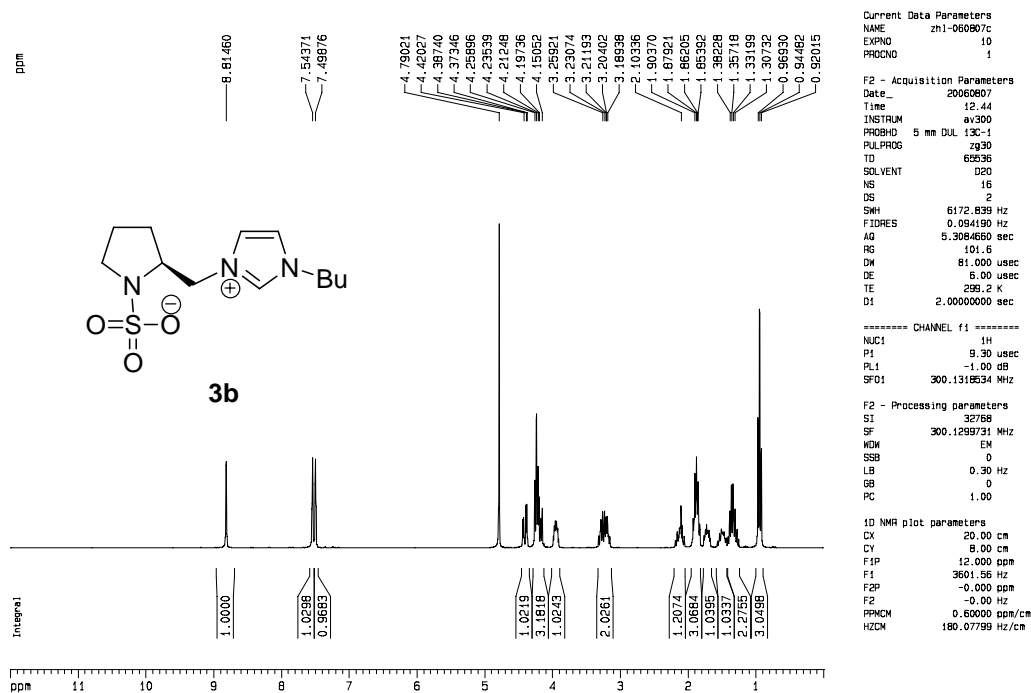
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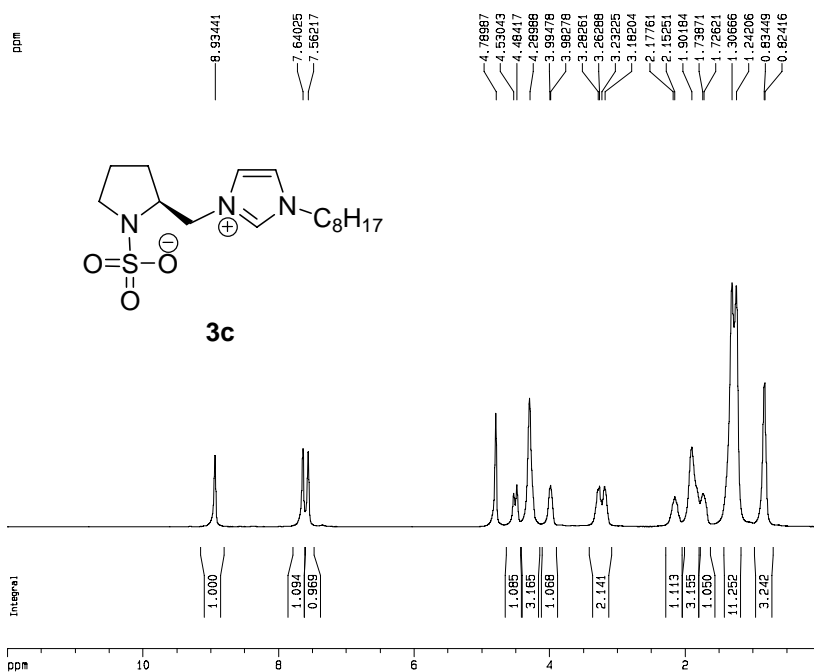
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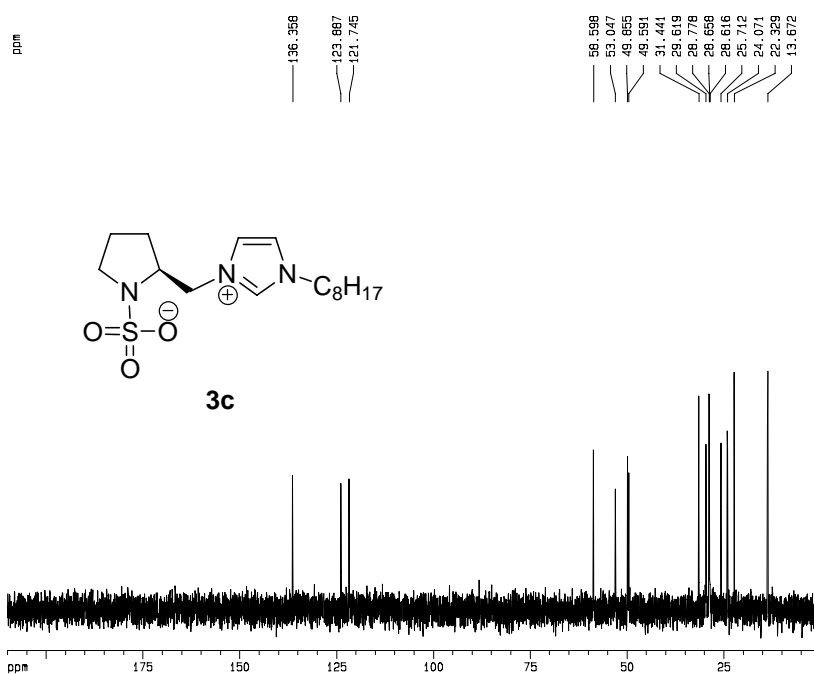
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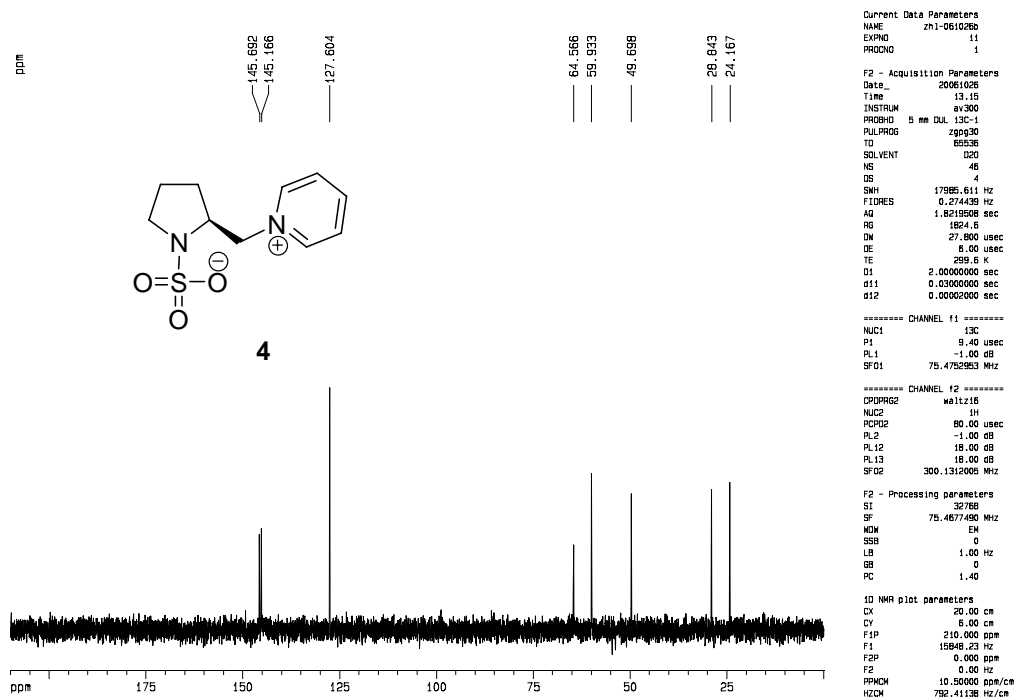
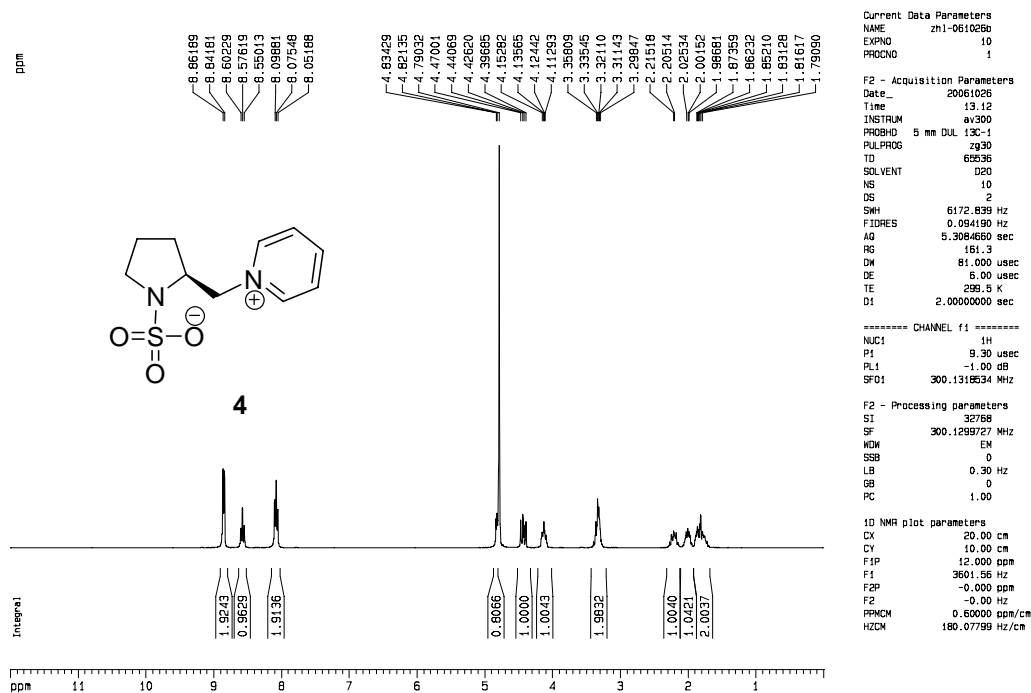
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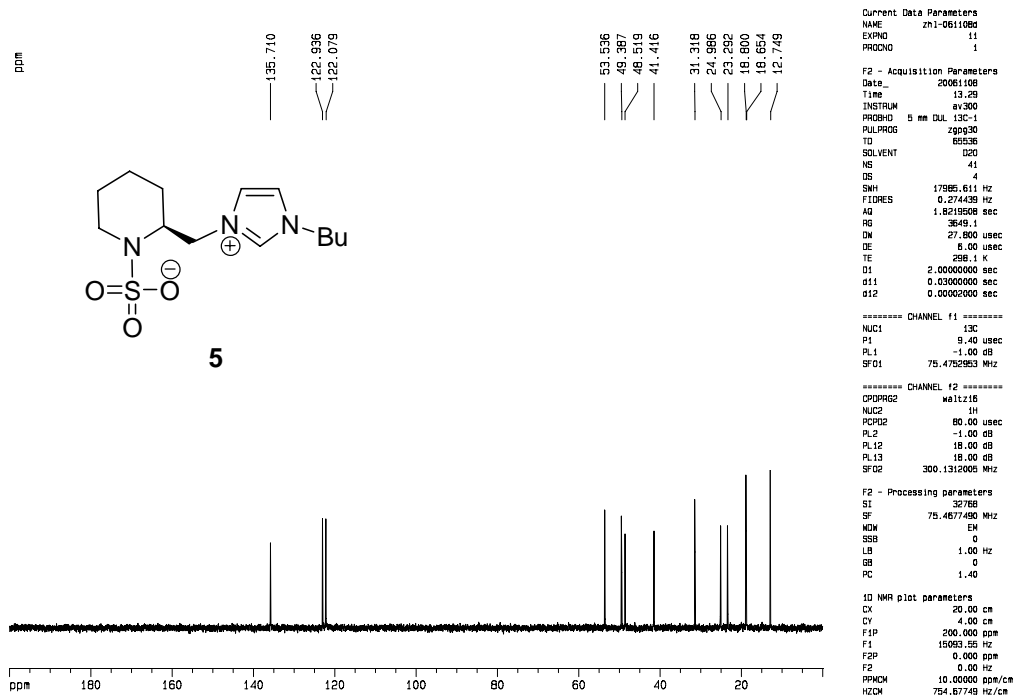
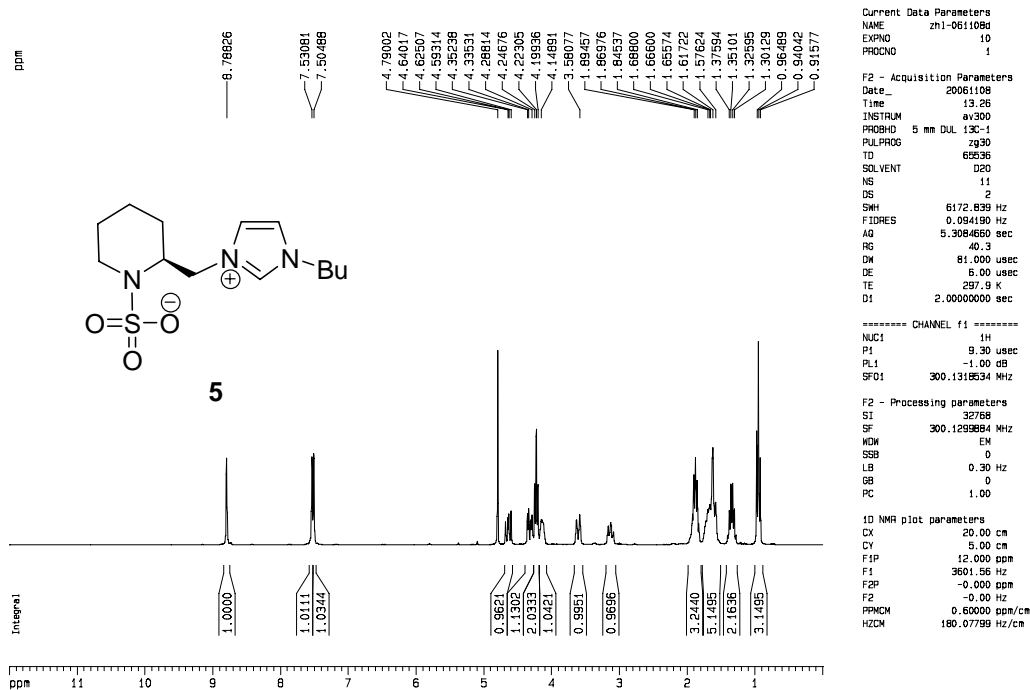
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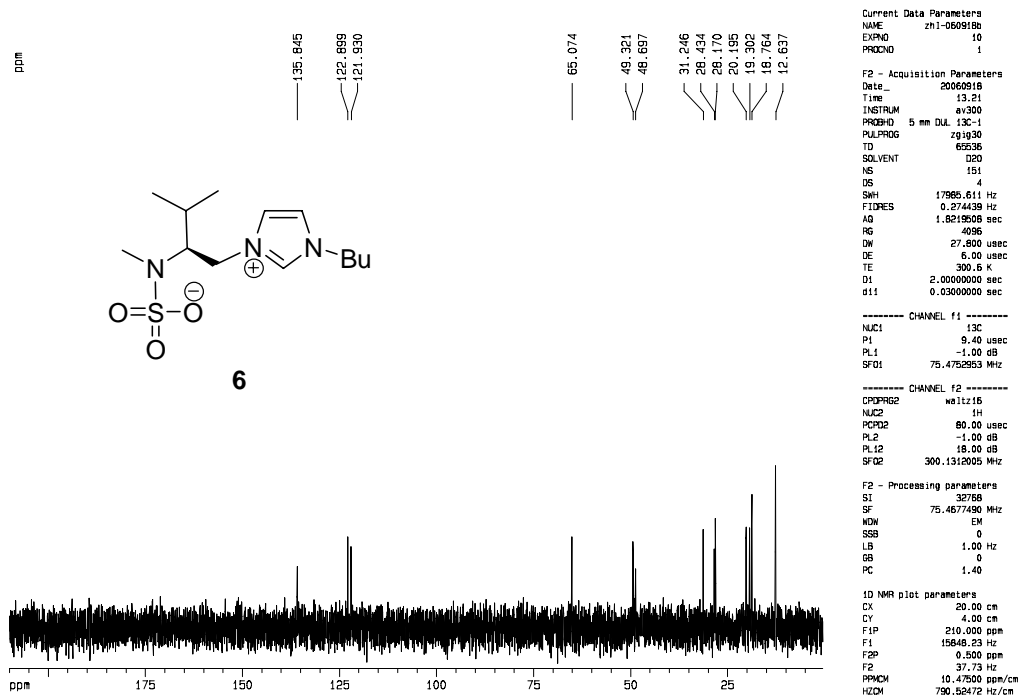
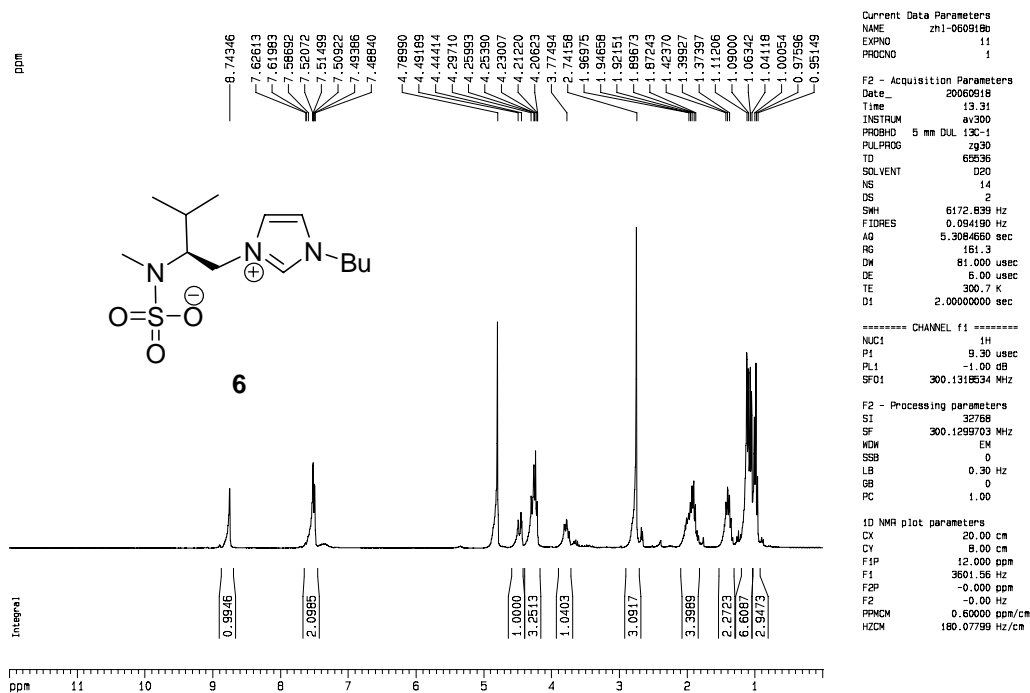
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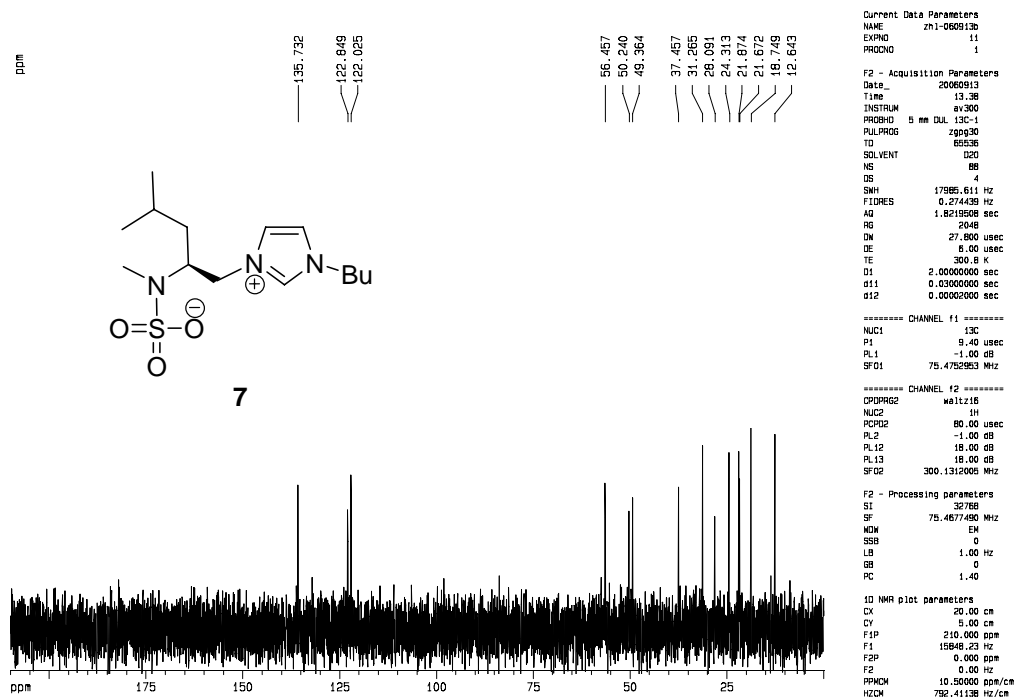
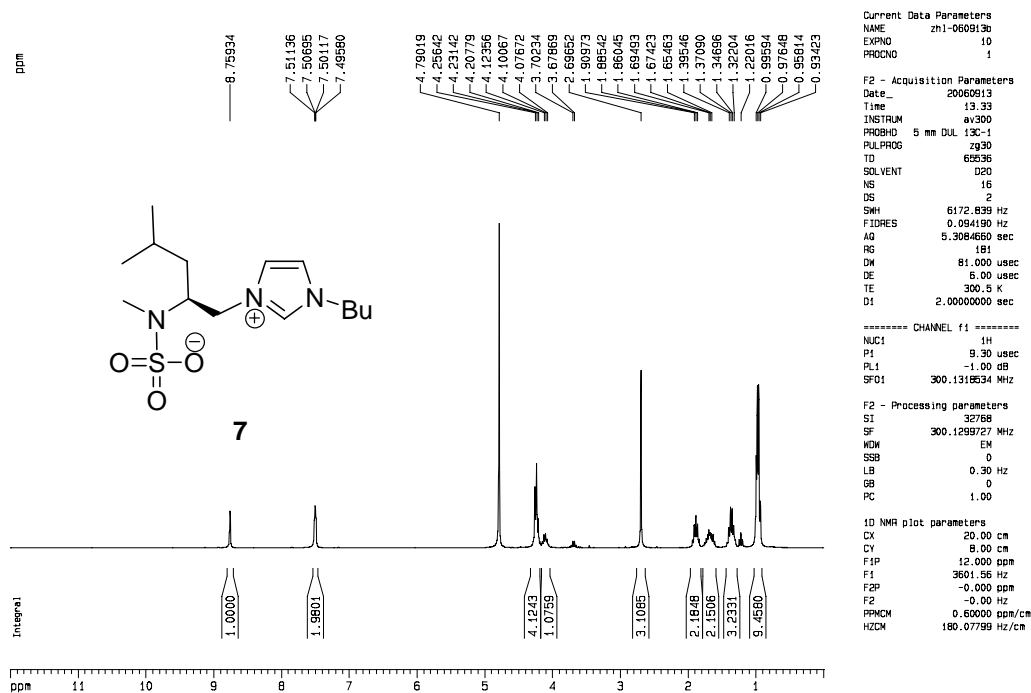
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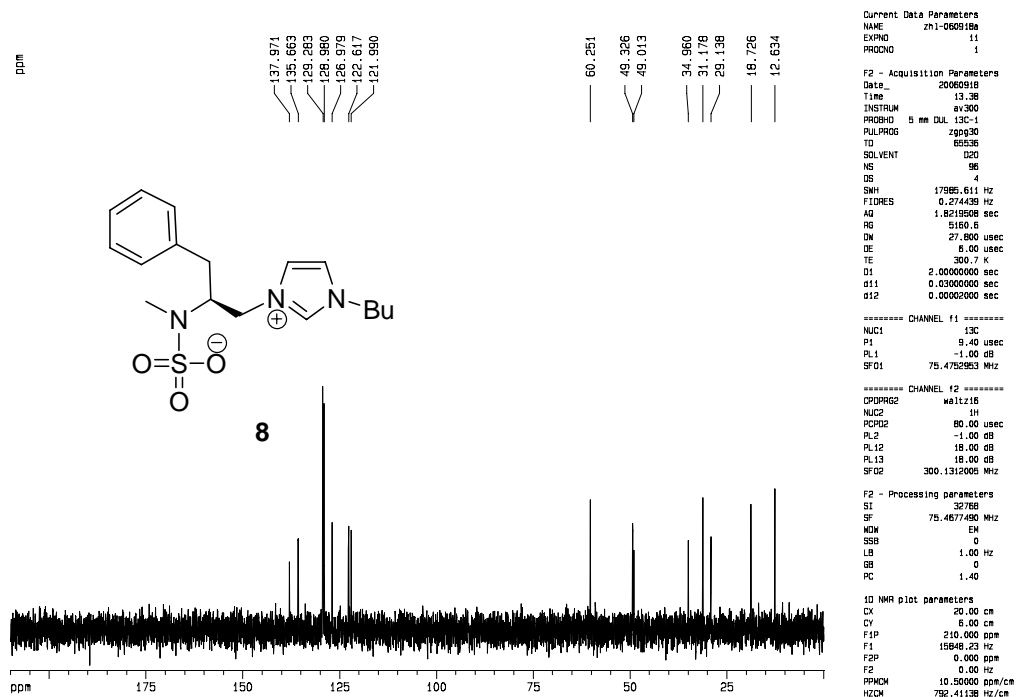
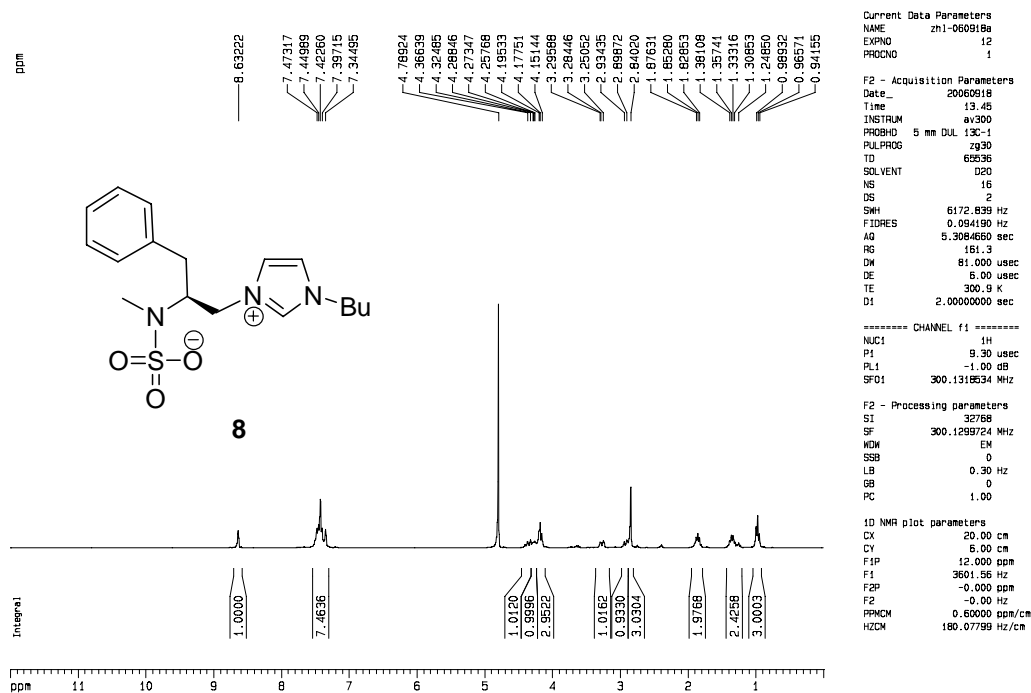


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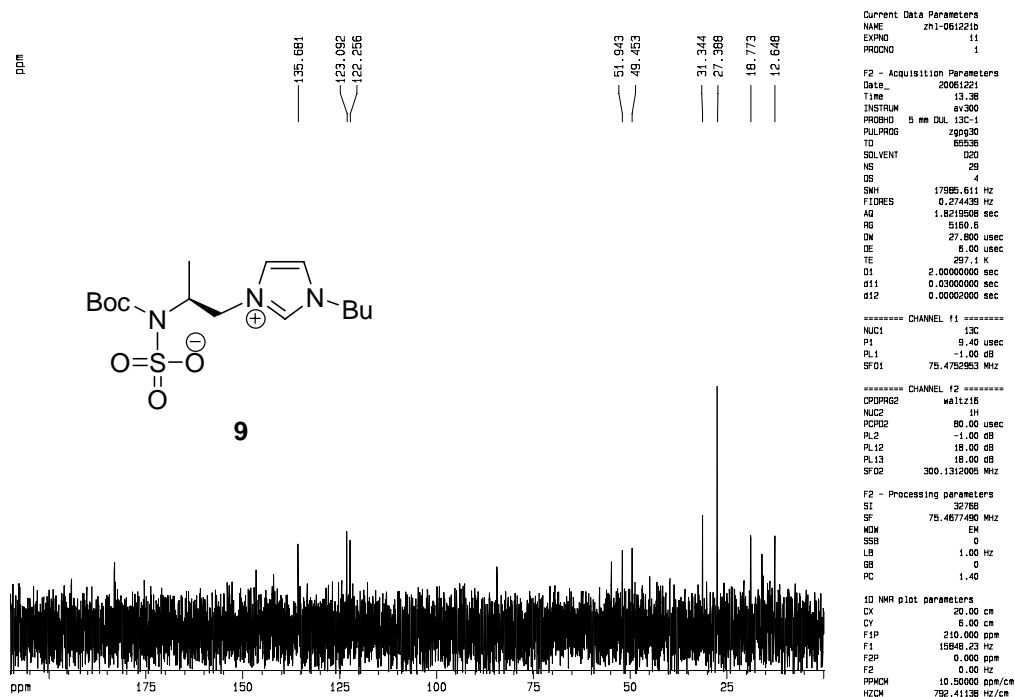
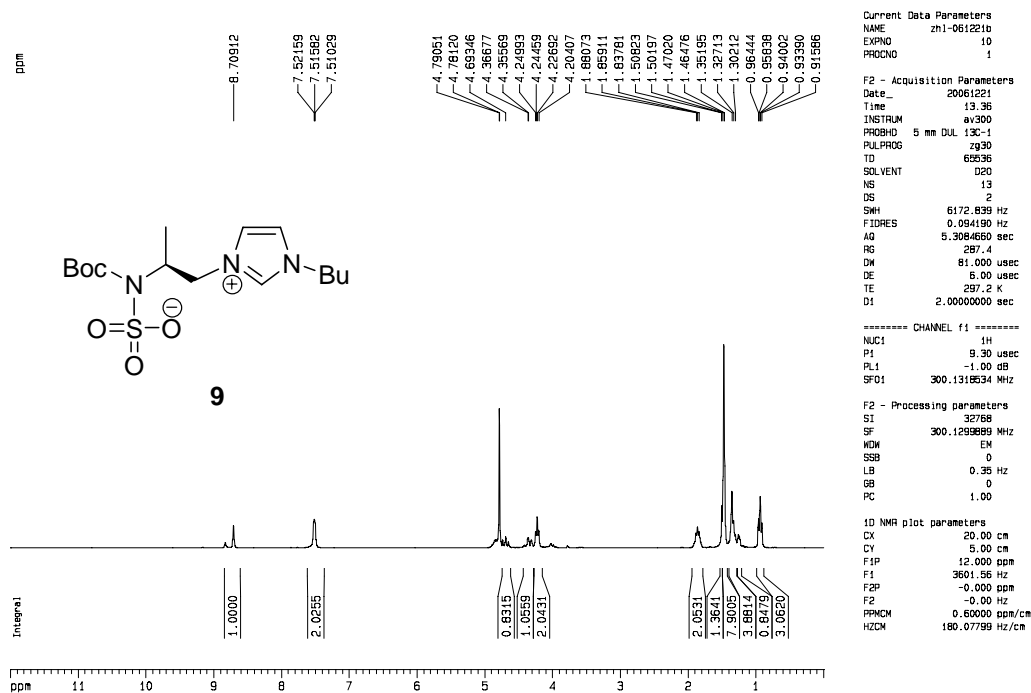




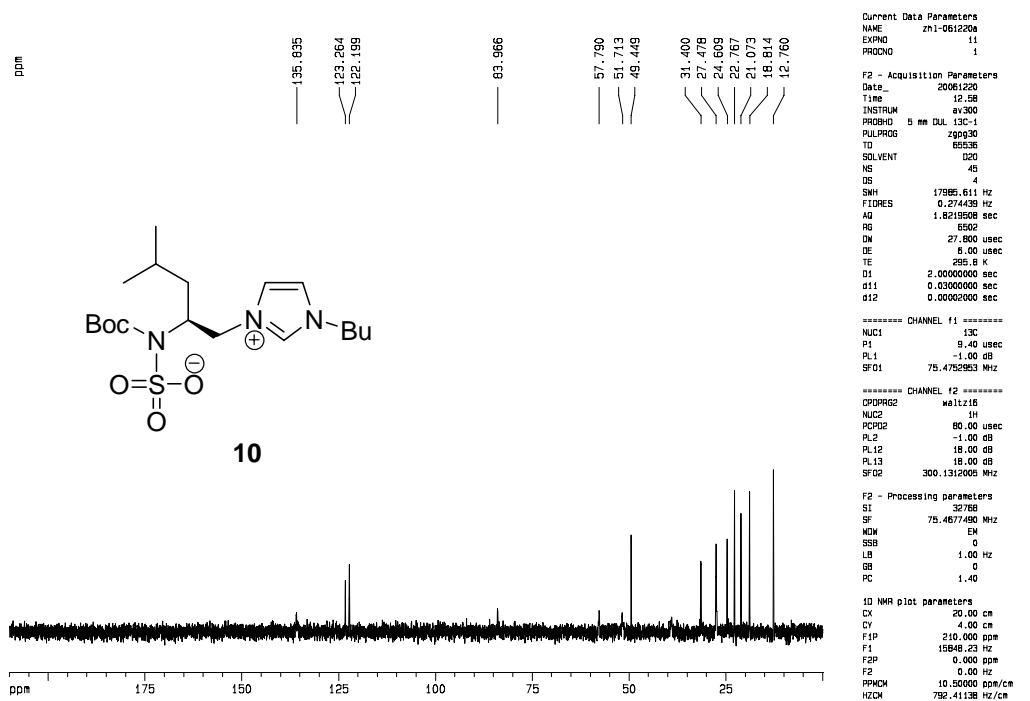
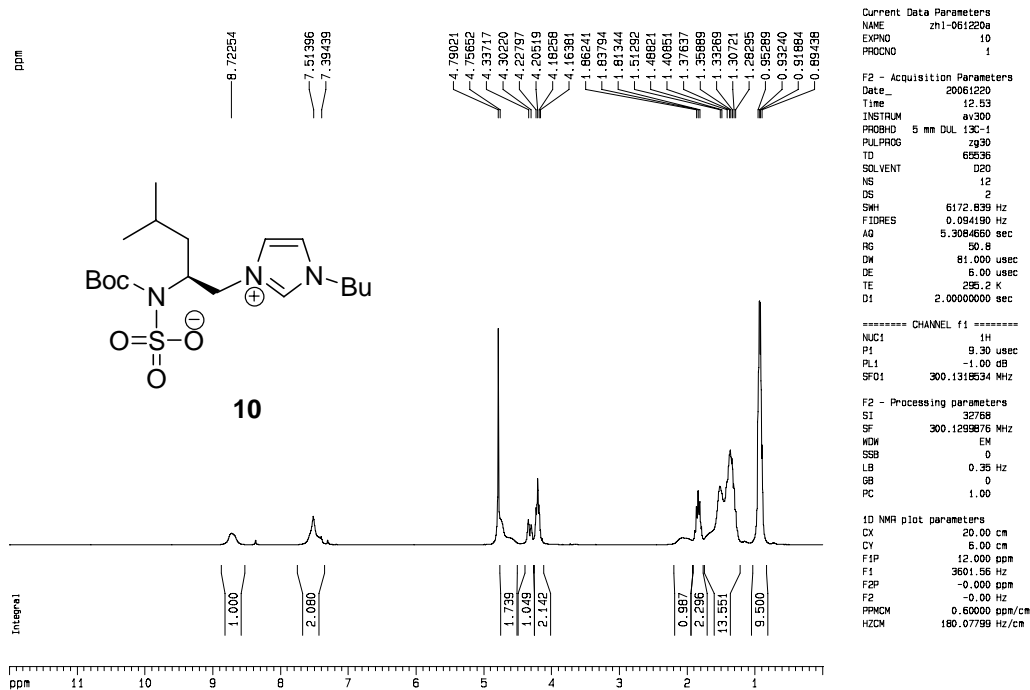
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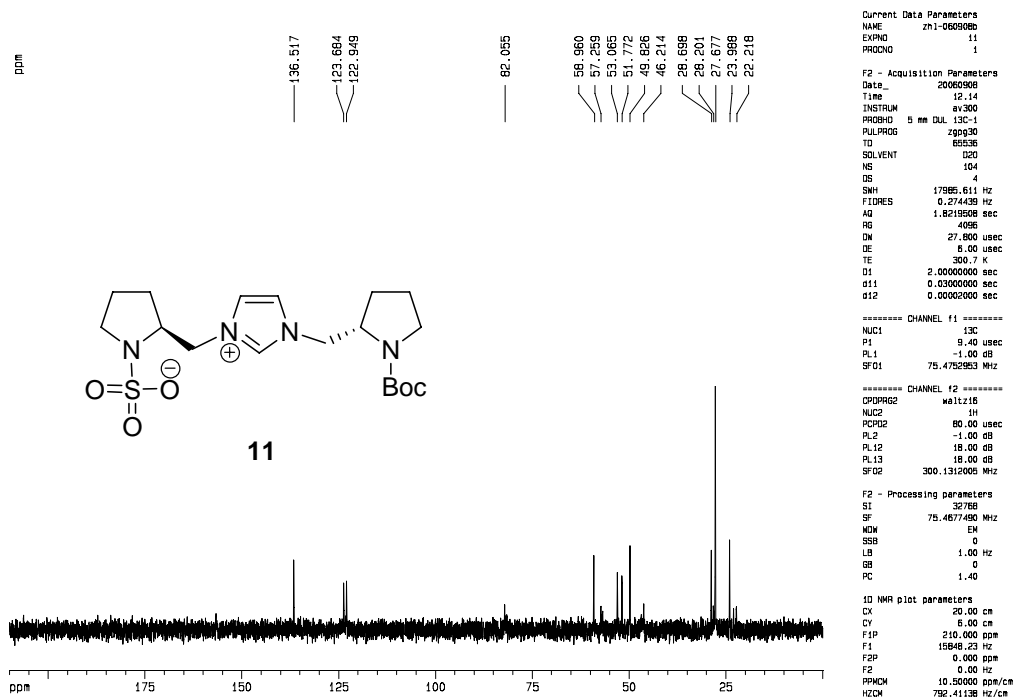
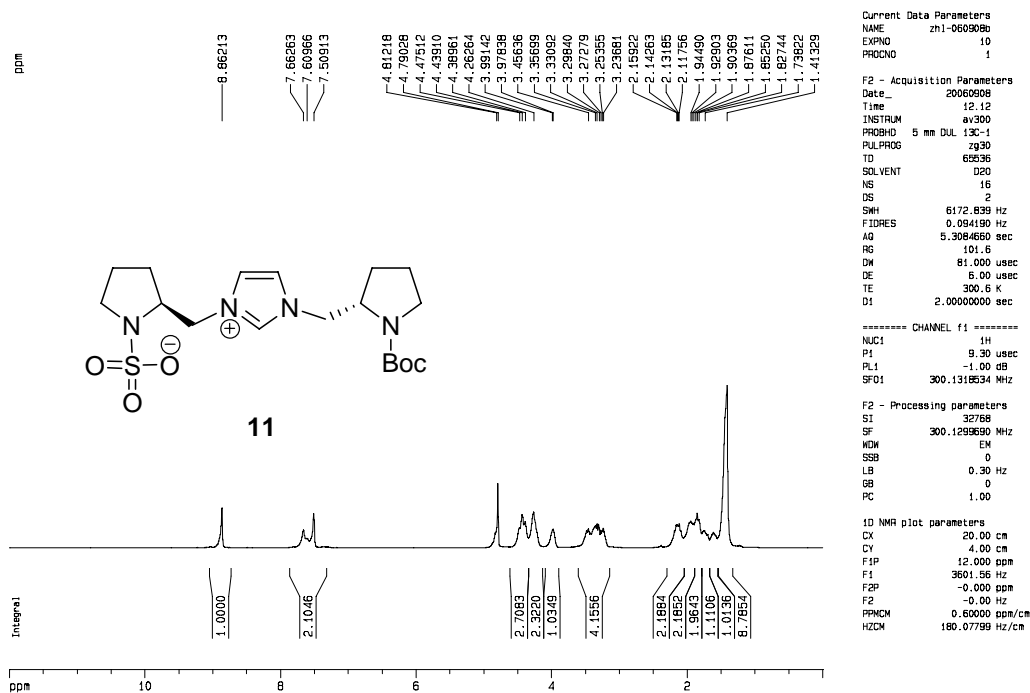
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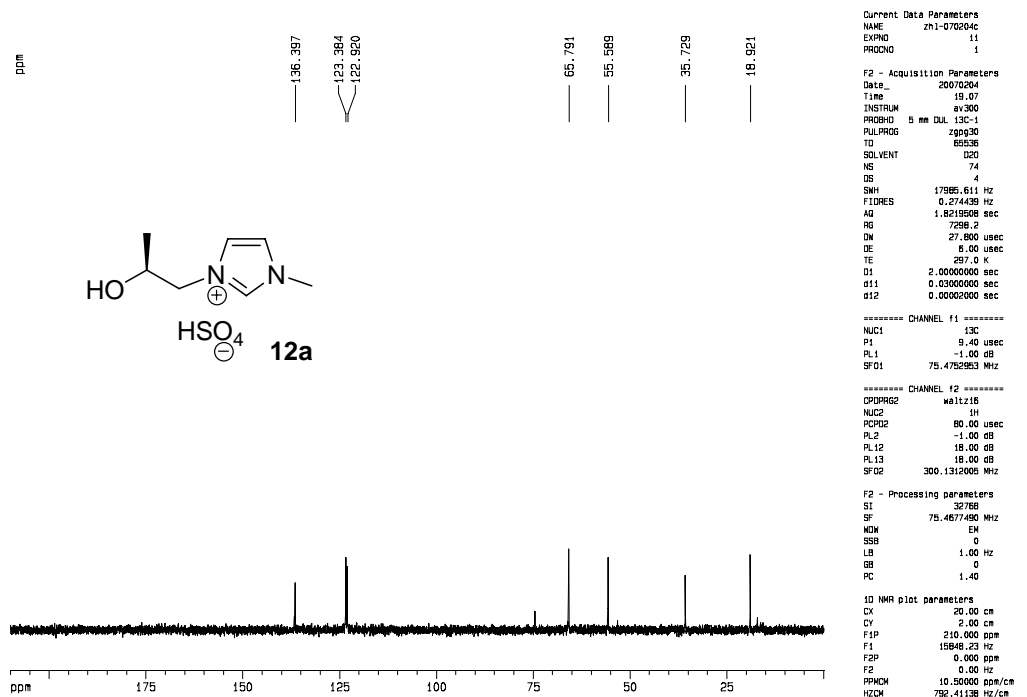
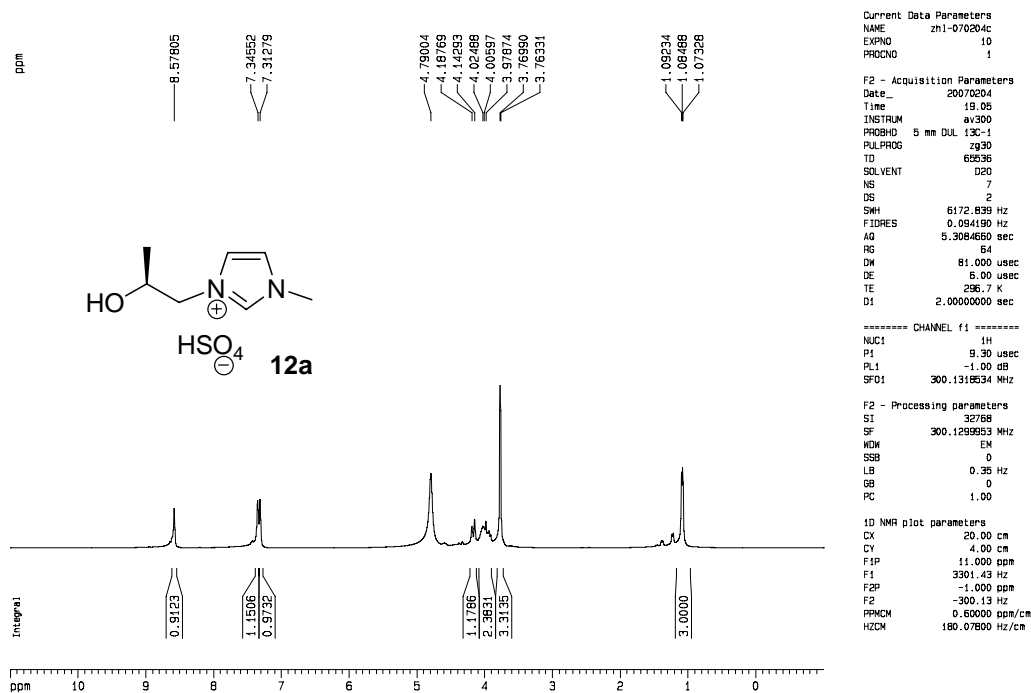
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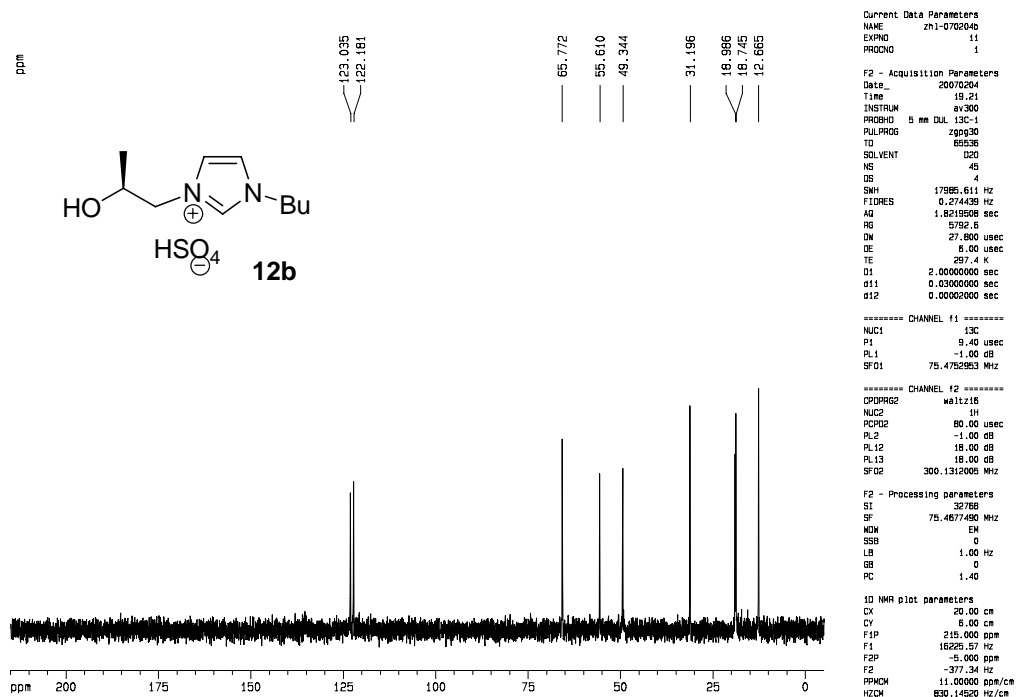
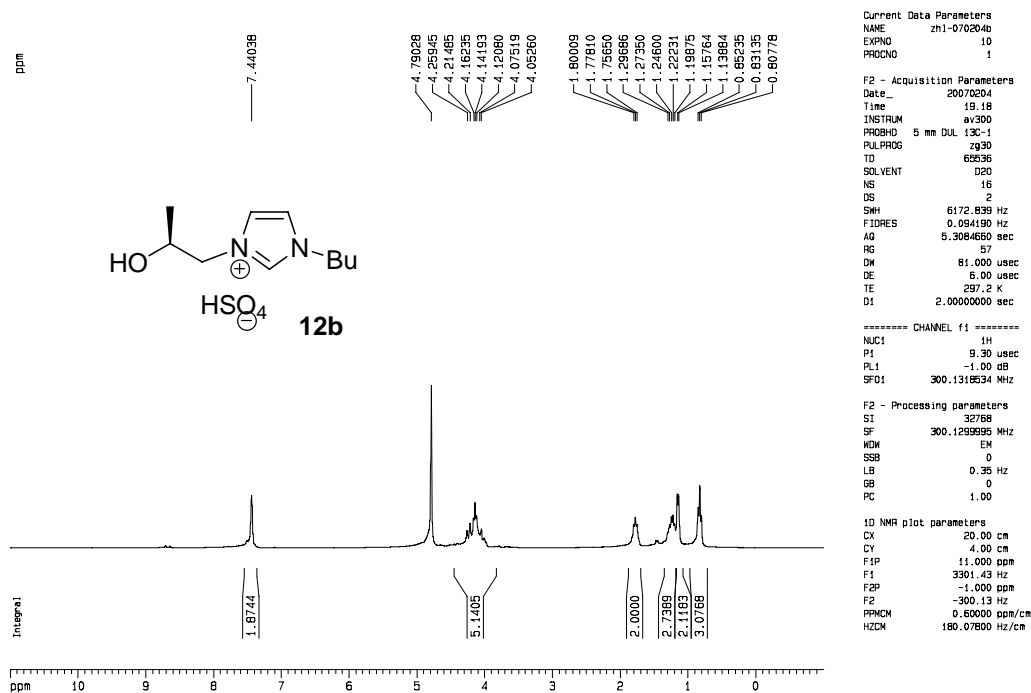
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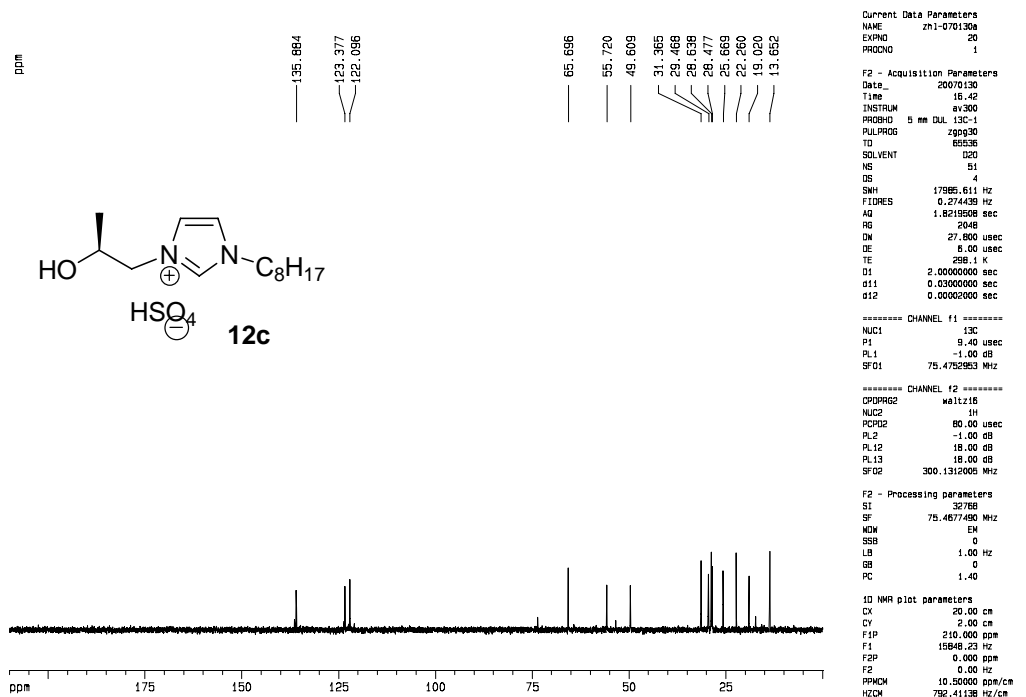
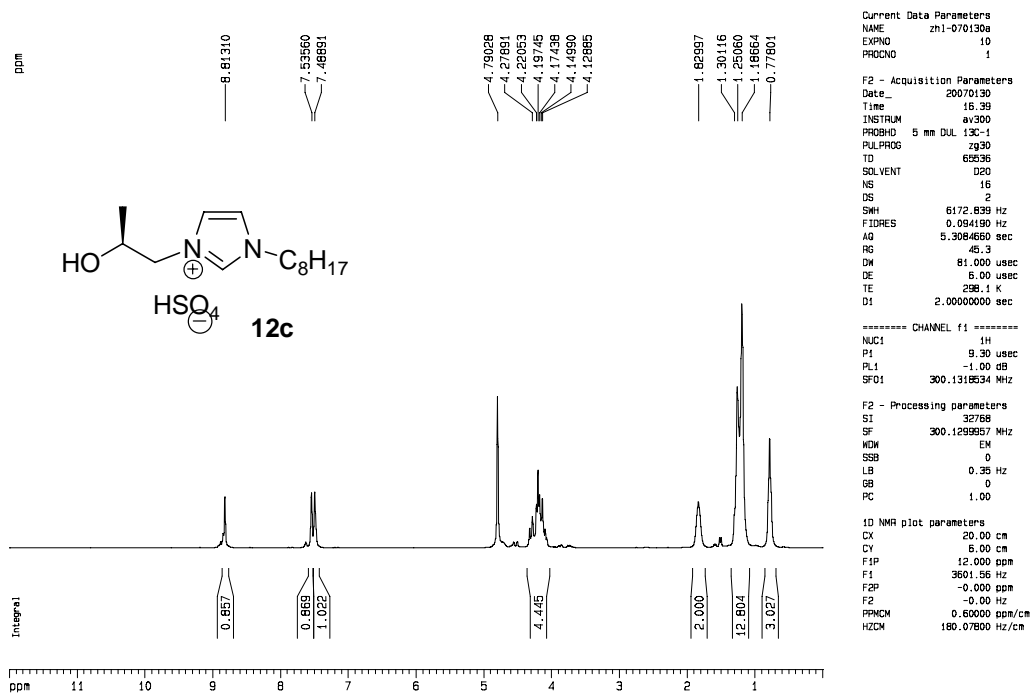
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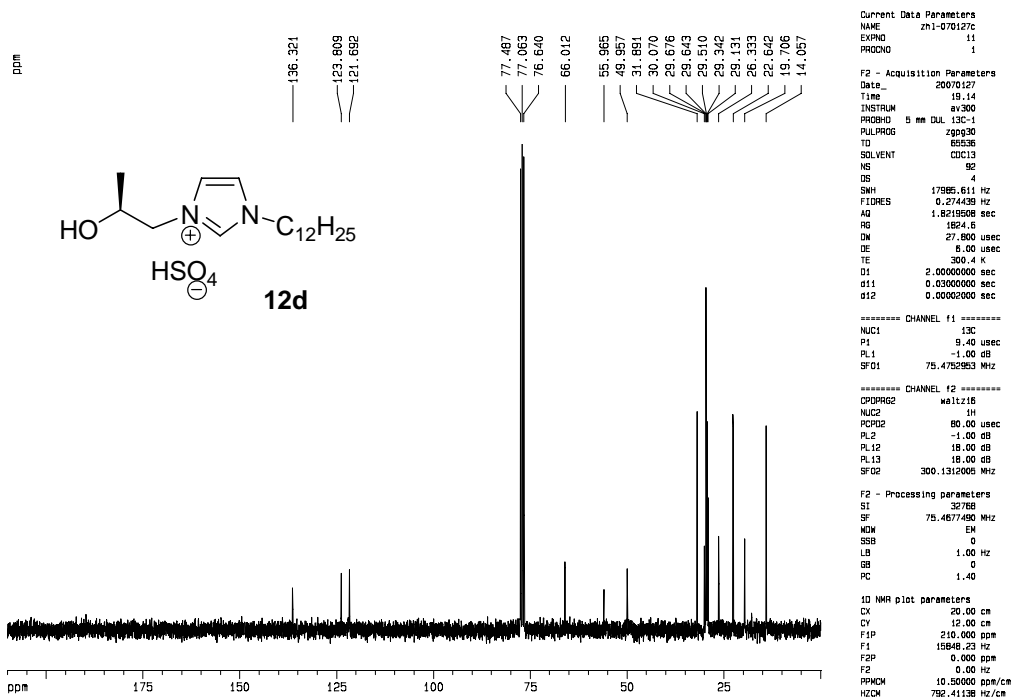
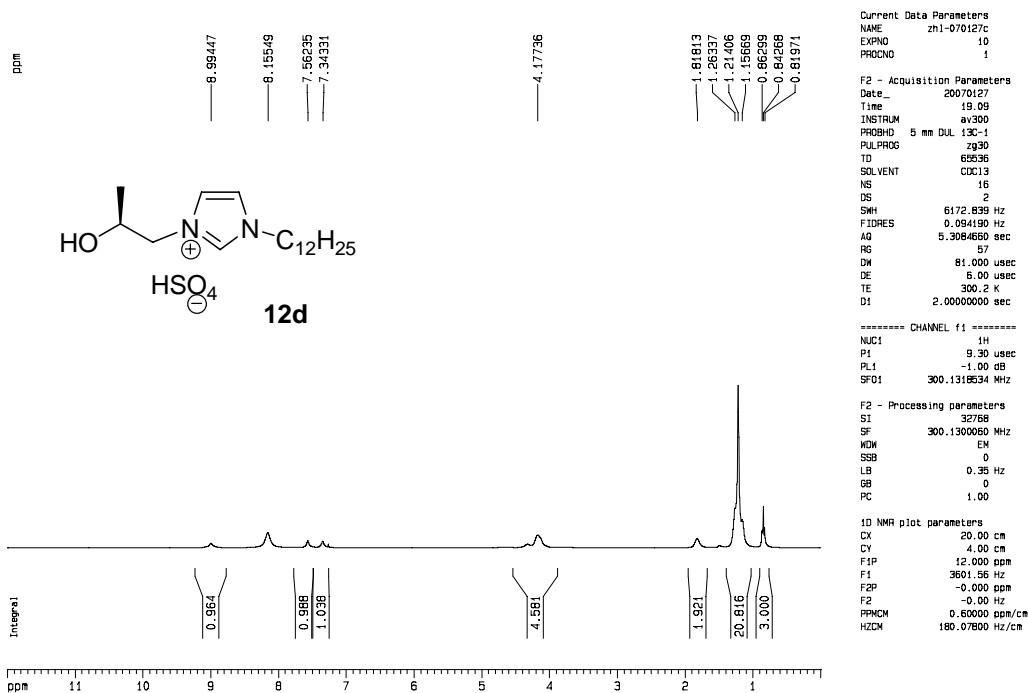
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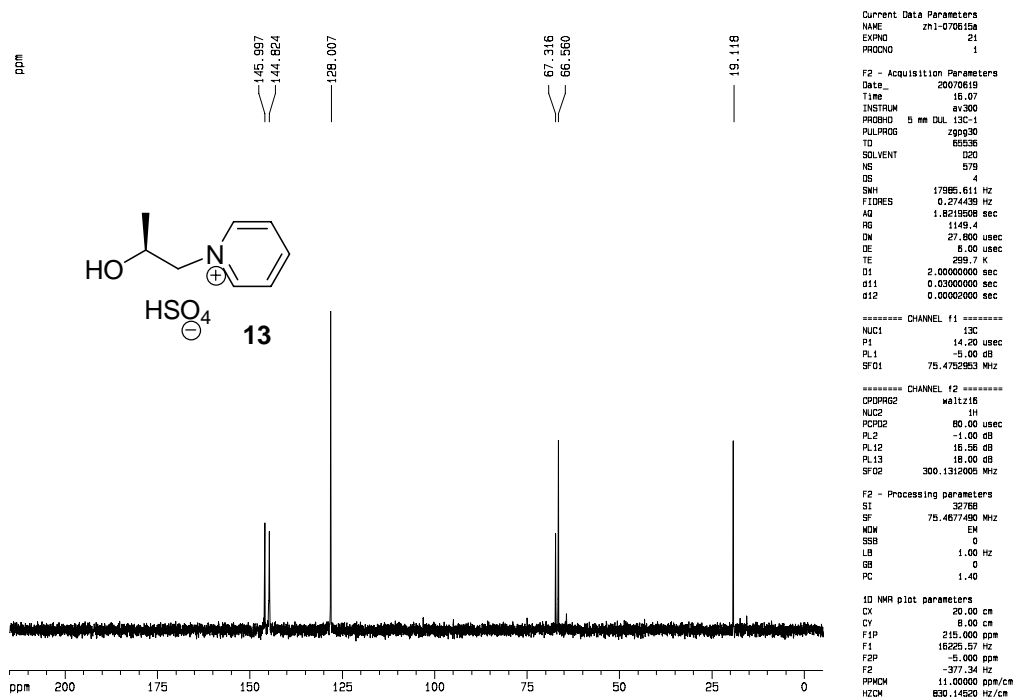
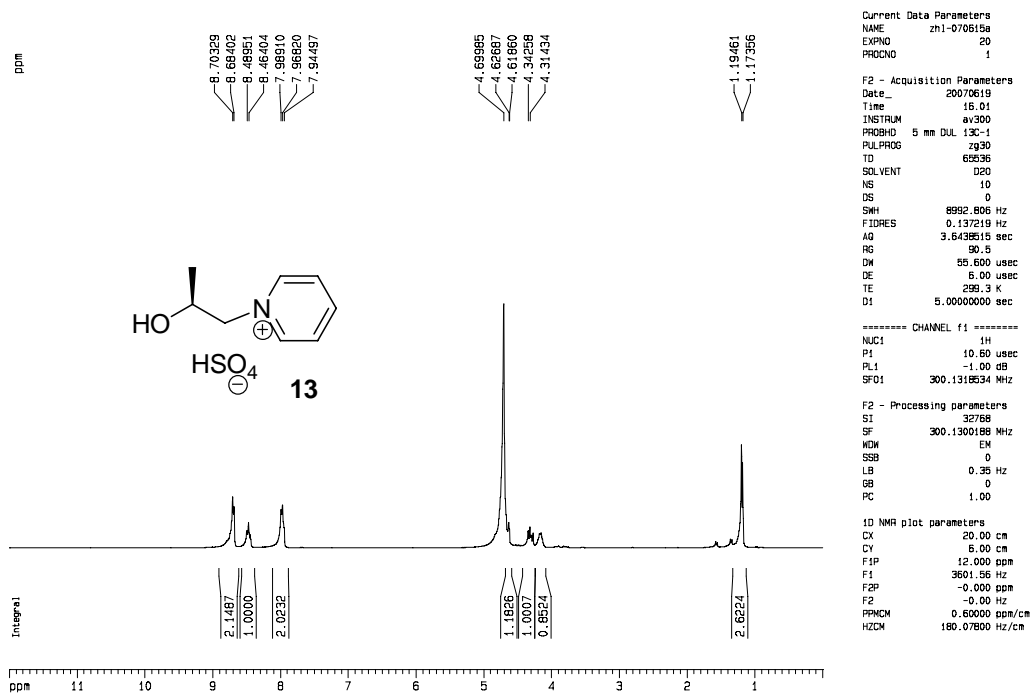


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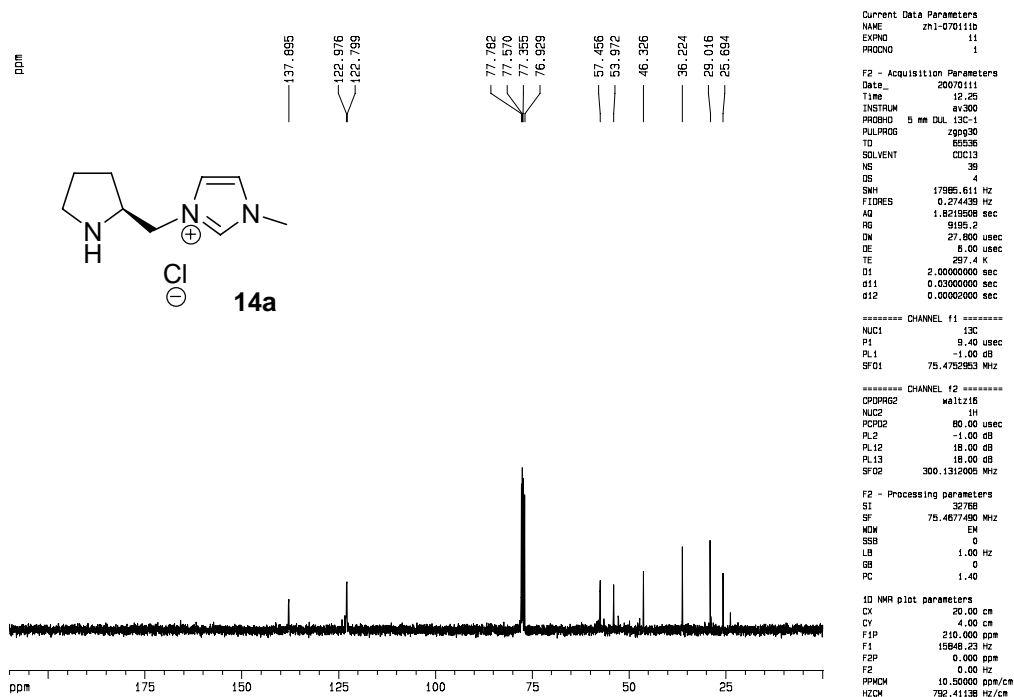
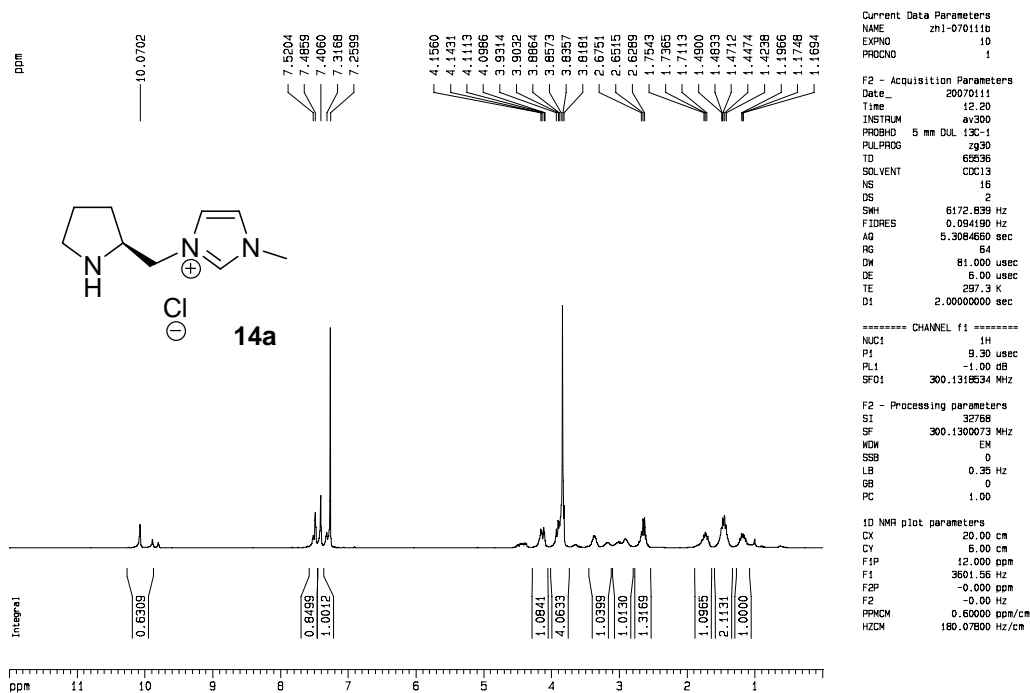




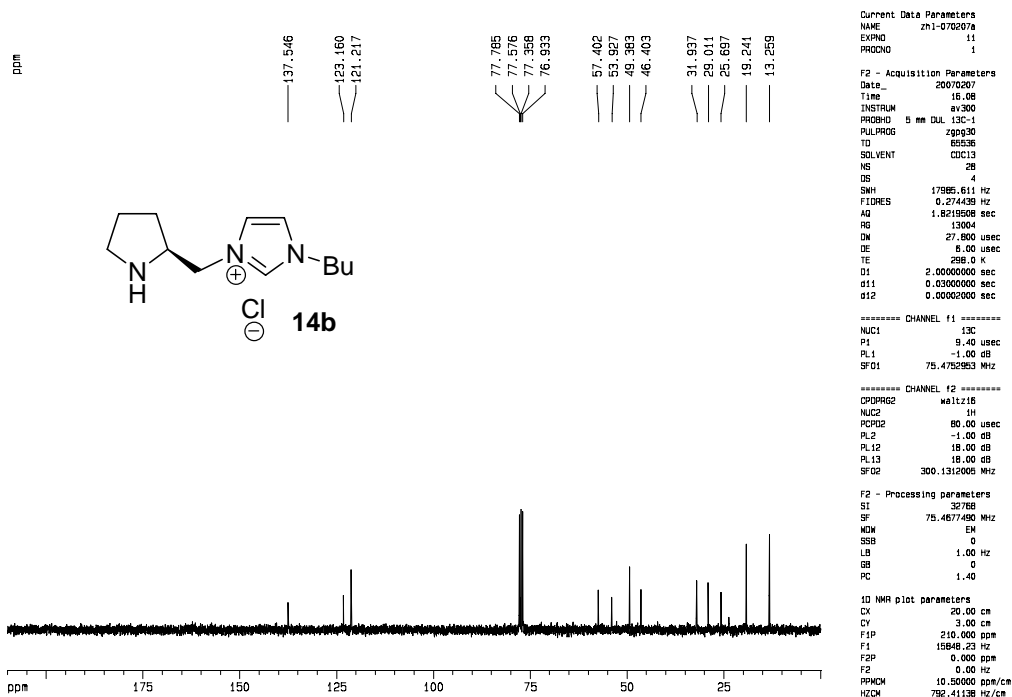
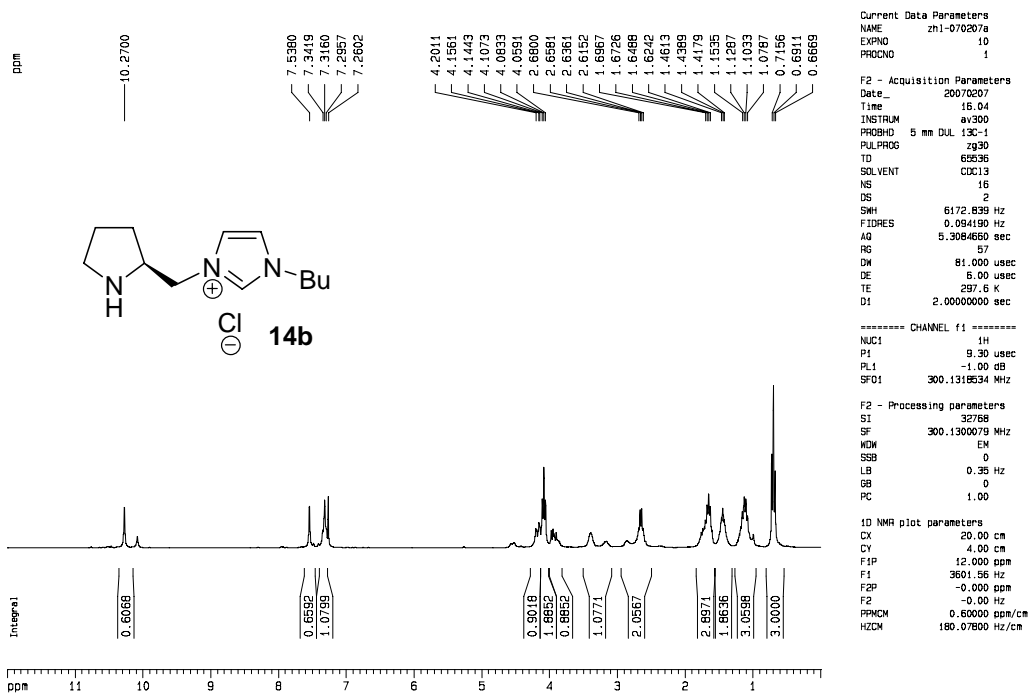
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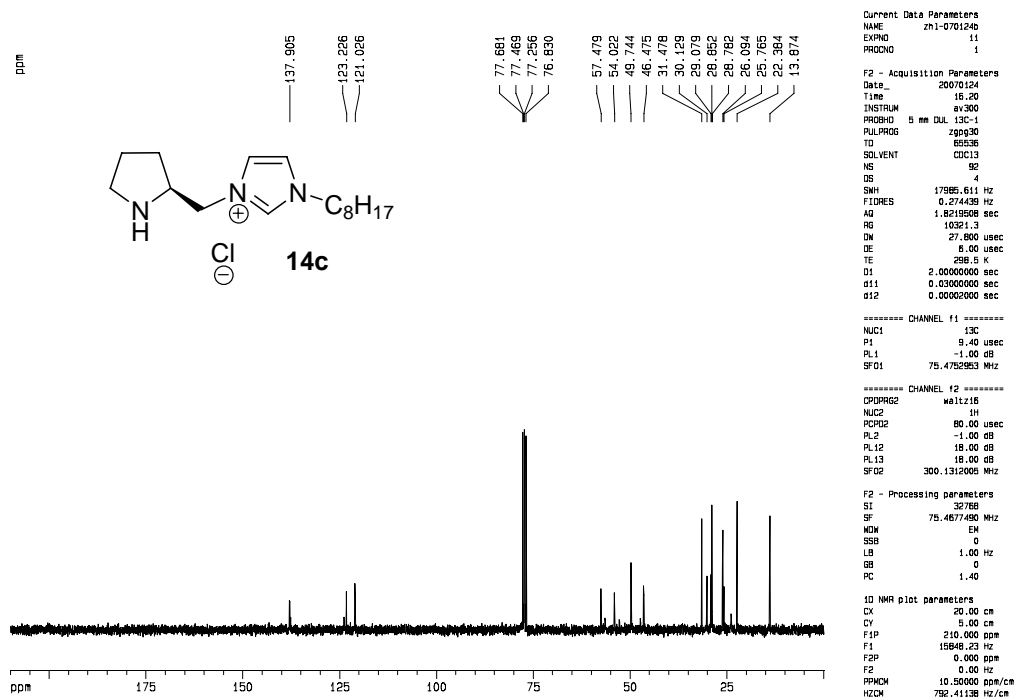
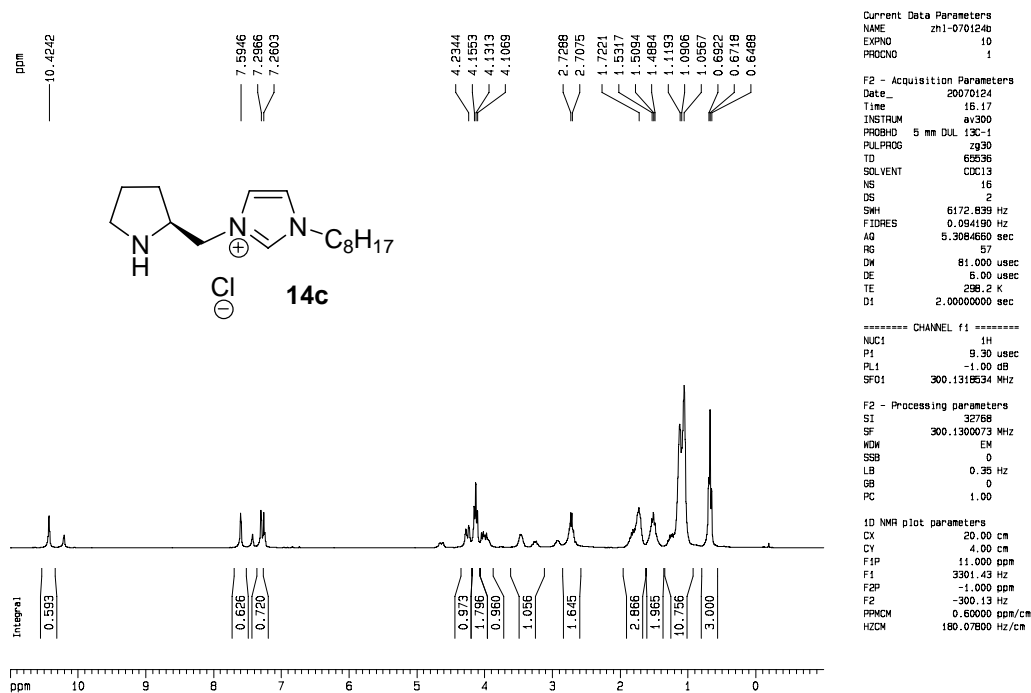
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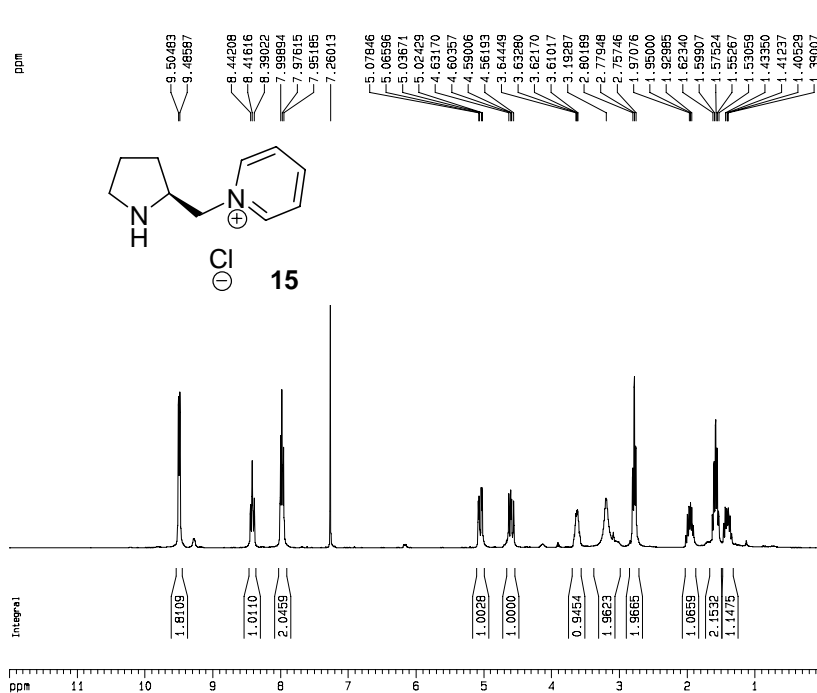
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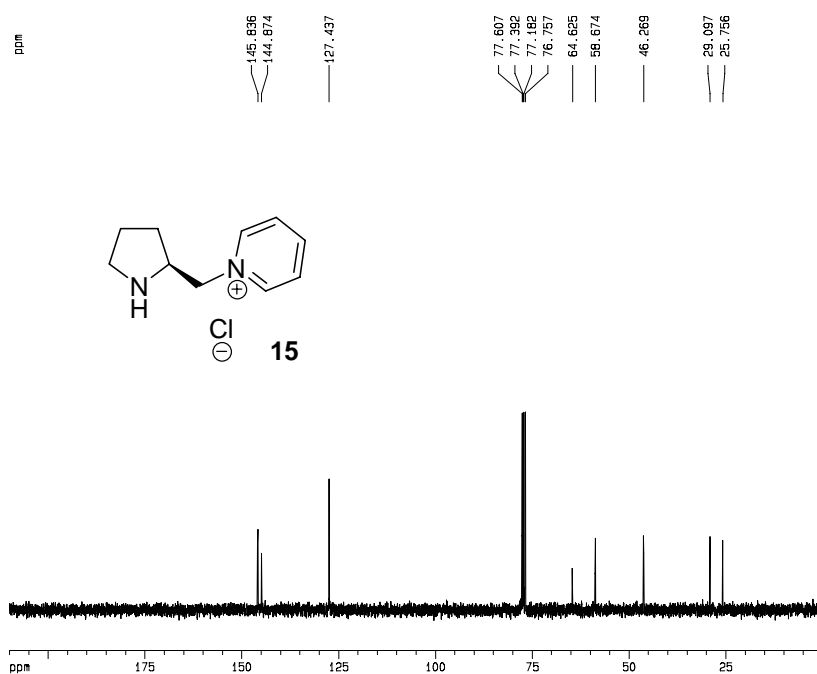
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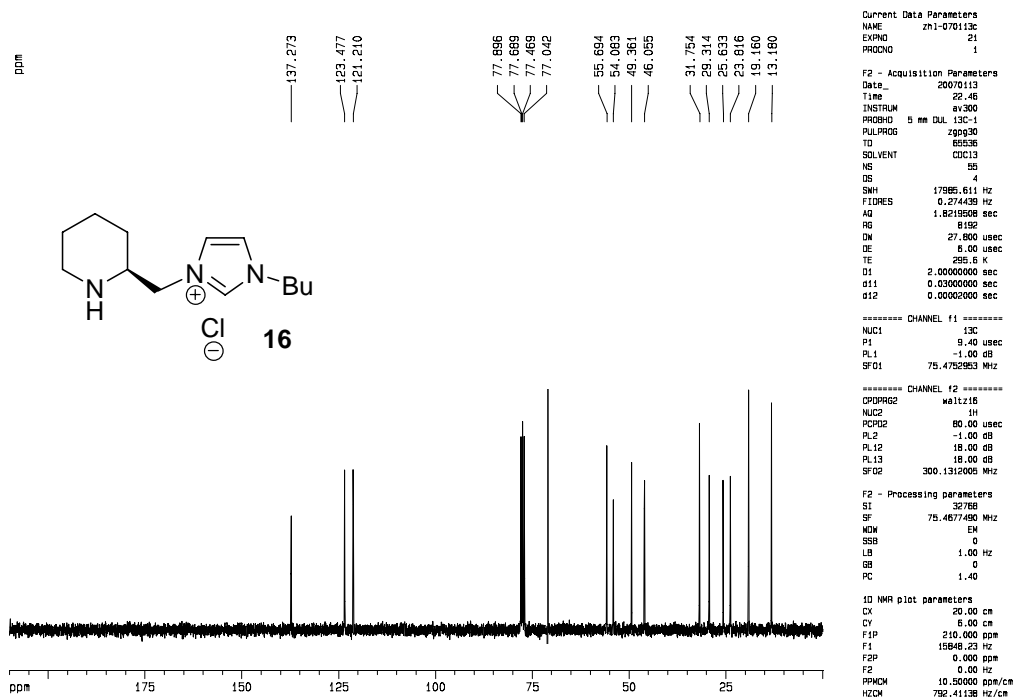
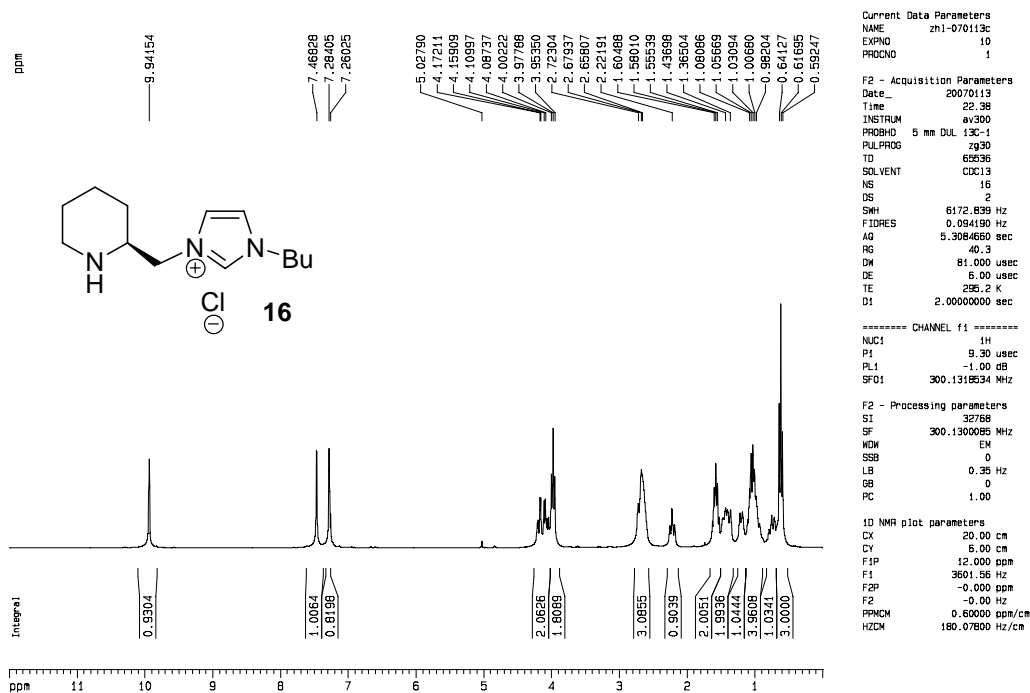
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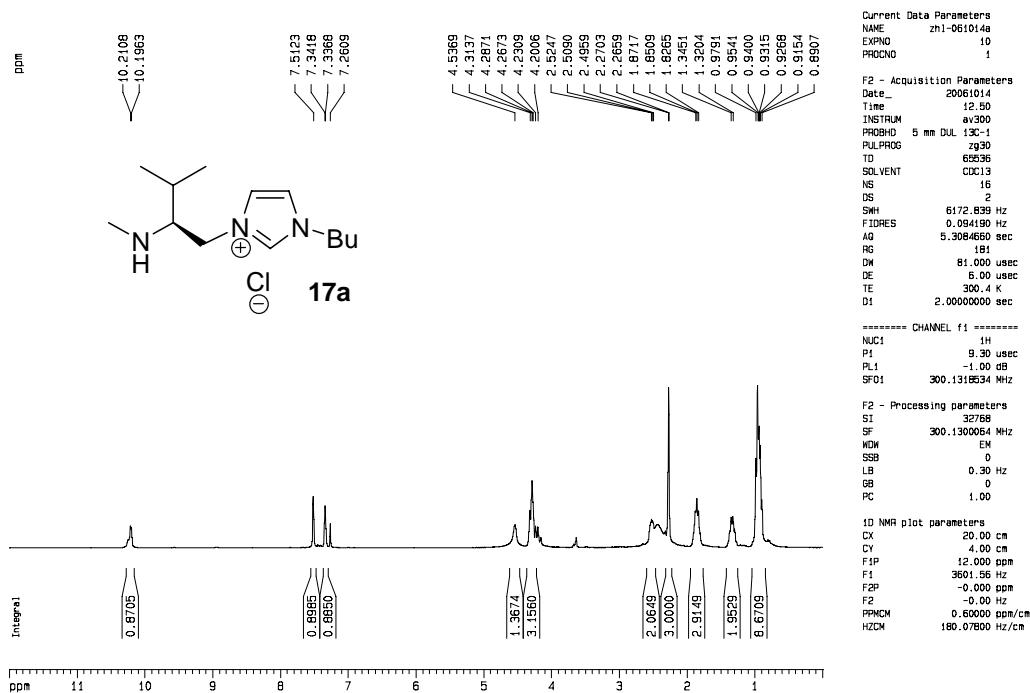
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 F2 0.00 Hz  
 FPCMC 10.50000 ppm/cm  
 HZCM 792.41138 Hz/cm

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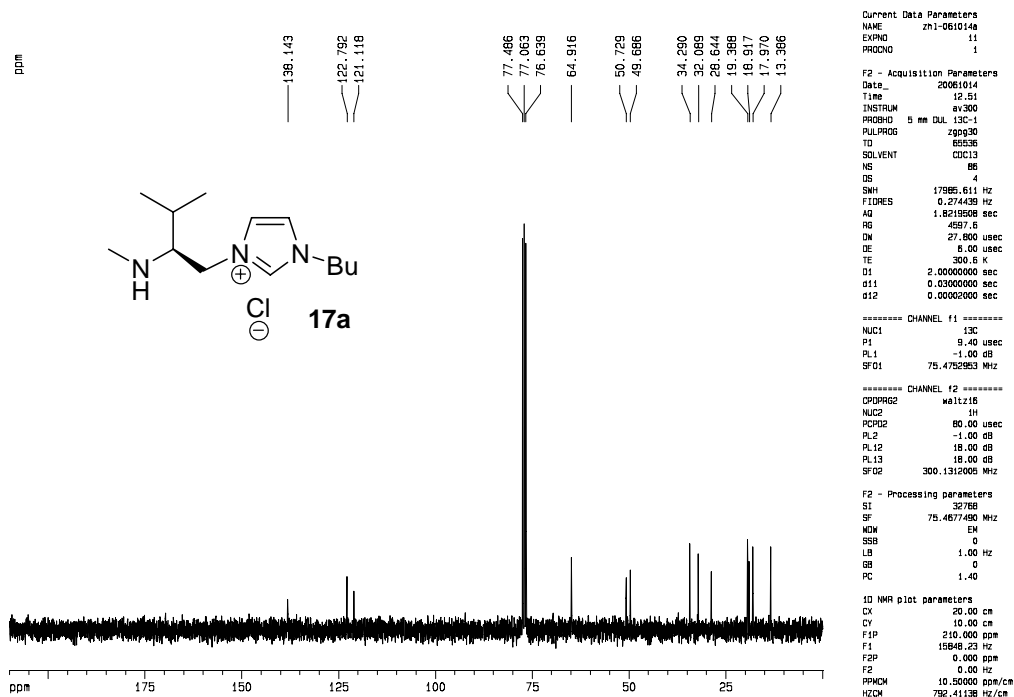
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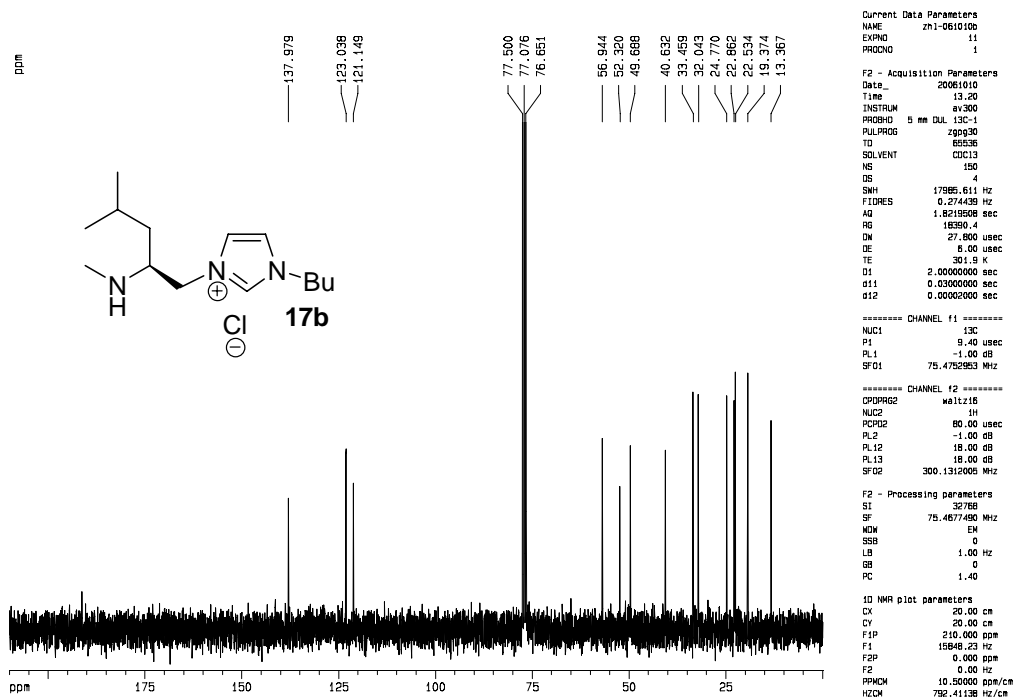
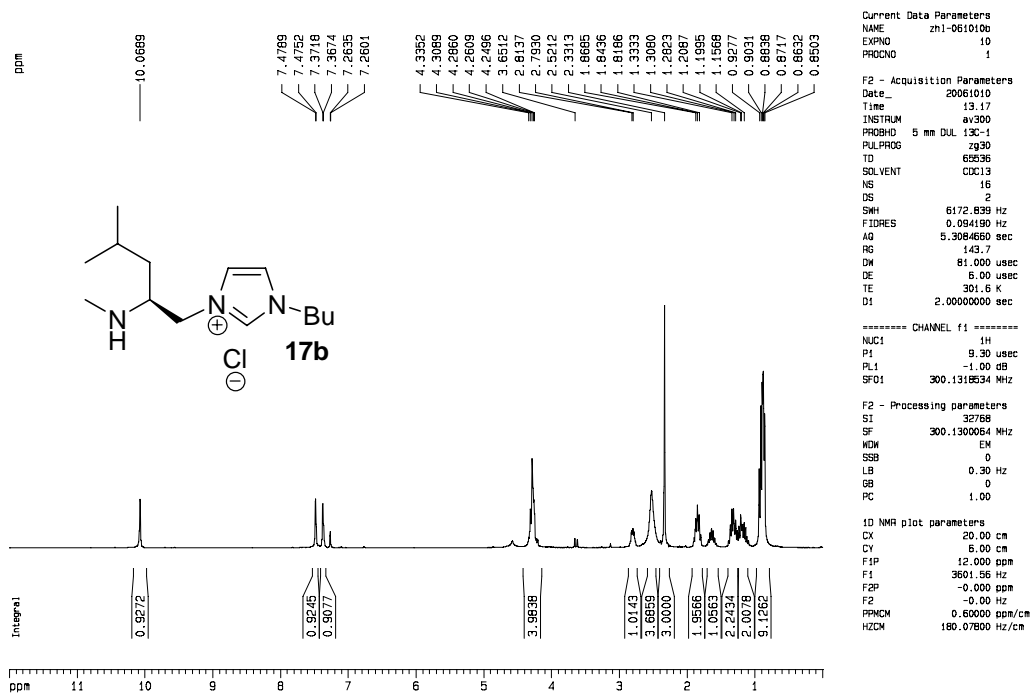
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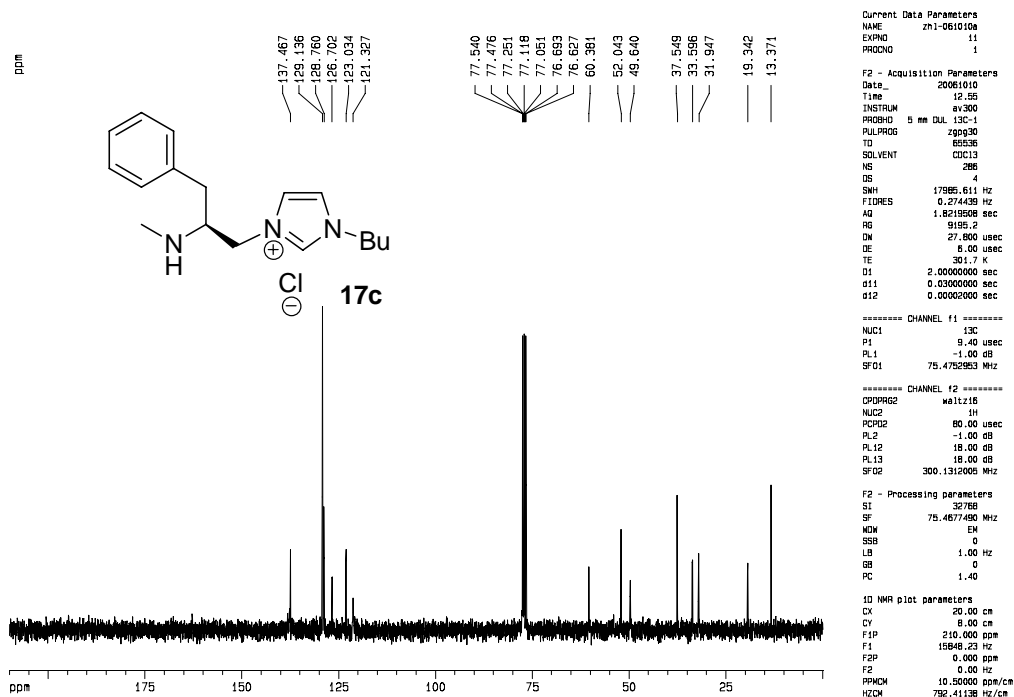
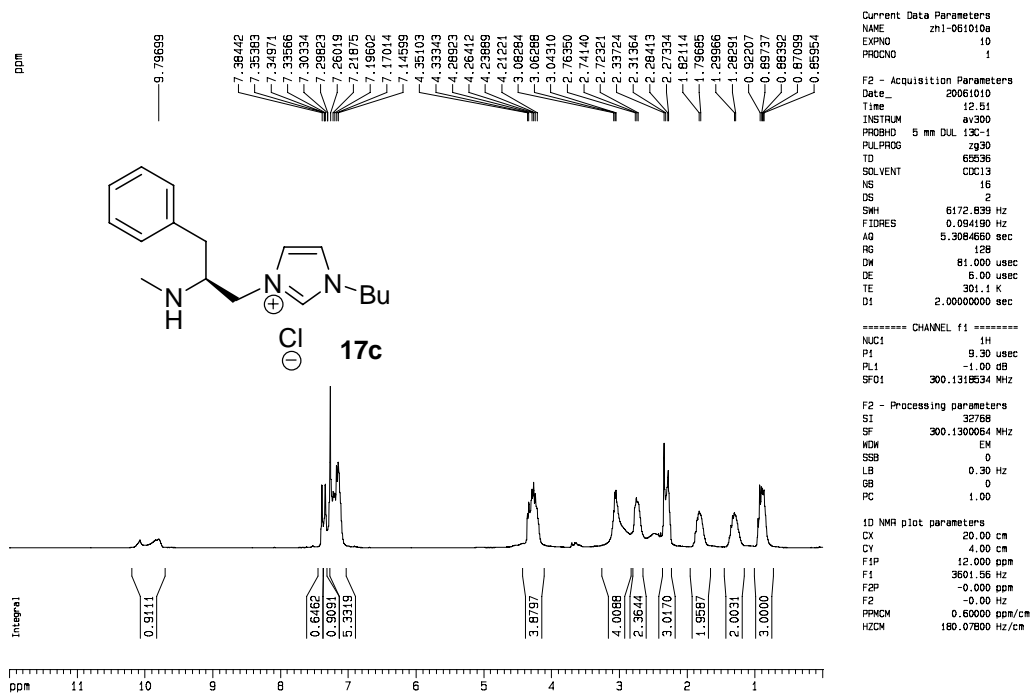
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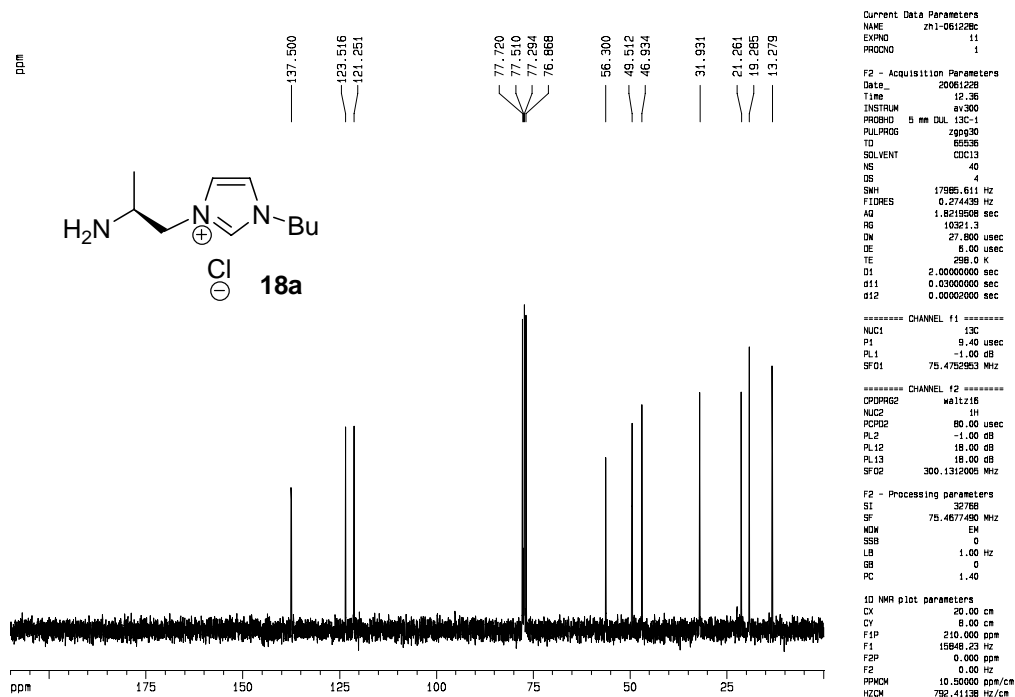
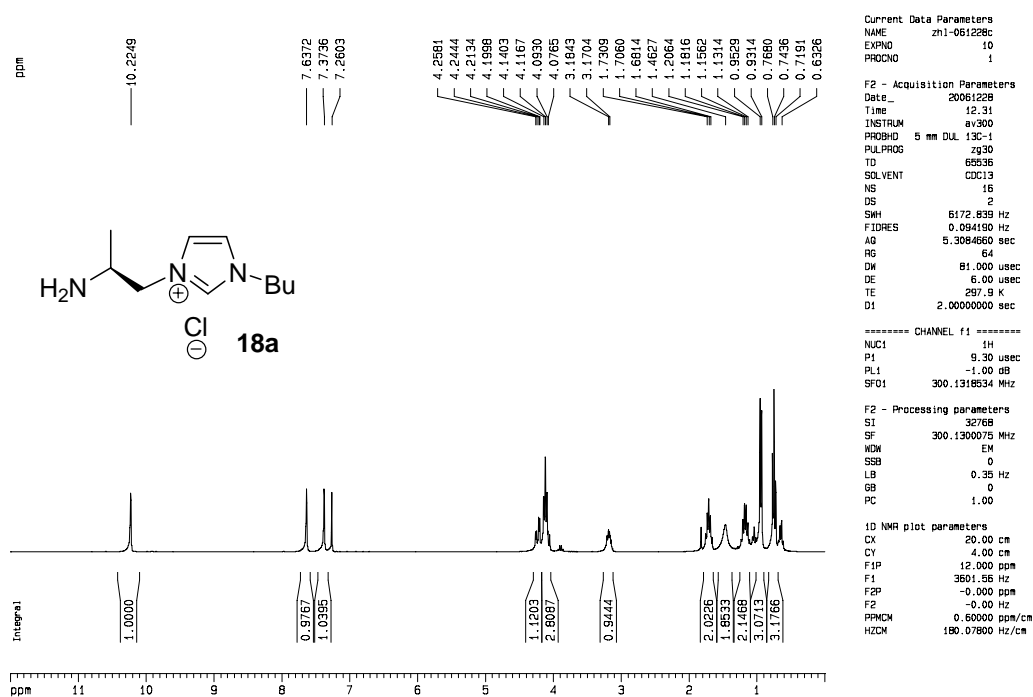




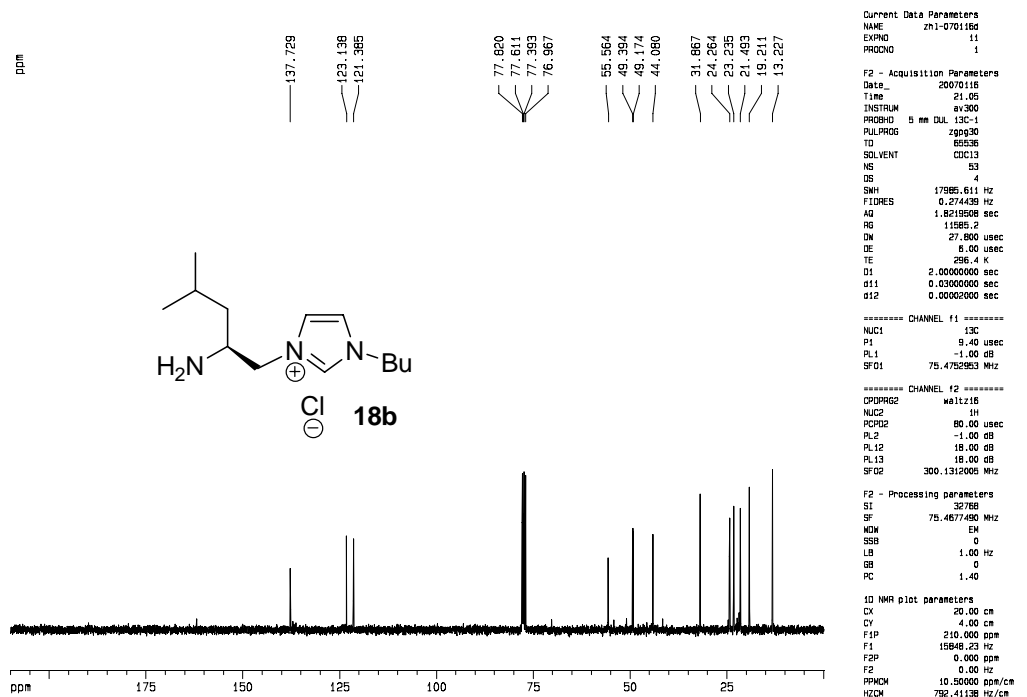
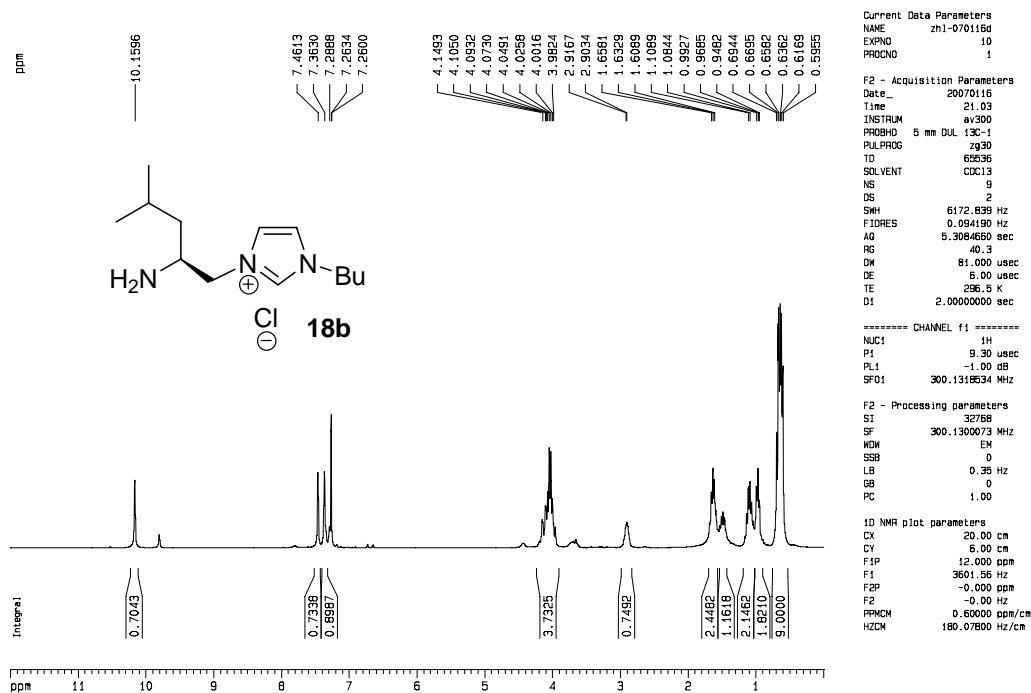
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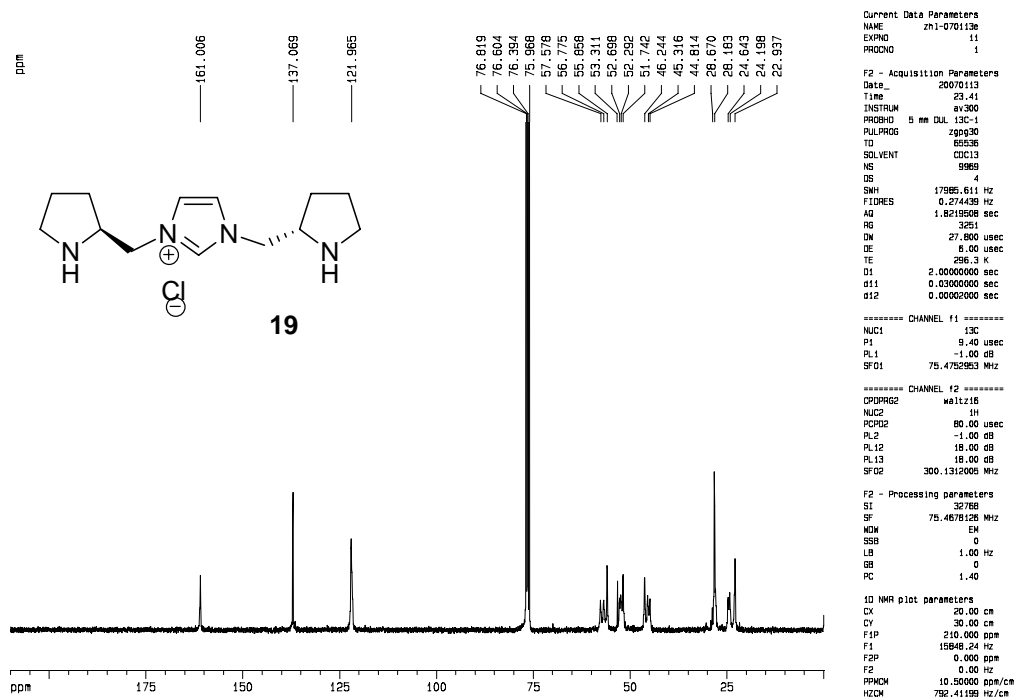
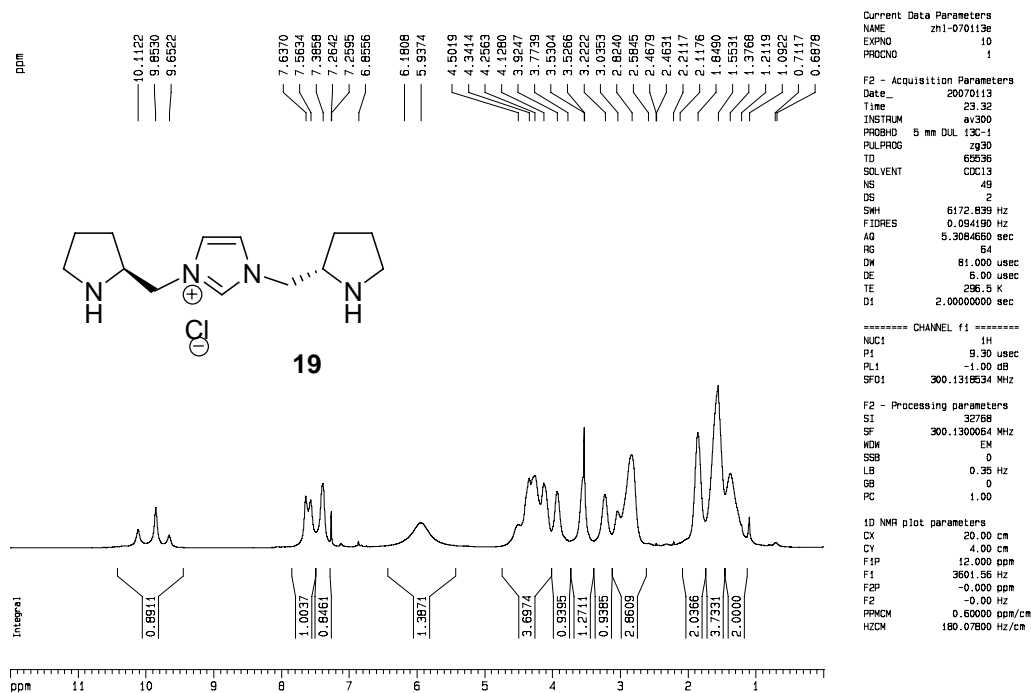
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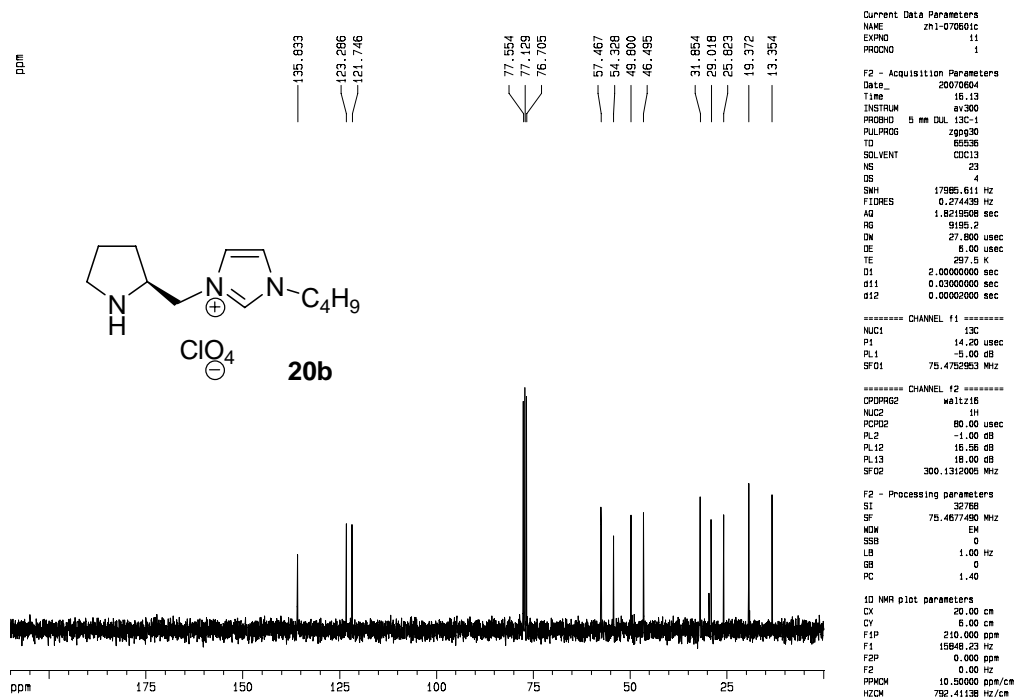
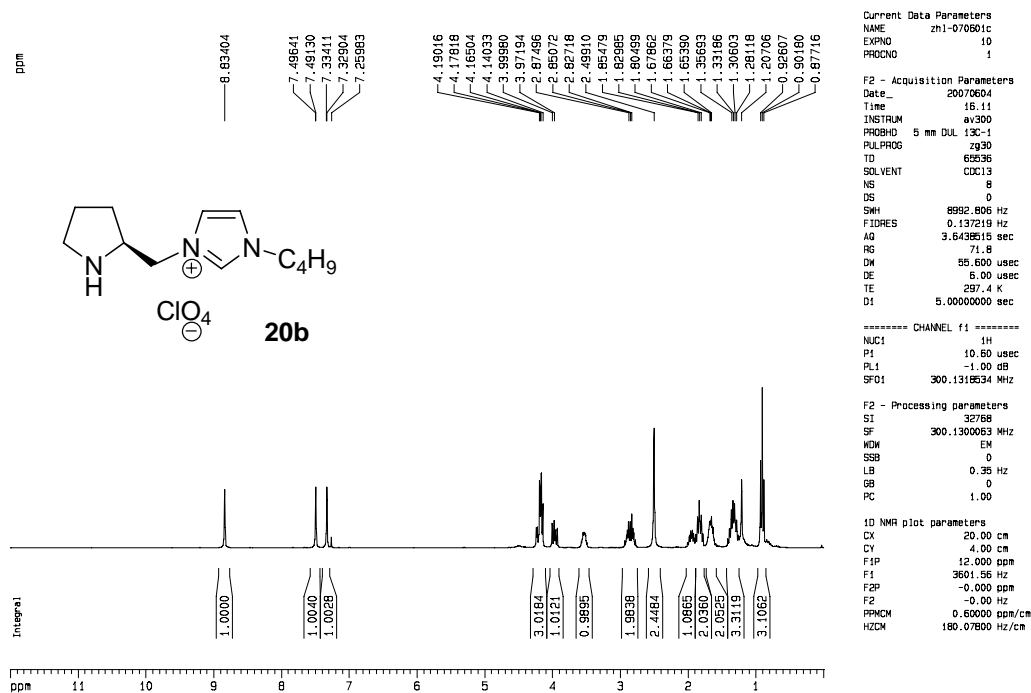
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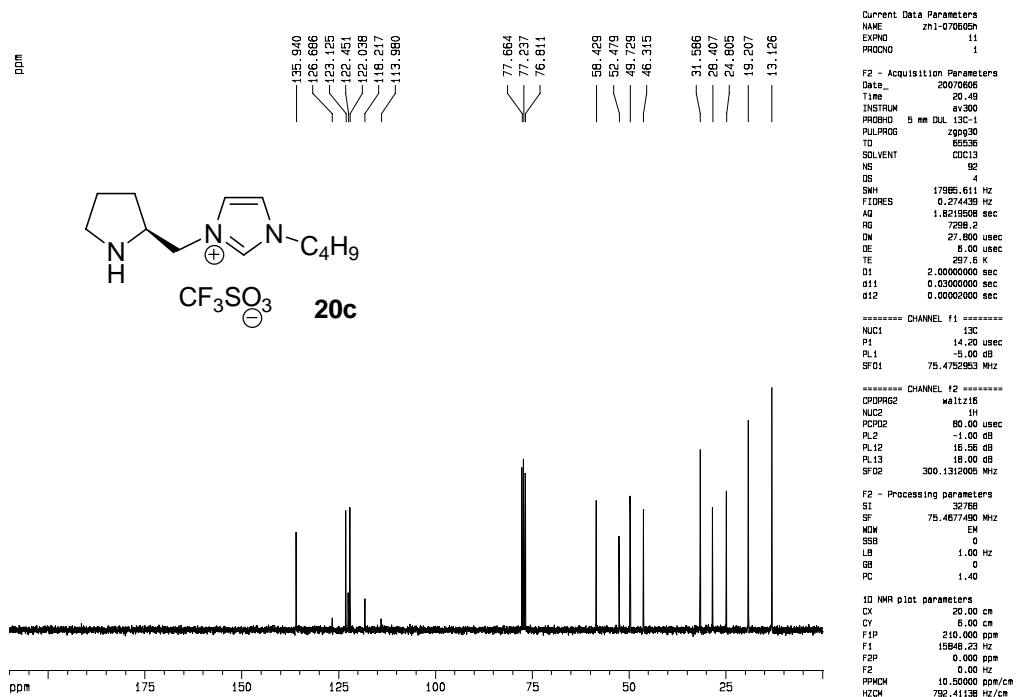
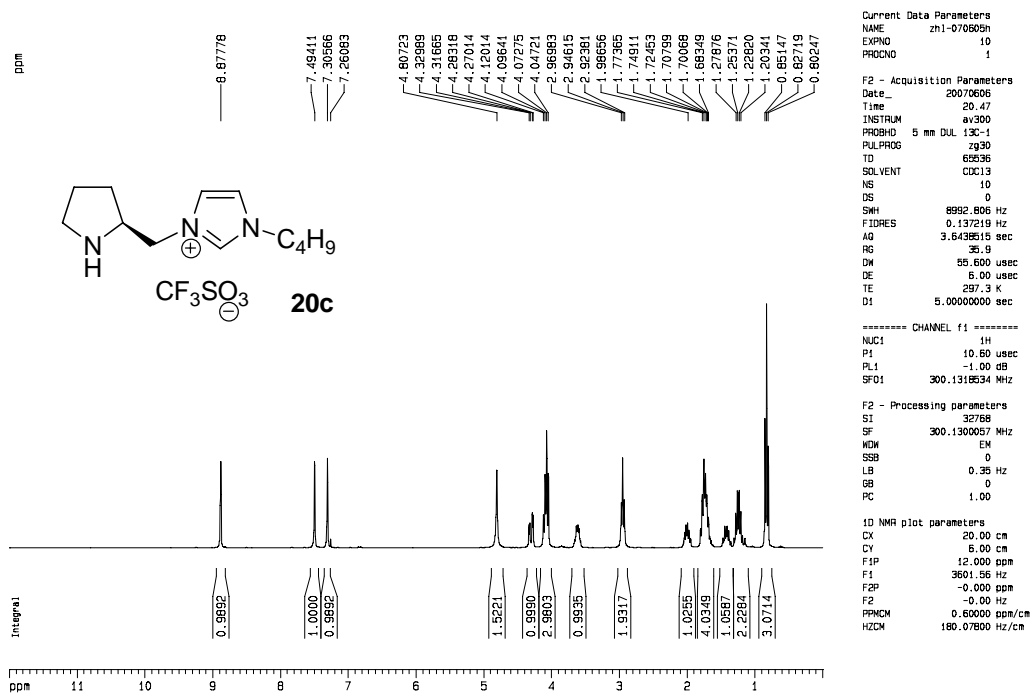
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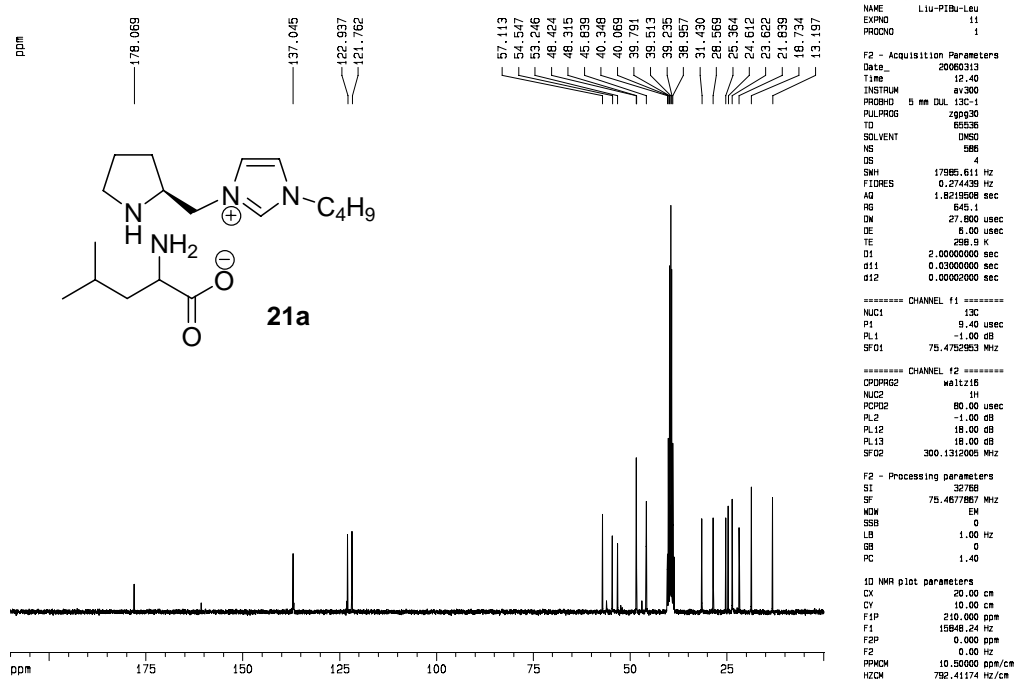
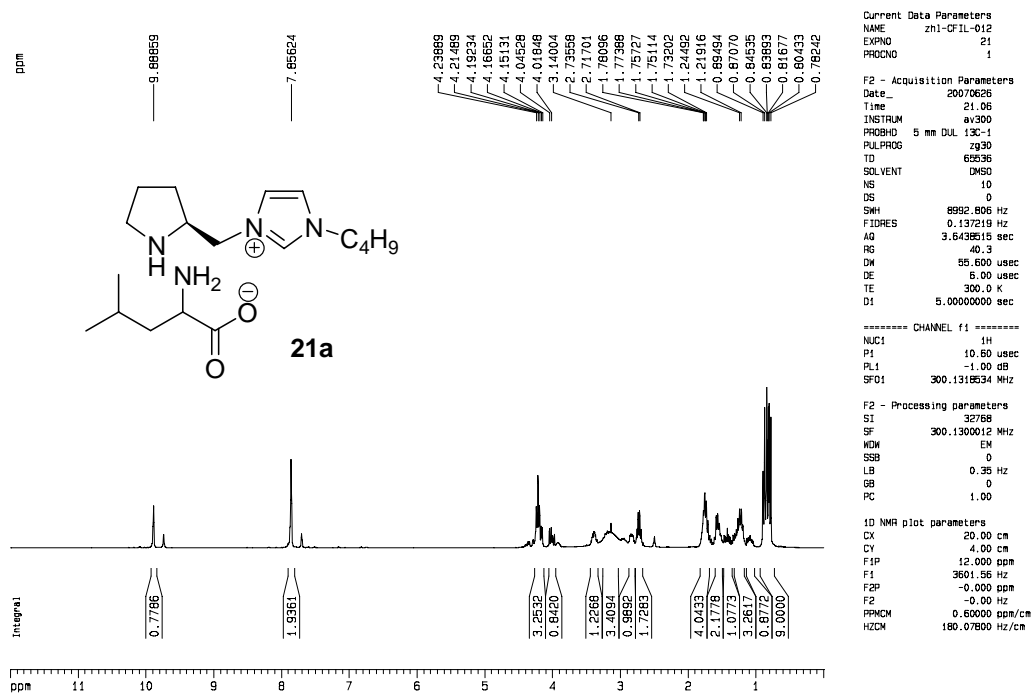
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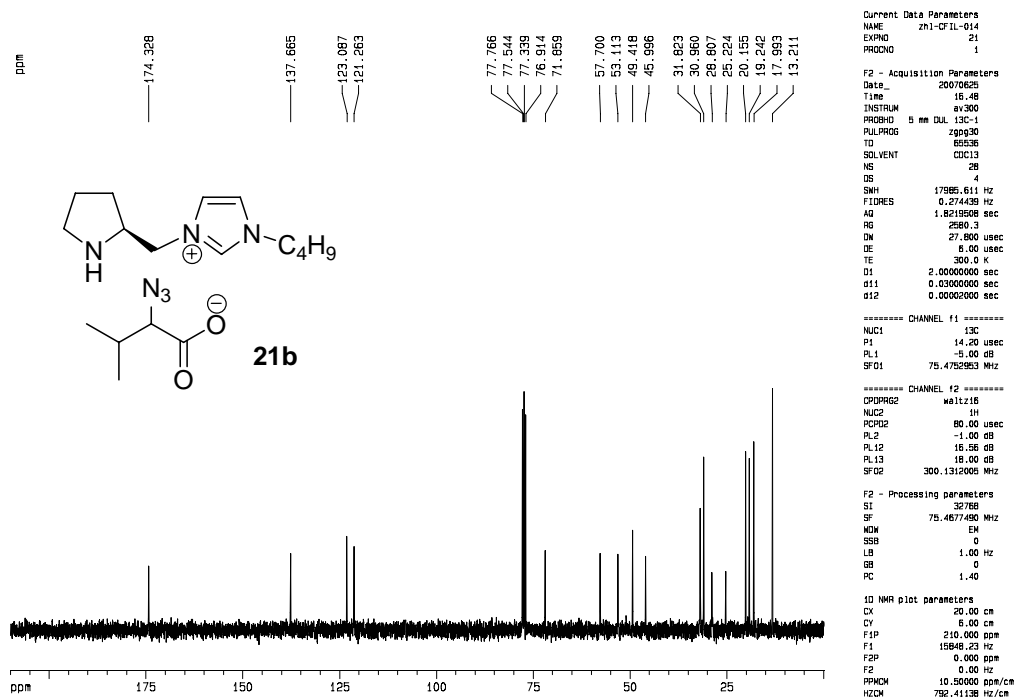
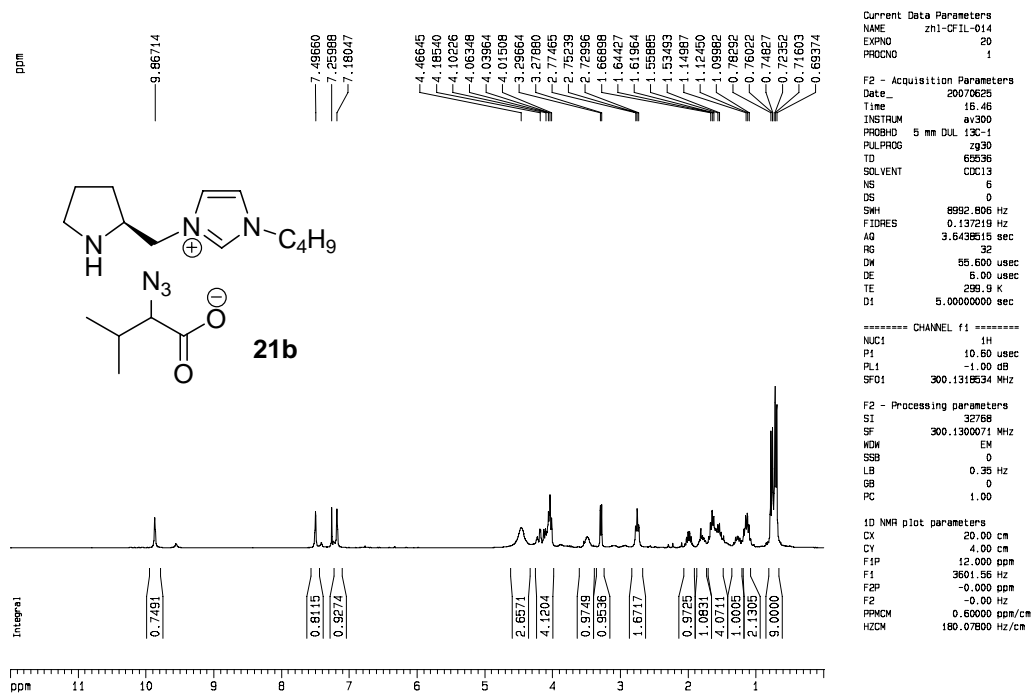
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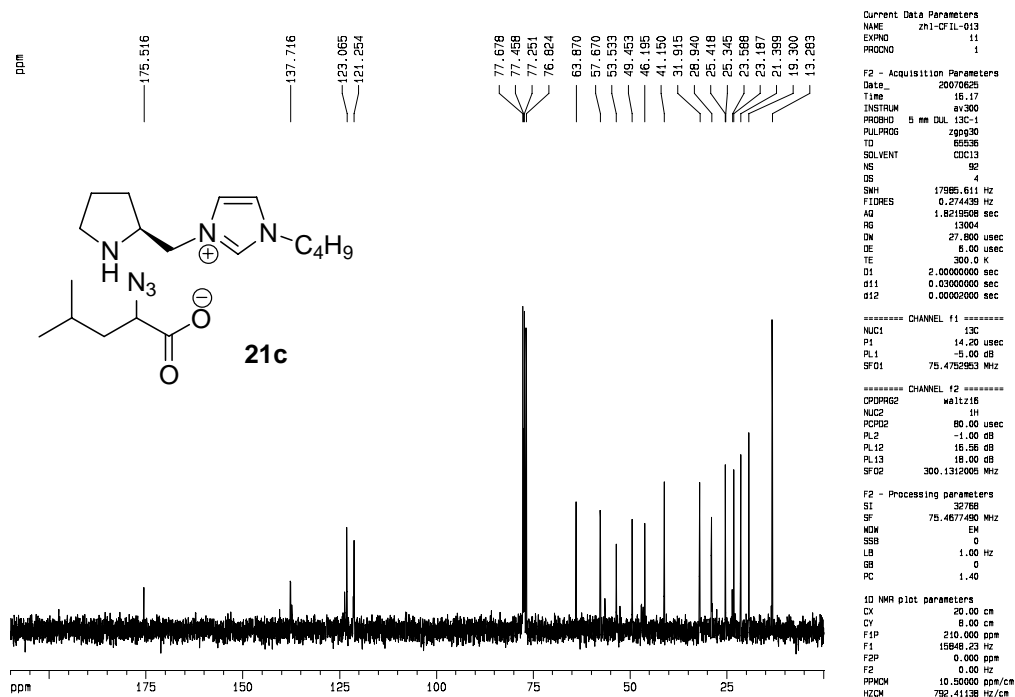
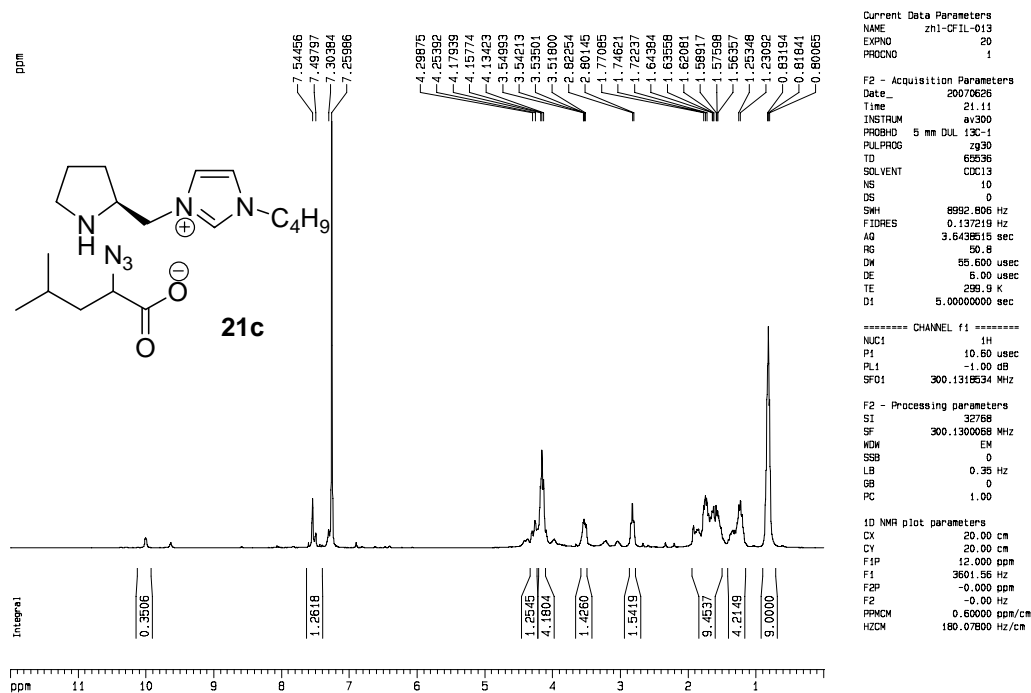


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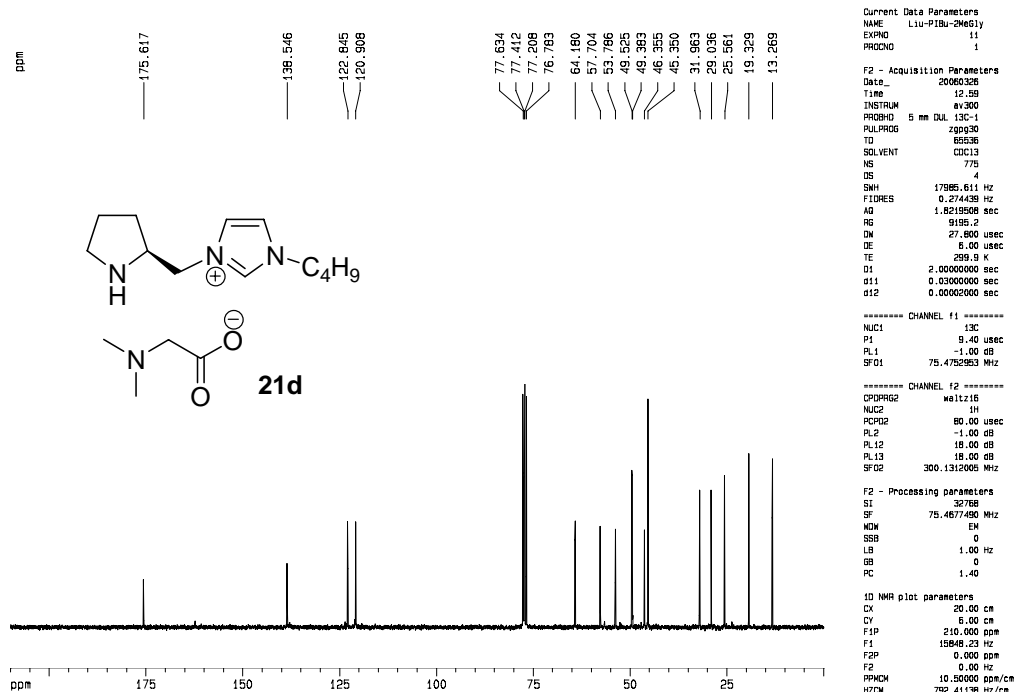
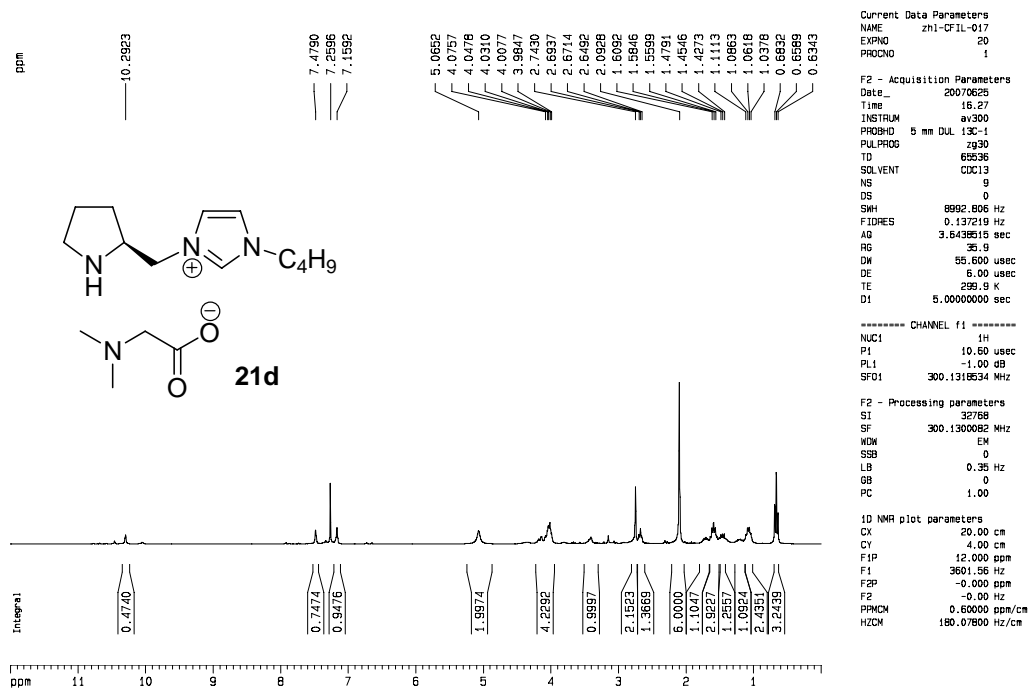




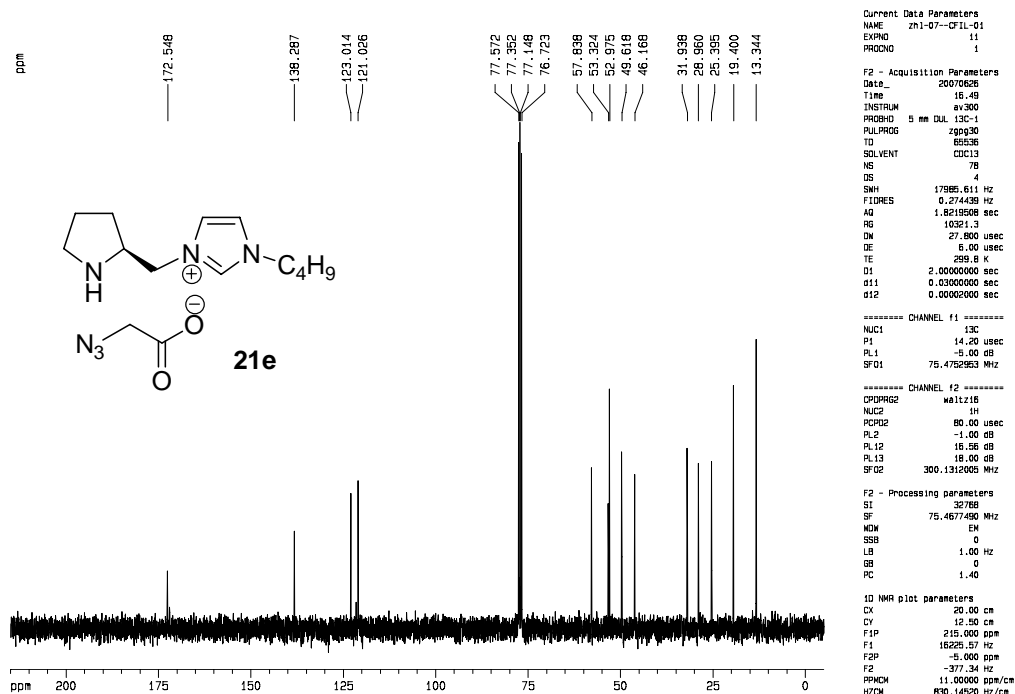
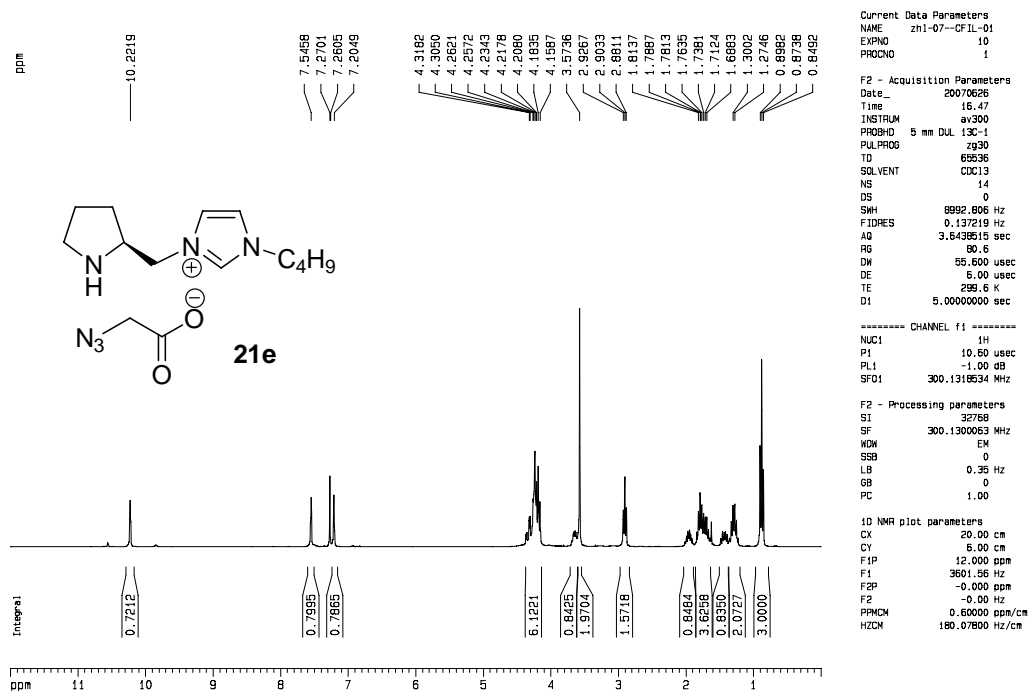
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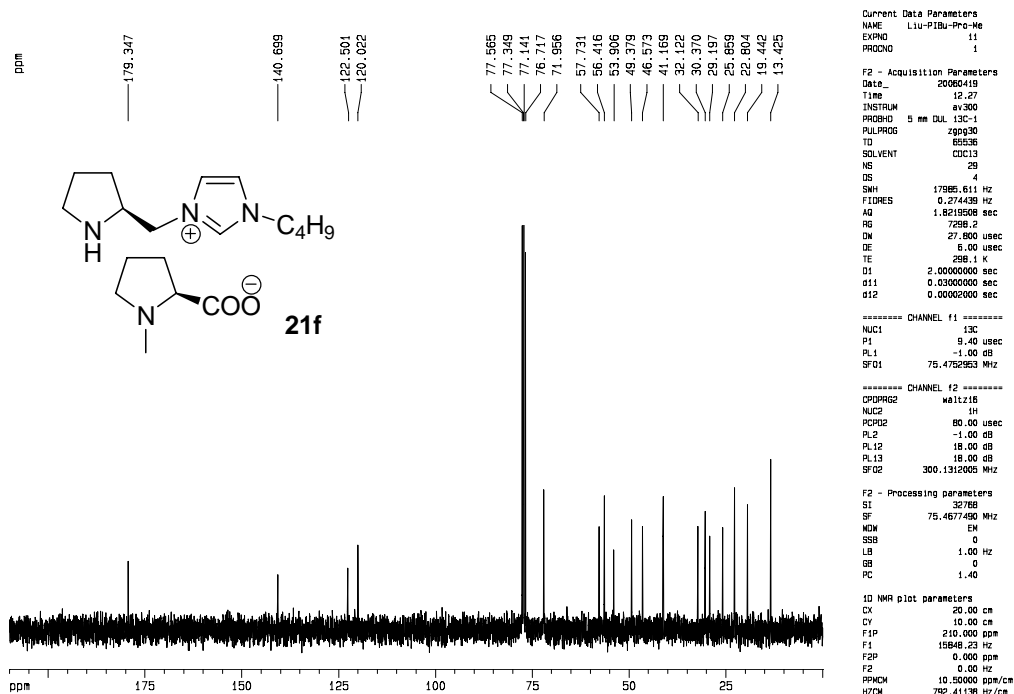
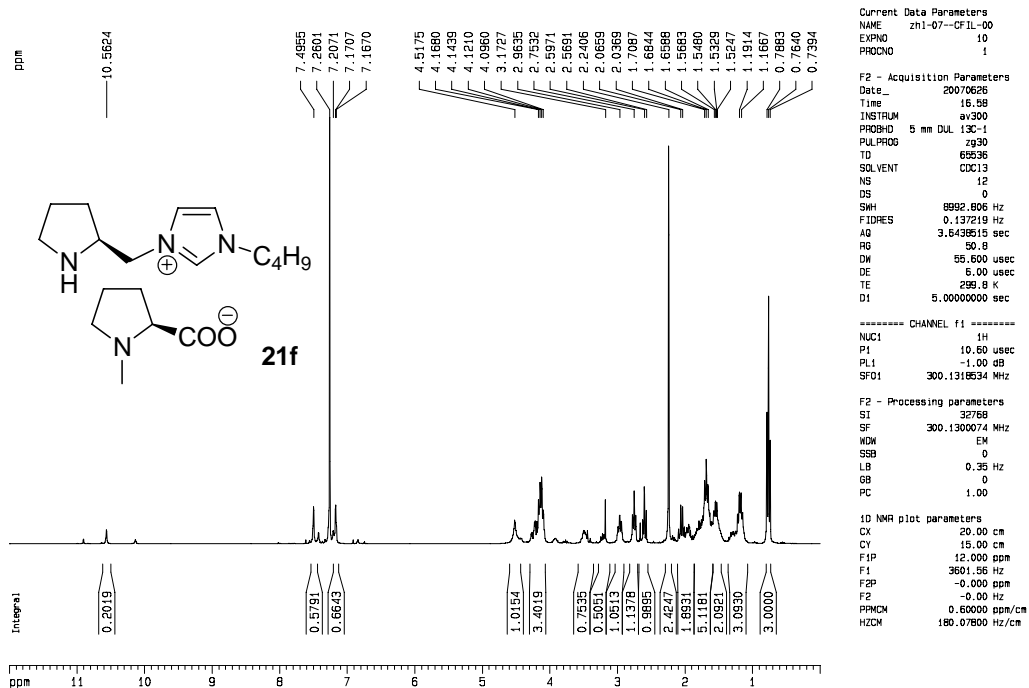
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