

## **Supporting Information**

### **Copper-Catalyzed Carbonylative Synthesis of Aliphatic Amides from Alkanes and Primary Amines *via* C<sub>(sp<sup>3</sup>)</sub>-H Bond Activation**

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## General Remarks

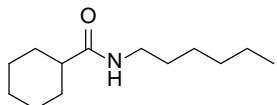
Most of chemicals were purchased from Sigma-Aldrich, Strem, Acros, TCI or Alfa Aesar and used as such unless stated otherwise. Solvents (Anhydrous and under inert atmosphere) were collected from The Solvent purification system by M BRAUN and used under standard schlenk technique. NMR spectra were recorded on Bruker Avance 300 and Bruker ARX 400 spectrometers. Chemical shifts (ppm) are given relative to solvent: references for  $\text{CDCl}_3$  were 7.26 ppm ( $^1\text{H}$  NMR) and 77.00 ppm ( $^{13}\text{C}$  NMR). Multiplets were assigned as s (singlet), d (doublet), t (triplet), q (quartet), p (pentet) dd (doublet of doublet), m (multiplet) and br. s (broad singlet). GC-yields were calculated using isoctane as internal standard. All measurements were carried out at room temperature unless otherwise stated. Electron impact (EI) mass spectra were recorded on AMD 402 mass spectrometer (70 eV). High resolution mass spectra (HRMS) were recorded on Agilent 6210. The data are given as mass units per charge (m/z). Gas chromatography analysis was performed on an Agilent HP-7890A instrument with a FID detector and HP-5 capillary column (polydimethylsiloxane with 5% phenyl groups, 30 m, 0.32 mm i.d., 0.25  $\mu\text{m}$  film thickness) using argon as carrier gas. The products were isolated from the reaction mixture by column chromatography on silica gel 60, 0.063-0.2 mm, 70-230 mesh (Merck).

## General Procedure

A 4 mL screw-cap vial was charged with  $\text{CuF}_2$  (5.05 mg, 10 mol%), 1,10-phenanthroline hydrate (9.9 mg, 10 mol%), aniline(0.5 mmol), cyclohexane (1.5 mL)and an oven-dried stirring bar. The vial was closed by Teflon septum and phenolic cap and connected with atmosphere with a needle. After cyclohexane (1.5 mL), DTBP (0.75 mmol) were injected by syringe, the vial was fixed in an alloy plate and put into Paar 4560 series autoclave (500 mL) under argon atmosphere. At room temperature, the autoclave is flushed with carbon monoxide for three times and 20 bar of carbon monoxide was charged. The autoclave was placed on a heating plate equipped with magnetic stirring and an aluminum block. The reaction is allowed to be heated under 120 °C for 24 hours. Afterwards, the autoclave is cooled to room temperature and the pressure was carefully released. After removal of solvent under reduced pressure, pure product was obtained by column chromatography on silica gel (eluent: pentane/ethyl acetate = 10:1).

## Analytic Data of Products

### N-Hexylcyclohexanecarboxamide



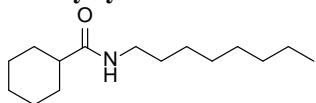
<sup>1</sup>H NMR (300 MHz, Chloroform-*d*) δ 5.81 – 5.58 (m, 1H), 3.19 (td, *J* = 7.2, 5.7 Hz, 2H), 2.04 (tt, *J* = 11.8, 3.4 Hz, 1H), 1.89 – 1.69 (m, 4H), 1.63 (dtt, *J* = 8.2, 3.5, 1.6 Hz, 1H), 1.52 – 1.13 (m, 13H), 0.92 – 0.76 (m, 3H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 176.11, 45.61, 39.33, 31.50, 29.75, 29.65, 26.58, 25.78, 22.56, 22.55, 14.01.

GC-MS (EI, 70ev): m/z(%) = 211(M+,23), 182(18), 168(30), 156(65), 141(28), 128(29), 112(15), 111(36), 100(15), 86(15), 83(100), 73(10), 55(56), 44(16), 43(30), 41(30)

HRMS(ESI): calcd. for [C<sub>13</sub>H<sub>25</sub>NO + H]<sup>+</sup>: 212.20089, found: 212.20145, calcd. for [C<sub>13</sub>H<sub>25</sub>NO + Na]<sup>+</sup>: 234.18284, found: 234.1832

### N-Octylcyclohexanecarboxamide



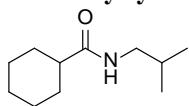
<sup>1</sup>H NMR (300 MHz, Chloroform-*d*) δ 5.72 (d, *J* = 7.1 Hz, 1H), 3.27 – 3.10 (m, 2H), 2.03 (tt, *J* = 11.8, 3.4 Hz, 1H), 1.88 – 1.68 (m, 4H), 1.62 (tt, *J* = 6.1, 2.7 Hz, 1H), 1.53 – 1.10 (m, 17H), 0.91 – 0.76 (m, 3H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 176.08, 45.60, 39.33, 31.79, 29.75, 29.70, 29.29, 29.21, 26.93, 25.78, 22.64, 14.08.

GC-MS (EI, 70ev): m/z(%) = 239(M+,32), 210(13), 184(60), 182(15), 168(31), 156(23), 154(11), 141(35), 128(17), 114(13), 112(14), 111(37), 100(25), 86(17), 84(10), 83(100), 73(11), 71(10), 55(58), 44(15), 41(34).

HRMS(ESI): calcd. for [C<sub>15</sub>H<sub>29</sub>NO + H]<sup>+</sup>: 240.23219, found: 198.14892, calcd. for [C<sub>15</sub>H<sub>29</sub>NO + Na]<sup>+</sup>: 262.21414, found: 262.21444

### N-Isobutylcyclohexanecarboxamide



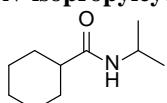
<sup>1</sup>H NMR (300 MHz, Chloroform-*d*) δ 5.82 (s, 1H), 3.01 (dd, *J* = 6.9, 6.0 Hz, 2H), 2.05 (tt, *J* = 11.8, 3.4 Hz, 1H), 1.87 – 1.56 (m, 6H), 1.49 – 1.31 (m, 2H), 1.30 – 1.09 (m, 3H), 0.85 (d, *J* = 6.7 Hz, 6H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 176.25, 46.57, 45.64, 29.79, 28.54, 25.77, 20.07.

GC-MS (EI, 70ev): m/z(%) = 183(M+,26), 128(75), 112(18), 111(51), 83(100), 58(15), 57(18), 55(42), 41(27).

HRMS(ESI): calcd. for [C<sub>11</sub>H<sub>21</sub>NO + H]<sup>+</sup>: 184.16959, found: 184.16993, calcd. for [C<sub>11</sub>H<sub>21</sub>NO + Na]<sup>+</sup>: 206.15154, found: 206.15183

### N-isopropylcyclohexanecarboxamide



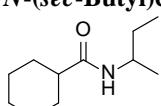
<sup>1</sup>H NMR (300 MHz, Chloroform-*d*) δ 5.41 (s, 1H), 4.03 (dp, *J* = 8.0, 6.6 Hz, 1H), 1.99 (tt, *J* = 11.8, 3.4 Hz, 1H), 1.87 – 1.68 (m, 4H), 1.63 (dtt, *J* = 9.1, 3.1, 1.7 Hz, 1H), 1.49 – 1.30 (m, 2H), 1.30 – 1.15 (m, 3H), 1.10 (d, *J* = 6.6 Hz, 6H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 175.27, 45.64, 40.91, 29.70, 25.77, 22.84.

GC-MS (EI, 70ev): m/z(%) = 169(M+,48), 154(15), 140(13), 128(19), 114(100), 111(11), 101(29), 86(26), 83(75), 72(13), 58(11), 55(61), 44(65), 43(49), 41(39), 39(13).

HRMS(ESI): calcd. for [C<sub>10</sub>H<sub>19</sub>NO + H]<sup>+</sup>: 170.15394, found: 170.15394, calcd. for [C<sub>10</sub>H<sub>19</sub>NO + Na]<sup>+</sup>: 192.13589, found: 192.13591

### N-(sec-Butyl)cyclohexanecarboxamide

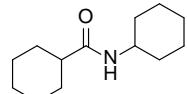


<sup>1</sup>H NMR (300 MHz, Chloroform-*d*) δ 5.35 (d, *J* = 8.6 Hz, 1H), 3.87 (dq, *J* = 8.5, 6.6 Hz, 1H), 2.01 (tt, *J* = 11.8, 3.4 Hz, 1H), 1.87 – 1.69 (m, 4H), 1.63 (dtp, *J* = 8.3, 3.4, 1.7 Hz, 1H), 1.50 – 1.32 (m, 4H), 1.31 – 1.13 (m, 3H), 1.07 (d, *J* = 6.6 Hz, 3H), 0.85 (t, *J* = 7.4 Hz, 3H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 175.48, 46.08, 45.75, 29.90, 29.76, 29.68, 25.80, 25.78, 25.76, 20.57, 10.36.  
GC-MS (EI, 70ev): m/z(%) = 183(M+,23), 154(30), 111(16), 83(79), 72(11), 58(21), 57(18), 55(43), 44(100), 41(26).

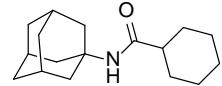
HRMS(ESI): calcd. for [C<sub>11</sub>H<sub>21</sub>NO + H]<sup>+</sup>: 184.16959, found: 184.1699, calcd. for [C<sub>11</sub>H<sub>21</sub>NO + Na]<sup>+</sup>: 206.15154, found: 206.1519

#### N-Cyclohexylcyclohexanecarboxamide



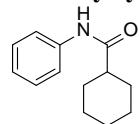
<sup>1</sup>H NMR (300 MHz, Chloroform-*d*) δ 5.48 (d, *J* = 8.4 Hz, 1H), 3.79 – 3.62 (m, 1H), 1.99 (tt, *J* = 11.8, 3.4 Hz, 1H), 1.92 – 1.50 (m, 10H), 1.47 – 0.97 (m, 10H).  
<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 175.15, 47.70, 45.66, 33.23, 29.75, 25.77, 25.57, 24.91.  
GC-MS (EI, 70ev): m/z(%) = 209(M+,29), 154(30), 128(100), 83(39), 67(10), 56(21), 55(43), 41(25).  
HRMS(ESI): calcd. for [C<sub>13</sub>H<sub>23</sub>NO + H]<sup>+</sup>: 210.18524, found: 210.18559, calcd. for [C<sub>13</sub>H<sub>23</sub>NO + Na]<sup>+</sup>: 232.16719, found: 232.16753

#### N-((3s,5s,7s)-Adamantan-1-yl)cyclohexanecarboxamide



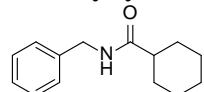
<sup>1</sup>H NMR (300 MHz, Chloroform-*d*) δ 5.09 (s, 1H), 2.09 – 1.87 (m, 10H), 1.77 (tdd, *J* = 13.9, 5.8, 3.1 Hz, 4H), 1.65 (t, *J* = 3.1 Hz, 7H), 1.46 – 1.13 (m, 5H).  
<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 175.45, 51.46, 46.37, 41.74, 36.42, 29.84, 29.48, 25.81.  
GC-MS (EI, 70ev): m/z(%) = 265(M+,33), 220(14), 207(11), 206(71), 204(21), 136(11), 135(100), 94(23), 93(17), 83(13), 79(17), 67(12), 55(27), 41(18).  
HRMS(ESI): calcd. for [C<sub>17</sub>H<sub>27</sub>NO + H]<sup>+</sup>: 262.21654, found: 262.21701, calcd. for [C<sub>17</sub>H<sub>27</sub>NO + Na]<sup>+</sup>: 284.19849, found: 284.19888

#### N-Phenylcyclohexanecarboxamide



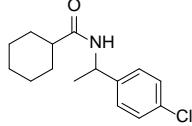
<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) δ 9.78 (s, 1H), 7.65 – 7.56 (m, 2H), 7.34 – 7.21 (m, 2H), 7.00 (tt, *J* = 7.5, 1.2 Hz, 1H), 2.32 (tt, *J* = 11.8, 3.4 Hz, 1H), 1.78 (dddd, *J* = 17.0, 11.9, 5.2, 2.6 Hz, 4H), 1.69 – 1.59 (m, 1H), 1.49 – 1.34 (m, 2H), 1.33 – 1.15 (m, 3H).  
<sup>13</sup>C NMR (101 MHz, DMSO) δ 174.22, 139.45, 128.52, 122.77, 118.98, 44.82, 29.10, 25.37, 25.21.  
GC-MS (EI, 70ev): m/z(%) = 203(M+,12), 93(100), 83(32), 55(26), 41(12).  
HRMS(ESI): calcd. for [C<sub>13</sub>H<sub>17</sub>NO + H]<sup>+</sup>: 204.13829, found: 214.21707, calcd. for [C<sub>13</sub>H<sub>17</sub>NO + Na]<sup>+</sup>: 226.12024, found: 226.12041

#### N-Benzylcyclohexanecarboxamide



<sup>1</sup>H NMR (300 MHz, Chloroform-*d*) δ 7.37 – 7.06 (m, 1H), 5.98 (d, *J* = 7.3 Hz, 0H), 4.32 (d, *J* = 5.7 Hz, 0H), 2.04 (tt, *J* = 11.7, 3.4 Hz, 0H), 1.86 – 1.64 (m, 1H), 1.58 (dqd, *J* = 10.4, 3.5, 1.9 Hz, 0H), 1.46 – 1.28 (m, 0H), 1.26 – 1.06 (m, 1H).  
<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 176.09, 138.66, 128.66, 127.67, 127.37, 45.52, 43.30, 29.74, 25.77.  
GC-MS (EI, 70ev): m/z(%) = 217(M+,59), 176(11), 162(55), 149(21), 107(17), 106(27), 92(12), 91(100), 83(26), 65(12), 55(38), 41(18).

**N-(1-(4-Chlorophenyl)ethyl)cyclohexanecarboxamide**



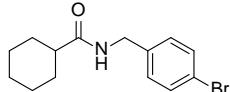
<sup>1</sup>H NMR (300 MHz, Chloroform-*d*) δ 7.26 – 7.08 (m, 4H), 5.82 (d, *J* = 8.0 Hz, 1H), 5.07 – 4.90 (m, 1H), 2.00 (tt, *J* = 11.8, 3.4 Hz, 1H), 1.83 – 1.65 (m, 4H), 1.58 (dd, *J* = 8.0, 6.4, 3.3, 2.0 Hz, 1H), 1.46 – 1.26 (m, 5H), 1.25 – 1.06 (m, 3H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 175.28, 142.19, 132.84, 128.69, 127.48, 47.73, 45.45, 29.73, 29.59, 25.73, 25.70, 21.80.

GC-MS (EI, 70ev): m/z(%) = 265(M+, 52), 210(13), 154(18), 142(22), 141(37), 140(83), 139(100), 138(46), 127(12), 112(10), 111(20), 103(48), 83(70), 77(25), 72(13), 55(52), 41(22)

HRMS(ESI): calcd. for [C<sub>15</sub>H<sub>21</sub>ClNO + H]<sup>+</sup>: 266.13062, found: 266.13094, calcd. for [C<sub>15</sub>H<sub>21</sub>ClNO + Na]<sup>+</sup>: 288.11256, found: 288.11289

**N-(4-Bromobenzyl)cyclohexanecarboxamide**



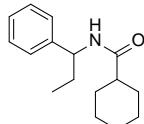
<sup>1</sup>H NMR (300 MHz, Chloroform-*d*) δ 7.46 – 7.35 (m, 2H), 7.14 – 7.05 (m, 2H), 6.22 – 6.05 (m, 1H), 4.32 (d, *J* = 5.9 Hz, 2H), 2.10 (tt, *J* = 11.8, 3.4 Hz, 1H), 1.92 – 1.70 (m, 4H), 1.65 (dtp, *J* = 8.7, 3.7, 1.9 Hz, 1H), 1.42 (qd, *J* = 12.1, 2.7 Hz, 2H), 1.33 – 1.12 (m, 3H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 176.22, 137.77, 131.69, 131.67, 129.31, 121.17, 45.45, 42.61, 29.71, 25.73.

GC-MS (EI, 70ev): m/z(%) = 295(M+, 62), 297(60), 282(10), 280(11), 256(12), 254(14), 242(56), 240(61), 229(22), 227(22), 186(11), 184(14), 171(93), 169(97), 106(33), 90(48), 89(36), 83(96), 81(11), 77(18), 67(11), 55(100), 41(40), 39(14).

HRMS(ESI): calcd. for [C<sub>14</sub>H<sub>18</sub>BrNO + H]<sup>+</sup>: 296.06445, found: 296.06476, calcd. for [C<sub>14</sub>H<sub>18</sub>BrNO + Na]<sup>+</sup>: 318.0464, found: 318.0469

**N-(1-Phenylpropyl)cyclohexanecarboxamide**



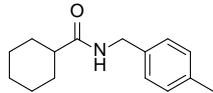
<sup>1</sup>H NMR (300 MHz, Chloroform-*d*) δ 7.30 – 7.10 (m, 5H), 5.79 (d, *J* = 8.7 Hz, 1H), 4.81 (q, *J* = 7.4 Hz, 1H), 2.01 (tt, *J* = 11.9, 3.5 Hz, 1H), 1.86 – 1.63 (m, 6H), 1.58 (ttd, *J* = 7.0, 3.4, 2.0 Hz, 1H), 1.48 – 1.26 (m, 2H), 1.25 – 1.04 (m, 3H), 0.81 (t, *J* = 7.4 Hz, 3H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 175.37, 142.53, 128.58, 127.17, 126.57, 54.39, 45.63, 29.86, 29.66, 29.23, 25.78, 25.75, 10.81.

GC-MS (EI, 70ev): m/z(%) = 245(M+, 8), 118(19), 106(100), 91(30), 83(30), 55(20), 41(11), 41(27).

HRMS(ESI): calcd. for [C<sub>16</sub>H<sub>23</sub>NO + H]<sup>+</sup>: 246.18524, found: 246.1853, calcd. for [C<sub>16</sub>H<sub>23</sub>NO + Na]<sup>+</sup>: 268.16719, found: 268.16725

**N-(4-Methylbenzyl)cyclohexanecarboxamide**



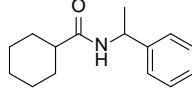
<sup>1</sup>H NMR (300 MHz, Chloroform-*d*) δ 7.19 – 7.07 (m, 4H), 5.82 (s, 1H), 4.37 (d, *J* = 5.6 Hz, 2H), 2.33 (s, 3H), 2.09 (tt, *J* = 11.8, 3.4 Hz, 1H), 1.94 – 1.72 (m, 4H), 1.66 (ddd, *J* = 8.3, 3.7, 1.8 Hz, 1H), 1.45 (qd, *J* = 11.9, 3.3 Hz, 2H), 1.35 – 1.12 (m, 3H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 175.92, 137.11, 135.56, 129.36, 127.76, 45.58, 43.15, 29.75, 25.78, 21.13.

GC-MS (EI, 70ev): m/z(%) = 231(M+, 46), 176(27), 120(13), 106(34), 105(100), 83(19), 79(10), 77(13), 55(26), 41(13).

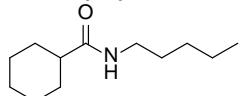
HRMS(ESI): calcd. for [C<sub>15</sub>H<sub>21</sub>NO + H]<sup>+</sup>: 232.16959, found: 232.16964, calcd. for [C<sub>15</sub>H<sub>21</sub>NO + Na]<sup>+</sup>: 254.15154, found: 254.15161

**N-(1-Phenylethyl)cyclohexanecarboxamide**



<sup>1</sup>H NMR (300 MHz, Chloroform-*d*) δ 7.32 – 7.10 (m, 5H), 5.79 (d, *J* = 8.2 Hz, 1H), 5.12 – 4.95 (m, 1H), 2.08 – 1.92 (m, 1H), 1.87 – 1.63 (m, 4H), 1.58 (dtt, *J* = 8.3, 3.5, 1.9 Hz, 1H), 1.48 – 1.26 (m, 5H), 1.26 – 1.06 (m, 3H).  
<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 175.16, 143.53, 128.63, 127.22, 126.13, 48.26, 45.55, 29.73, 29.66, 25.77, 21.81.  
GC-MS (EI, 70ev): m/z(%) = 231(M+, 55), 176(13), 121(10), 120(25), 106(75), 105(100), 104(40), 83(36), 79(17), 77(20), 55(32), 41(15).

### N-Pentylcyclohexanecarboxamide



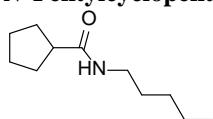
<sup>1</sup>H NMR (300 MHz, Chloroform-*d*) δ 5.69 (s, 1H), 3.19 (td, *J* = 7.2, 5.7 Hz, 2H), 2.03 (tt, *J* = 11.8, 3.4 Hz, 1H), 1.78 (dddd, *J* = 19.8, 10.0, 5.5, 2.9 Hz, 4H), 1.63 (dtq, *J* = 8.4, 3.6, 1.5 Hz, 1H), 1.52 – 1.12 (m, 11H), 0.90 – 0.81 (m, 3H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 176.10, 45.61, 39.30, 29.75, 29.38, 29.08, 25.78, 22.38, 14.00.

GC-MS (EI, 70ev): m/z(%) = 197(M+, 20), 168(36), 142(68), 141(25), 114(25), 112(15), 111(35), 100(10), 86(12), 84(10), 83(100), 73(10), 71(12), 55(56), 44(17), 43(26), 41(34), 29(12).

HRMS(ESI): calcd. for [C<sub>12</sub>H<sub>23</sub>NO + H]<sup>+</sup>: 198.18524, found: 198.18534, calcd. for [C<sub>12</sub>H<sub>23</sub>NO + Na]<sup>+</sup>: 220.16719, found: 220.16729

### N-Pentylcyclopentanecarboxamide



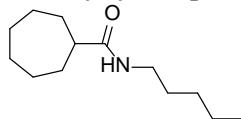
<sup>1</sup>H NMR (300 MHz, Chloroform-*d*) δ 5.62 (s, 1H), 3.16 (td, *J* = 7.2, 5.8 Hz, 2H), 2.51 – 2.35 (m, 1H), 1.86 – 1.58 (m, 6H), 1.58 – 1.35 (m, 4H), 1.33 – 1.12 (m, 4H), 0.89 – 0.74 (m, 3H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 176.18, 45.95, 39.49, 30.49, 29.41, 29.10, 25.93, 25.92, 22.38, 14.01.

GC-MS (EI, 70ev): m/z(%) = 183(M+, 20), 154(28), 142(100), 127(15), 114(20), 98(14), 97(42), 86(21), 69(92), 55(14), 44(14), 43(20), 41(50), 39(16), 29(13).

HRMS(ESI): calcd. for [C<sub>11</sub>H<sub>21</sub>NO + H]<sup>+</sup>: 184.16959, found: 184.1699, calcd. for [C<sub>11</sub>H<sub>21</sub>NO + Na]<sup>+</sup>: 206.15154, found: 206.15194

### N-Pentylcycloheptanecarboxamide



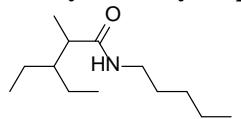
<sup>1</sup>H NMR (300 MHz, Chloroform-*d*) δ 5.63 (s, 1H), 3.18 (td, *J* = 7.2, 5.7 Hz, 2H), 2.17 (tt, *J* = 10.0, 3.9 Hz, 1H), 1.90 – 1.16 (m, 18H), 0.93 – 0.79 (m, 3H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 177.25, 47.62, 39.32, 31.79, 29.41, 29.09, 28.13, 26.66, 22.38, 14.01.

GC-MS (EI, 70ev): m/z(%) = 211(M+, 28), 196(18), 182(48), 169(13), 168(50), 156(19), 155(21), 143(11), 142(83), 129(27), 126(12), 125(22), 114(33), 112(13), 99(13), 97(74), 87(15), 86(13), 73(13), 71(15), 69(14), 67(12), 55(100), 44(13), 43(36), 42(10), 41(47), 39(19), 29(23).

HRMS(ESI): calcd. for [C<sub>13</sub>H<sub>25</sub>NO + H]<sup>+</sup>: 212.20089, found: 212.20131, calcd. for [C<sub>13</sub>H<sub>25</sub>NO + Na]<sup>+</sup>: 234.18284, found: 234.1834

### 3-Ethyl-2-methyl-N-pentylpentanamide

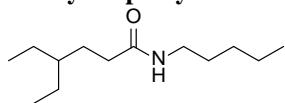


<sup>1</sup>H NMR (400 MHz, Chloroform-*d*) δ 5.49 (s, 1H), 3.23 (dp, *J* = 19.6, 6.5 Hz, 2H), 2.14 (p, *J* = 7.1 Hz, 1H), 1.56 – 1.39 (m, 4H), 1.38 – 1.13 (m, 8H), 1.06 (d, *J* = 6.9 Hz, 2H), 0.93 – 0.74 (m, 9H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 176.40, 43.63, 43.27, 39.32, 29.41, 29.11, 23.10, 22.37, 21.20, 14.34, 14.01, 11.34, 10.24.

GC-MS (EI, 70ev): m/z(%) = 213(M+, 5), 198(14), 184(26), 144(10), 143(99), 142(16), 114(100), 101(26), 100(15), 87(45), 86(16), 71(16), 57(51), 56(11), 55(24), 44(13), 43(46), 41(38), 29(23).

HRMS(ESI): calcd. for [C<sub>13</sub>H<sub>27</sub>NO + H]<sup>+</sup>: 214.21654, found: 214.21707, calcd. for [C<sub>13</sub>H<sub>27</sub>NO + Na]<sup>+</sup>: 236.19849, found: 236.19885

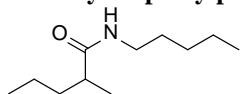
**4-Ethyl-N-pentylhexanamide**

<sup>1</sup>H NMR (400 MHz, Chloroform-d) δ 5.39 (s, 1H), 3.17 (td, *J* = 7.2, 5.6 Hz, 2H), 2.14 – 2.02 (m, 2H), 1.57 – 1.48 (m, 2H), 1.43 (p, *J* = 7.3 Hz, 2H), 1.30 – 1.10 (m, 9H), 0.80 (dt, *J* = 18.8, 7.0 Hz, 9H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 173.36, 40.08, 39.52, 34.38, 29.41, 29.11, 28.76, 25.16, 22.39, 14.01, 10.81.

GC-MS (EI, 70ev): m/z(%) = 213(M+,7), 184(76), 143(11), 142(100), 129(70), 128(11), 127(17), 114(50), 109(36), 100(17), 87(40), 86(42), 73(43), 72(22), 71(30), 58(12), 57(27), 56(10), 55(40), 44(28), 43(72), 42(14), 41(53), 39(14), 30(26), 29(23).

HRMS(ESI): calcd. for [C<sub>13</sub>H<sub>27</sub>NO + H]<sup>+</sup>: 214.21654, found: 214.21707, calcd. for [C<sub>13</sub>H<sub>27</sub>NO + Na]<sup>+</sup>: 236.19849, found: 236.19885

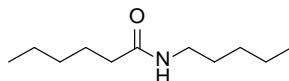
**2-Methyl-N-pentylpentanamide**

<sup>1</sup>H NMR (300 MHz, Chloroform-d) δ 5.48 (s, 1H), 3.23 (tdd, *J* = 7.1, 5.7, 3.7 Hz, 2H), 2.21 – 2.06 (m, 1H), 1.69 – 1.41 (m, 3H), 1.37 – 1.21 (m, 7H), 1.11 (d, *J* = 6.9 Hz, 3H), 0.89 (ddd, *J* = 7.4, 5.3, 2.3 Hz, 6H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 176.50, 41.55, 39.33, 36.60, 29.43, 29.09, 22.38, 20.69, 17.97, 14.09, 14.02.

GC-MS (EI, 70ev): m/z(%) = 185(M+,5), 170(10), 156(60), 143(85), 142(10), 114(87), 101(27), 100(38), 99(31), 87(41), 86(17), 72(11), 71(100), 58(10), 56(11), 55(24), 44(23), 43(99), 42(14), 41(47), 39(15), 30(46), 29(23).

HRMS(ESI): calcd. for [C<sub>11</sub>H<sub>23</sub>NO + H]<sup>+</sup>: 186.18524, found: 186.18524, calcd. for [C<sub>11</sub>H<sub>23</sub>NO + Na]<sup>+</sup>: 208.16719, found: 208.16757

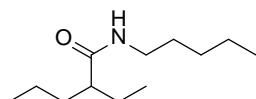
**N-Pentylhexanamide**

<sup>1</sup>H NMR (300 MHz, Chloroform-d) δ 5.42 (s, 1H), 3.23 (td, *J* = 7.2, 5.7 Hz, 2H), 2.21 – 2.08 (m, 2H), 1.62 (dddd, *J* = 9.6, 8.7, 7.2, 3.5 Hz, 2H), 1.54 – 1.43 (m, 2H), 1.37 – 1.23 (m, 8H), 0.94 – 0.84 (m, 6H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 173.07, 39.49, 36.95, 31.51, 29.40, 29.10, 25.56, 22.45, 22.39, 14.03, 13.99.

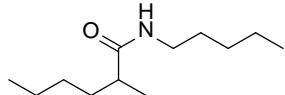
GC-MS (EI, 70ev): m/z(%) = 185(M+,52), 156(40), 142(33), 129(83), 128(10), 116(11), 114(47), 100(30), 99(55), 87(47), 86(45), 73(55), 72(24), 71(56), 58(17), 55(23), 44(40), 43(100), 42(15), 41(39), 30(46), 29(23).

HRMS(ESI): calcd. for [C<sub>11</sub>H<sub>23</sub>NO + H]<sup>+</sup>: 186.18524, found: 186.18524, calcd. for [C<sub>11</sub>H<sub>23</sub>NO + Na]<sup>+</sup>: 208.16719, found: 208.16757

**2-Ethyl-N-Pentylpentanamide**

<sup>1</sup>H NMR (300 MHz, Chloroform-d) δ 5.47 – 5.27 (m, 1H), 3.19 (td, *J* = 7.1, 5.8 Hz, 2H), 1.83 (tt, *J* = 9.5, 4.8 Hz, 1H), 1.61 – 1.38 (m, 4H), 1.23 (tdd, *J* = 9.4, 5.3, 2.0 Hz, 8H), 0.82 (td, *J* = 7.3, 2.8 Hz, 9H).

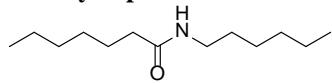
<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 175.72, 49.83, 39.32, 35.09, 31.49, 29.78, 26.60, 26.15, 22.58, 20.89, 14.17, 14.03, 12.18.

**2-Methyl-N-Pentylhexanamide**

<sup>1</sup>H NMR (300 MHz, Chloroform-d) δ 5.45 (s, 1H), 3.23 (tdd, *J* = 7.0, 5.8, 4.5 Hz, 2H), 2.19 – 2.03 (m, 1H), 1.69 – 1.56 (m, 1H), 1.47 (td, *J* = 8.3, 7.7, 3.6 Hz, 2H), 1.40 – 1.21 (m, 9H), 1.12 (dd, *J* = 6.9, 1.7 Hz, 6H), 0.94 – 0.84 (m, 6H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 176.51, 41.80, 39.35, 34.17, 31.50, 29.77, 29.71, 26.59, 22.75, 22.59, 18.02, 14.03.

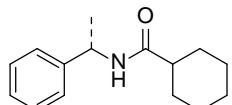
**N-Hexylheptanamide**



<sup>1</sup>H NMR (300 MHz, Chloroform-*d*) δ 5.42 (d, *J* = 7.8 Hz, 1H), 3.24 (td, *J* = 7.2, 5.7 Hz, 2H), 2.22 – 2.09 (m, 2H), 1.69 – 1.56 (m, 3H), 1.55 – 1.42 (m, 2H), 1.30 (ddd, *J* = 13.4, 8.5, 6.4 Hz, 10H), 0.88 (ddt, *J* = 7.3, 4.9, 2.7 Hz, 6H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 173.10, 39.53, 37.00, 31.59, 31.51, 29.68, 29.01, 26.62, 25.84, 22.59, 22.55, 14.07, 14.05.

**(S)-N-(1-Phenylethyl)cyclohexanecarboxamide**

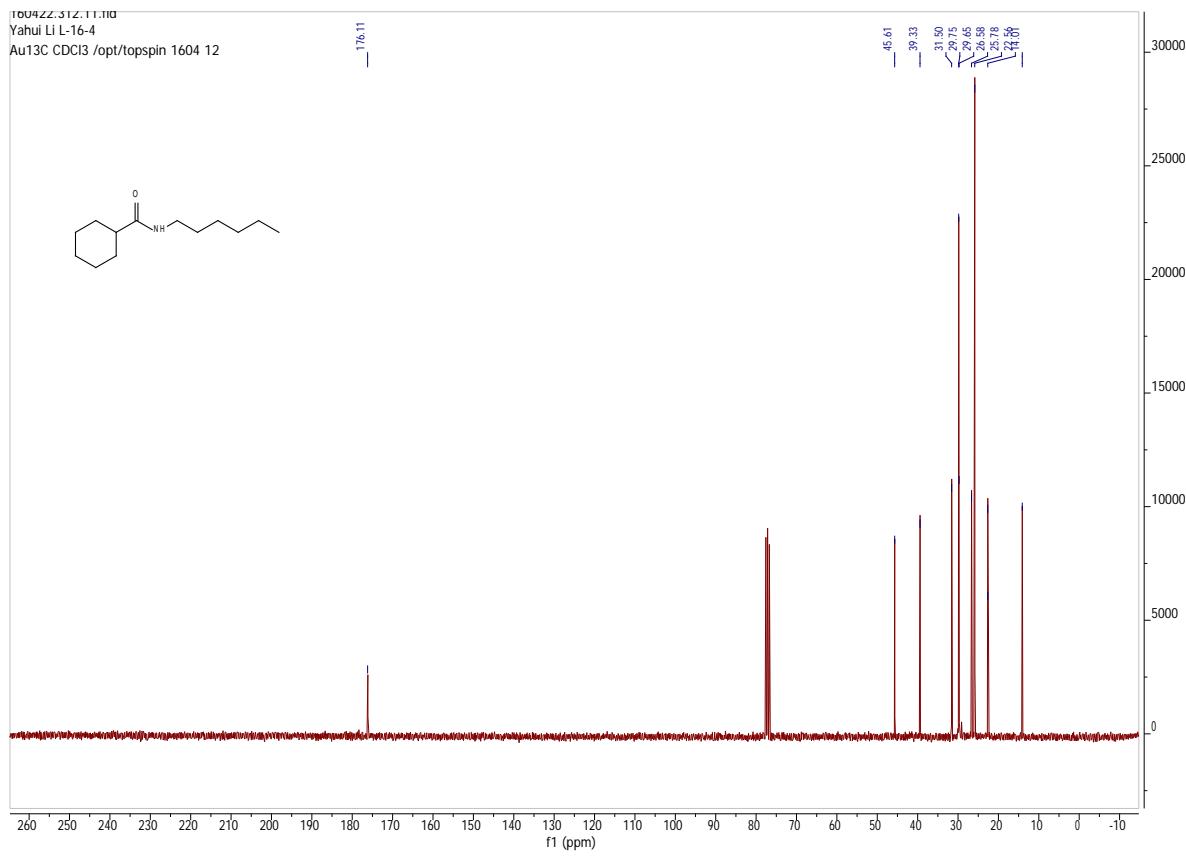
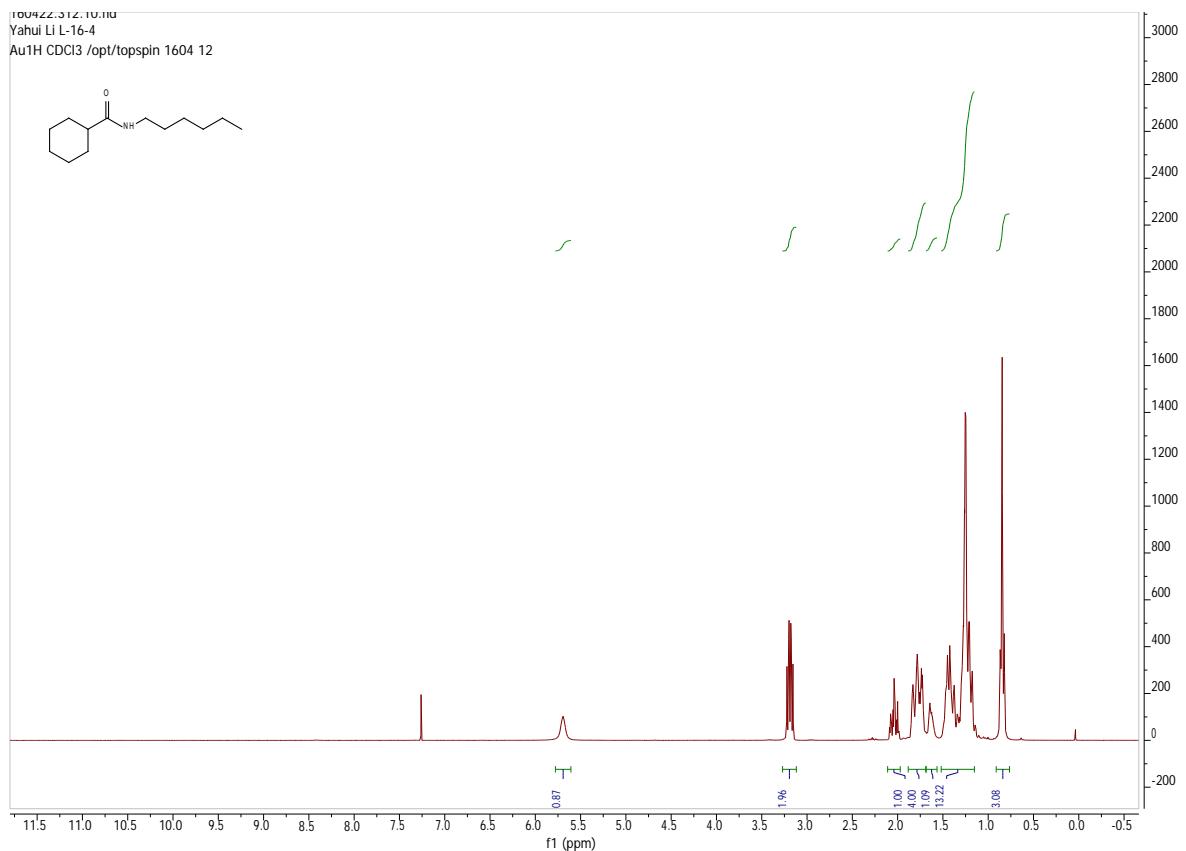


<sup>1</sup>H NMR (300 MHz, Chloroform-*d*) δ 7.31 – 7.11 (m, 5H), 5.79 (d, *J* = 7.8 Hz, 1H), 5.12 – 4.97 (m, 1H), 2.07 – 1.92 (m, 1H), 1.85 – 1.51 (m, 4H), 1.49 – 1.26 (m, 5H), 1.26 – 1.04 (m, 3H).

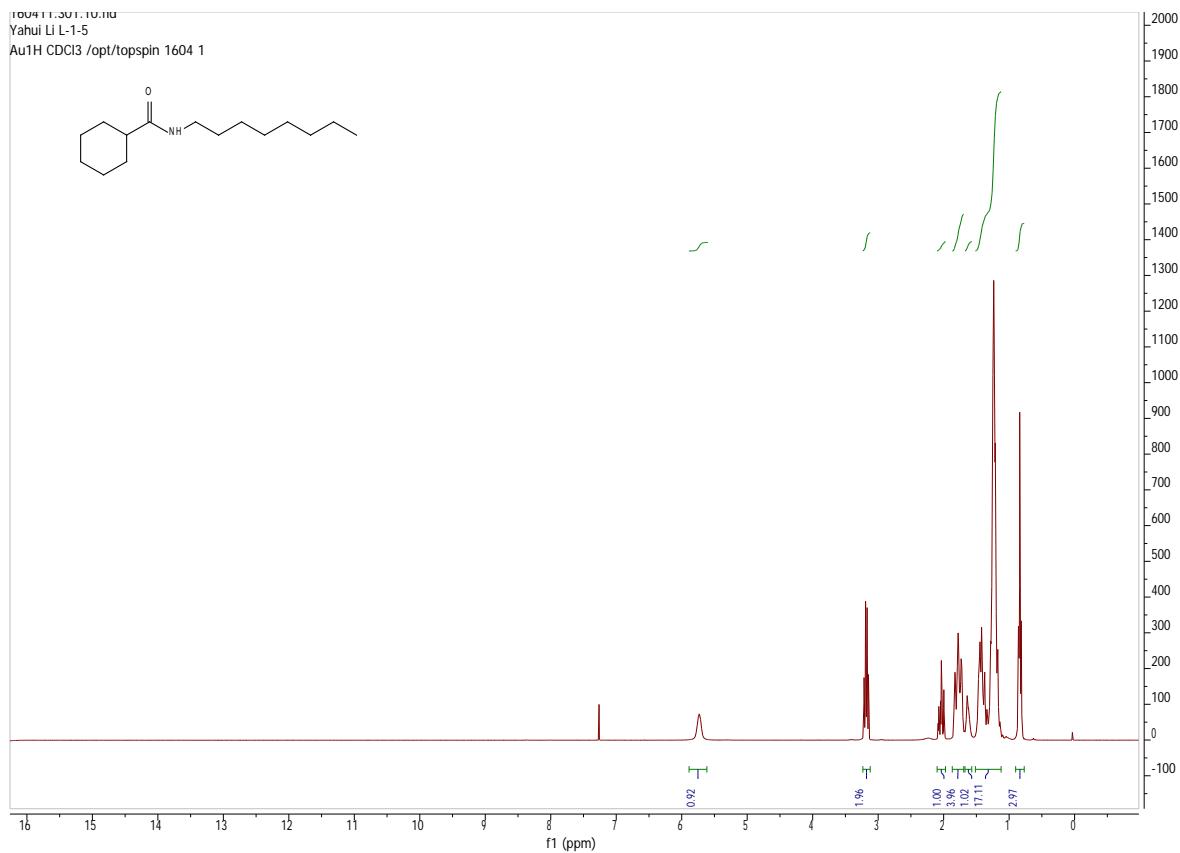
<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 175.16, 143.53, 128.63, 127.22, 126.13, 48.26, 45.55, 29.73, 29.66, 25.77, 21.81.

HRMS(ESI): calcd. for [C<sub>15</sub>H<sub>21</sub>NO + H]<sup>+</sup>: 232.16959, found: 232.17003, calcd. for [C<sub>15</sub>H<sub>21</sub>NO + Na]<sup>+</sup>:

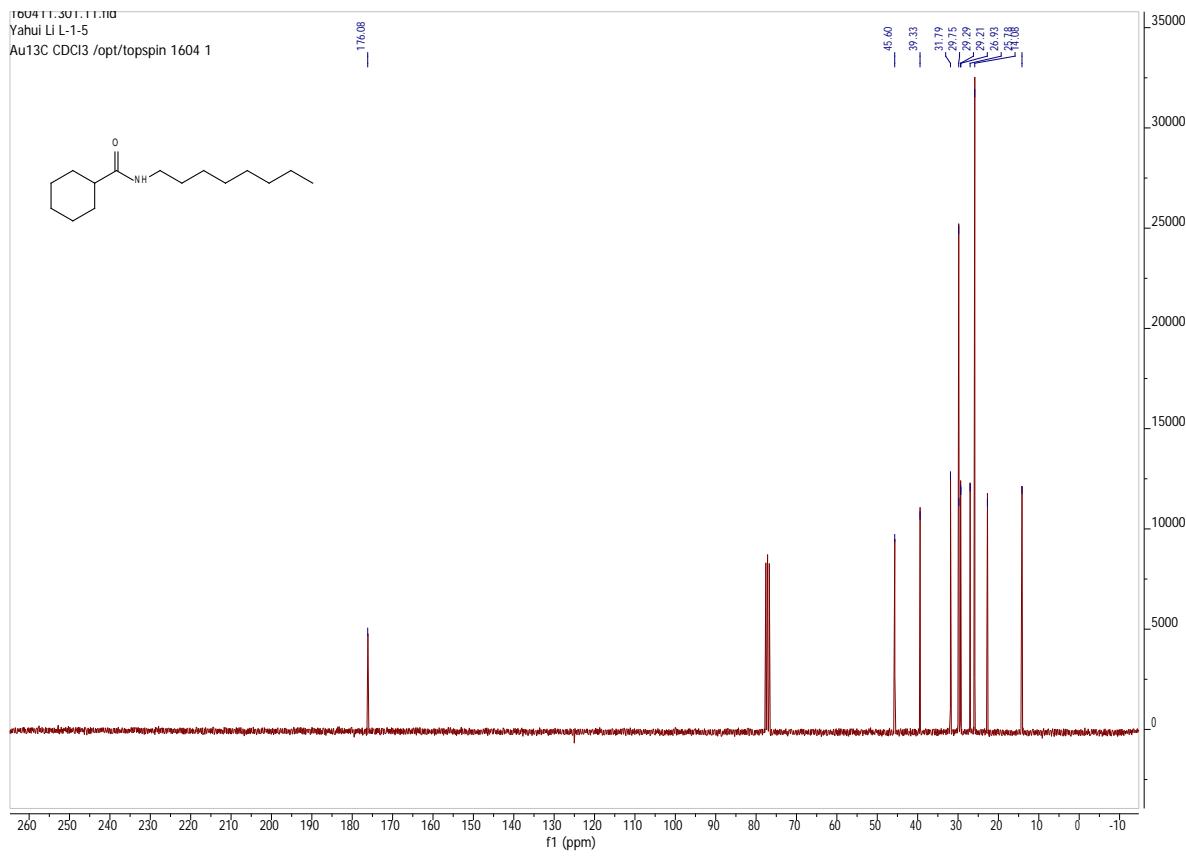
254.15154, found: 254.15207

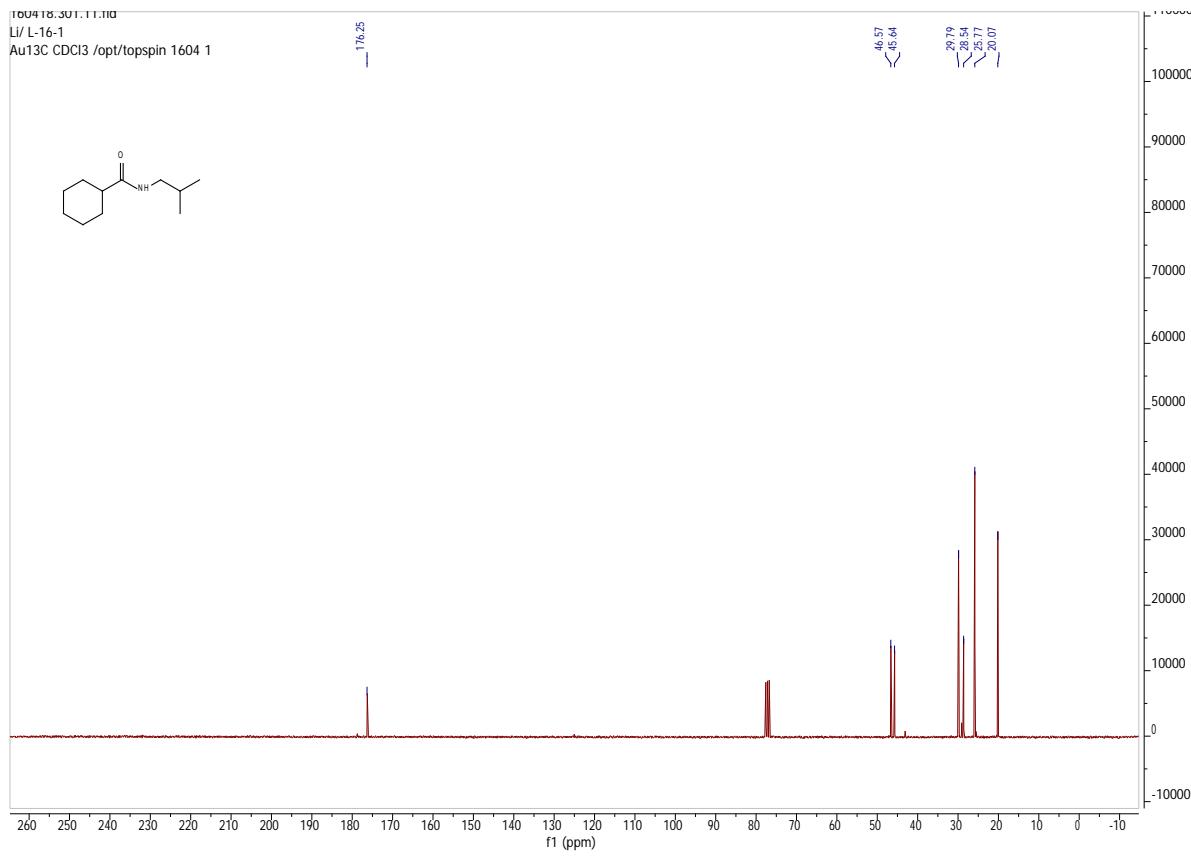
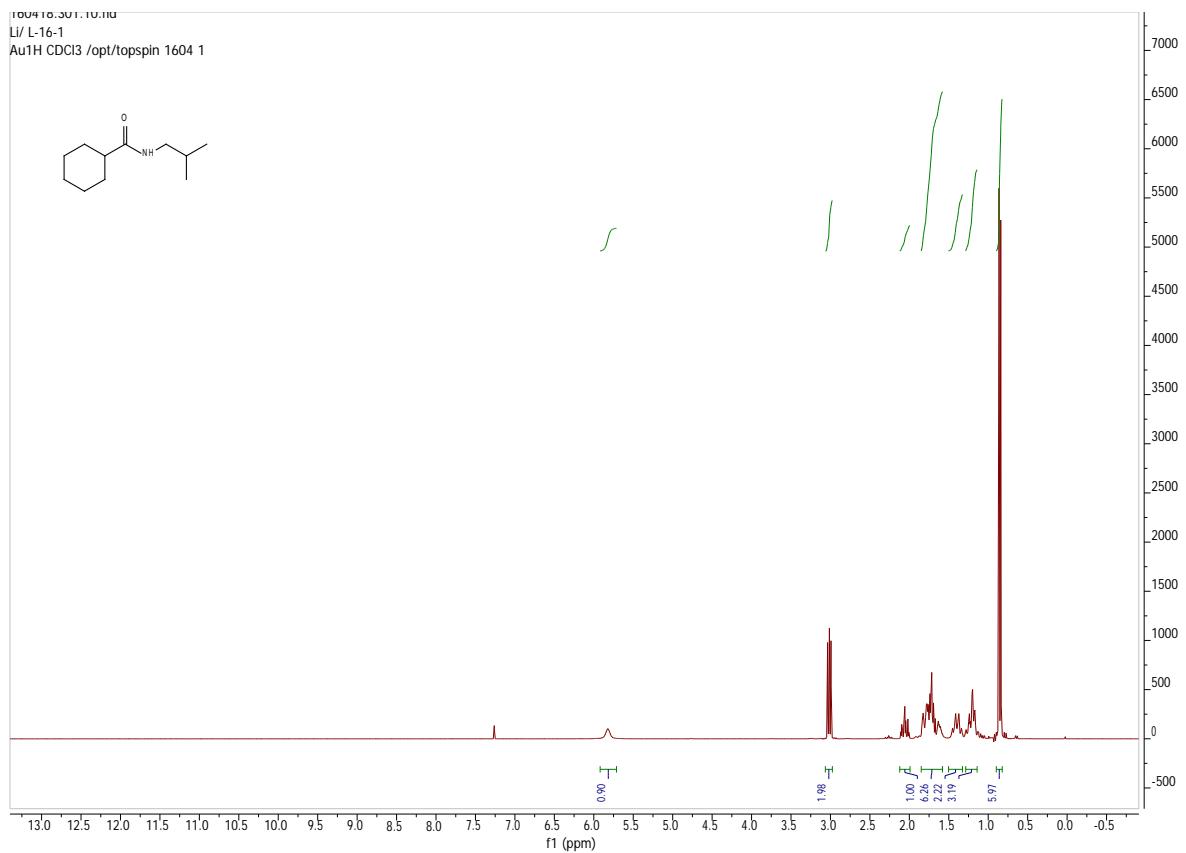


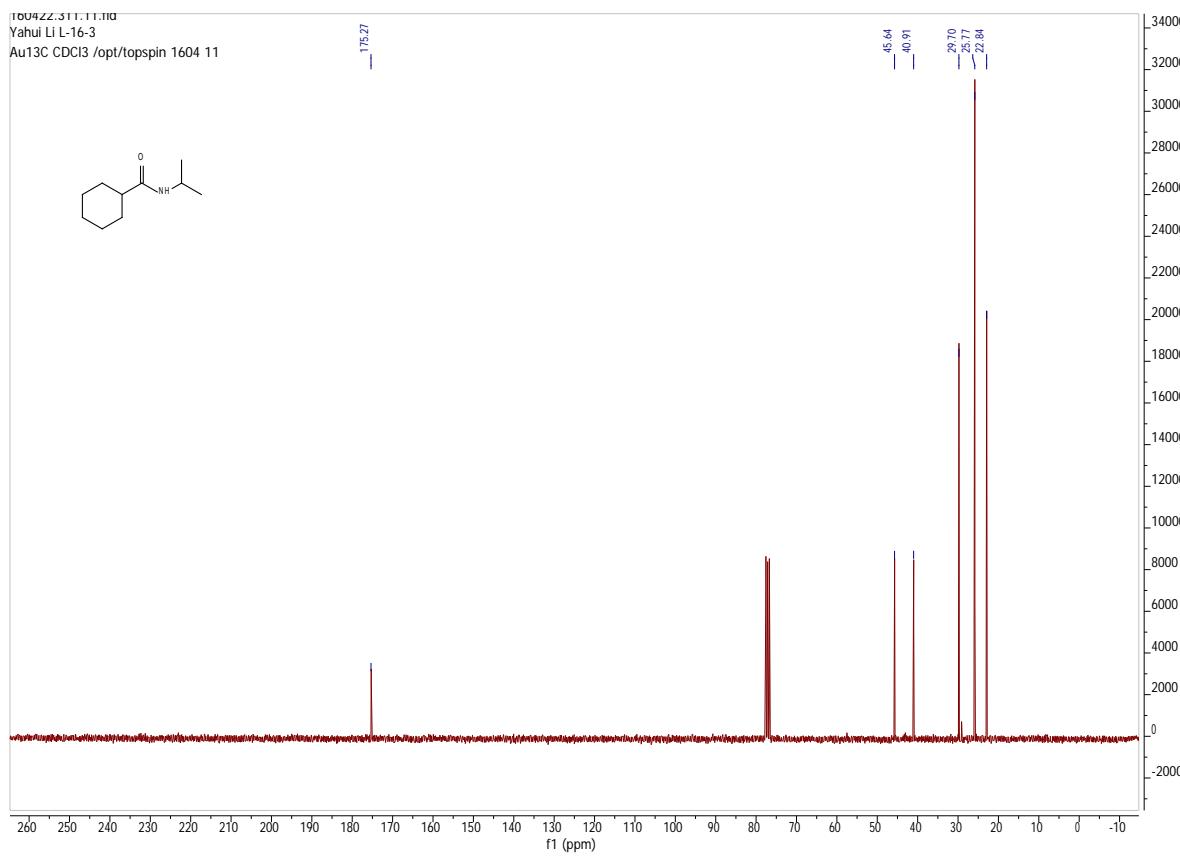
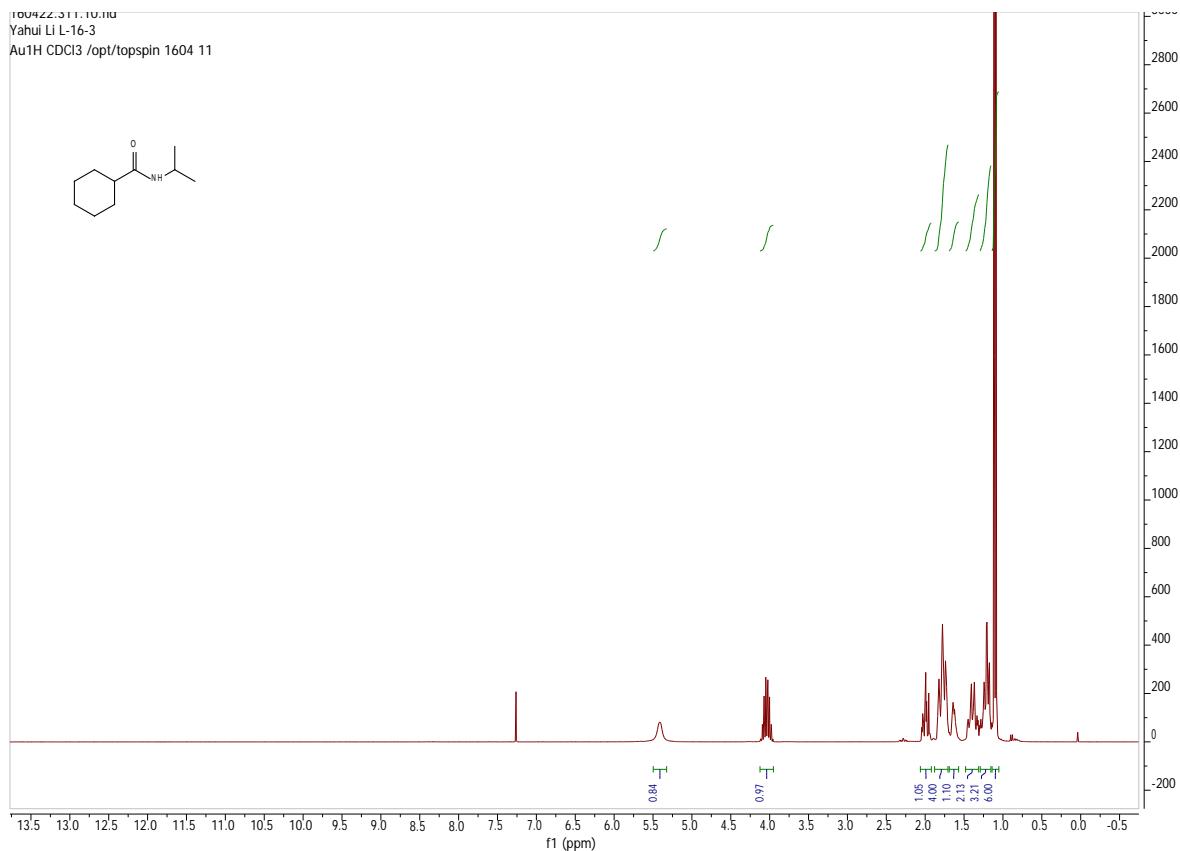
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Yahui Li L-1-5  
Au<sup>1</sup>H CDCl<sub>3</sub> /opt/topspin 1604 1



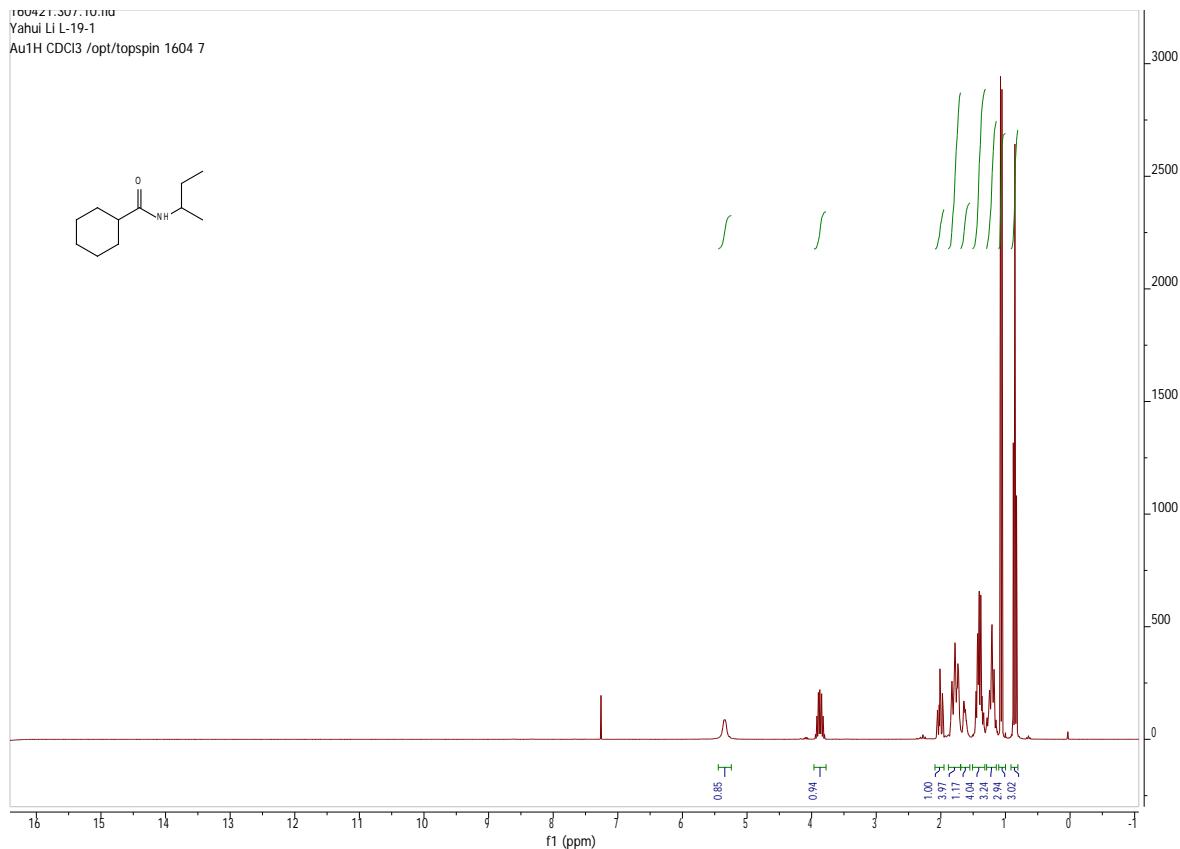
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Au<sup>13</sup>C CDCl<sub>3</sub> /opt/topspin 1604 1



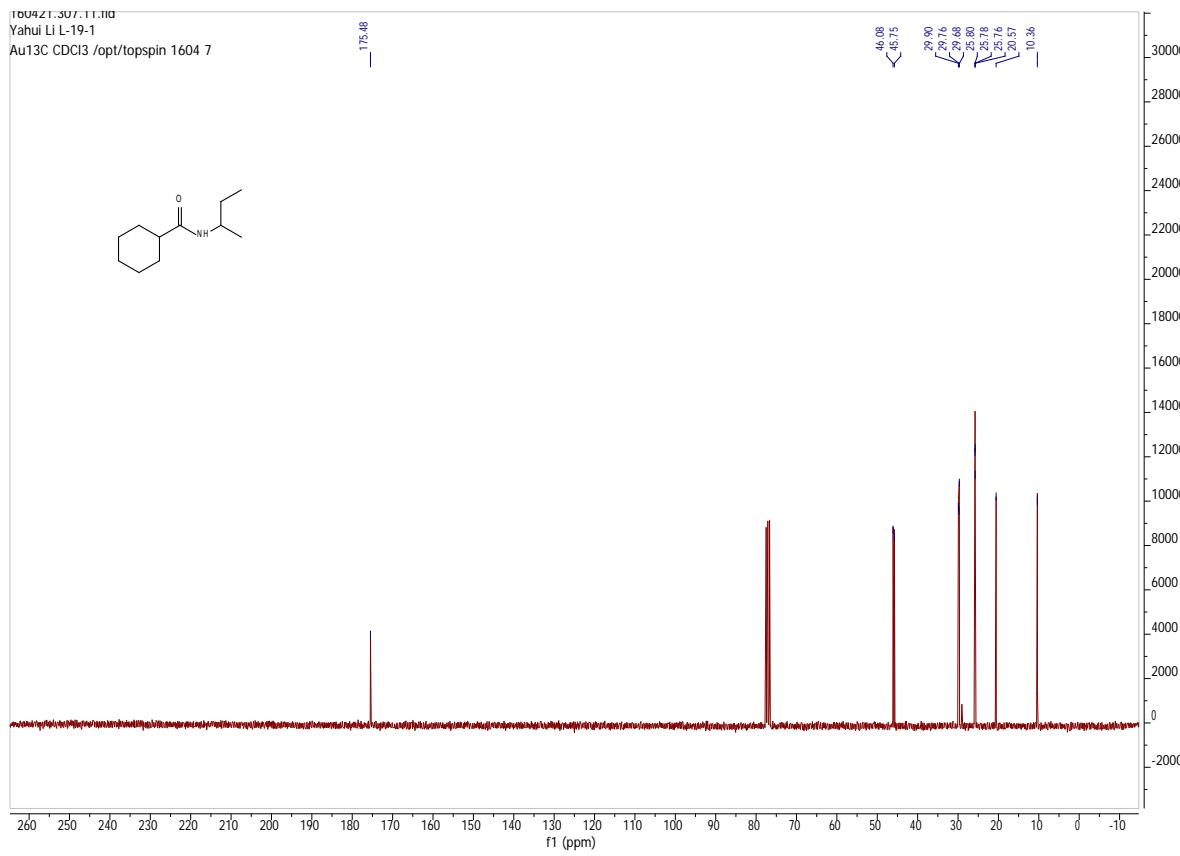




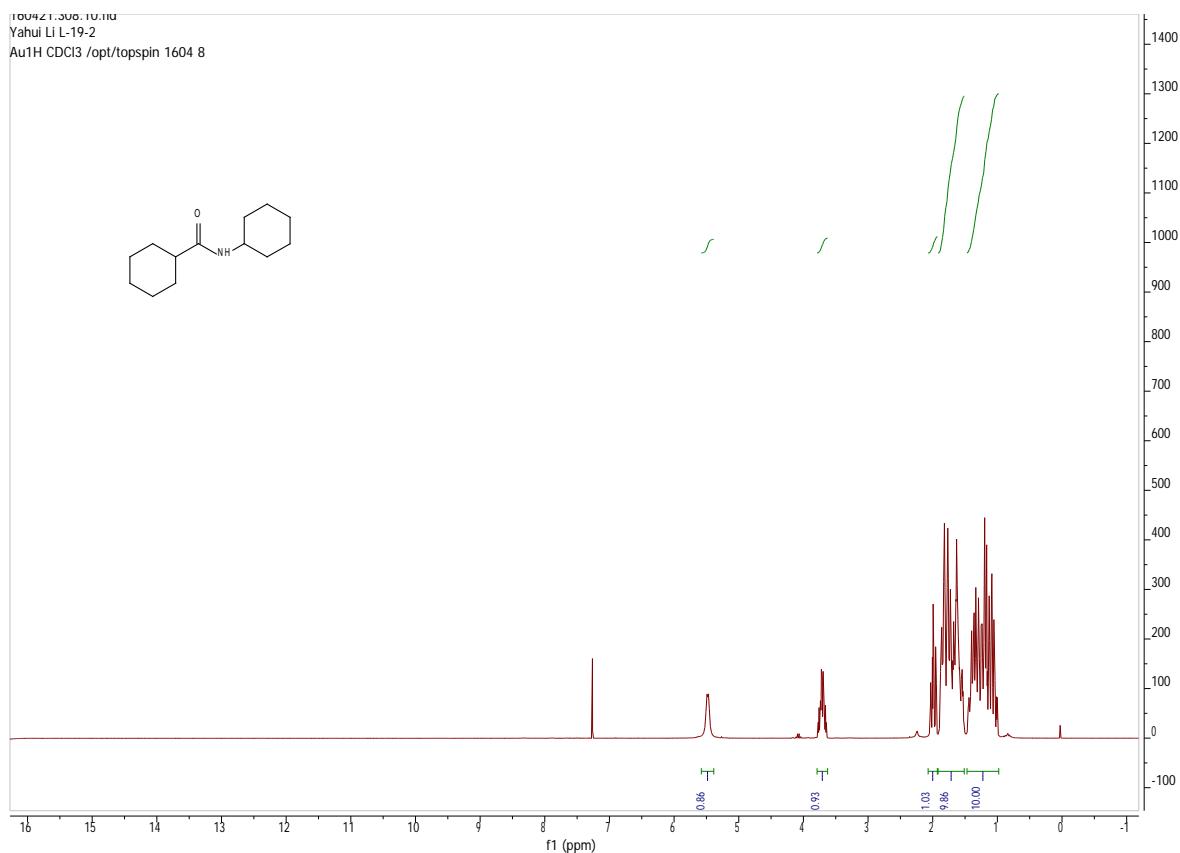
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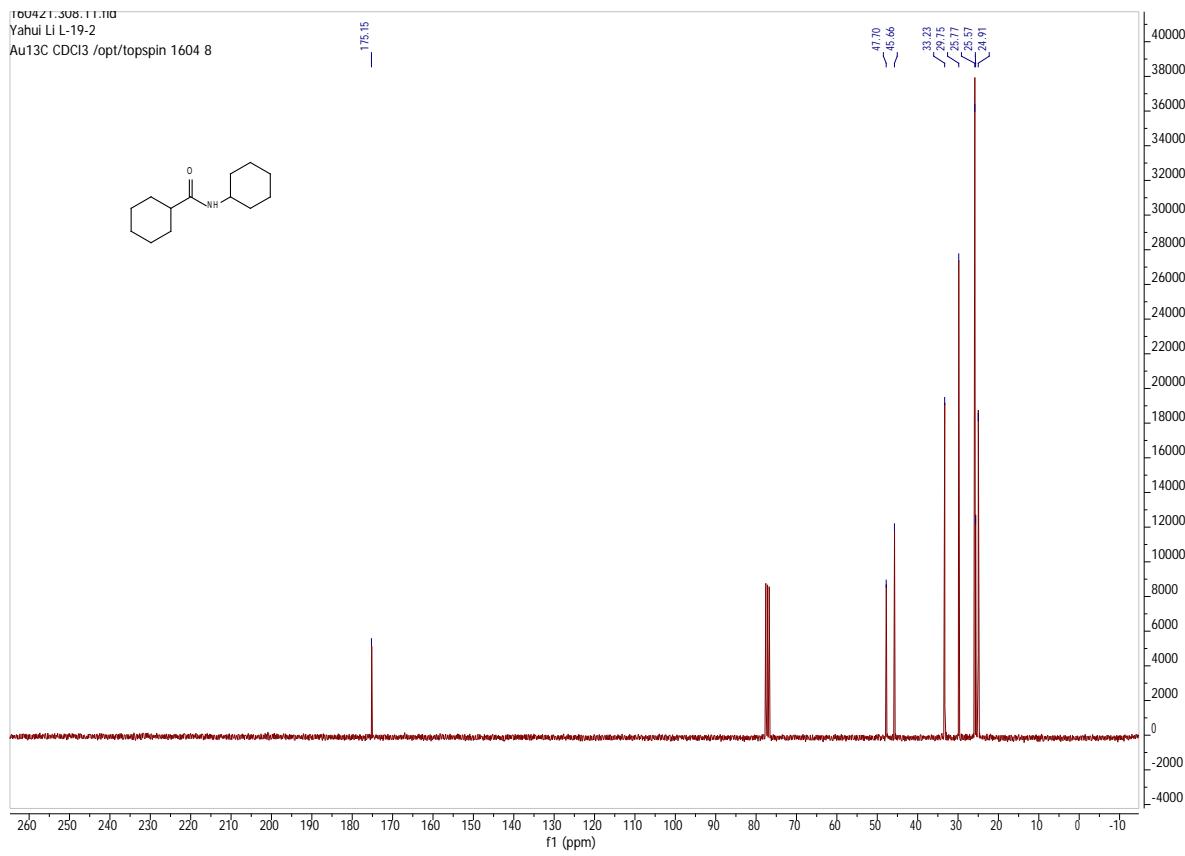
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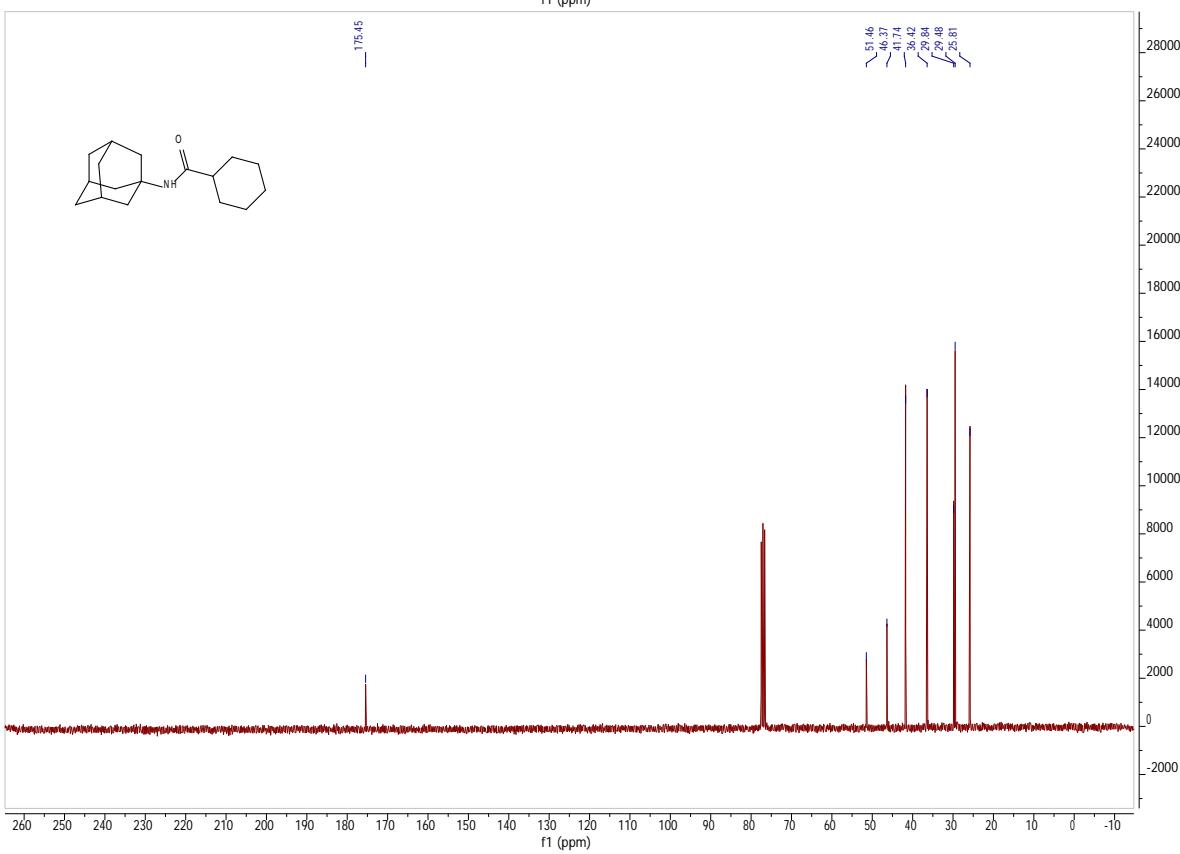
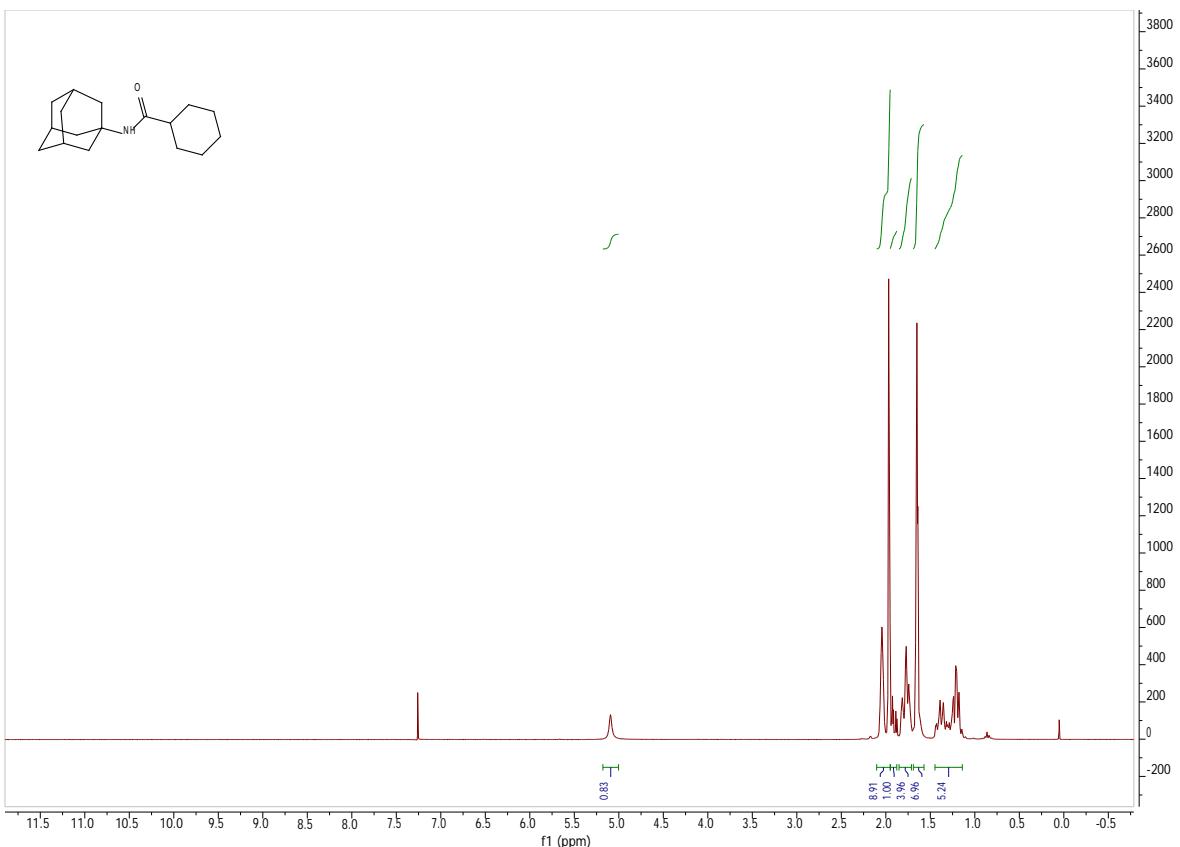


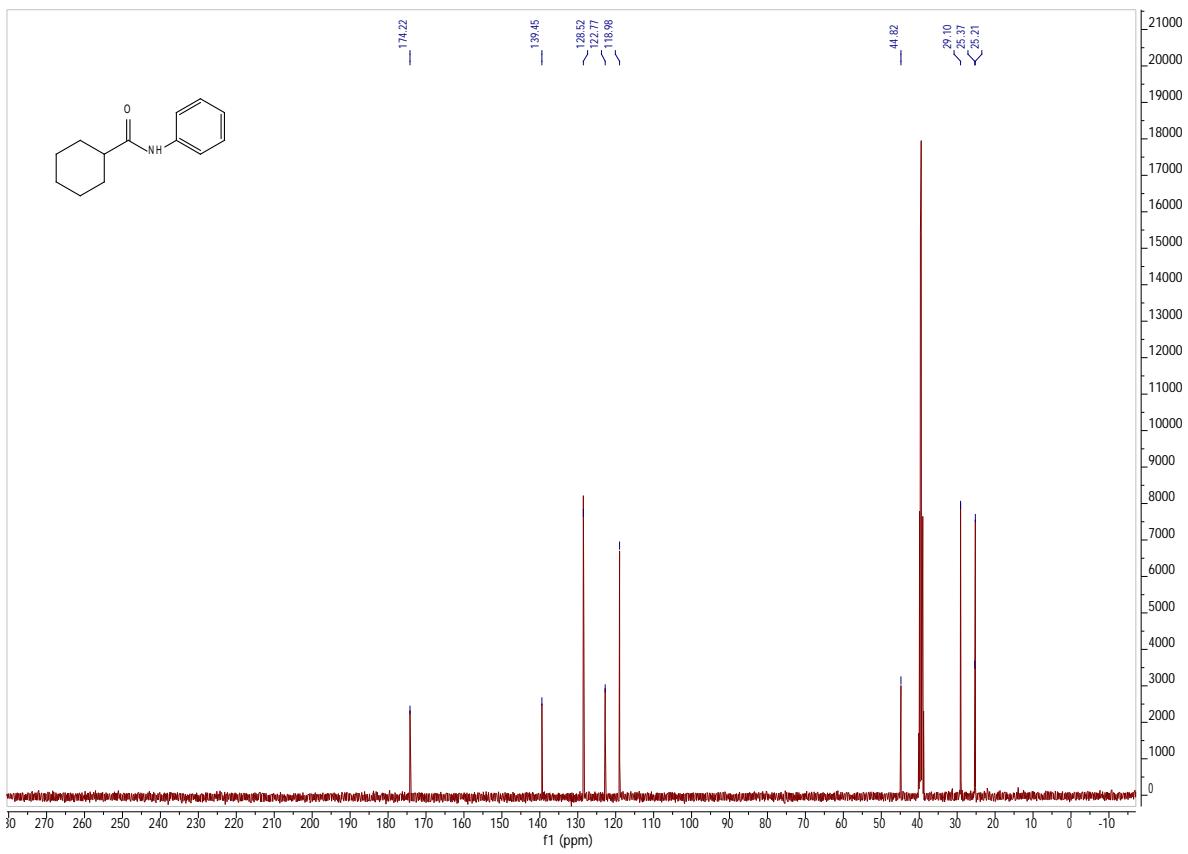
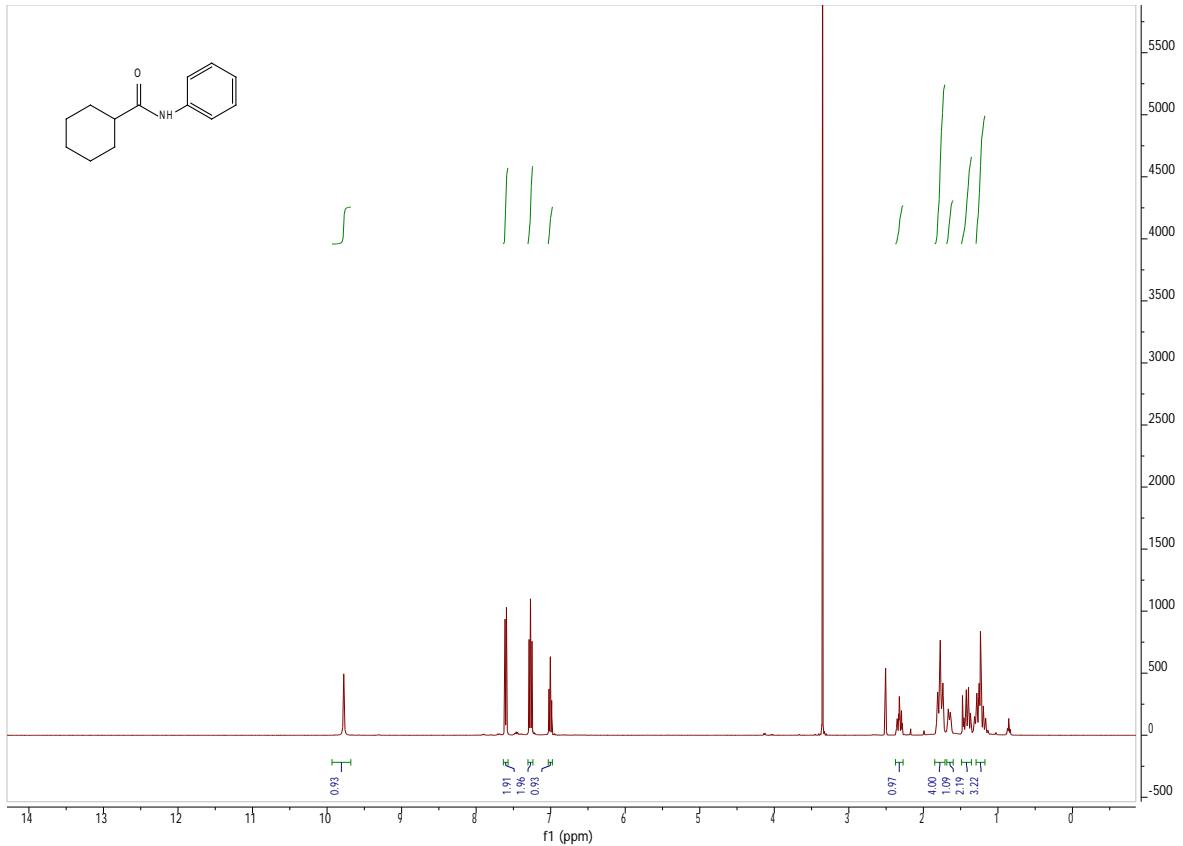
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Au1H CDCl<sub>3</sub> /opt/topspin 1604 8

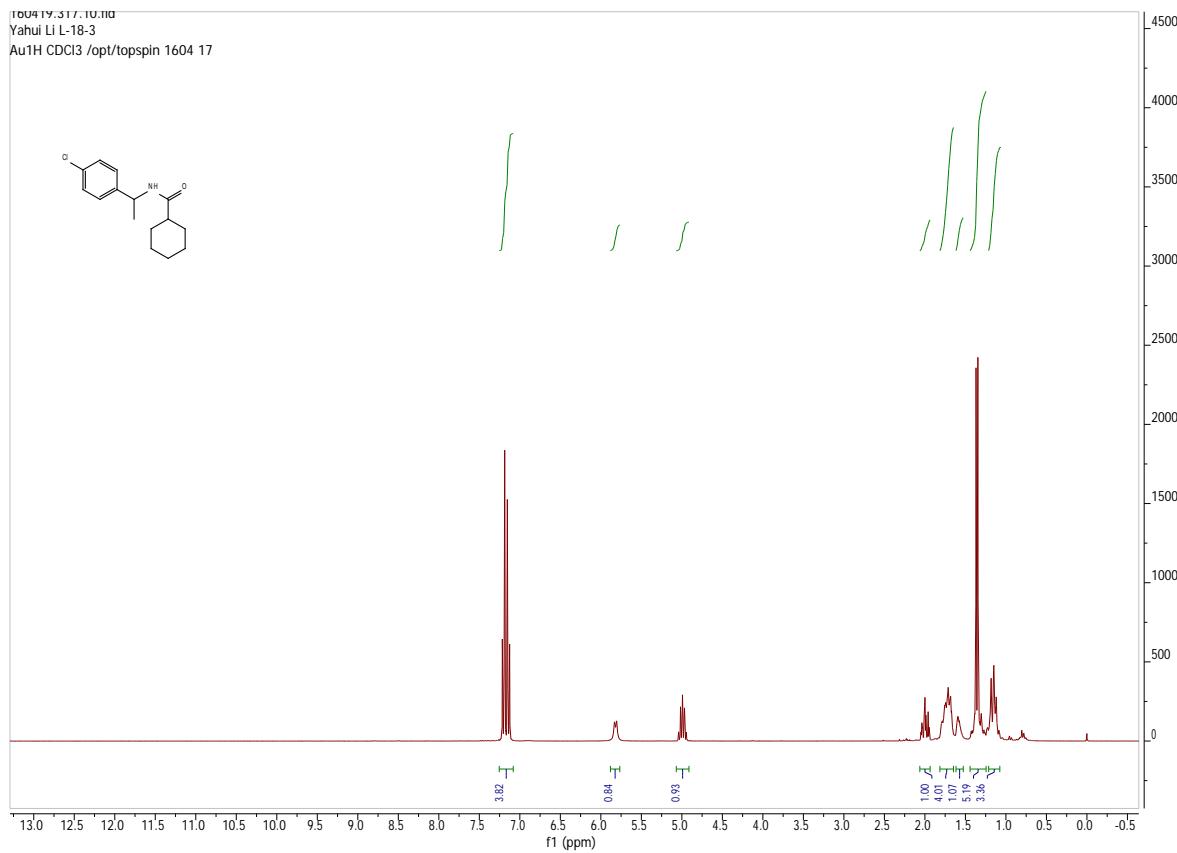
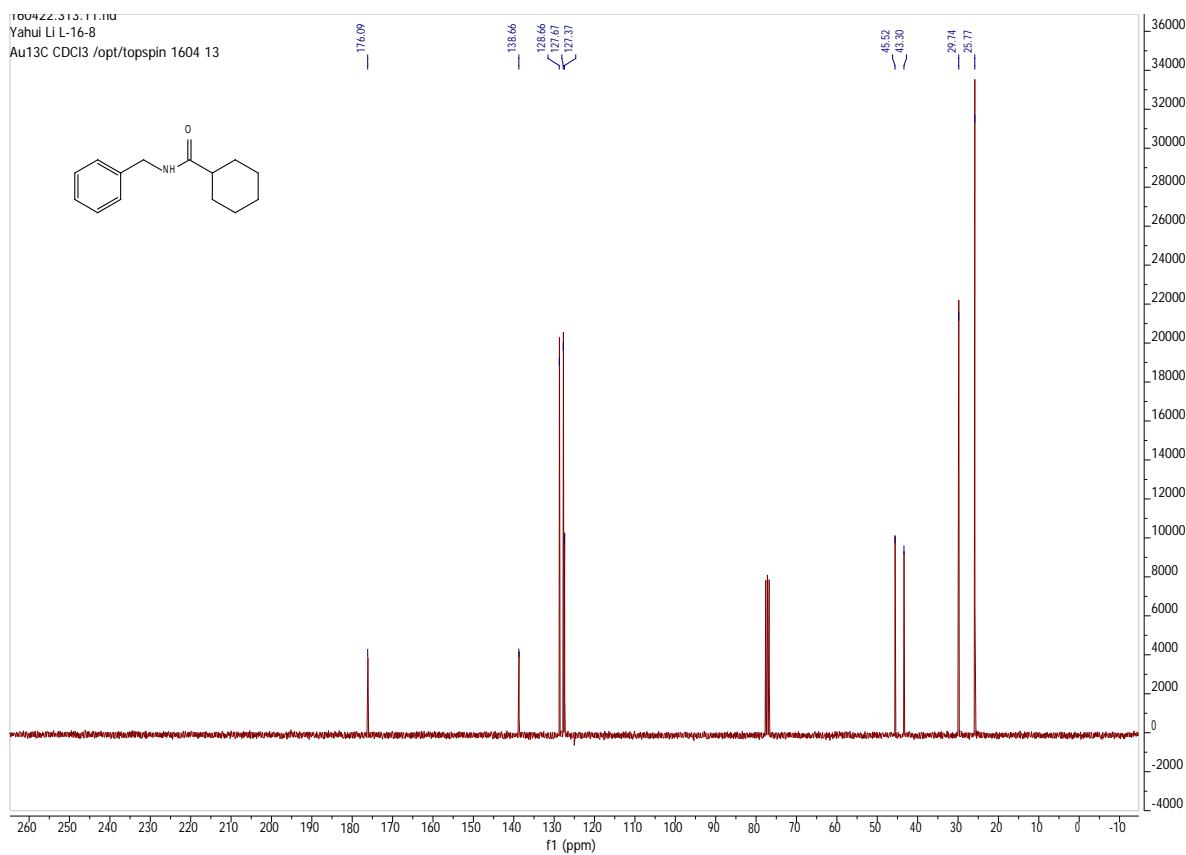


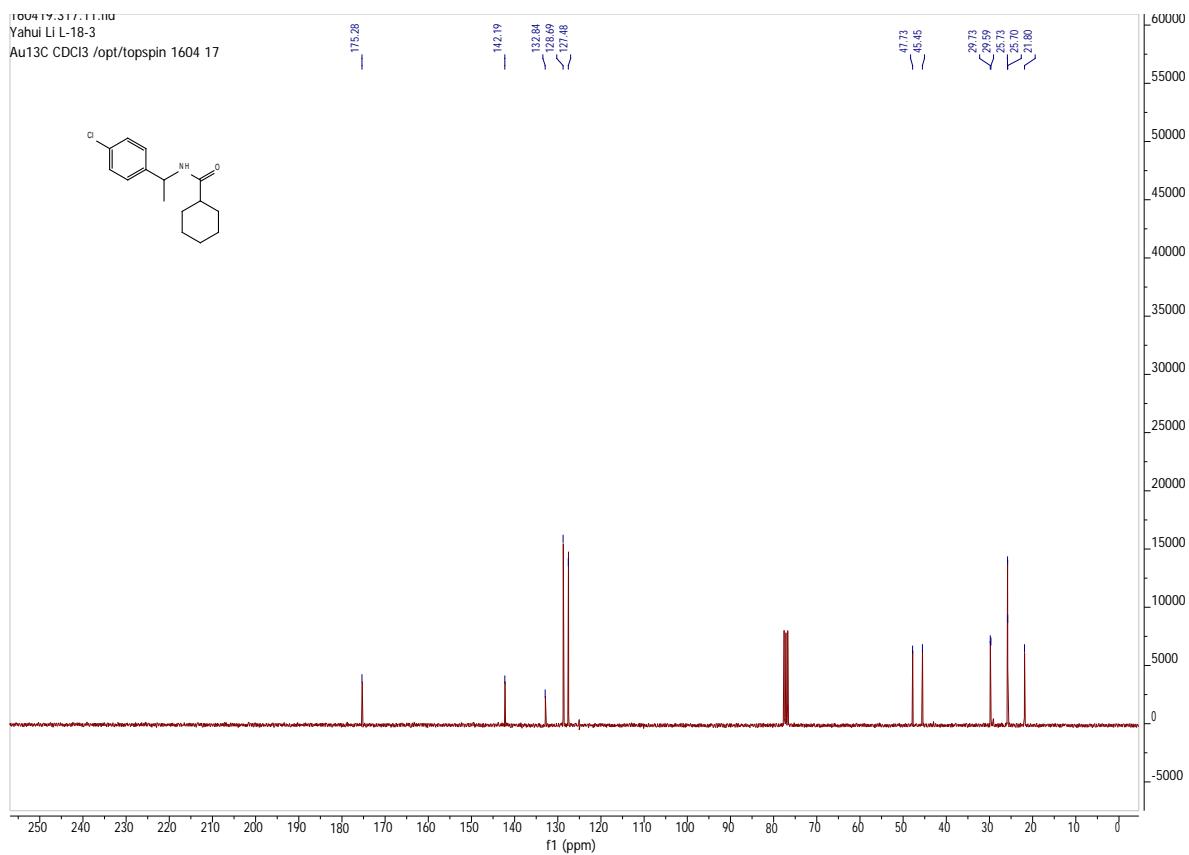
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Au13C CDCl<sub>3</sub> /opt/topspin 1604 8

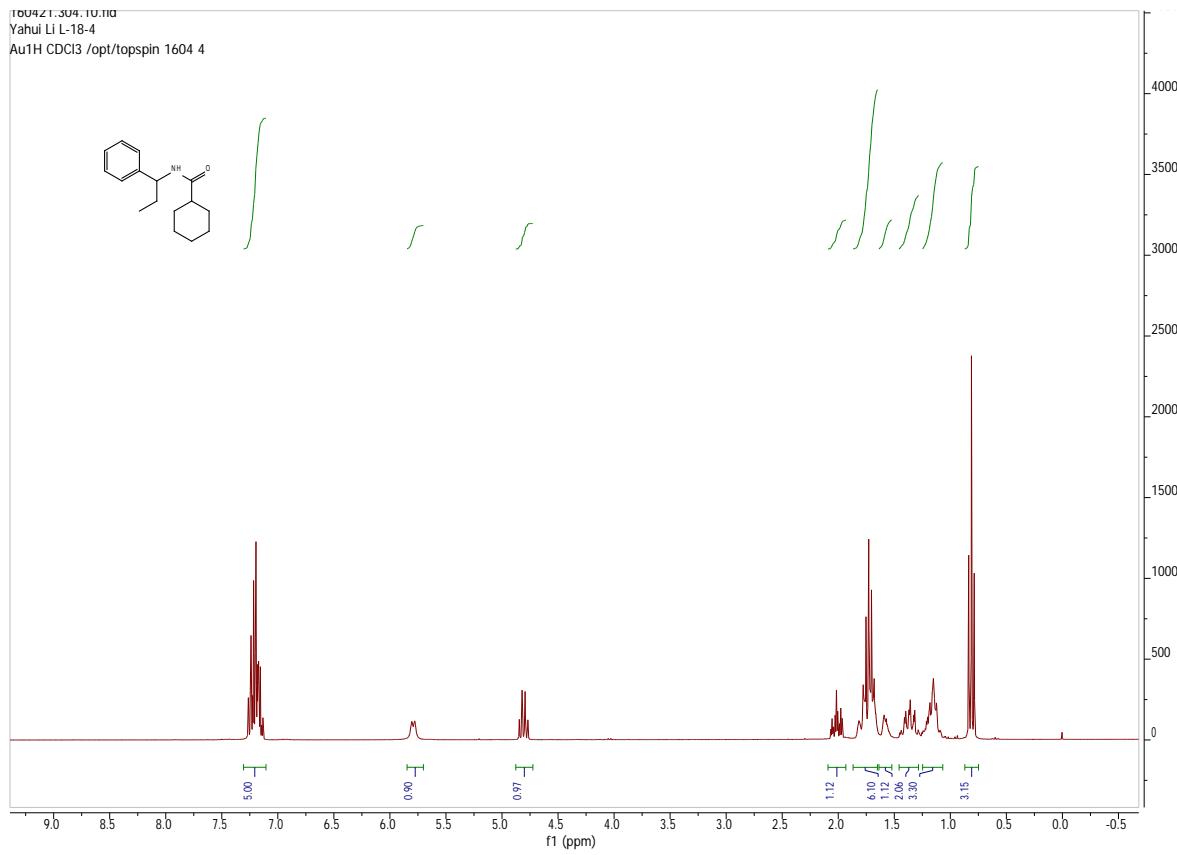
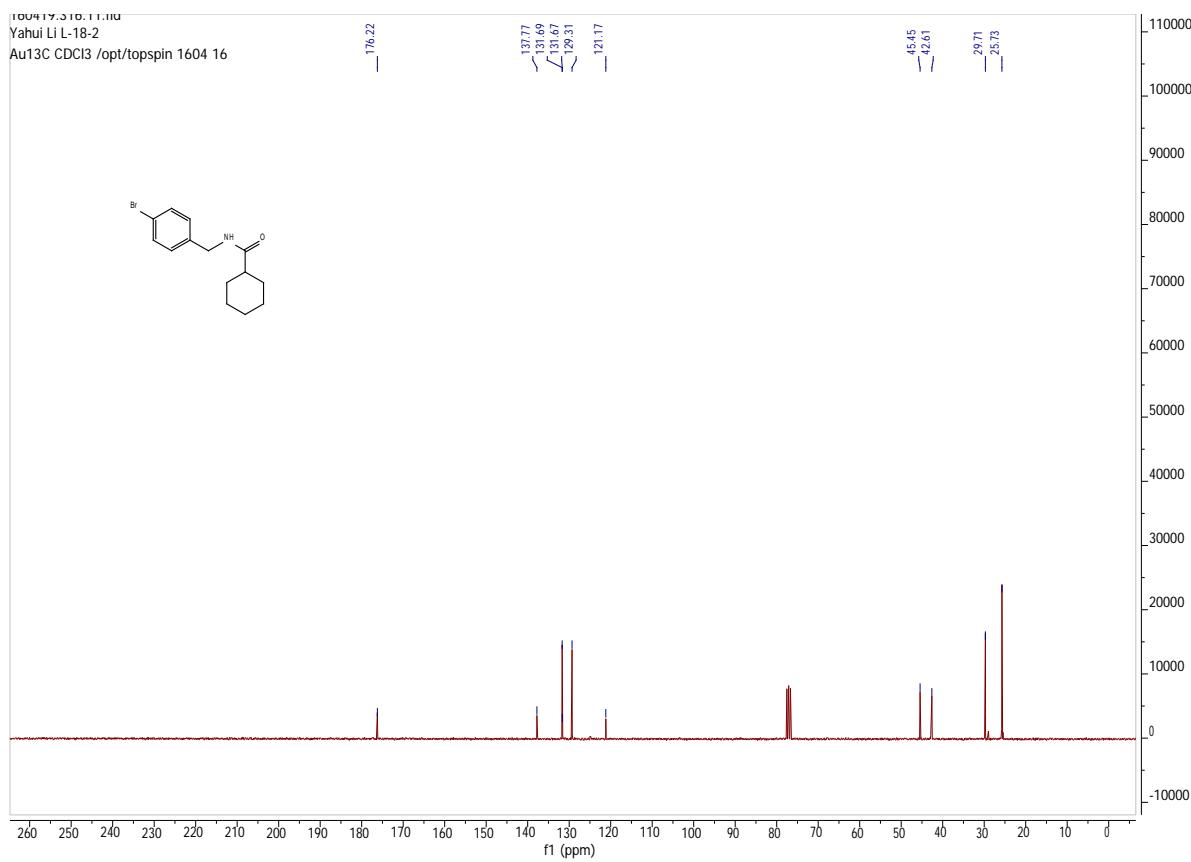


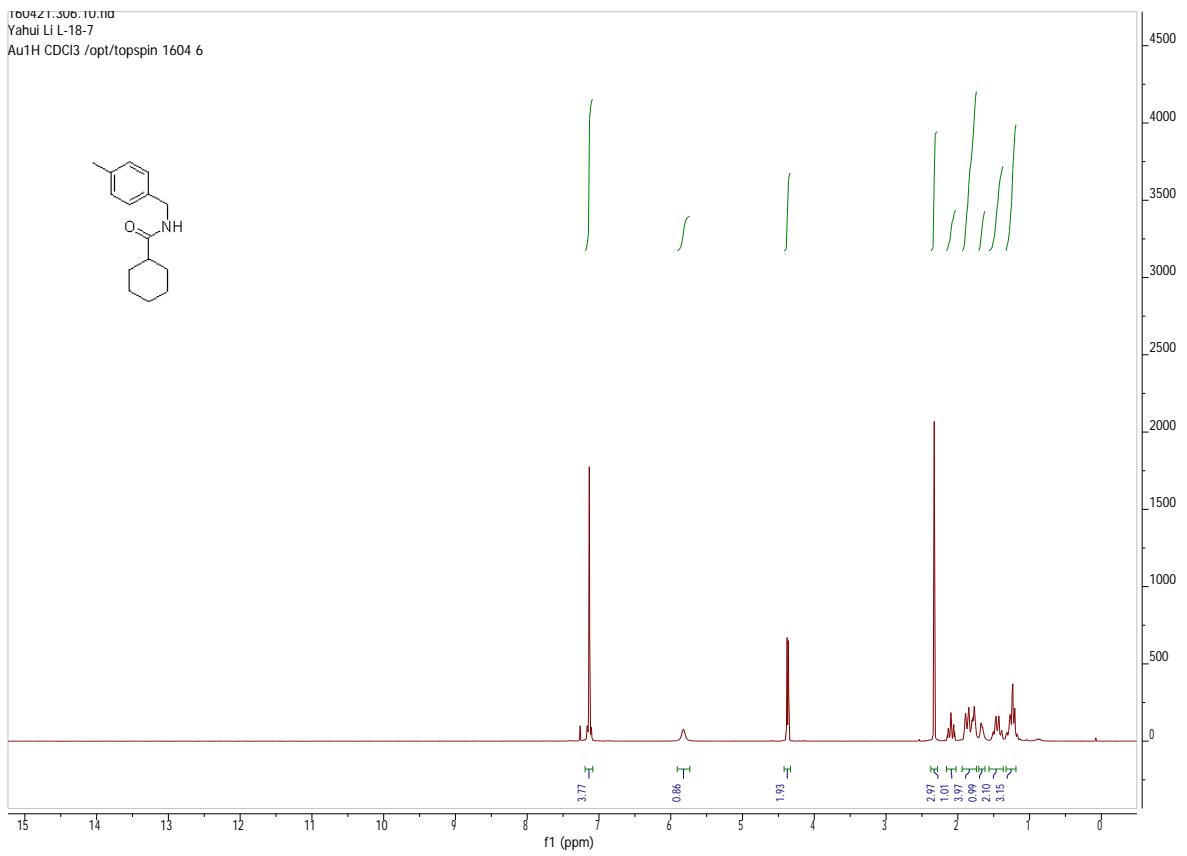
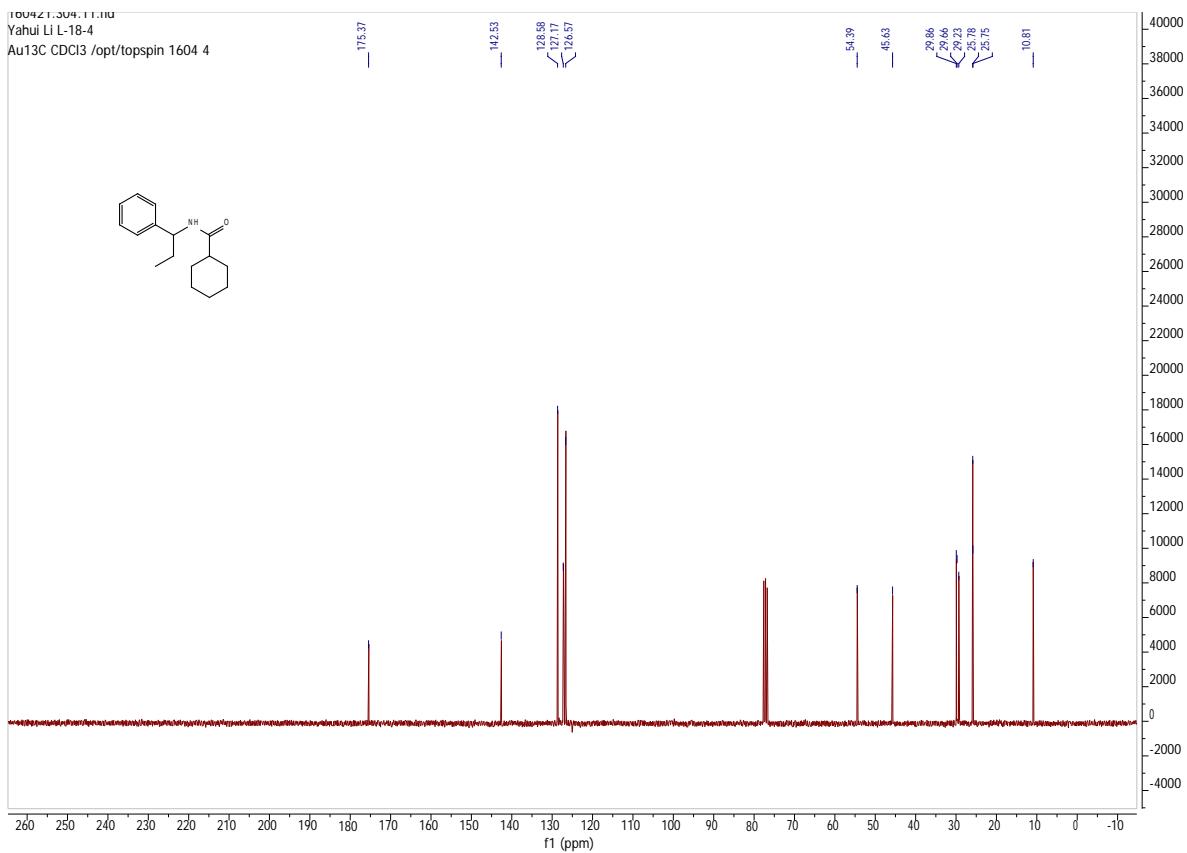


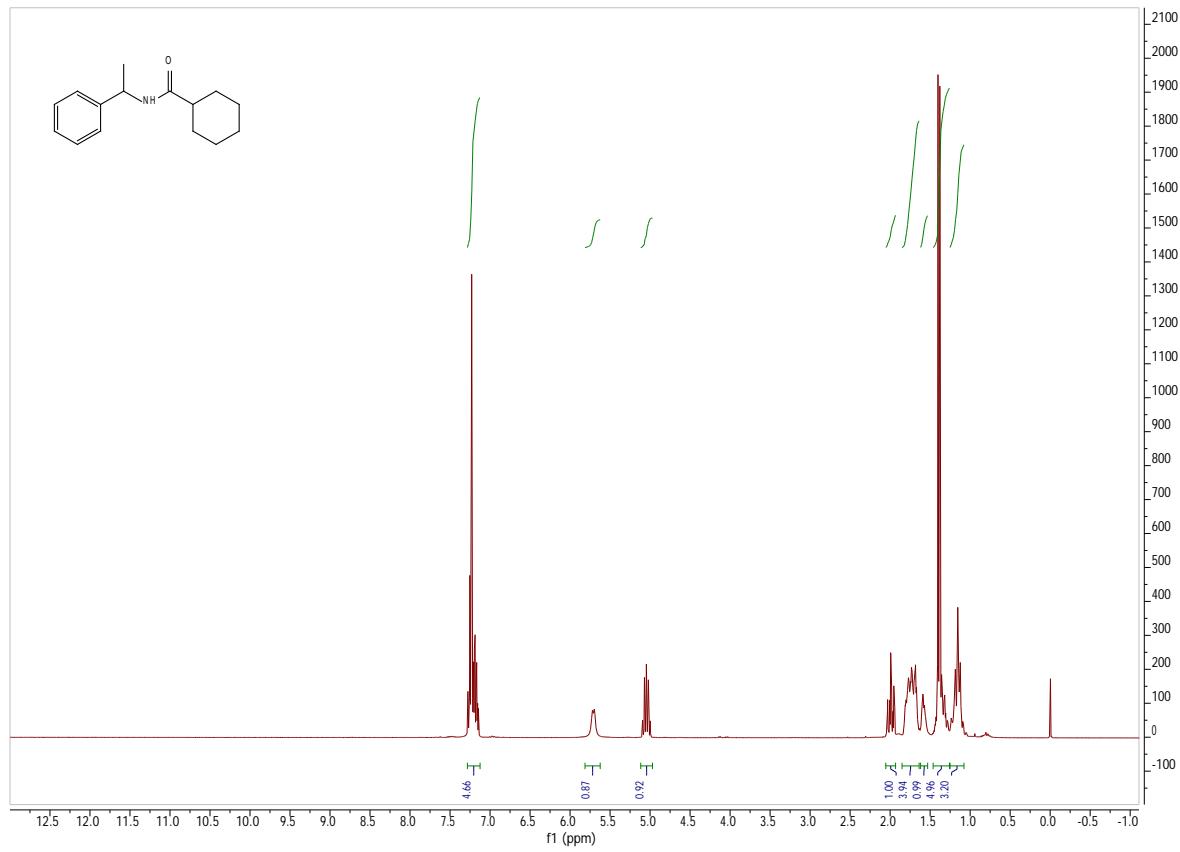
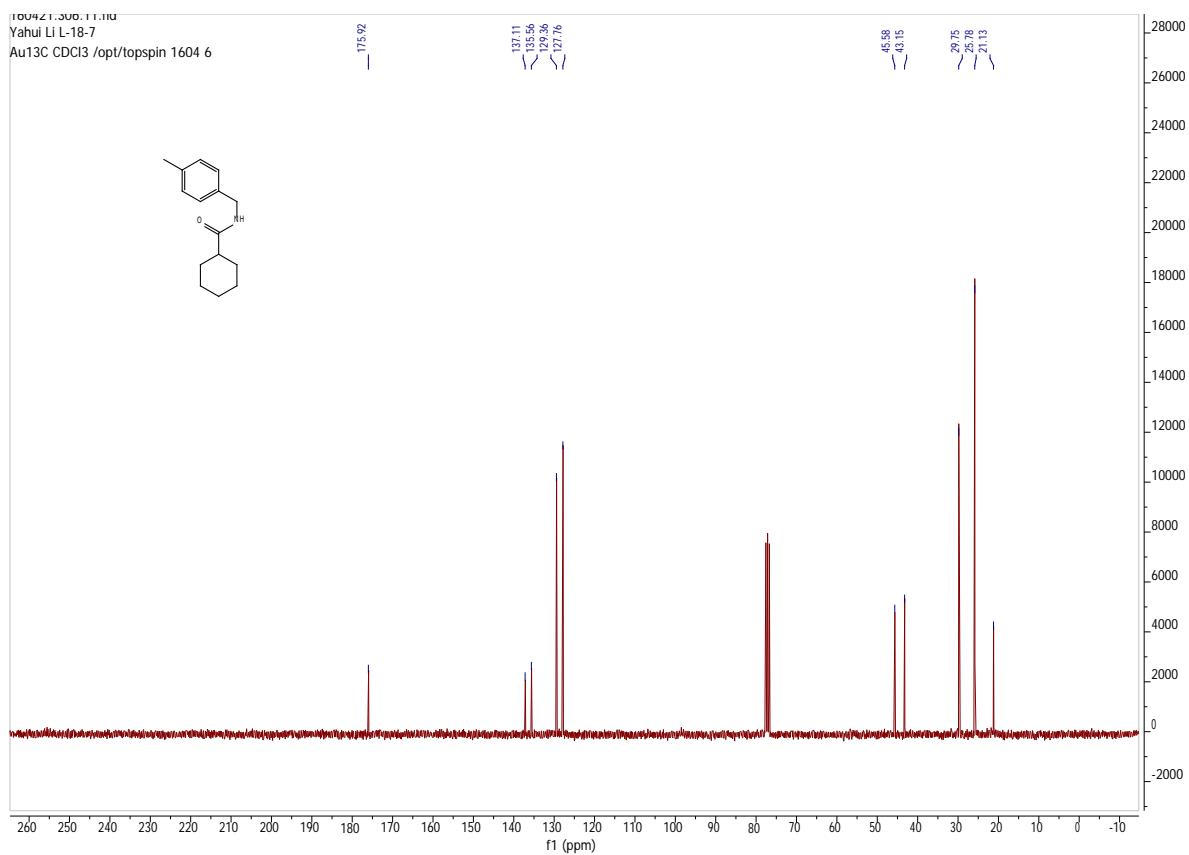


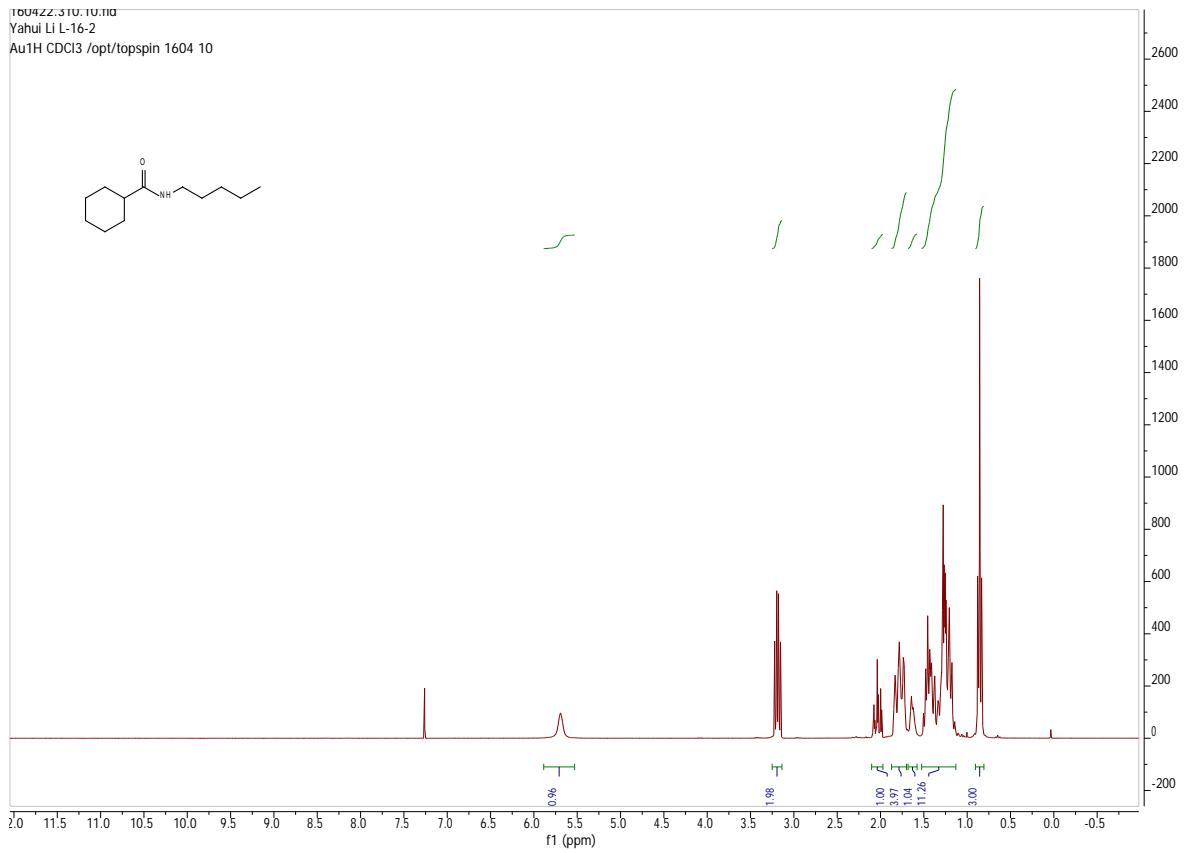
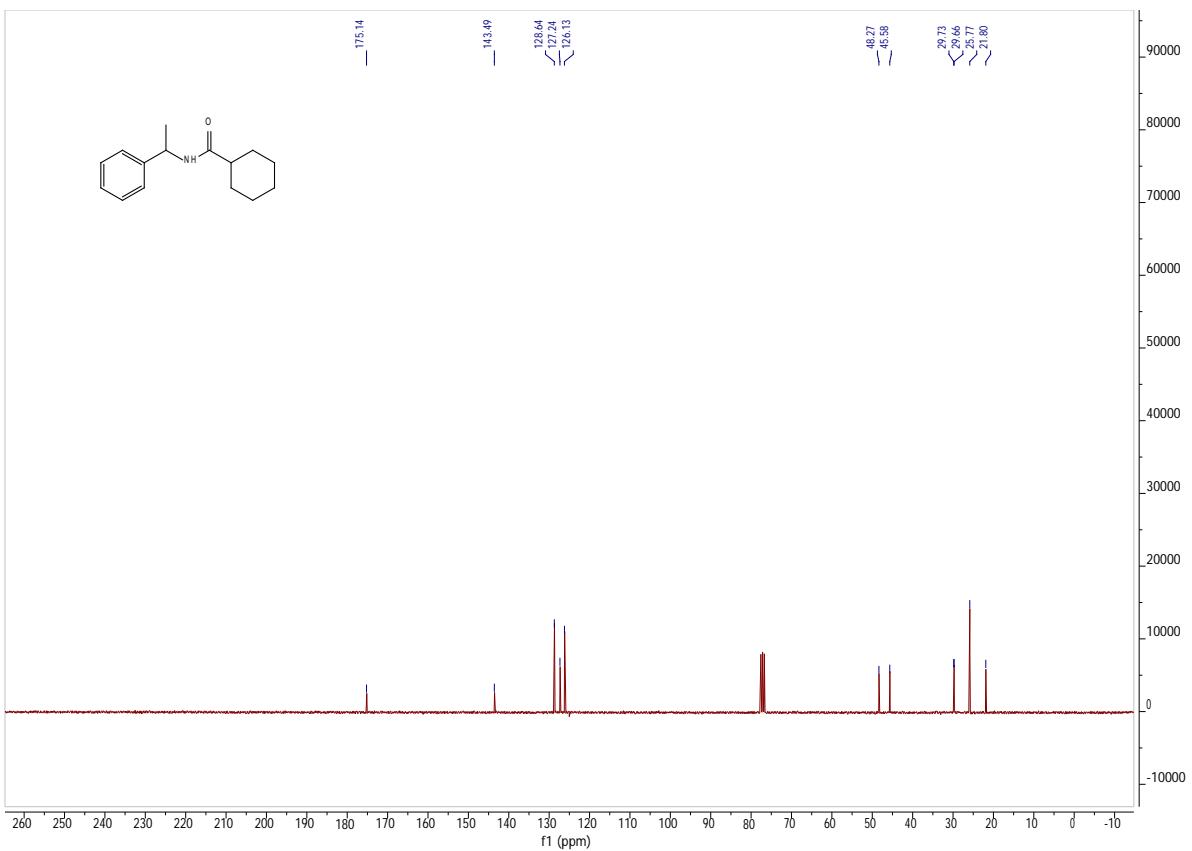


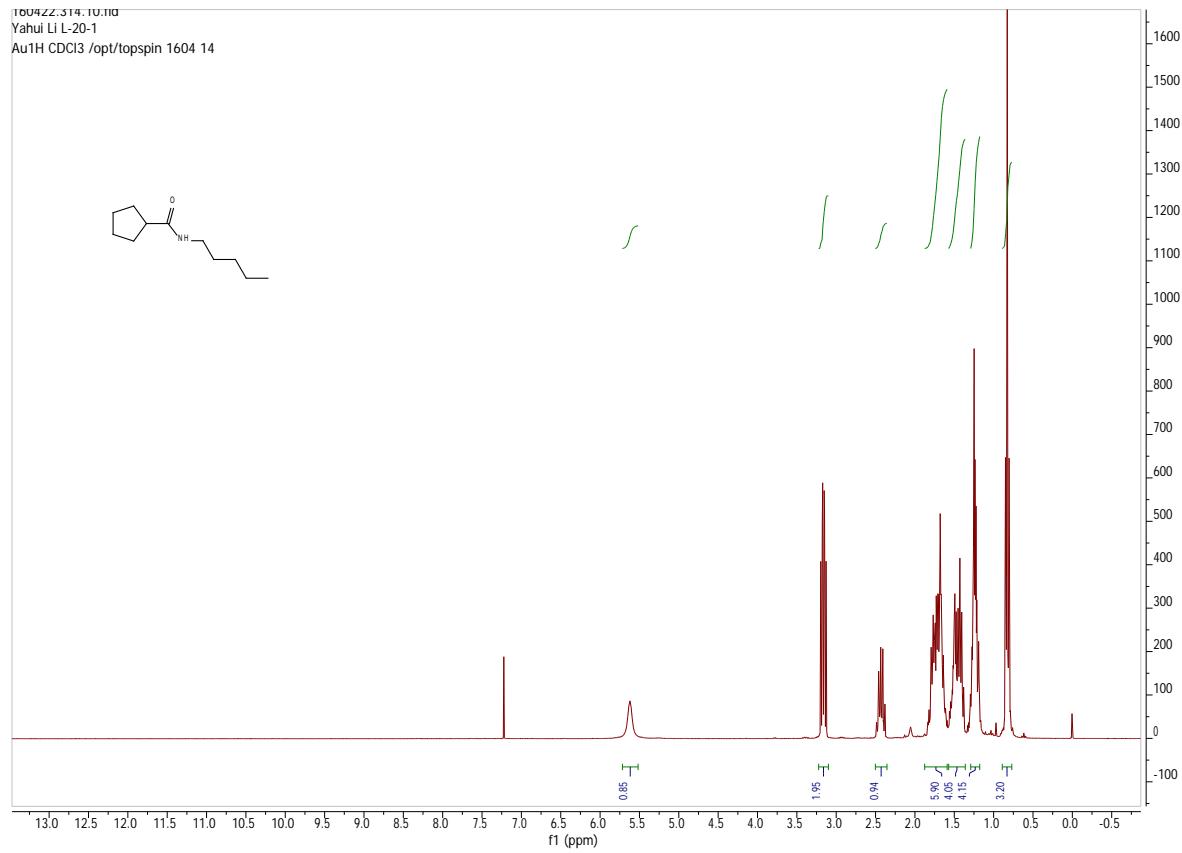
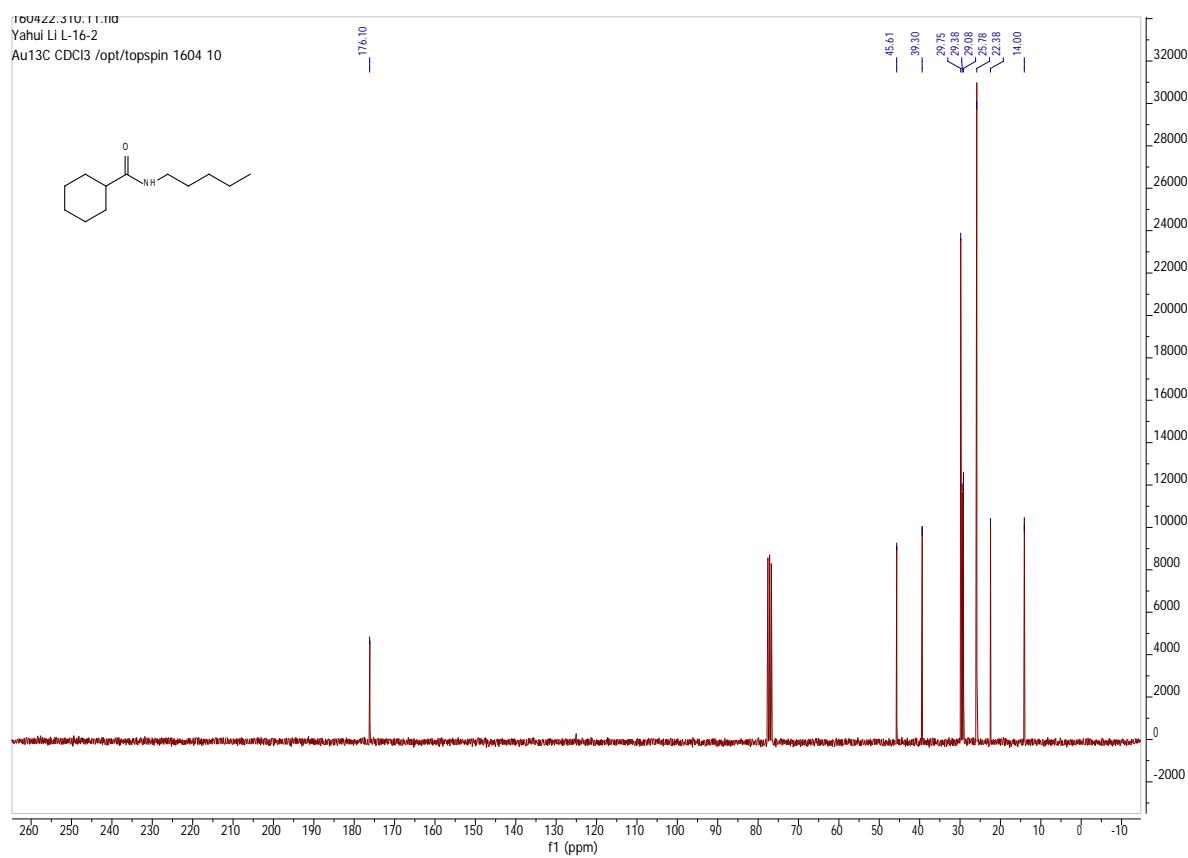


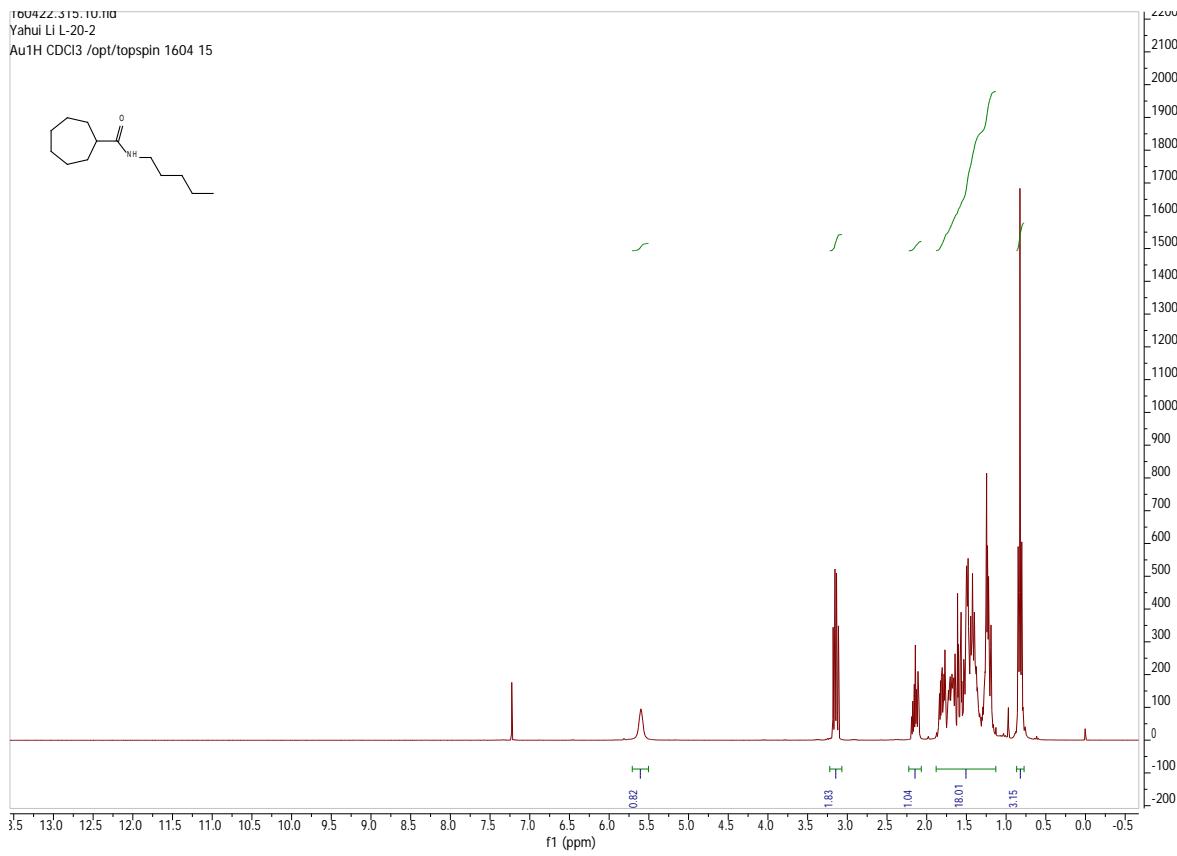
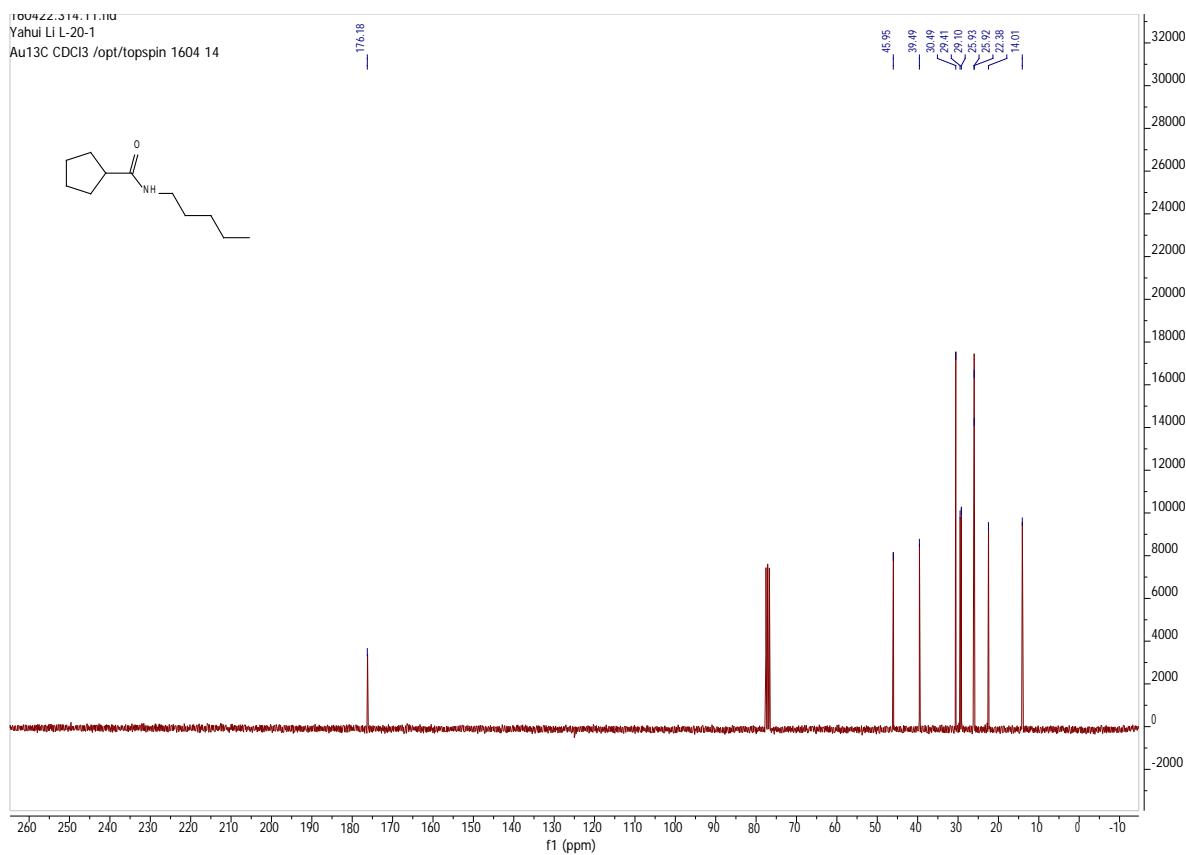


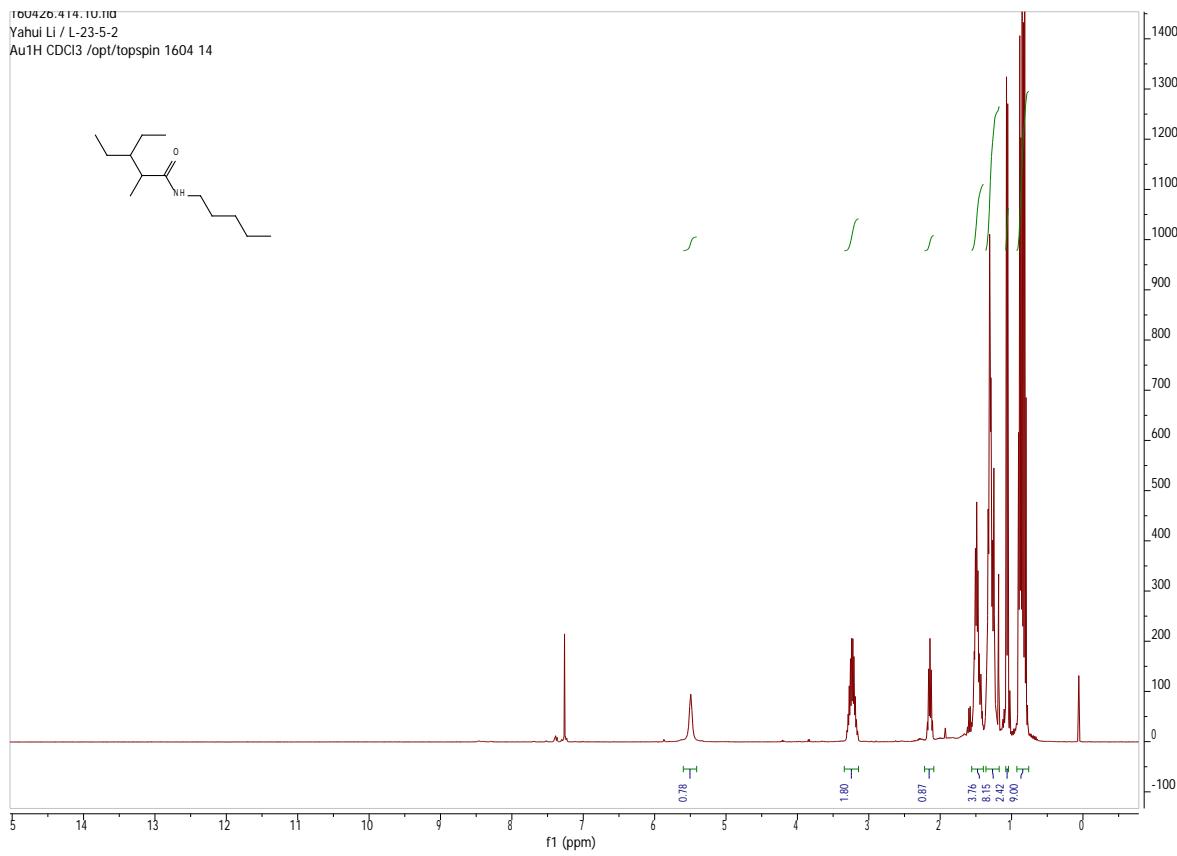
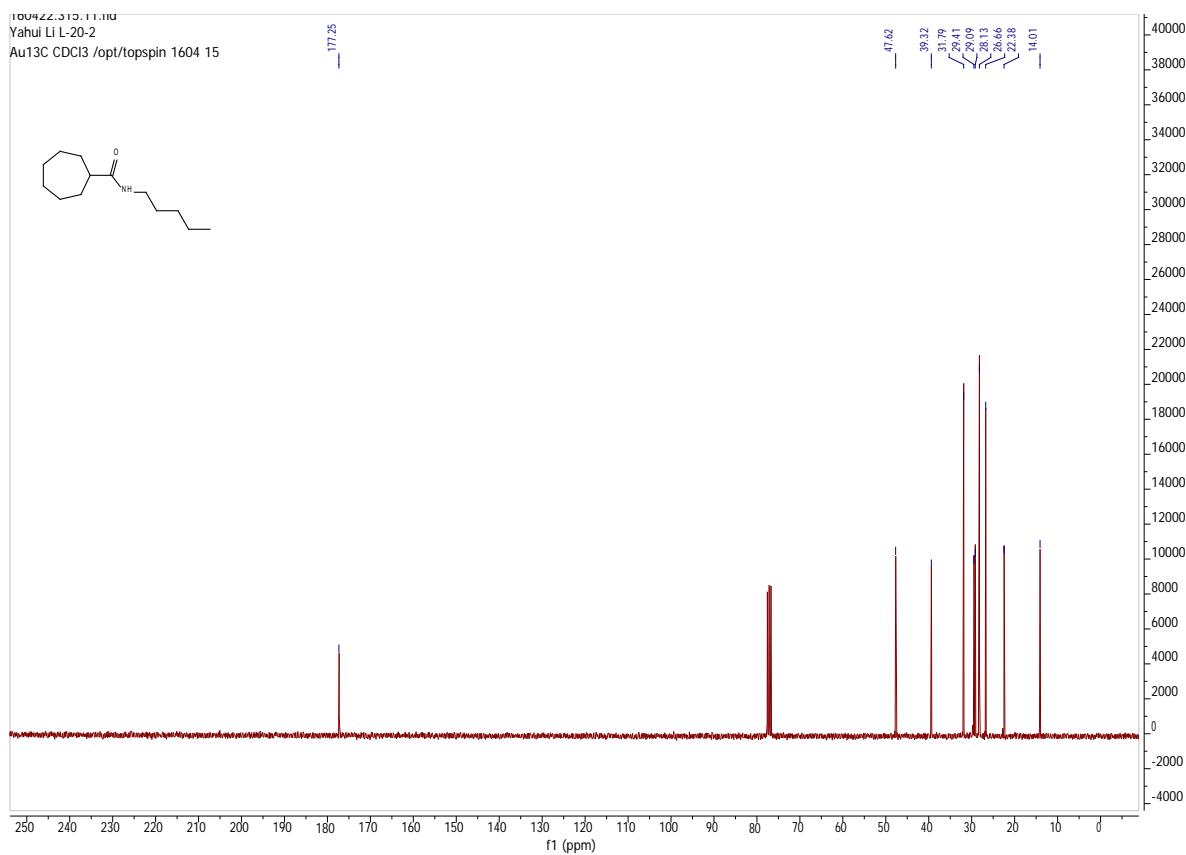


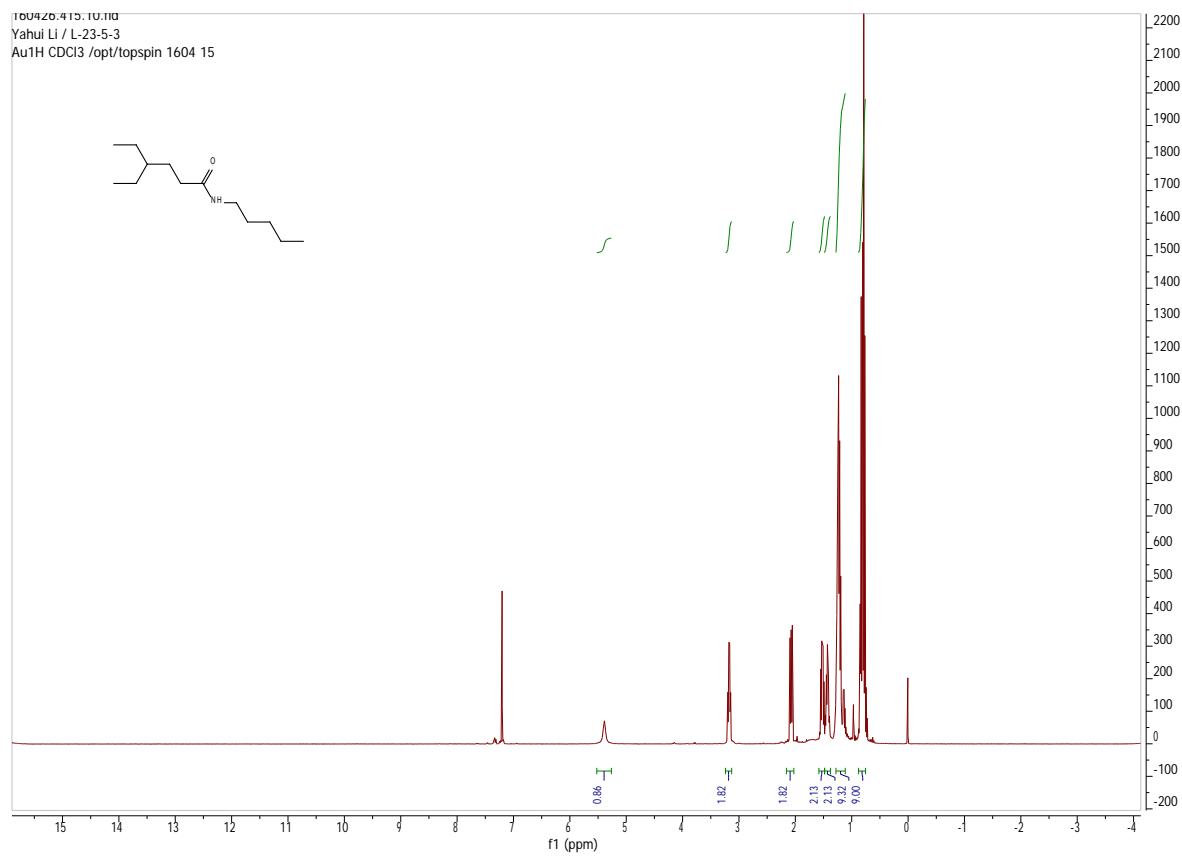
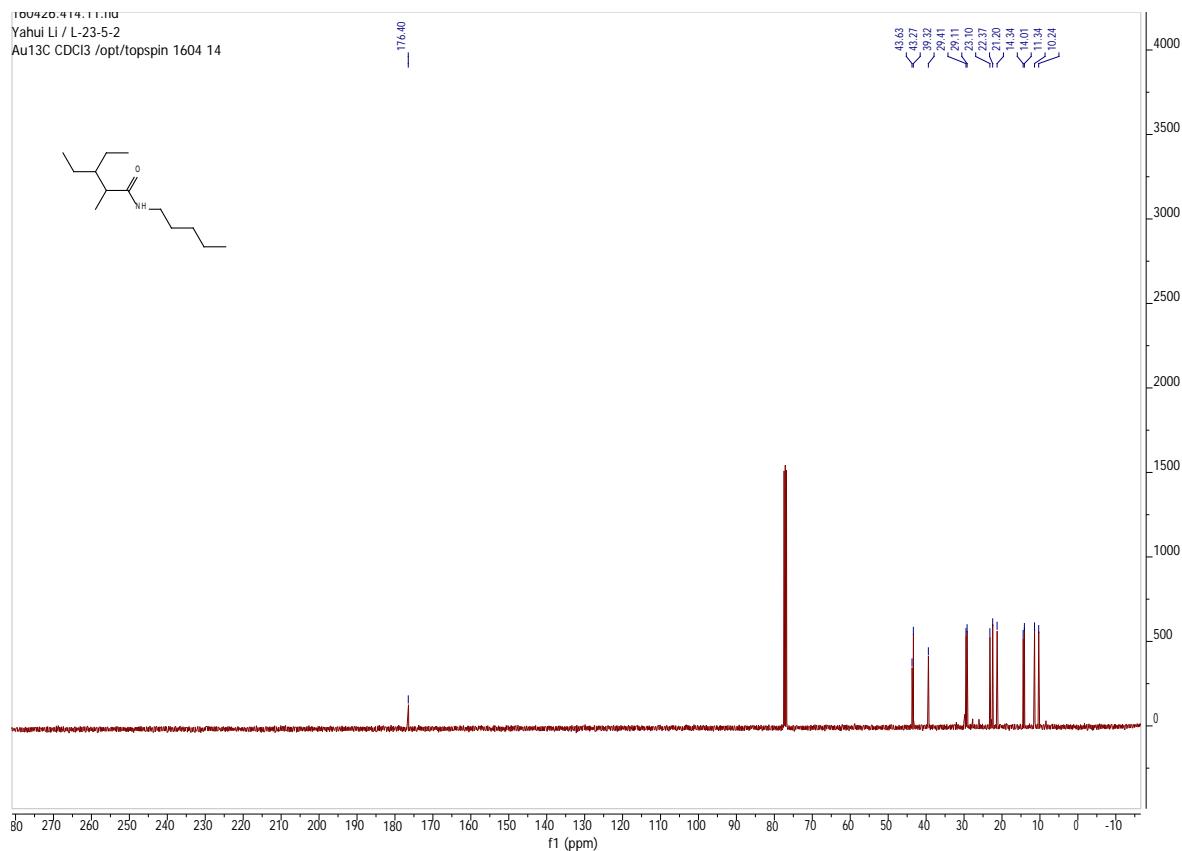


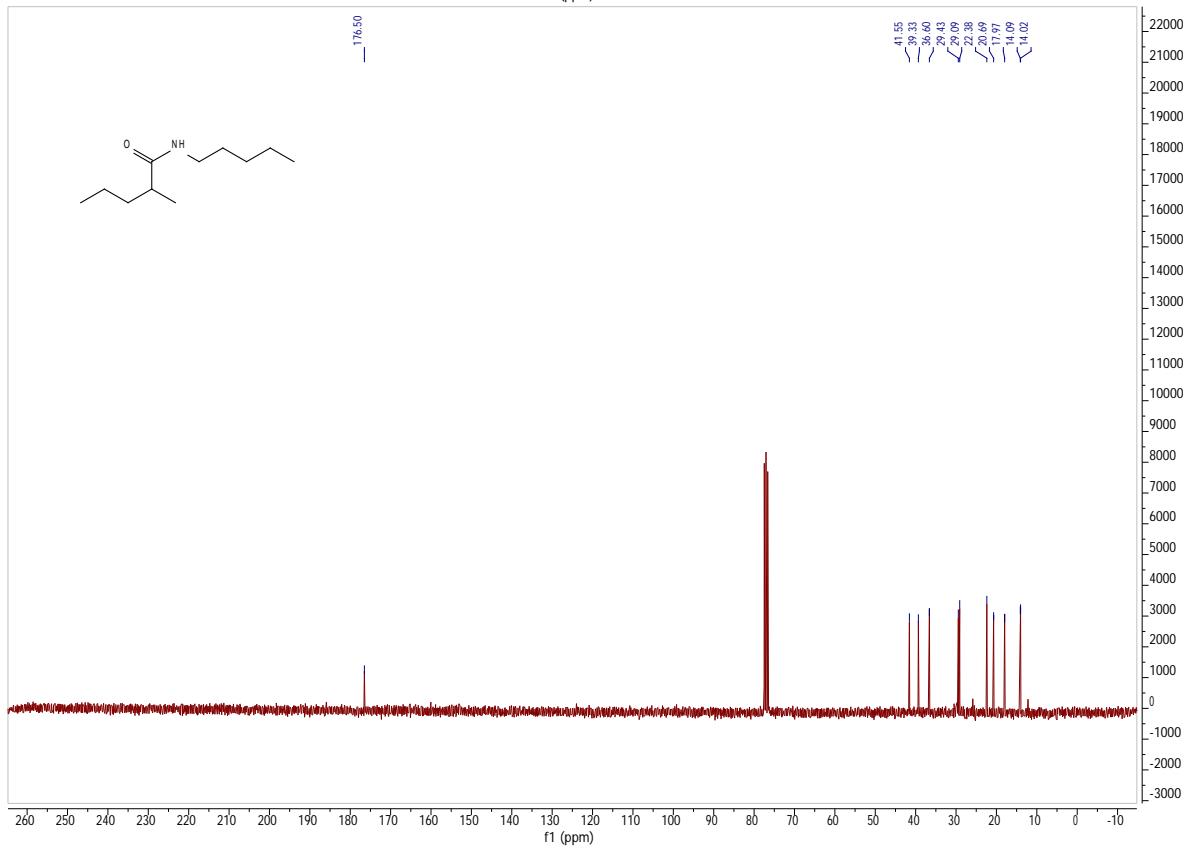
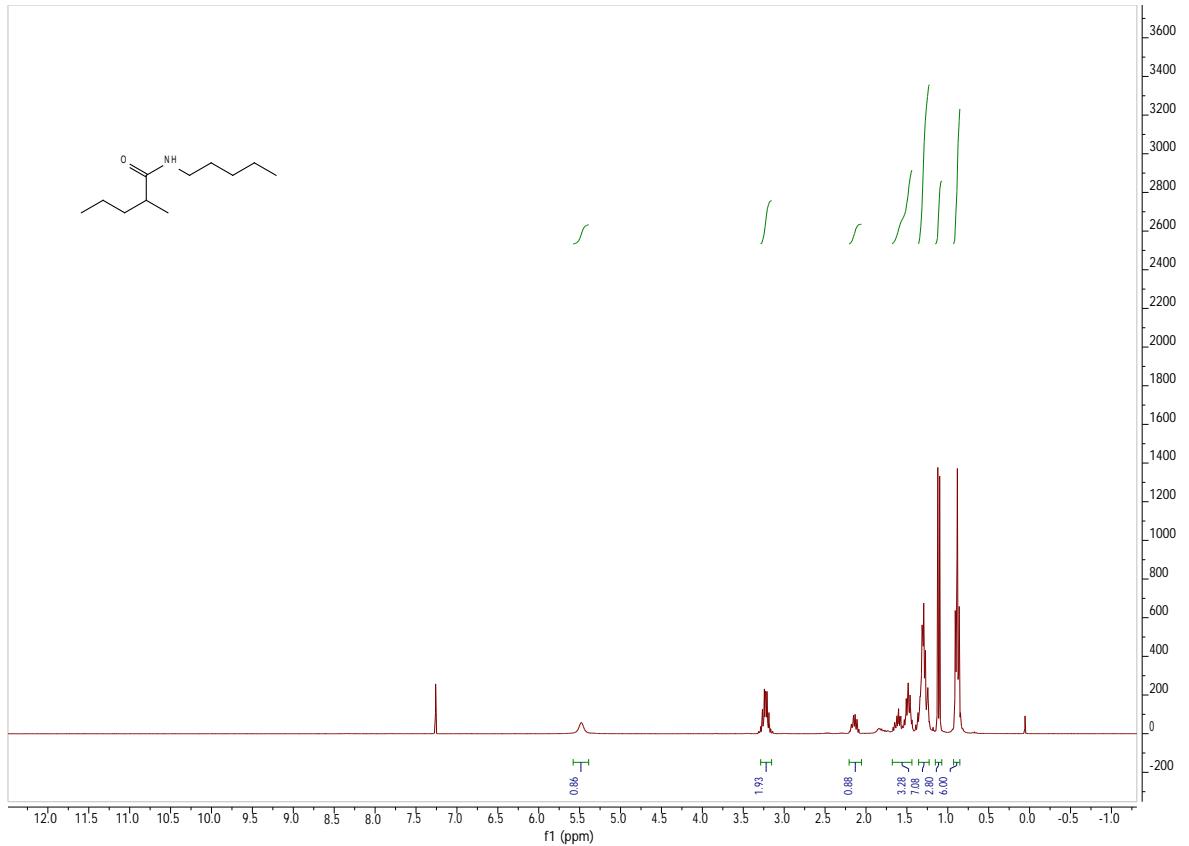


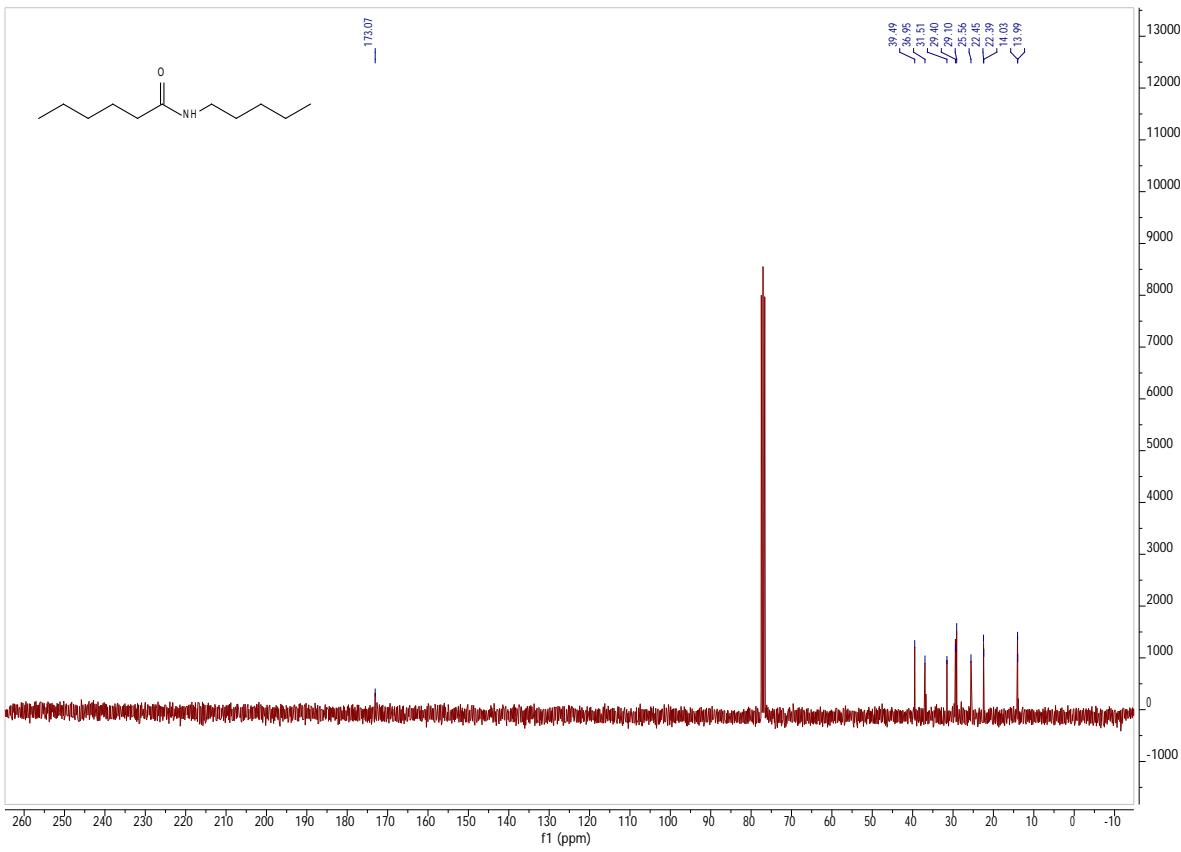
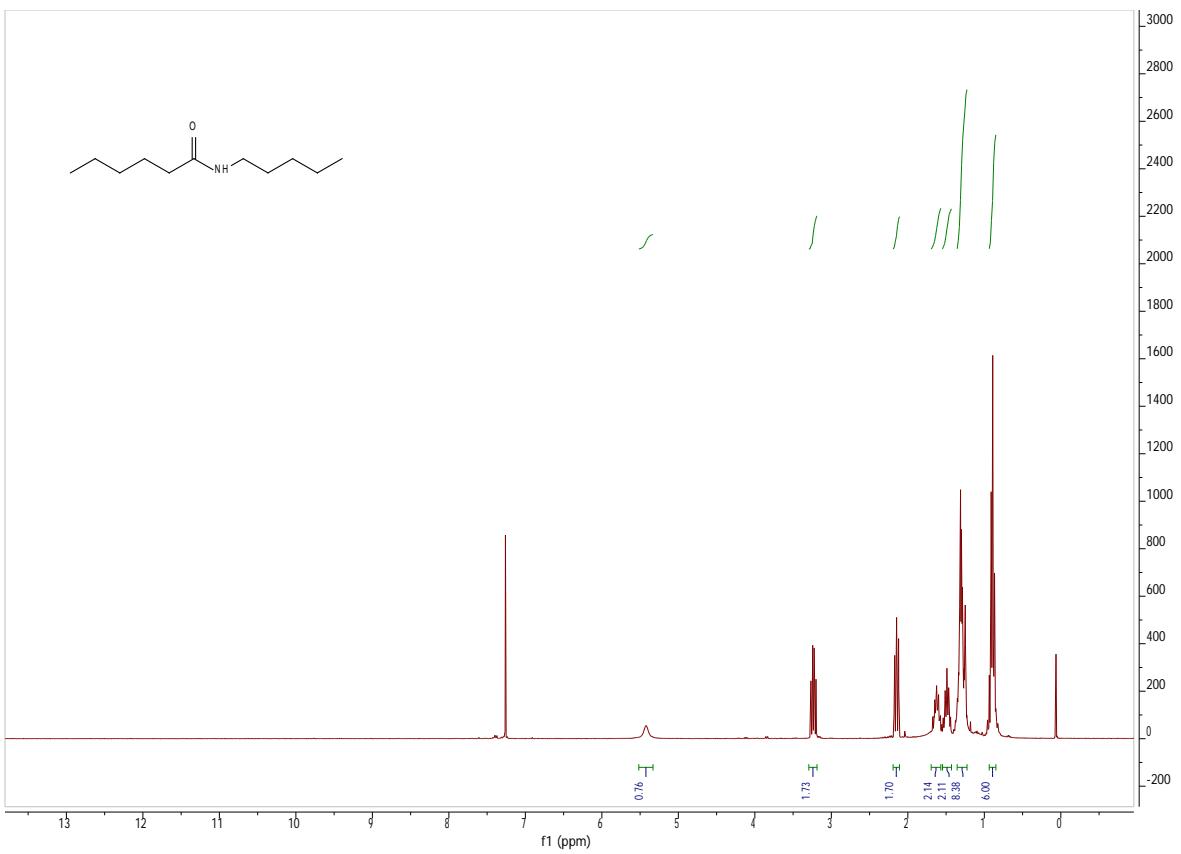


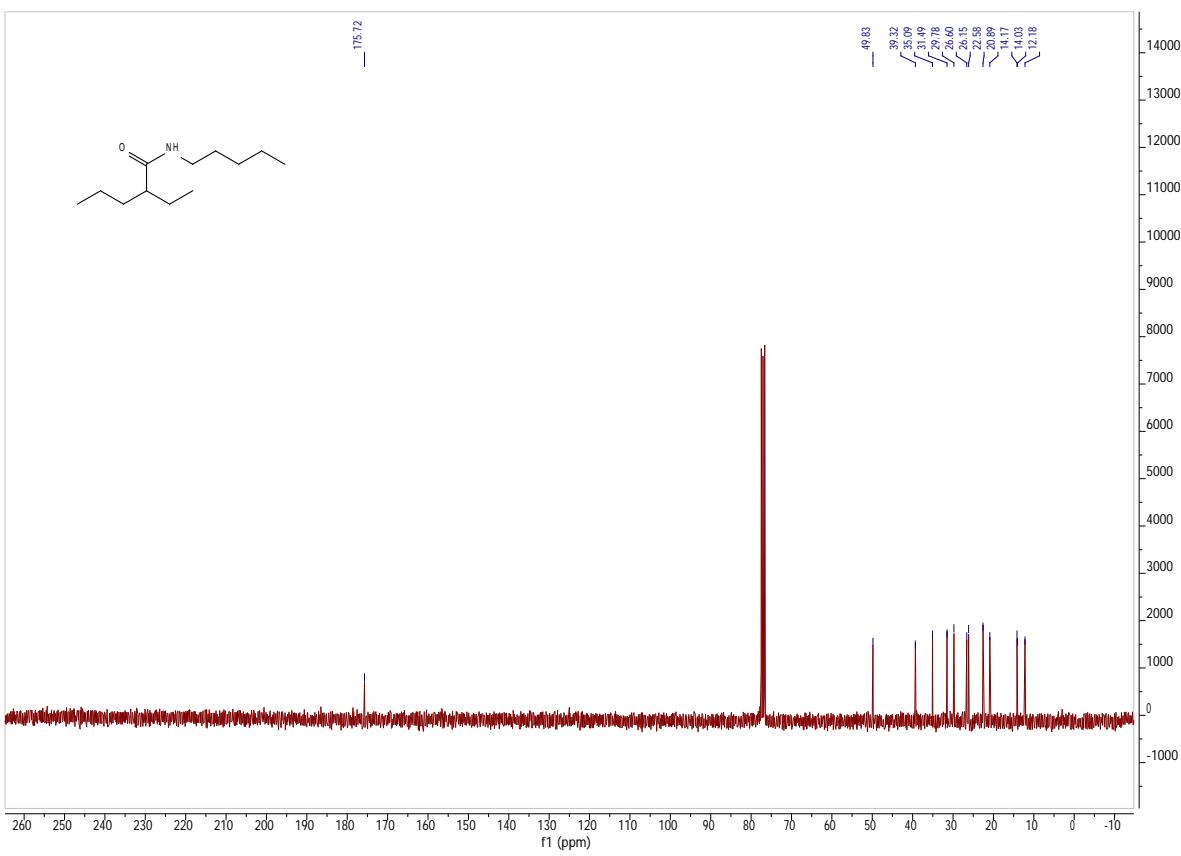
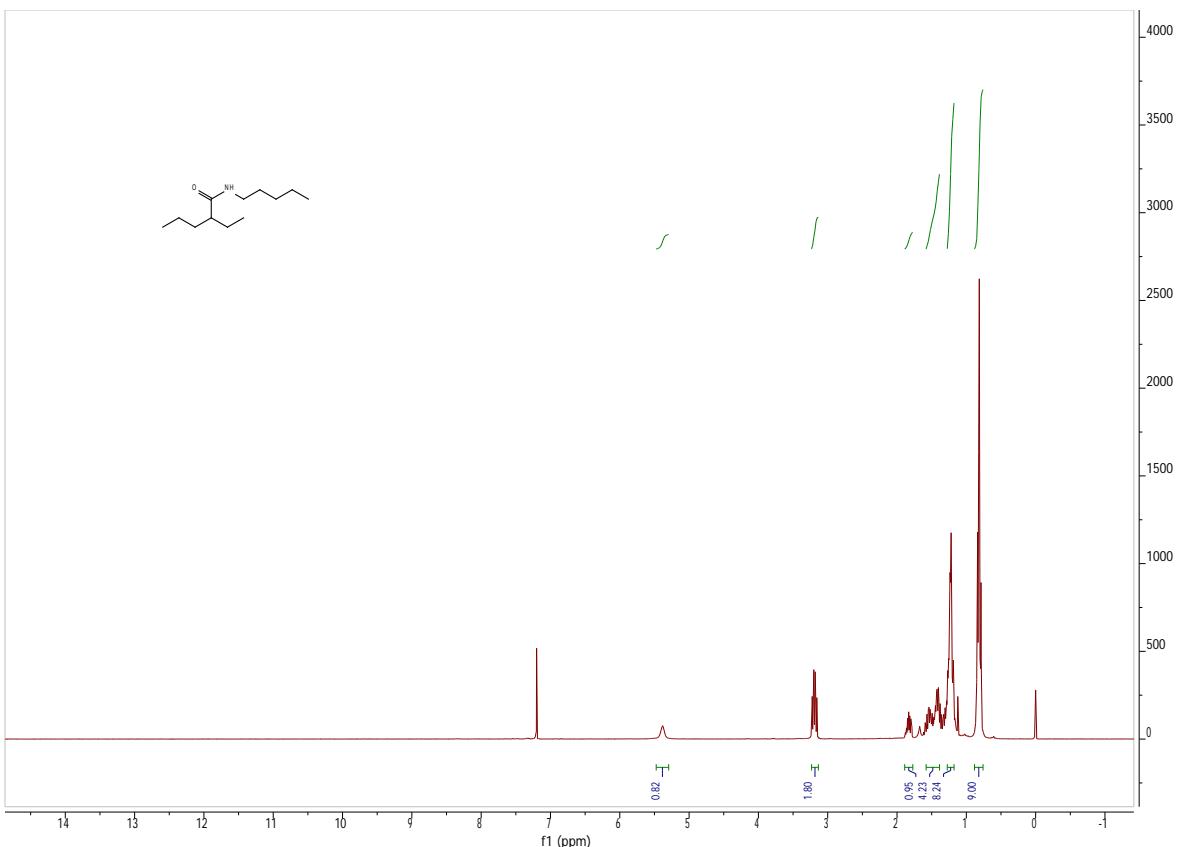


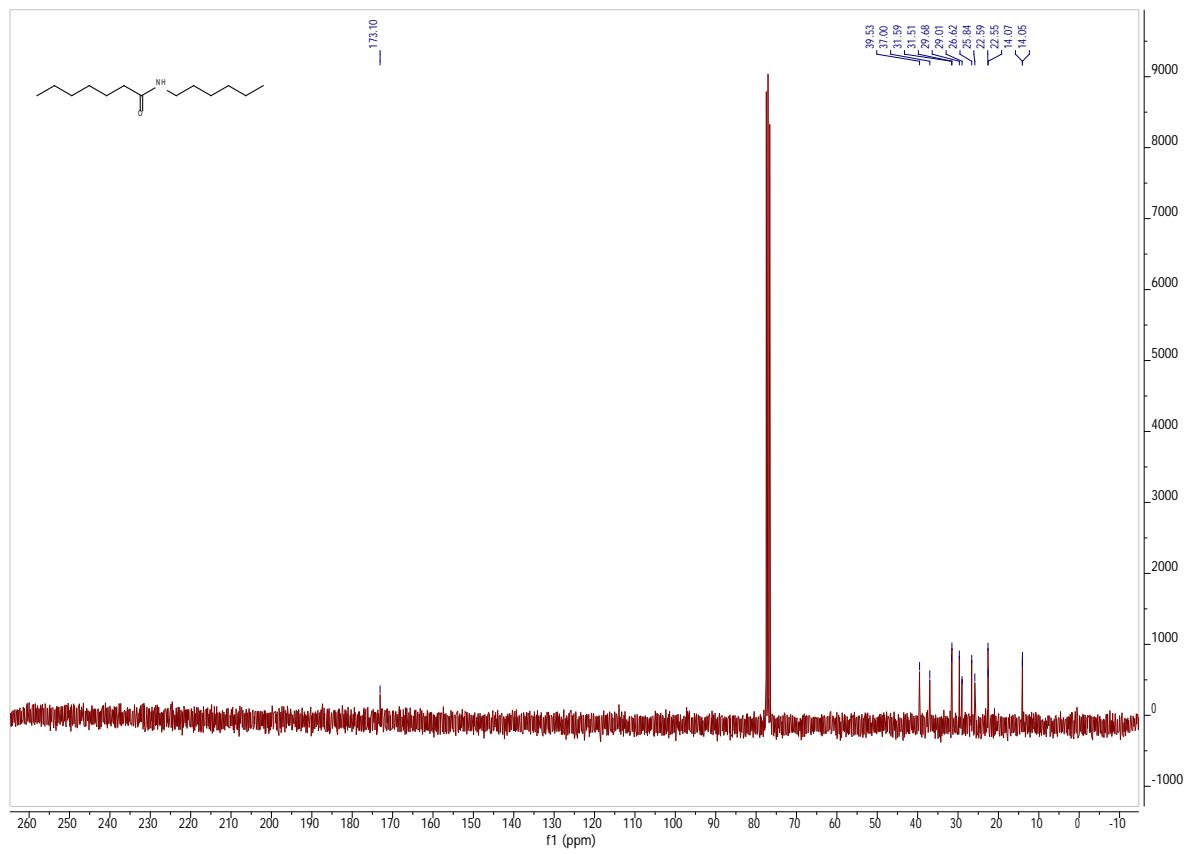
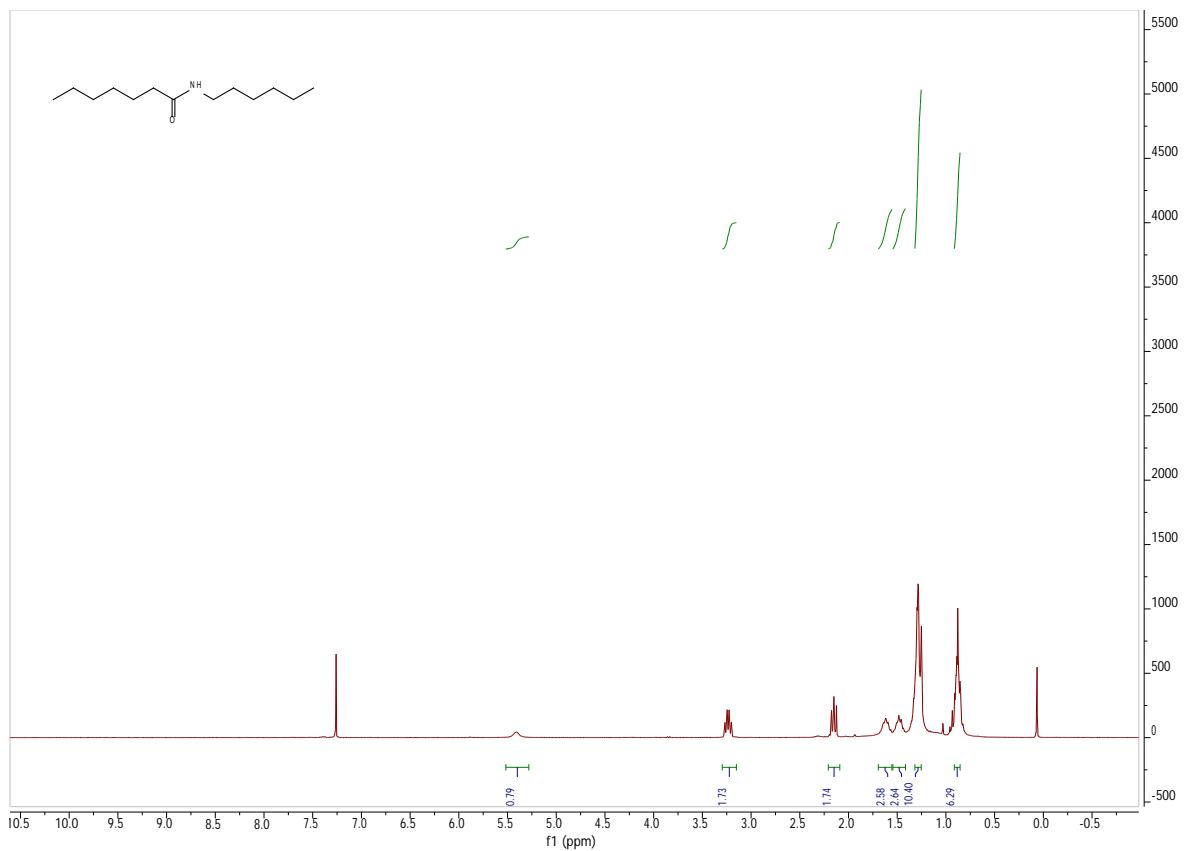


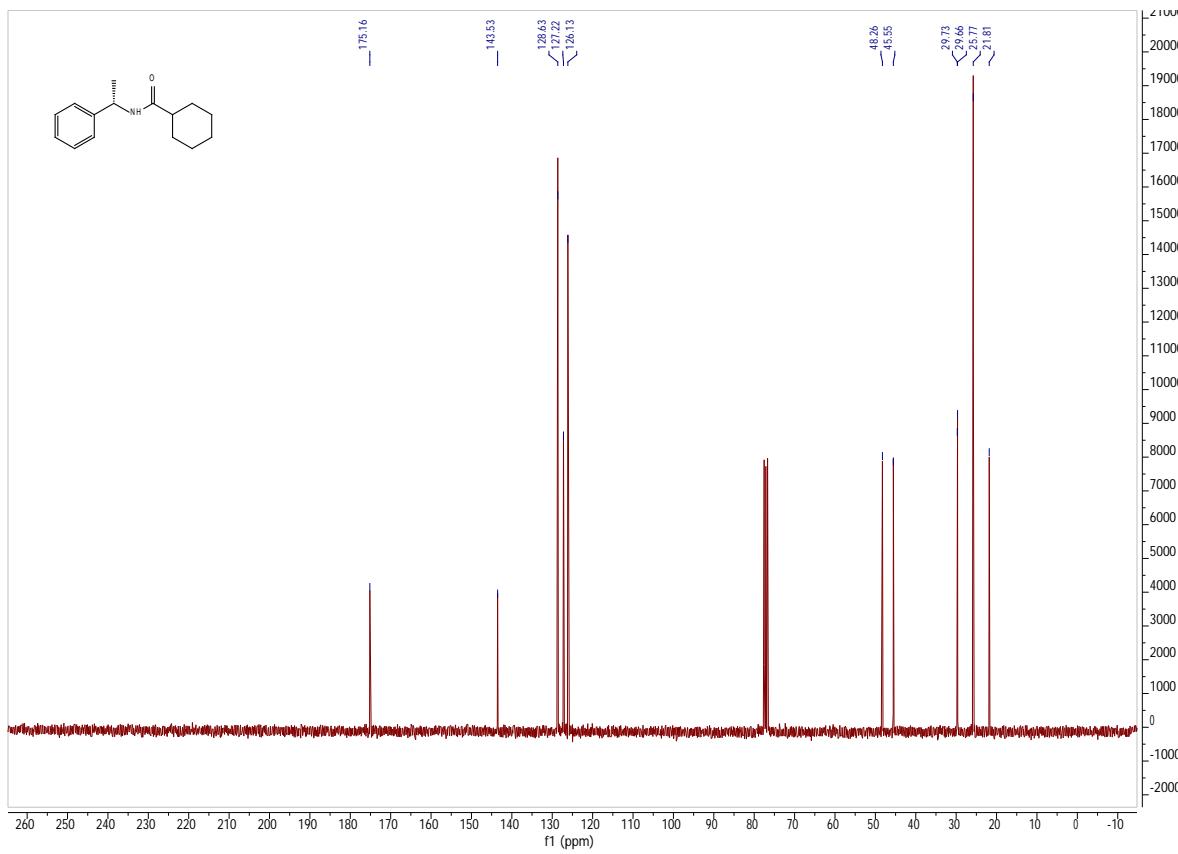
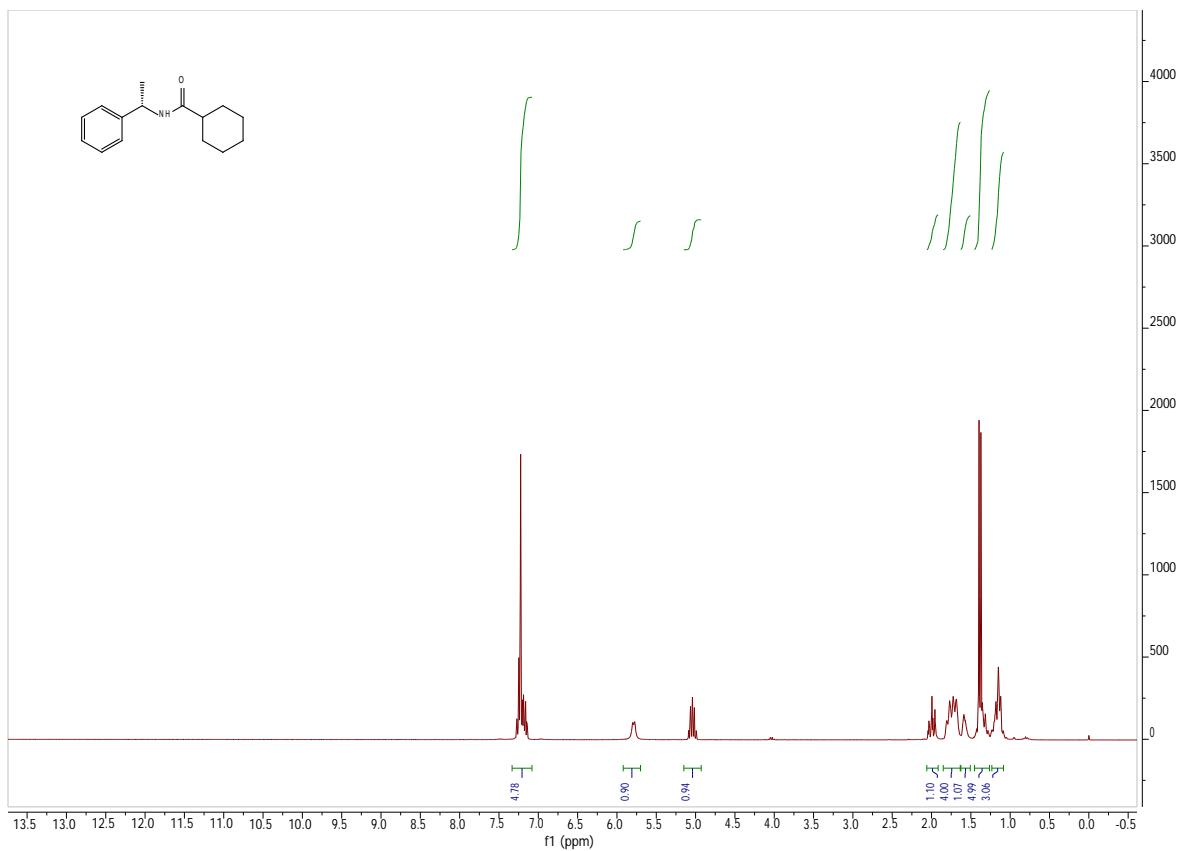






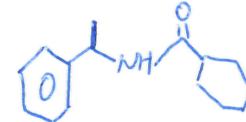




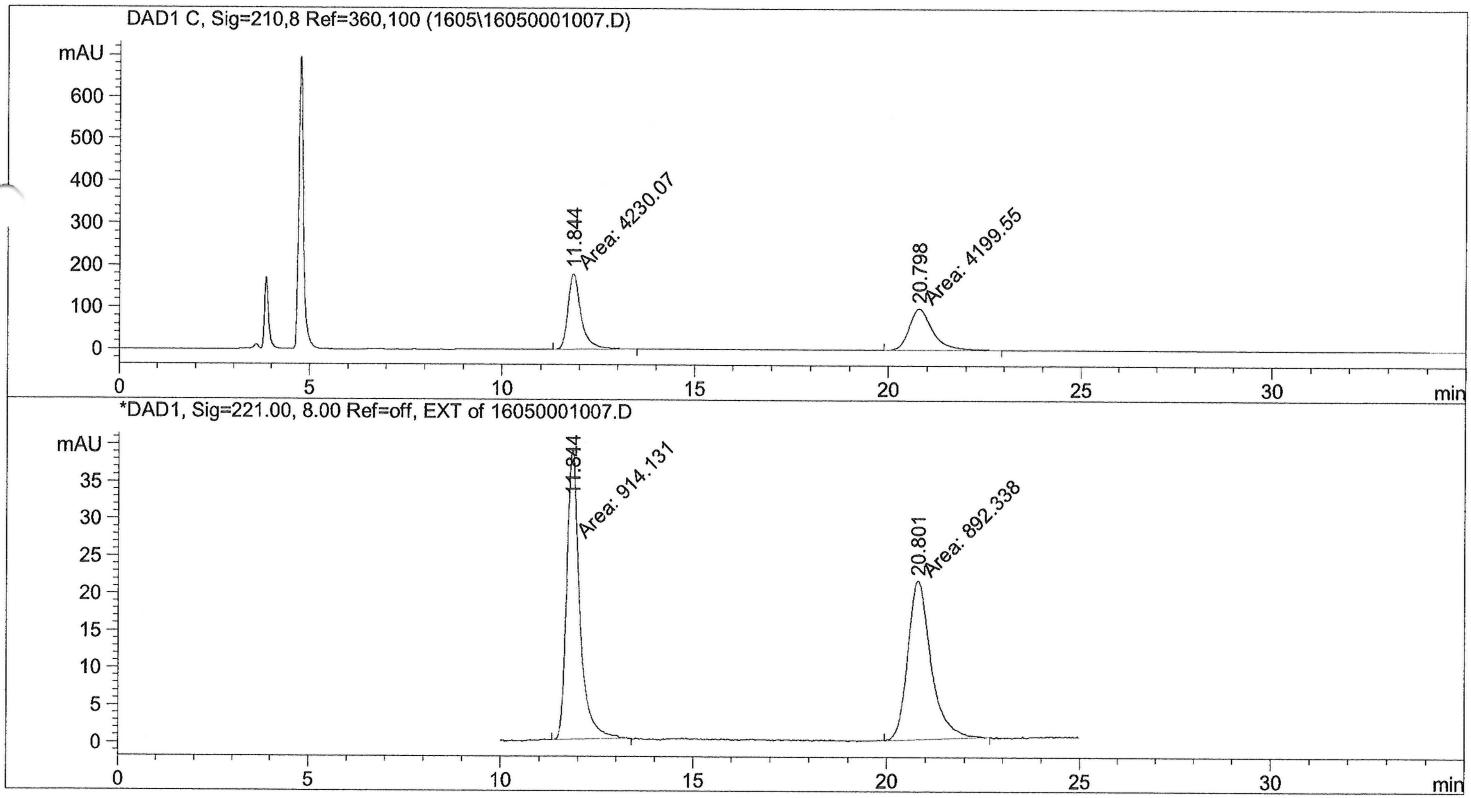


Sample Name: L37-1

```
=====
Acq. Operator   :                               Seq. Line : 5
Acq. Instrument : LC 2                      Location  : Vial 12
Injection Date  : 5/10/2016 1:50:33 PM        Inj       : 1
                                                Inj Volume : 0.1 µl
Acq. Method     : C:\CHEM32\2\METHODS\FISCHER.M
Last changed    : 5/10/2016 2:31:42 PM
                  (modified after loading)
Analysis Method : C:\CHEM32\2\METHODS\FISCHER.M
Last changed    : 5/10/2016 3:15:38 PM
                  (modified after loading)
Method Info     : Cellulose2, Heptan/EtOH 95:5, Fluß: 0.5ml/min
```



Additional Info : Peak(s) manually integrated



## ===== Area Percent Report =====

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

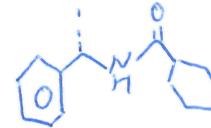
Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.844	MM	0.3913	4230.07227	180.15289	50.1811
2	20.798	MM	0.7070	4199.54639	99.00576	49.8189

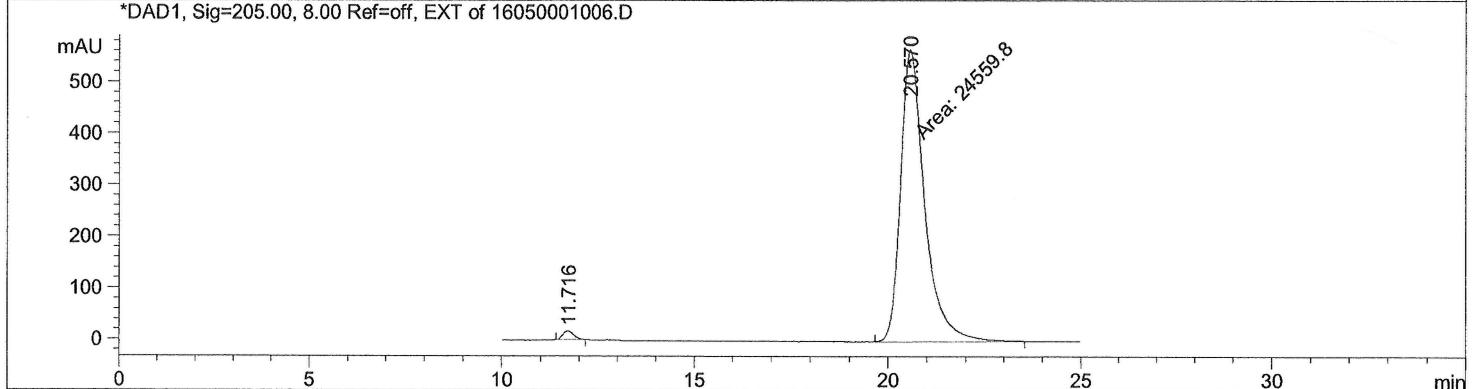
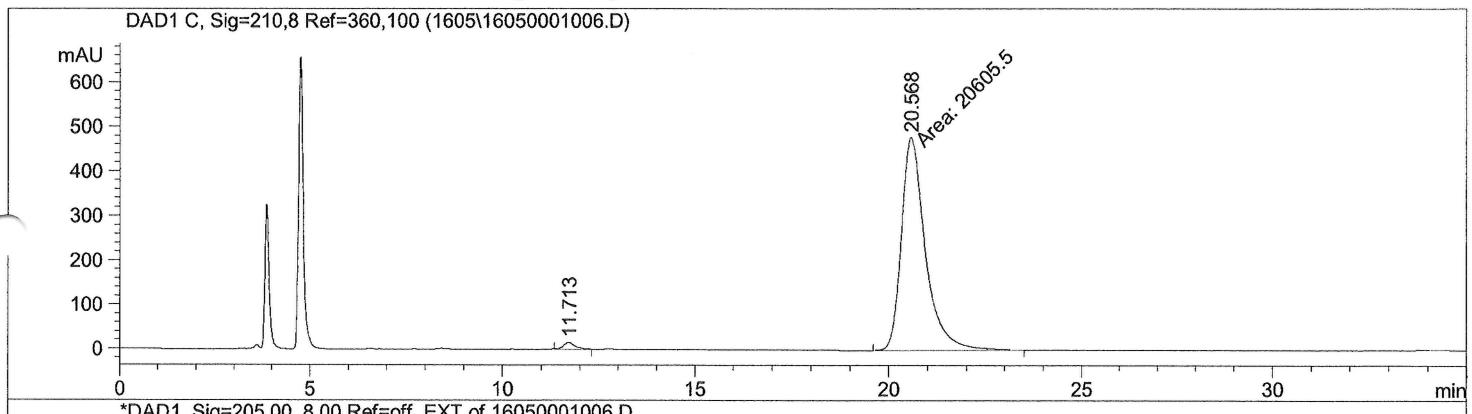
Totals : 8429.61865 279.15865

Sample Name: L18-1

```
=====
Acq. Operator   :                               Seq. Line :    4
Acq. Instrument : LC 2                      Location : Vial 11
Injection Date  : 5/10/2016 1:04:27 PM        Inj :    1
                                                Inj Volume : 0.1 µl
Acq. Method     : C:\CHEM32\2\METHODS\FISCHER.M
Last changed    : 5/10/2016 10:45:17 AM
Analysis Method : C:\CHEM32\2\METHODS\FISCHER.M
Last changed    : 5/10/2016 3:15:38 PM
                    (modified after loading)
Method Info     : Cellulose2, Heptan/EtOH 95:5, Fluß: 0.5ml/min
```



Additional Info : Peak(s) manually integrated



```
=====
Area Percent Report
=====
```

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.713	BB	0.3287	327.52512	15.12213	1.5646
2	20.568	MM	0.7186	2.06055e4	477.88281	98.4354

Totals : 2.09330e4 493.00494