

## Supporting Information

### One Pot Tandem Dual C=C and C=O Bond Reductions in $\beta$ -Alkylation of Secondary Alcohols with Primary Alcohols by Ruthenium Complexes of Amido and Picolyl Functionalized N-Heterocyclic Carbenes

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### Synthesis of 1-mesityl imidazole<sup>1</sup>

To a mixture of mesitylamine (4.46 g, 33.0 mmol), 38 % aq. glyoxal (10.1 g, 66.0 mmol) and 35 % aq. formaldehyde (5.66 g, 66.0 mmol) in MeOH (*ca.* 60 mL) CH<sub>3</sub>CO<sub>2</sub>NH<sub>4</sub> (5.08 g, 66.0 mmol) was added. The reaction mixture was refluxed for 12 hours during which the initially formed yellow solid becomes black colored solution. The reaction mixture was then cooled to room temperature and the volatiles were evaporated under reduced pressure. The residue thus obtained was dissolved in ethyl acetate and washed with 10 % aq. NaOH solution followed by brine solution. The combined organic layers were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated. The crude mass so obtained was finally purified by silica gel column chromatography using ethyl acetate/petroleum ether (1:4 v/v) mixed medium to give the product as a light brown solid (3.46 g, 56 %). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 25 °C): δ ppm, 7.43 (s, 1H, NCHN), 7.23 (s, 1H, NCHCHN), 6.97 (s, 2H, 2,4,6-(CH<sub>3</sub>)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>), 6.89 (s, 1H, NCHCHN), 2.34 (s, 3H, 2,4,6-(CH<sub>3</sub>)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>), 1.98 (s, 6H, 2,4,6-(CH<sub>3</sub>)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>). <sup>13</sup>C{<sup>1</sup>H} NMR (CDCl<sub>3</sub>, 125 MHz, 25 °C): δ ppm, 139.0 (NCHN), 137.6 (*ipso*-2,4,6-(CH<sub>3</sub>)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>), 135.5 (2,4,6-(CH<sub>3</sub>)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>), 133.5 (2,4,6-(CH<sub>3</sub>)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>), 129.6 (NCHCHN), 129.1 (2,4,6-(CH<sub>3</sub>)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>), 120.2 (NCHCHN), 21.1 (2,4,6-(CH<sub>3</sub>)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>), 17.4 (2,4,6-(CH<sub>3</sub>)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>).

### Synthesis of 2-chloro-N-(2,6-Me<sub>2</sub>-phenyl)acetamide<sup>2</sup>

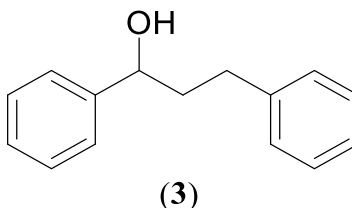
To a solution of 2,6-dimethylaniline (2.00 g, 16.5 mmol) in CHCl<sub>3</sub> (*ca.* 25 mL), 2-chloroacetyl chloride (3.73 g, 33.0 mmol) was added drop wise at 0 °C and the reaction mixture was stirred at room temperature for 8 hours. After the completion of reaction, the reaction mixture was washed with saturated NaHCO<sub>3</sub> solution (*ca.* 3 × 50 mL). The organic layer was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated. The residue so obtained was vacuum dried to give the product as a white solid (2.75 g, 84 %). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz, 25 °C): δ ppm,

7.86 (br, 1H, NH), 7.16-7.13 (m, 1H, 2,6-(CH<sub>3</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>), 7.11-7.09 (m, 2H, 2,6-(CH<sub>3</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>), 4.25 (s, 2H, CH<sub>2</sub>), 2.24 (s, 6H, 2,6-(CH<sub>3</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>). <sup>13</sup>C{<sup>1</sup>H} NMR (CDCl<sub>3</sub>, 125 MHz, 25 °C): δ ppm, 164.5 (CO), 135.5 (*o*-2,6-(CH<sub>3</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>), 132.8 (*ipso*-2,6-(CH<sub>3</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>), 128.5 (*m*-2,6-(CH<sub>3</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>), 128.1 (*p*-2,6-(CH<sub>3</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>), 42.9 (CH<sub>2</sub>), 18.5 (2,6-(CH<sub>3</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>).

### Synthesis of 1-(2,6-Me<sub>2</sub>-phenyl)imidazole<sup>3</sup>

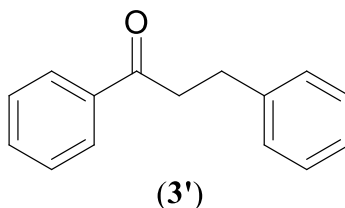
To a mixture of 2,6-dimethylaniline (4.00 g, 33.0 mmol), 38 % aq. glyoxal (10.1 g, 66.0 mmol) and 35 % aq. formaldehyde (5.66 g, 66.0 mmol) in MeOH (*ca.* 60 mL) CH<sub>3</sub>CO<sub>2</sub>NH<sub>4</sub> (5.08 g, 66.0 mmol) was added. The reaction mixture was refluxed for 12 hours during which the initially formed yellow solid becomes black colored solution. The reaction mixture was then cooled to room temperature and the volatiles were evaporated under reduced pressure. The residue thus obtained was dissolved in ethyl acetate and washed with 10 % aq. NaOH solution followed by brine solution. The combined organic layers were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated. The crude mass so obtained was finally purified by silica gel column chromatography using ethyl acetate/petroleum ether (1:4 *v/v*) mixed medium to give the product as a light brown solid (1.96 g, 34 %). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz, 25 °C): δ ppm, 7.46 (s, 1H, NCHN), 7.27-7.24 (m, 1H, 2,6-(CH<sub>3</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>), 7.25 (t, 1H, <sup>3</sup>*J*<sub>HH</sub> = 1 Hz, NCHCHN), 7.17-7.15 (m, 2H, 2,6-(CH<sub>3</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>), 6.92 (t, 1H, <sup>3</sup>*J*<sub>HH</sub> = 1 Hz, NCHCHN), 2.04 (s, 6H, 2,6-(CH<sub>3</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>). <sup>13</sup>C{<sup>1</sup>H} NMR (CDCl<sub>3</sub>, 125 MHz, 25 °C): δ ppm, 137.4 (NCHN), 136.1 (*ipso*-2,6-(CH<sub>3</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>), 135.9 (2,6-(CH<sub>3</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>), 129.8 (2,6-(CH<sub>3</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>), 129.1 (NCHCHN), 128.5 (2,6-(CH<sub>3</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>), 120.0 (NCHCHN), 17.6 (2,6-(CH<sub>3</sub>)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>).

### 1,3-Diphenylpropan-1-ol (3)<sup>4</sup>



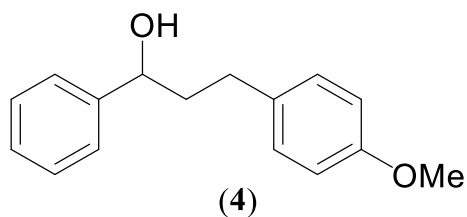
Colorless liquid [0.153 g, 72 % isolated yield (**1c**); 0.144 g, 68 % isolated yield (**2c**)] <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 25 °C): δ 7.37–7.36 (m, 4H, C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 7.32–7.28 (m, 3H, C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 7.22–7.19 (m, 3H, C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 4.71–4.68 (m, 1H, C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 2.81–2.65 (m, 2H, C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 2.20–2.00 (m, 2H, C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 1.88 (b, 1H, C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>). <sup>13</sup>C {<sup>1</sup>H} NMR (CDCl<sub>3</sub>, 100 MHz, 25 °C): δ 144.7 (*ipso*-C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 141.9 (C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>-*ipso*-C<sub>6</sub>H<sub>5</sub>), 128.7 (*o*-C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 128.6 (C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>-*o*-C<sub>6</sub>H<sub>5</sub>), 128.5 (*m*-C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 127.8 (*p*-C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 126.1 (C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>-*m*-C<sub>6</sub>H<sub>5</sub>), 126.0 (C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>-*p*-C<sub>6</sub>H<sub>5</sub>), 74.0 (C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 40.6 (C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 32.2 (C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>). GCMS (ESI): [M]<sup>+</sup> *m/z* = 212. Anal. Calcd. for C<sub>15</sub>H<sub>16</sub>O: C, 84.87; H, 7.60; Found: C, 84.17; H, 7.43.

### 1,3-Diphenylpropan-1-one (3')<sup>4</sup>



Colorless liquid [0.023 g, 11 % isolated yield (**1c**); 0.027 g, 13 % isolated yield (**2c**)]  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz, 25 °C):  $\delta$  8.01 (dd, 2H,  $^3J_{\text{H-H}} = 8$  Hz,  $^4J_{\text{H-H}} = 1$  Hz,  $\text{C}_6\text{H}_5\text{COCH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 7.60 (dt, 1H,  $^3J_{\text{H-H}} = 8$  Hz,  $^4J_{\text{H-H}} = 1$  Hz,  $\text{C}_6\text{H}_5\text{COCH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 7.50 (t, 2H,  $^3J_{\text{H-H}} = 8$  Hz,  $\text{C}_6\text{H}_5\text{COCH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 7.37-7.34 (m, 2H,  $\text{C}_6\text{H}_5\text{COCH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 7.32-7.31 (m, 2H,  $\text{C}_6\text{H}_5\text{COCH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 7.26 (t, 1H,  $^3J_{\text{H-H}} = 7$  Hz,  $\text{C}_6\text{H}_5\text{COCH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 3.35 (t, 2H,  $^3J_{\text{H-H}} = 8$  Hz,  $\text{C}_6\text{H}_5\text{COCH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 3.13 (t, 2H,  $^3J_{\text{H-H}} = 8$  Hz,  $\text{C}_6\text{H}_5\text{COCH}_2\text{CH}_2\text{C}_6\text{H}_5$ ).  $^{13}\text{C}\{^1\text{H}\}$  NMR ( $\text{CDCl}_3$ , 125 MHz, 25 °C):  $\delta$  199.3 ( $\text{C}_6\text{H}_5\text{COCH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 141.4 (*ipso*- $\text{C}_6\text{H}_5\text{COCH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 137.0 ( $\text{C}_6\text{H}_5\text{COCH}_2\text{CH}_2$ -*ipso*- $\text{C}_6\text{H}_5$ ), 133.2 (*p*- $\text{C}_6\text{H}_5\text{COCH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 128.7 (*o*- $\text{C}_6\text{H}_5\text{COCH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 128.6 (*m*- $\text{C}_6\text{H}_5\text{COCH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 128.5 ( $\text{C}_6\text{H}_5\text{COCH}_2\text{CH}_2$ -*m*- $\text{C}_6\text{H}_5$ ), 128.1 ( $\text{C}_6\text{H}_5\text{COCH}_2\text{CH}_2$ -*o*- $\text{C}_6\text{H}_5$ ), 126.2 ( $\text{C}_6\text{H}_5\text{COCH}_2\text{CH}_2$ -*p*- $\text{C}_6\text{H}_5$ ), 40.5 ( $\text{C}_6\text{H}_5\text{COCH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 30.2 ( $\text{C}_6\text{H}_5\text{COCH}_2\text{CH}_2\text{C}_6\text{H}_5$ ). GCMS (ESI):  $[\text{M}]^+ m/z = 210$ .

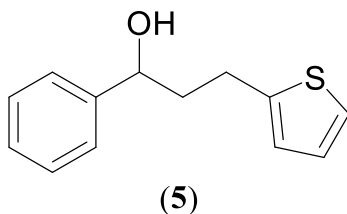
### 3-(4-Methoxyphenyl)-1-phenylpropan-1-ol (**4**)<sup>4</sup>



Colorless liquid [0.215 g, 89 % isolated yield (**1c**); 0.208 g, 86 % isolated yield (**2c**)]  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz, 25 °C):  $\delta$  7.36–7.35 (m, 4H,  $\text{C}_6\text{H}_5\text{CH}(\text{OH})\text{CH}_2\text{CH}_2$ -4-OCH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>), 7.31–7.28 (m, 1H,  $\text{C}_6\text{H}_5\text{CH}(\text{OH})\text{CH}_2\text{CH}_2$ -4-OCH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>), 7.12 (d, 2H,  $^3J_{\text{H-H}} = 9$  Hz,  $\text{C}_6\text{H}_5\text{CH}(\text{OH})\text{CH}_2\text{CH}_2$ -4-OCH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>), 6.84 (d, 2H,  $^3J_{\text{H-H}} = 8$  Hz,  $\text{C}_6\text{H}_5\text{CH}(\text{OH})\text{CH}_2\text{CH}_2$ -4-OCH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>), 4.69–4.66 (m, 1H,  $\text{C}_6\text{H}_5\text{CH}(\text{OH})\text{CH}_2\text{CH}_2$ -4-OCH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>), 3.79 (s, 3H,  $\text{C}_6\text{H}_5\text{CH}(\text{OH})\text{CH}_2\text{CH}_2$ -4-OCH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>), 2.74–2.58 (m, 2H,  $\text{C}_6\text{H}_5\text{CH}(\text{OH})\text{CH}_2\text{CH}_2$ -4-OCH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>), 2.16–1.97 (m, 2H,  $\text{C}_6\text{H}_5\text{CH}(\text{OH})\text{CH}_2\text{CH}_2$ -4-OCH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>), 1.96 (b, 1H,

$C_6H_5CH(OH)CH_2CH_2-4-OCH_3-C_6H_4$ ).  $^{13}C\{^1H\}$  NMR ( $CDCl_3$ , 100 MHz, 25 °C):  $\delta$  157.9 ( $C_6H_5CH(OH)CH_2CH_2-4-OCH_3-p-C_6H_4$ ), 144.8 (*ipso*- $C_6H_5CH(OH)CH_2CH_2-4-OCH_3-C_6H_4$ ), 134.0 ( $C_6H_5CH(OH)CH_2CH_2-4-OCH_3-ipso-C_6H_4$ ), 129.5 ( $C_6H_5CH(OH)CH_2CH_2-4-OCH_3-o-C_6H_4$ ), 128.7 (*m*- $C_6H_5CH(OH)CH_2CH_2-4-OCH_3-C_6H_4$ ), 127.8 (*p*- $C_6H_5CH(OH)CH_2CH_2-4-OCH_3-C_6H_4$ ), 126.1 (*o*- $C_6H_5CH(OH)CH_2CH_2-4-OCH_3-C_6H_4$ ), 114.0 ( $C_6H_5CH(OH)CH_2CH_2-4-OCH_3-o-C_6H_4$ ), 74.0 ( $C_6H_5CH(OH)CH_2CH_2-4-OCH_3-C_6H_4$ ), 55.4 ( $C_6H_5CH(OH)CH_2CH_2-4-OCH_3-C_6H_4$ ), 40.8 ( $C_6H_5CH(OH)CH_2CH_2-4-OCH_3-C_6H_4$ ), 31.3 ( $C_6H_5CH(OH)CH_2CH_2-4-OCH_3-C_6H_4$ ). GCMS (ESI):  $[M]^+$   $m/z = 242$ . Anal. Calcd. for  $C_{16}H_{18}O_2$ : C, 79.31; H, 7.49; Found: C, 78.97; H, 7.02.

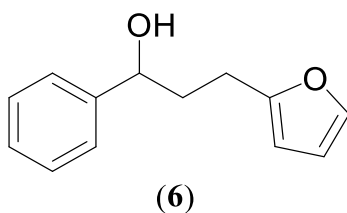
### 1-Phenyl-3-(thiophen-2-yl)propan-1-ol (**5**)<sup>4</sup>



Colorless liquid [0.161 g, 74 % isolated yield (**1c**); 0.137 g, 63 % isolated yield (**2c**)]  $^1H$  NMR ( $CDCl_3$ , 500 MHz, 25 °C):  $\delta$  7.40–7.39 (m, 4H,  $C_6H_5CH(OH)CH_2CH_2C_4H_3S$ ), 7.34–7.31 (m, 1H,  $C_6H_5CH(OH)CH_2CH_2C_4H_3S$ ), 7.15 (dd, 1H,  $^3J_{H-H} = 5$  Hz,  $^4J_{H-H} = 1$  Hz,  $C_6H_5CH(OH)CH_2CH_2C_4H_3S$ ), 6.95 (dd, 1H,  $^3J_{H-H} = 5$  Hz,  $^4J_{H-H} = 2$  Hz,  $C_6H_5CH(OH)CH_2CH_2C_4H_3S$ ), 6.84–6.83 (m, 1H,  $C_6H_5CH(OH)CH_2CH_2C_4H_3S$ ), 4.78–4.76 (m, 1H,  $C_6H_5CH(OH)CH_2CH_2C_4H_3S$ ), 2.98–2.93 (m, 2H,  $C_6H_5CH(OH)CH_2CH_2C_4H_3S$ ), 2.26–2.18 (m, 1H,  $C_6H_5CH(OH)CH_2CH_2C_4H_3S$ ), 2.15–2.08 (m, 1H,  $C_6H_5CH(OH)CH_2CH_2C_4H_3S$ ), 1.66 (b, 1H,  $C_6H_5CH(OH)CH_2CH_2C_4H_3S$ ).  $^{13}C\{^1H\}$  NMR ( $CDCl_3$ , 125 MHz, 25 °C):  $\delta$  144.8 (*ipso*- $C_6H_5CH(OH)CH_2CH_2C_4H_3S$ ), 144.5 ( $C_6H_5CH(OH)CH_2CH_2-ipso-C_4H_3S$ ), 128.8 (*m*-

$\underline{C}_6H_5CH(OH)CH_2CH_2C_4H_3S$ ), 127.9 ( $C_6H_5CH(OH)CH_2CH_2\underline{C}_4H_3S$ ), 126.9  
 $(C_6H_5CH(OH)CH_2CH_2\underline{C}_4H_3S)$ , 126.1 ( $o$ - $\underline{C}_6H_5CH(OH)CH_2CH_2C_4H_3S$ ), 124.5 ( $p$ -  
 $C_6H_5CH(OH)CH_2CH_2C_4H_3S$ ), 123.3 ( $C_6H_5CH(OH)CH_2CH_2\underline{C}_4H_3S$ ), 73.7  
 $(C_6H_5\underline{C}H(OH)CH_2CH_2C_4H_3S)$ , 40.9 ( $C_6H_5CH(OH)\underline{C}H_2CH_2C_4H_3S$ ), 26.4  
 $(C_6H_5CH(OH)CH_2\underline{C}H_2C_4H_3S)$ . GCMS (ESI):  $[M]^+$   $m/z$  = 218. Anal. Calcd. for  $C_{13}H_{14}OS$ : C, 71.52; H, 6.46; Found: C, 69.81; H, 8.02.

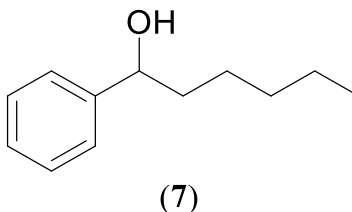
### 3-(Furan-2-yl)-1-phenylpropan-1-ol (**6**)<sup>4</sup>



Colorless liquid [0.138 g, 68 % isolated yield (**1c**); 0.141 g, 70 % isolated yield (**2c**)]  $^1H$  NMR  
 ( $CDCl_3$ , 500 MHz, 25 °C):  $\delta$  7.30–7.29 (m, 4H,  $C_6H_5CH(OH)CH_2CH_2C_4H_3O$ ), 7.24–7.23 (m,  
 1H,  $C_6H_5CH(OH)CH_2CH_2C_4H_3O$ ), 7.23–7.21 (m, 1H,  $C_6H_5CH(OH)CH_2CH_2C_4H_3O$ ), 6.21 (dd,  
 1H,  $^3J_{H-H} = 4$  Hz,  $^4J_{H-H} = 2$  Hz,  $C_6H_5CH(OH)CH_2CH_2C_4H_3O$ ), 5.94–5.93 (m, 1H,  
 $C_6H_5CH(OH)CH_2CH_2C_4H_3O$ ), 4.66–4.64 (m, 1H,  $C_6H_5CH(OH)CH_2CH_2C_4H_3O$ ), 2.70–2.63 (m,  
 2H,  $C_6H_5CH(OH)CH_2CH_2C_4H_3O$ ), 2.10–1.96 (m, 2H,  $C_6H_5CH(OH)CH_2CH_2C_4H_3O$ ), 1.82 (b,  
 1H,  $C_6H_5CH(OH)CH_2CH_2C_4H_3O$ ).  $^{13}C\{^1H\}$  NMR ( $CDCl_3$ , 125 MHz, 25 °C):  $\delta$  155.7  
 $(C_6H_5CH(OH)CH_2CH_2$ -*ipso*- $C_4H_3O)$ , 144.5 (*ipso*- $C_6H_5CH(OH)CH_2CH_2C_4H_3O$ ), 141.1  
 $(C_6H_5CH(OH)CH_2CH_2\underline{C}_4H_3O)$ , 128.7 ( $m$ - $\underline{C}_6H_5CH(OH)CH_2CH_2C_4H_3O$ ), 127.9 ( $p$ -  
 $\underline{C}_6H_5CH(OH)CH_2CH_2C_4H_3O$ ), 126.1 ( $o$ - $\underline{C}_6H_5CH(OH)CH_2CH_2C_4H_3O$ ), 110.3  
 $(C_6H_5CH(OH)CH_2CH_2\underline{C}_4H_3O)$ , 105.2 ( $C_6H_5CH(OH)CH_2CH_2\underline{C}_4H_3O$ ), 73.9  
 $(C_6H_5\underline{C}H(OH)CH_2CH_2C_4H_3O)$ , 37.3 ( $C_6H_5CH(OH)\underline{C}H_2CH_2C_4H_3O$ ), 24.6

(C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>4</sub>H<sub>9</sub>O). GCMS (ESI): [M]<sup>+</sup> *m/z* = 202. Anal. Calcd. for C<sub>13</sub>H<sub>14</sub>O<sub>2</sub>: C, 77.20; H, 6.98; Found: C, 77.71; H, 7.35.

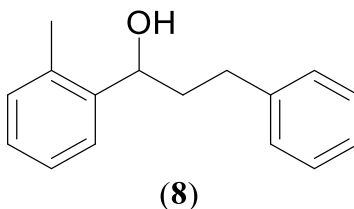
### 1-Phenylhexan-1-ol (7)<sup>4</sup>



Colorless liquid [0.126 g, 71 % isolated yield (**1c**); 0.123 g, 69 % isolated yield (**2c**)] <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz, 25 °C): δ 7.27–7.26 (m, 4H, C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 7.21–7.18 (m, 1H, C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 4.59–4.55 (m, 1H, C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 1.85 (b, 1H, C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 1.76–1.57 (m, 2H, C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 1.35–1.18 (m, 6H, C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>) 0.81–0.78 (m, 3H, C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>). <sup>13</sup>C{<sup>1</sup>H} NMR (CDCl<sub>3</sub>, 100 MHz, 25 °C): δ 145.1 (*ipso*-C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 128.6 (*m*-C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 127.6 (*p*-C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 126.1 (*o*-C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 74.9 (C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 39.2 (C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 31.9 (C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 25.7 (C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 22.7 (C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 14.2 (C<sub>6</sub>H<sub>5</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>). GCMS (ESI): [M]<sup>+</sup> *m/z* = 178. Anal. Calcd. for C<sub>12</sub>H<sub>18</sub>O: C, 80.85; H, 10.18; Found: C, 80.05; H, 10.07.

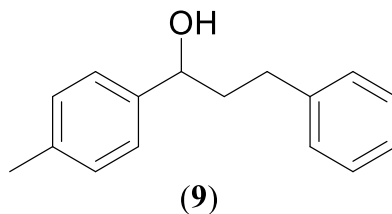
### 3-Phenyl-1-(*o*-tolyl)propan-1-ol (8)<sup>4</sup>





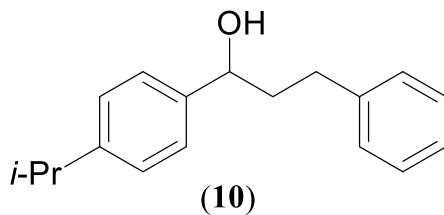
Colorless liquid [0.164 g, 73 % isolated yield (**1c**); 0.176 g, 78 % isolated yield (**2c**)]  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz, 25 °C):  $\delta$  7.53 (dd, 1H,  $^3J_{\text{HH}} = 8$  Hz,  $^4J_{\text{HH}} = 1$  Hz, 2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 7.33–7.30 (m, 2H, 2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 7.28–7.14 (m, 6H, 2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 4.97–4.95 (m, 1H, 2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 2.91–2.85 (m, 1H, 2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 2.80–2.74 (m, 1H, 2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 2.27 (s, 3H, 2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 2.14–1.99 (m, 2H, 2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 1.68 (b, 1H, 2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>).  $^{13}\text{C}\{^1\text{H}\}$  NMR ( $\text{CDCl}_3$ , 125 MHz, 25 °C):  $\delta$  142.9 (2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>-*ipso*-C<sub>6</sub>H<sub>5</sub>), 142.0 (2-CH<sub>3</sub>-*ipso*-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 134.6 (2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>-*p*-C<sub>6</sub>H<sub>5</sub>), 130.6 (2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>-*p*-C<sub>6</sub>H<sub>5</sub>), 128.6 (2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>-*m*-C<sub>6</sub>H<sub>5</sub>), 128.5 (2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>-*o*-C<sub>6</sub>H<sub>5</sub>), 127.4 (2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 126.5 (2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 126.1 (2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 125.3 (2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 70.1 (2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 39.6 (2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 32.5 (2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 19.1 (2-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>). GCMS (ESI):  $[\text{M}]^+$   $m/z$  = 226. Anal. Calcd. for C<sub>16</sub>H<sub>18</sub>O: C, 84.91; H, 8.02; Found: C, 84.72; H, 8.21.

### 3-Phenyl-1-(*p*-tolyl)propan-1-ol (**9**)<sup>4</sup>



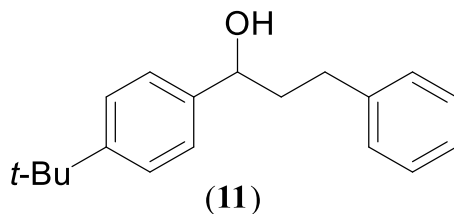
Colorless liquid [0.168 g, 74 % isolated yield (**1c**); 0.163 g, 72 % isolated yield (**2c**)]  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz, 25 °C):  $\delta$  7.32-7.27 (m, 4H, 4- $\text{CH}_3\text{-C}_6\text{H}_4\text{CH(OH)CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 7.23-7.19 (m, 5H, 4- $\text{CH}_3\text{-C}_6\text{H}_4\text{CH(OH)CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 4.70-4.67 (m, 1H, 4- $\text{CH}_3\text{-C}_6\text{H}_4\text{CH(OH)CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 2.80-2.66 (m, 2H, 4- $\text{CH}_3\text{-C}_6\text{H}_4\text{CH(OH)CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 2.38 (s, 3H, 4- $\text{CH}_3\text{-C}_6\text{H}_4\text{CH(OH)CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 2.20-2.13 (m, 1H, 4- $\text{CH}_3\text{-C}_6\text{H}_4\text{CH(OH)CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 2.09-2.01 (m, 1H, 4- $\text{CH}_3\text{-C}_6\text{H}_4\text{CH(OH)CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 1.63 (b, 1H, 4- $\text{CH}_3\text{-C}_6\text{H}_4\text{CH(OH)CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ).  $^{13}\text{C}\{^1\text{H}\}$  NMR ( $\text{CDCl}_3$ , 100 MHz, 25 °C):  $\delta$  142.0 (4- $\text{CH}_3\text{-C}_6\text{H}_4\text{CH(OH)CH}_2\text{CH}_2\text{-}i\text{pr}$ o- $\text{C}_6\text{H}_5$ ), 141.8 (4- $\text{CH}_3\text{-}i\text{pr}$ o- $\text{C}_6\text{H}_4\text{CH(OH)CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 137.5 (4- $\text{CH}_3\text{-C}_6\text{H}_4\text{CH(OH)CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 129.4 (4- $\text{CH}_3\text{-C}_6\text{H}_4\text{CH(OH)CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 128.6 (4- $\text{CH}_3\text{-C}_6\text{H}_4\text{CH(OH)CH}_2\text{CH}_2\text{-}m\text{-C}_6\text{H}_5$ ), 128.5 (4- $\text{CH}_3\text{-C}_6\text{H}_4\text{CH(OH)CH}_2\text{CH}_2\text{-}o\text{-C}_6\text{H}_5$ ), 126.1 (4- $\text{CH}_3\text{-C}_6\text{H}_4\text{CH(OH)CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 126.0 (4- $\text{CH}_3\text{-C}_6\text{H}_4\text{CH(OH)CH}_2\text{CH}_2\text{-}p\text{-C}_6\text{H}_5$ ), 73.9 (4- $\text{CH}_3\text{-C}_6\text{H}_4\text{CH(OH)CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 40.6 (4- $\text{CH}_3\text{-C}_6\text{H}_4\text{CH(OH)CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 32.3 (4- $\text{CH}_3\text{-C}_6\text{H}_4\text{CH(OH)CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 21.3 (4- $\text{CH}_3\text{-C}_6\text{H}_4\text{CH(OH)CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ). GCMS (ESI):  $[\text{M}]^+ m/z = 226$ . Anal. Calcd. for  $\text{C}_{16}\text{H}_{18}\text{O}$ : C, 84.91; H, 8.02; Found: C, 84.83; H, 7.83.

#### 1-(4-*iso*-Propylphenyl)-3-phenylpropan-1-ol (**10**)<sup>4</sup>



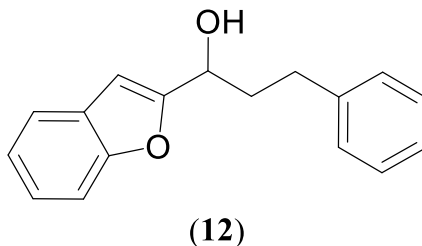
Colorless liquid [0.188 g, 74 % isolated yield (**1c**); 0.178 g, 70 % isolated yield (**2c**)]  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz, 25 °C):  $\delta$  7.30-7.26 (m, 4H, 4-(CH(CH<sub>3</sub>)<sub>2</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 7.23-7.19 (m, 5H, 4-(CH(CH<sub>3</sub>)<sub>2</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 4.68–4.65 (m, 1H, 4-(CH(CH<sub>3</sub>)<sub>2</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 2.92 (sept, 1H,  $^3J_{\text{HH}} = 7$  Hz, 4-(CH(CH<sub>3</sub>)<sub>2</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 2.81–2.64 (m, 2H, 4-(CH(CH<sub>3</sub>)<sub>2</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 2.18–1.99 (m, 2H, 4-(CH(CH<sub>3</sub>)<sub>2</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 1.74 (b, 1H, 4-(CH(CH<sub>3</sub>)<sub>2</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 1.26 (d, 6H,  $^3J_{\text{HH}} = 7$  Hz, 4-(CH(CH<sub>3</sub>)<sub>2</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>).  $^{13}\text{C}\{^1\text{H}\}$  NMR ( $\text{CDCl}_3$ , 100 MHz, 25 °C):  $\delta$  148.6 (4-(CH(CH<sub>3</sub>)<sub>2</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 142.1 (4-(CH(CH<sub>3</sub>)<sub>2</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>-*ipso*-C<sub>6</sub>H<sub>5</sub>), 142.0 (4-(CH(CH<sub>3</sub>)<sub>2</sub>)-*ipso*-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 128.6 (4-(CH(CH<sub>3</sub>)<sub>2</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>-*m*-C<sub>6</sub>H<sub>5</sub>), 128.5 (4-(CH(CH<sub>3</sub>)<sub>2</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>-*o*-C<sub>6</sub>H<sub>5</sub>), 126.7 (4-(CH(CH<sub>3</sub>)<sub>2</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 126.1 (4-(CH(CH<sub>3</sub>)<sub>2</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 126.0 (4-(CH(CH<sub>3</sub>)<sub>2</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>-*p*-C<sub>6</sub>H<sub>5</sub>), 73.9 (4-(CH(CH<sub>3</sub>)<sub>2</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 40.5 (4-(CH(CH<sub>3</sub>)<sub>2</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 34.0 (4-(CH(CH<sub>3</sub>)<sub>2</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 32.3 (4-(CH(CH<sub>3</sub>)<sub>2</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 24.2 (4-(CH(CH<sub>3</sub>)<sub>2</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>). GCMS (ESI):  $[\text{M}]^+ m/z = 254$ . Anal. Calcd. for C<sub>18</sub>H<sub>22</sub>O: C, 84.99; H, 8.72; Found: C, 85.07; H, 8.15.

**1-(4-(*tert*-Butyl)phenyl)-3-phenylpropan-1-ol (**11**)<sup>4</sup>**



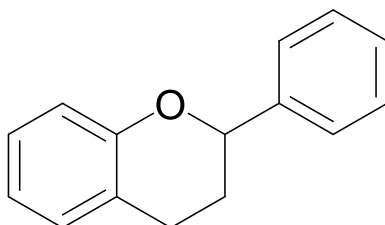
Colorless liquid [0.192 g, 72 % isolated yield (**1c**); 0.202 g, 75 % isolated yield (**2c**)]  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz, 25 °C):  $\delta$  7.40 (d, 2H,  $^3J_{\text{HH}} = 8$  Hz, 4-(C(CH<sub>3</sub>)<sub>3</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 7.34-7.26 (m, 4H, 4-(C(CH<sub>3</sub>)<sub>3</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 7.23-7.18 (m, 3H, 4-(C(CH<sub>3</sub>)<sub>3</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 4.69-4.66 (m, 1H, 4-(C(CH<sub>3</sub>)<sub>3</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 2.81-2.66 (m, 2H, 4-(C(CH<sub>3</sub>)<sub>3</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 2.21-2.00 (m, 2H, 4-(C(CH<sub>3</sub>)<sub>3</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 1.88 (b, 1H, 4-(C(CH<sub>3</sub>)<sub>3</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 1.35 (s, 9H, 4-(C(CH<sub>3</sub>)<sub>3</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>).  $^{13}\text{C}\{^1\text{H}\}$  NMR ( $\text{CDCl}_3$ , 100 MHz, 25 °C):  $\delta$  150.7 (4-(C(CH<sub>3</sub>)<sub>3</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 142.0 (4-(C(CH<sub>3</sub>)<sub>3</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>-*ipso*-C<sub>6</sub>H<sub>5</sub>), 141.7 (4-(C(CH<sub>3</sub>)<sub>3</sub>)-*ipso*-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 128.6 (4-(C(CH<sub>3</sub>)<sub>3</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>-*m*-C<sub>6</sub>H<sub>5</sub>), 128.5 (4-(C(CH<sub>3</sub>)<sub>3</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>-*o*-C<sub>6</sub>H<sub>5</sub>), 126.0 (4-(C(CH<sub>3</sub>)<sub>3</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>-*p*-C<sub>6</sub>H<sub>5</sub>), 125.8 (4-(C(CH<sub>3</sub>)<sub>3</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 125.6 (4-(C(CH<sub>3</sub>)<sub>3</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 73.8 (4-(C(CH<sub>3</sub>)<sub>3</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 40.4 (4-(C(CH<sub>3</sub>)<sub>3</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 34.7 (4-(C(CH<sub>3</sub>)<sub>3</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 32.3 (4-(C(CH<sub>3</sub>)<sub>3</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>), 31.5 (4-(C(CH<sub>3</sub>)<sub>3</sub>)-C<sub>6</sub>H<sub>4</sub>CH(OH)CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>). GCMS (ESI):  $[\text{M}]^+$   $m/z = 268$ . Anal. Calcd. for C<sub>19</sub>H<sub>24</sub>O: C, 85.03; H, 9.01; Found: C, 84.33; H, 8.30.

#### 1-(Benzofuran-2-yl)-3-phenylpropan-1-ol (**12**)<sup>4</sup>



Colorless liquid [0.171 g, 68 % isolated yield (**1c**); 0.161 g, 64 % isolated yield (**2c**)]  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz, 25 °C):  $\delta$  7.55 (dd, 1H,  $^3J_{\text{HH}} = 8$  Hz,  $^4J_{\text{HH}} = 1$  Hz,  $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 7.47 (d, 1H,  $^3J_{\text{HH}} = 8$  Hz,  $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 7.32-7.19 (m, 7H,  $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 6.64 (s, 1H,  $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 4.83 (t, 1H,  $^3J_{\text{HH}} = 7$  Hz,  $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 2.83–2.77 (m, 2H,  $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 2.31–2.25 (m, 2H,  $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 1.65 (d, 1H,  $^3J_{\text{HH}} = 7$  Hz,  $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ).  $^{13}\text{C}\{^1\text{H}\}$  NMR ( $\text{CDCl}_3$ , 125 MHz, 25 °C):  $\delta$  159.3 (*ipso*- $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 155.0 ( $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 141.5 ( $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{-ipso-C}_6\text{H}_5$ ), 128.7 ( $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{-m-C}_6\text{H}_5$ ), 128.6 ( $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{-o-C}_6\text{H}_5$ ), 128.3 ( $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 126.2 ( $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{-p-C}_6\text{H}_5$ ), 124.4 ( $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 123.0 ( $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 121.2 ( $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 111.4 ( $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 102.9 ( $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 67.7 ( $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 37.2 ( $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ), 31.8 ( $\text{C}_8\text{H}_5\text{OCH}(\text{OH})\text{CH}_2\text{CH}_2\text{C}_6\text{H}_5$ ). GCMS (ESI):  $[\text{M}]^+$   $m/z = 252$ . Anal. Calcd. for  $\text{C}_{17}\text{H}_{16}\text{O}_2$ : C, 80.93; H, 6.39; Found: C, 81.13; H, 5.87.

### Synthesis of 2-Phenylchroman<sup>5</sup>

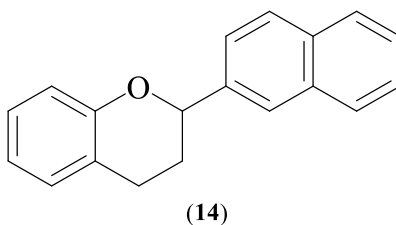


(13)

Colorless dense liquid [0.0346 g, 17 % isolated yield (**1c**); 0.0197 g, 10 % isolated yield (**2c**)].  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz, 25 °C):  $\delta$  7.47–7.45 (m, 2H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_5$ ), 7.42 (t, 2H,

$^3J_{\text{H-H}} = 8 \text{ Hz}$ ,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_5$ ), 7.35 (t, 1H,  $^3J_{\text{H-H}} = 8 \text{ Hz}$ ,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_5$ ), 7.17–7.11 (m, 2H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_5$ ), 6.95–6.89 (m, 2H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_5$ ), 5.10 (dd, 1H,  $^3J_{\text{H-H}} = 8 \text{ Hz}$ ,  $^1J_{\text{H-H}} = 2 \text{ Hz}$ ,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_5$ ), 3.07–3.00 (m, 1H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_5$ ), 2.86–2.81 (m, 1H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_5$ ), 2.27–2.22 (m, 1H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_5$ ), 2.17–2.08 (m, 1H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_5$ ).  $^{13}\text{C}\{^1\text{H}\}$  NMR ( $\text{CDCl}_3$ , 125 MHz, 25 °C):  $\delta$  ppm, 155.1, 141.8, 129.5, 128.5, 127.8, 127.3, 125.9, 121.8, 120.3, 116.9, 77.8, 29.9, 25.0. GCMS (ESI):  $[\text{M}]^+ m/z = 210$ . Anal. Calcd. for  $\text{C}_{15}\text{H}_{14}\text{O} \cdot 1/4\text{CH}_2\text{Cl}_2$ : C, 79.12.11; H, 6.31; Found: C, 79.34; H, 5.58 %.

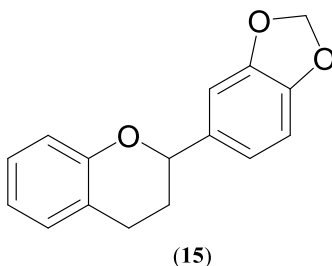
## 2-(Naphthalen-2-yl)chroman (14)<sup>5</sup>



Yellow Solid [0.0618 g, 36 % isolated yield (**1c**); 0.0473 g, 28 % isolated yield (**2c**).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz, 25 °C):  $\delta$  7.90–7.85 (m, 4H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_3\text{C}_4\text{H}_4$ ),  $\delta$  7.56–7.48 (m, 3H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_3\text{C}_4\text{H}_4$ ),  $\delta$  7.18–7.12 (m, 2H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_3\text{C}_4\text{H}_4$ ),  $\delta$  6.98–6.96 (m, 1H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_3\text{C}_4\text{H}_4$ ), 6.90 (t, 1H,  $^3J_{\text{H-H}} = 8 \text{ Hz}$ ,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_3\text{C}_4\text{H}_4$ ), 5.25 (dd, 1H,  $^3J_{\text{H-H}} = 8 \text{ Hz}$ ,  $^1J_{\text{H-H}} = 2 \text{ Hz}$ ,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_3\text{C}_4\text{H}_4$ ), 3.09–3.01 (m, 1H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_3\text{C}_4\text{H}_4$ ), 2.87–2.81 (m, 1H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_3\text{C}_4\text{H}_4$ ), 2.34–2.27 (m, 1H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_3\text{C}_4\text{H}_4$ ), 2.14–2.14 (m, 1H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_3\text{C}_4\text{H}_4$ ).  $^{13}\text{C}\{^1\text{H}\}$  NMR ( $\text{CDCl}_3$ , 125 MHz, 25 °C):  $\delta$  ppm, 155.1, 139.1, 133.3, 133.1, 129.6, 128.3, 128.1, 127.7, 127.4, 126.2, 125.9, 124.9, 124.0,

121.9, 120.4, 116.9, 77.9, 29.9, 25.2. GCMS (ESI):  $[M]^+$   $m/z = 260$ . Anal. Calcd. for  $C_{19}H_{16}O \cdot 1/4CH_2Cl_2$ : C, 82.12; H, 5.91; Found: C, 81.68; H, 5.66 %.

### 2-(Benzo[1,3]dioxol-5-yl)chroman (15)<sup>5</sup>



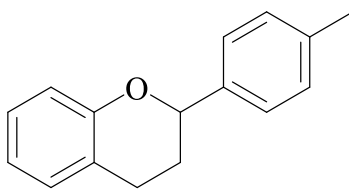
Colorless dense liquid [0.0301 g, 18 % isolated yield (**1c**); 0.0246 g, 15 % isolated yield (**2c**)].

$^1H$  NMR ( $CDCl_3$ , 500 MHz, 25 °C):  $\delta$  7.15–7.12 (m, 2H,  $C_6H_4CH_2CH_2CH(O)C_6H_3OCH_2O$ ),  $\delta$  6.97 (s, 1H,  $C_6H_4CH_2CH_2CH(O)C_6H_3OCH_2O$ ),  $\delta$  6.93–6.89 (m, 3H,  $C_6H_4CH_2CH_2CH(O)C_6H_3OCH_2O$ ),  $\delta$  6.85–6.84 (m, 1H,  $C_6H_4CH_2CH_2CH(O)C_6H_3OCH_2O$ ), 5.99 (s, 2H,  $C_6H_4CH_2CH_2CH(O)C_6H_3OCH_2O$ ), 4.99 (dd, 1H,  $^3J_{H-H} = 8$  Hz,  $^1J_{H-H} = 2$  Hz,  $C_6H_4CH_2CH_2CH(O)C_6H_3OCH_2O$ ), 3.05–2.98 (m, 1H,  $C_6H_4CH_2CH_2CH(O)C_6H_3OCH_2O$ ), 2.86–2.81 (m, 1H,  $C_6H_4CH_2CH_2CH(O)C_6H_3OCH_2O$ ), 2.22–2.17 (m, 1H,  $C_6H_4CH_2CH_2CH(O)C_6H_3OCH_2O$ ), 2.14–2.05 (m, 1H,  $C_6H_4CH_2CH_2CH(O)C_6H_3OCH_2O$ ).

$^{13}C\{^1H\}$  NMR ( $CDCl_3$ , 125 MHz, 25 °C):  $\delta$  ppm, 155.1, 147.8, 147.2, 135.6, 129.5, 127.4, 121.7, 120.3, 119.6, 116.9, 108.2, 106.7, 101.1, 77.7, 29.9, 25.2. GCMS (ESI):  $[M]^+$   $m/z = 254$ .

Anal. Calcd. for  $C_{16}H_{14}O_3$ : C, 75.58; H, 5.55; Found: C, 74.42; H, 4.29 %.

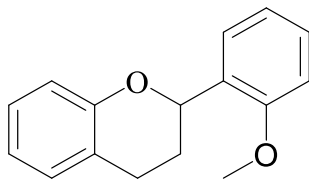
### 2-(4-Tolyl)chroman (16)<sup>5</sup>



(16)

Colorless solid [0.0175 g, 12 % isolated yield (**1c**); 0.0153 g, 11 % isolated yield (**2c**)]  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz, 25 °C):  $\delta$  7.32–7.31 (m, 2H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_4\text{CH}_3$ ), 7.20 (d, 2H,  $^3J_{\text{H-H}} = 8$  Hz,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_4\text{CH}_3$ ), 7.14–7.06 (m, 2H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_4\text{CH}_3$ ), 6.91–6.84 (m, 2H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_4\text{CH}_3$ ), 5.04 (m, 1H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_4\text{CH}_3$ ), 3.05–2.97 (m, 1H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_4\text{CH}_3$ ), 2.84–2.78 (m, 1H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_4\text{CH}_3$ ), 2.40 (s, 3H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_4\text{CH}_3$ ), 2.24–2.19 (m, 1H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_4\text{CH}_3$ ), 2.16–2.06 (m, 1H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_4\text{CH}_3$ ).  $^{13}\text{C}\{^1\text{H}\}$  NMR ( $\text{CDCl}_3$ , 400 MHz, 25 °C):  $\delta$  ppm, 155.2, 138.8, 137.2, 129.4, 129.1, 127.3, 125.9, 121.6, 120.2, 96.2, 77.5, 30.0, 25.2, 21.2. GCMS (ESI):  $[\text{M}]^+ m/z = 224$ . Anal. Calcd. for  $\text{C}_{16}\text{H}_{16}\text{O}$ : C, 85.68; H, 7.19; Found: C, 85.41; H, 6.35 %.

### 2-(2-Methoxyphenyl)chroman (17)<sup>5</sup>



(17)

Colorless solid [0.0214 g, 14 % isolated yield (**1c**); 0.0197 g, 13 % isolated yield (**2c**)]  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz, 25 °C):  $\delta$  7.53 (d, 1H,  $^3J_{\text{H-H}} = 8$  Hz,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_4\text{OCH}_3$ ), 7.31 (t, 1H,  $^3J_{\text{H-H}} = 8$  Hz,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_4\text{OCH}_3$ ), 7.17–7.11 (m, 2H,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_4\text{OCH}_3$ ), 7.03 (t, 1H,  $^3J_{\text{H-H}} = 8$  Hz,  $\text{C}_6\text{H}_4\text{CH}_2\text{CH}_2\text{CH}(\text{O})\text{C}_6\text{H}_4\text{OCH}_3$ ),



6.96-6.88 (m, 3H, C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CH<sub>2</sub>CH(O)C<sub>6</sub>H<sub>4</sub>OCH<sub>3</sub>), 5.48 (d, 1H, <sup>3</sup>J<sub>H-H</sub> = 10 Hz, C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CH<sub>2</sub>CH(O)C<sub>6</sub>H<sub>4</sub>OCH<sub>3</sub>), 3.87 (s, 3H, C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CH<sub>2</sub>CH(O)C<sub>6</sub>H<sub>4</sub>OCH<sub>3</sub>), 3.07–2.99 (m, 1H, C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CH<sub>2</sub>CH(O)C<sub>6</sub>H<sub>4</sub>OCH<sub>3</sub>), 2.82–2.76 (m, 1H, C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CH<sub>2</sub>CH(O)C<sub>6</sub>H<sub>4</sub>OCH<sub>3</sub>), 2.30–2.25 (m, 1H, C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CH<sub>2</sub>CH(O)C<sub>6</sub>H<sub>4</sub>OCH<sub>3</sub>), 2.03–1.93 (m, 1H, C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CH<sub>2</sub>CH(O)C<sub>6</sub>H<sub>4</sub>OCH<sub>3</sub>). <sup>13</sup>C{<sup>1</sup>H} NMR (CDCl<sub>3</sub>, 100 MHz, 25 °C): δ ppm, 155.9, 155.5, 130.3, 129.6, 128.5, 127.2, 126.5, 122.3, 120.8, 120.1, 116.9, 110.4, 72.4, 55.4, 28.6, 25.3. GCMS (ESI): [M]<sup>+</sup> *m/z* = 240. Anal. Calcd. for C<sub>16</sub>H<sub>14</sub>O<sub>2</sub>: C, 79.97; H, 6.71; Found: C, 79.21; H, 6.47 %.

NAME PG-APP-07-122-1-  
EXPNO 4  
PROCNO 1  
Date\_ 20160720  
Time 21.21  
INSTRUM spect  
PROBHD 5 mm SEI 1H/D-  
PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2999091 sec  
RG 101  
DW 60.800 usec  
DE 6.50 usec  
TE 297.2 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.75 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SFO1 400.1324710 MHz  
SI 32768  
SF 400.1300103 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

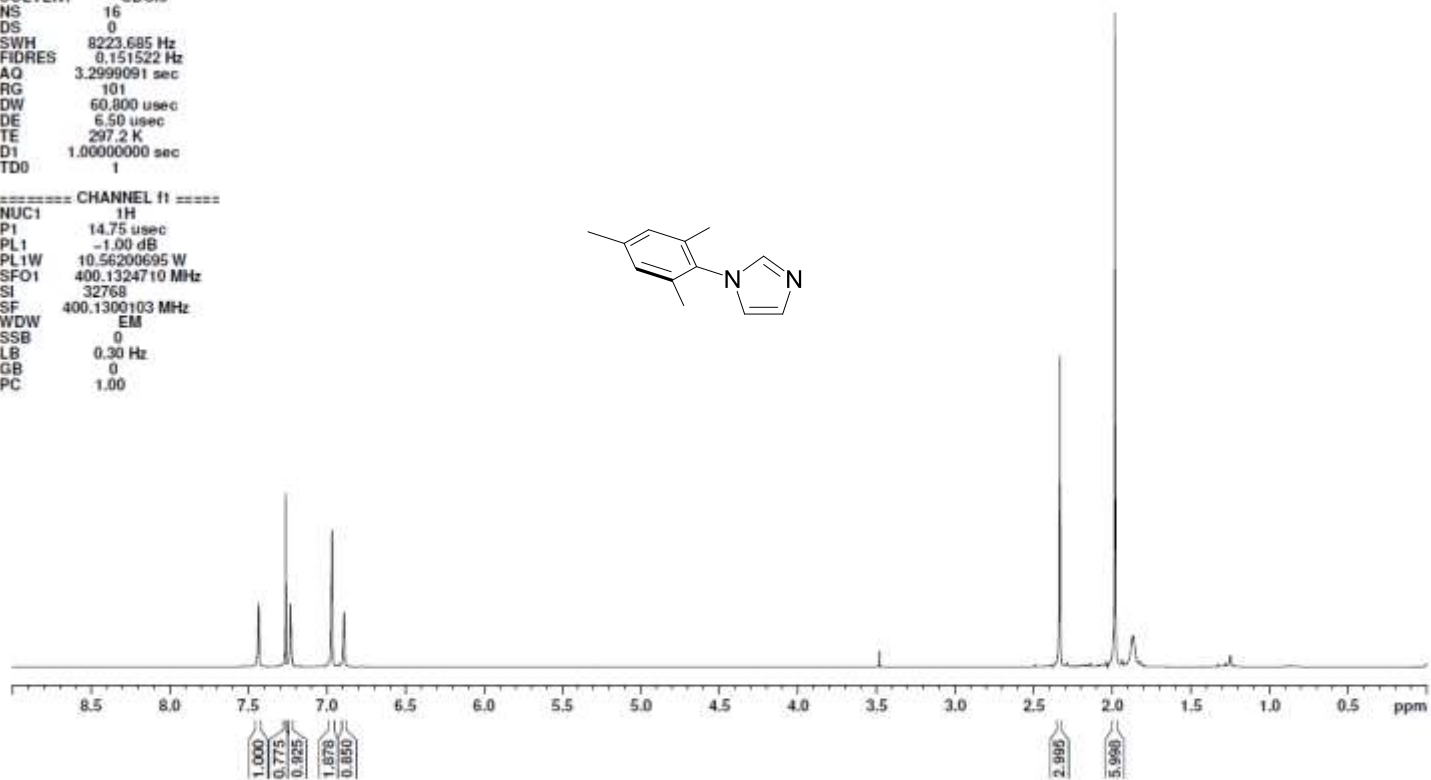
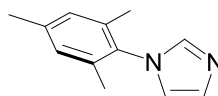
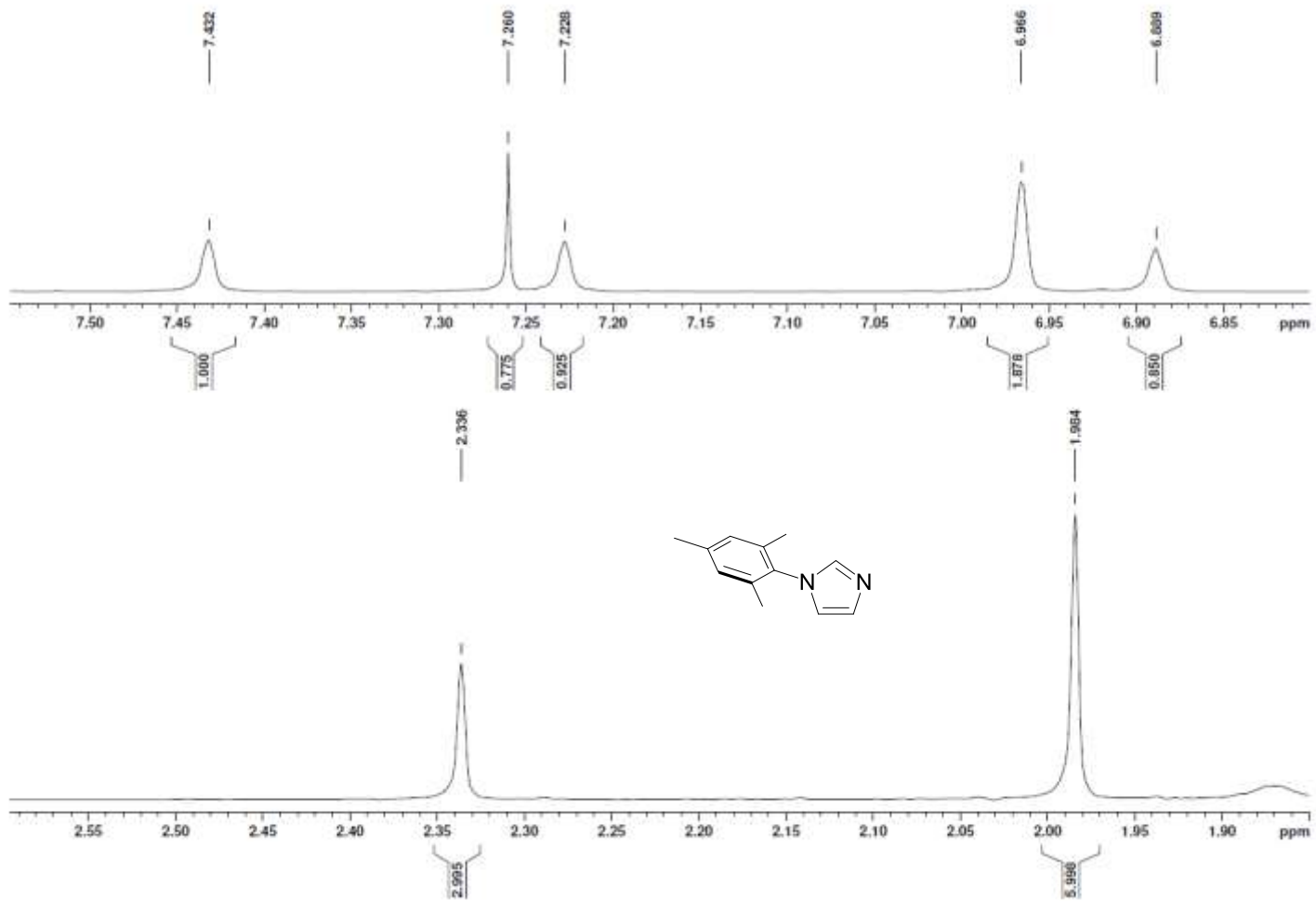


Figure S1. <sup>1</sup>H NMR spectrum of 1-Mesityl imidazole in CDCl<sub>3</sub>.



**Figure S2.** Expanded  $^1\text{H}$  NMR spectrum of 1-Mesityl imidazole in  $\text{CDCl}_3$ .

PG-APP-07-122-1-13C

Current Data Parameters  
NAME PG-APP-07-122-1-13C  
EXPNO 12  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20160721  
Time\_ 19.35  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 112  
DS 4  
SWH 29761.904 Hz  
FIDRES 0.454131 Hz  
AQ 1.1010048 sec  
RG 197.27  
DW 16.800 usec  
DE 6.50 usec  
TE 296.4 K  
D1 1.00000000 sec  
D11 0.03000000 sec  
TD0 1

----- CHANNEL f1 -----  
SFO1 125.7703637 MHz  
NUC1 13C  
P1 8.90 usec  
PLW1 103.00000000 W

----- CHANNEL f2 -----  
SFO2 500.1320005 MHz  
NUC2 1H  
CPDPRG(2) waltz16  
PCPD2 80.00 usec  
PLW2 13.00000000 W  
PLW12 0.34327999 W  
PLW13 0.17267001 W

F2 - Processing parameters  
SI 32768  
SF 125.7577739 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

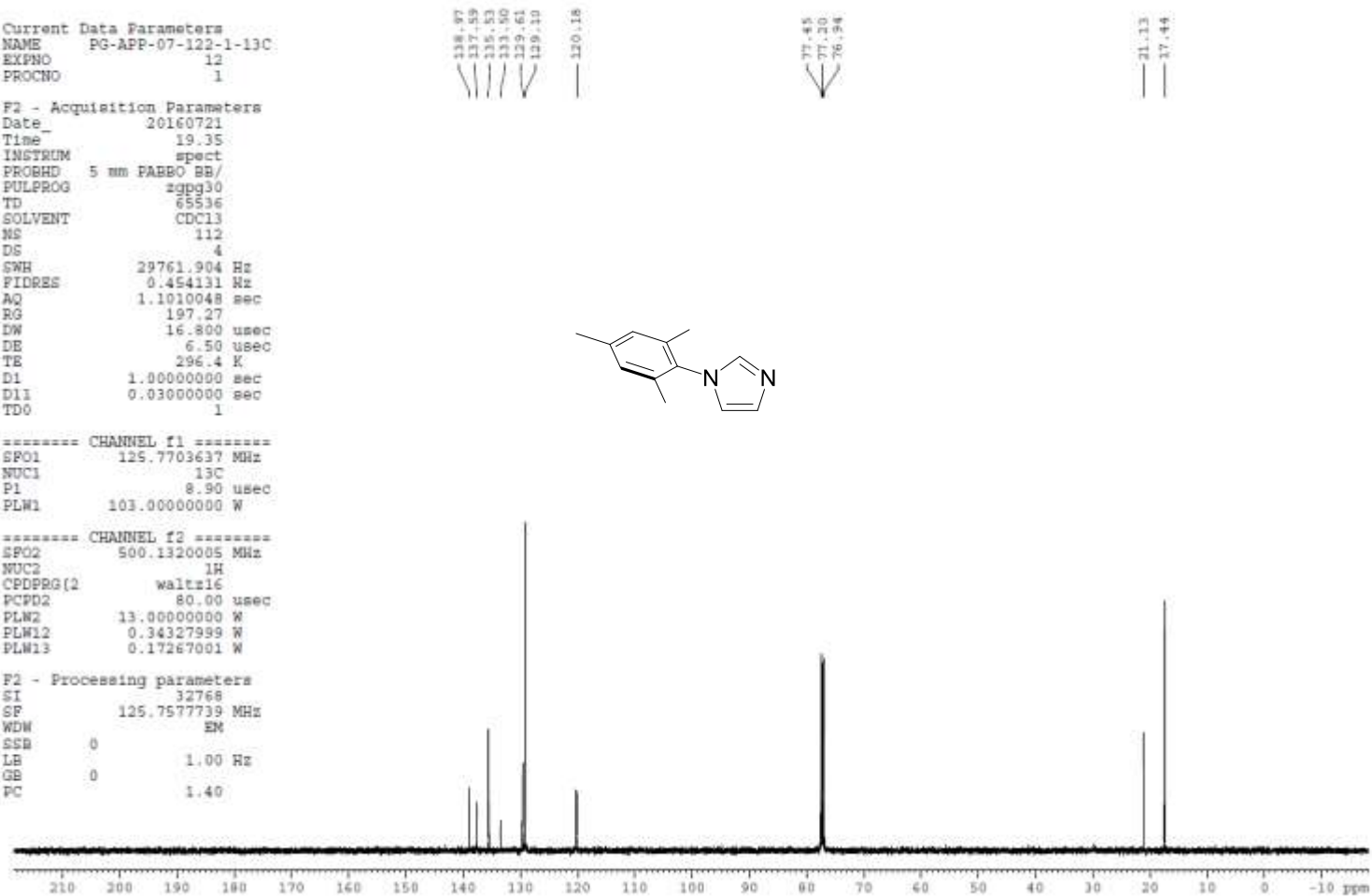
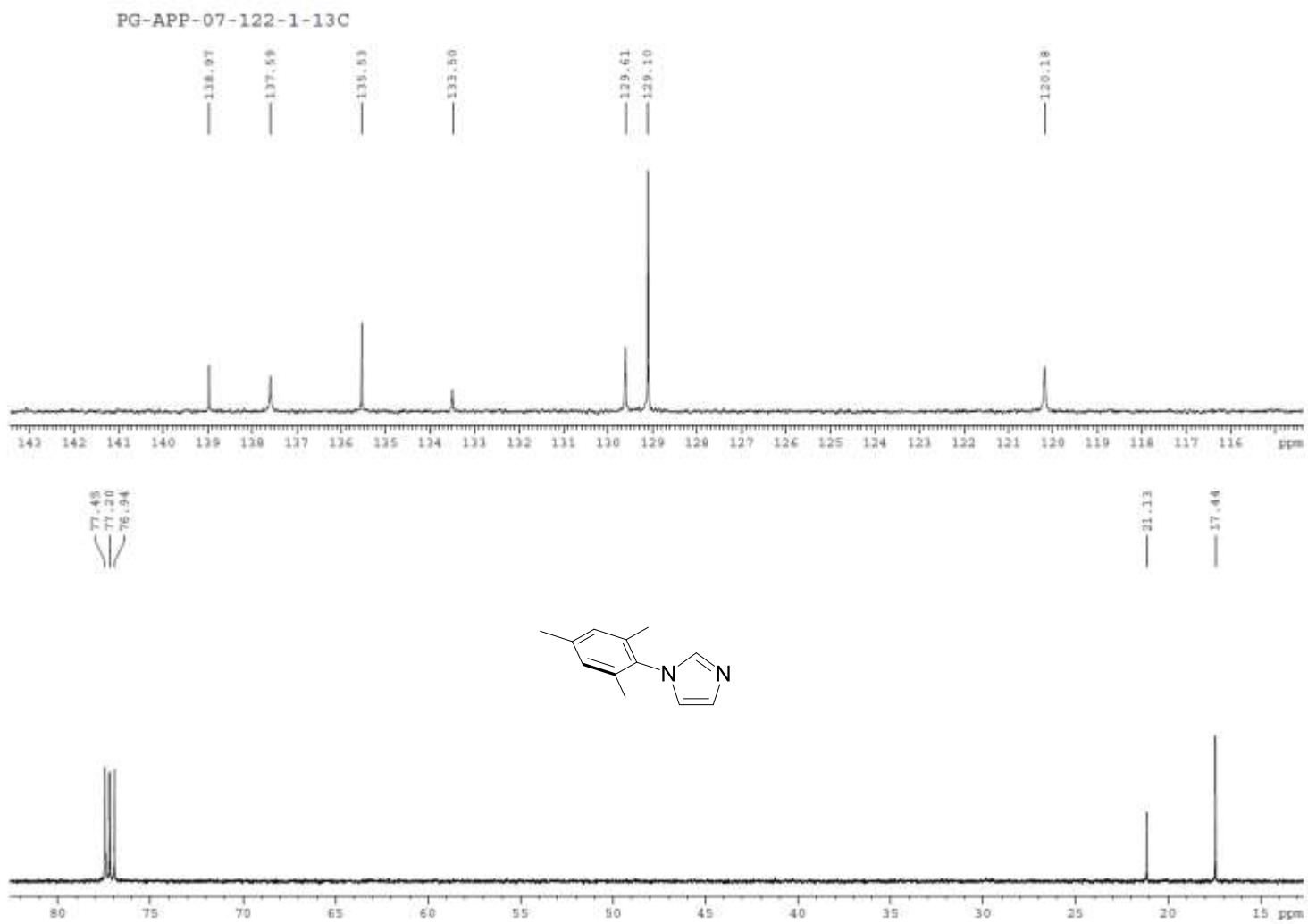


Figure S3.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of 1-Mesityl imidazole in  $\text{CDCl}_3$ .



**Figure S4.** Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of 1-Mesityl imidazole in  $\text{CDCl}_3$ .

PG-APP-09-209-1-1H

Current Data Parameters  
NAME PG-APP-09-209-1-1H  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date 20171208  
Time 8.28  
INSTRUM spect  
PROBHD 5 mm PANBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 98.91  
DW 50.000 usec  
DE 6.50 usec  
TE 295.4 K  
D1 1.00000000 sec  
TD0 1

----- CHANNEL f1 -----  
SF01 500.133085 MHz  
NUC1 1H  
P1 13.35 usec  
PLM1 16.00000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1300134 MHz  
WUM EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

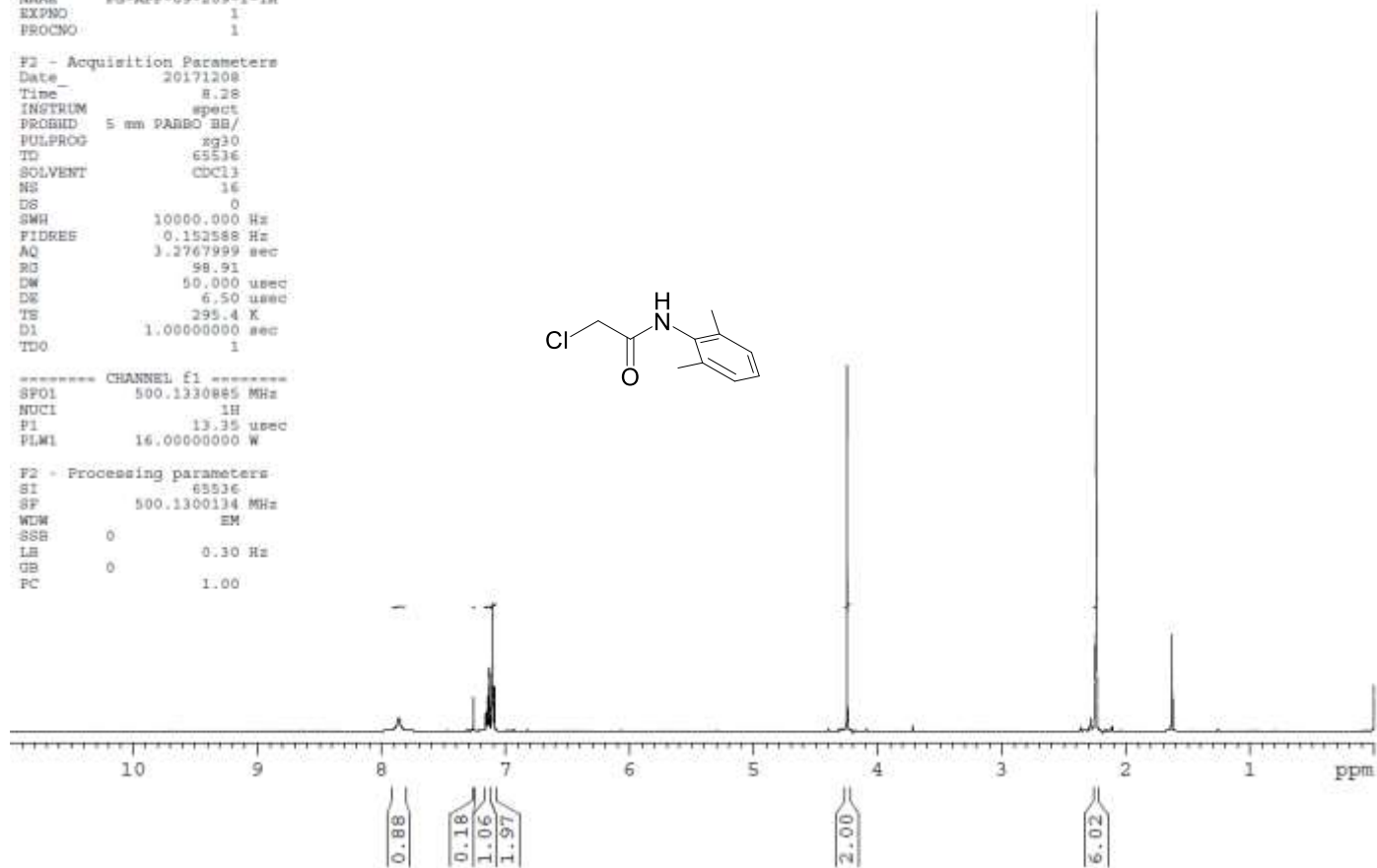
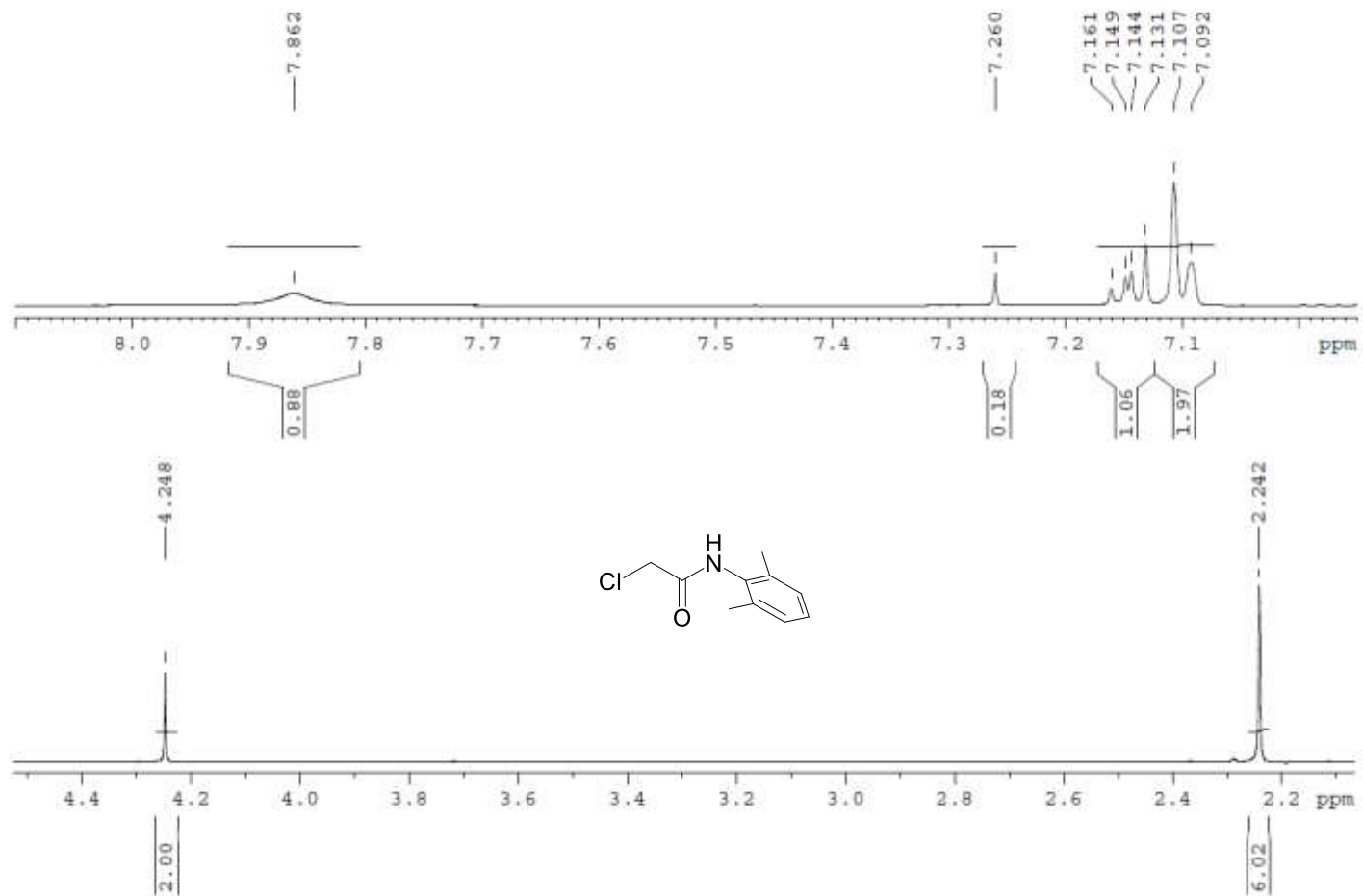


Figure S5. <sup>1</sup>H NMR spectrum of 2-chloro-N-(2,6-Me<sub>2</sub>-phenyl)acetamide in CDCl<sub>3</sub>.

PG-APP-09-209-1-1H



**Figure S6.** Expanded <sup>1</sup>H NMR spectrum of 2-chloro-*N*-(2,6-Me<sub>2</sub>-phenyl)acetamide in CDCl<sub>3</sub>.

PG-APP-09-209-1-13C

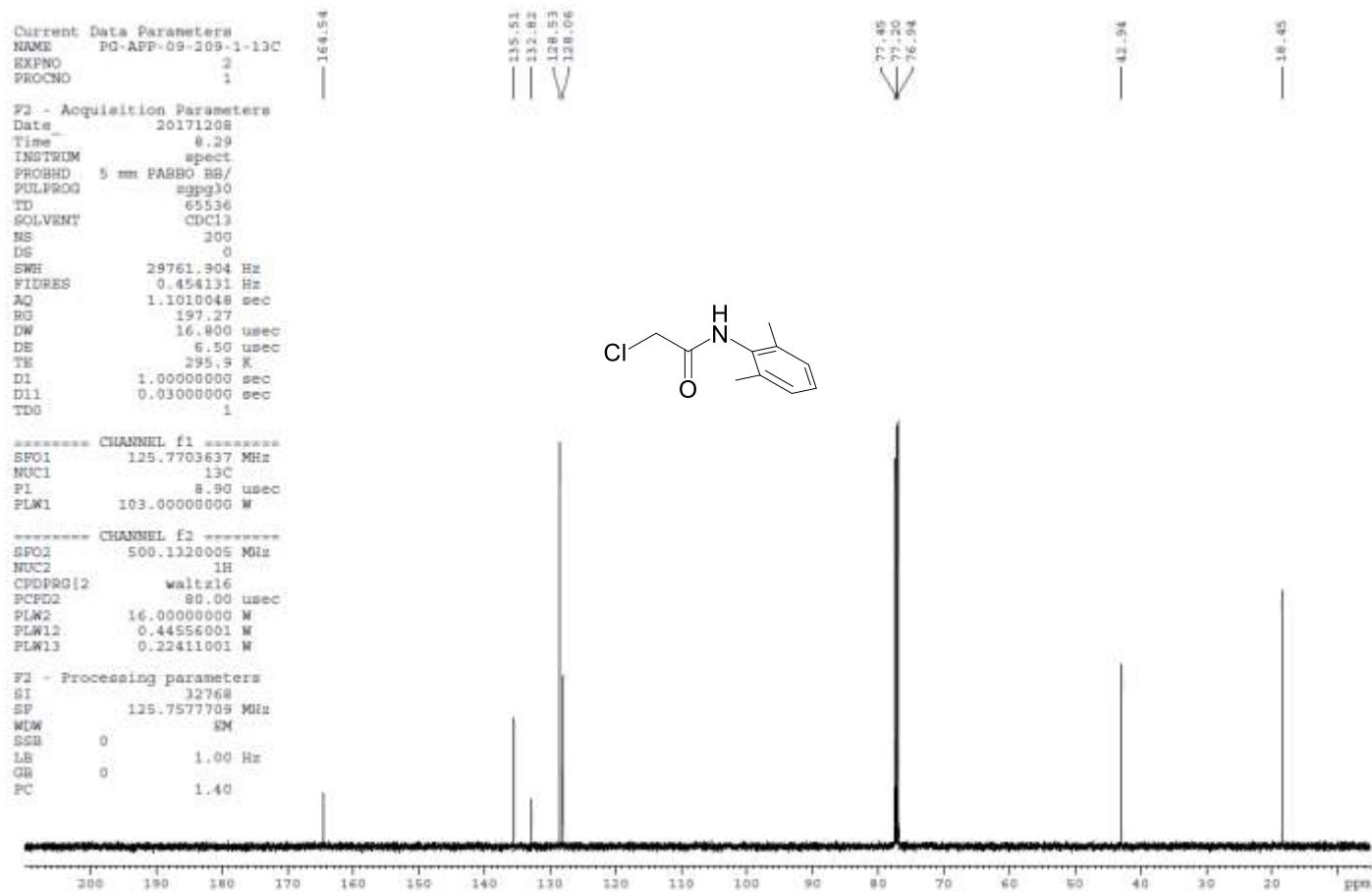
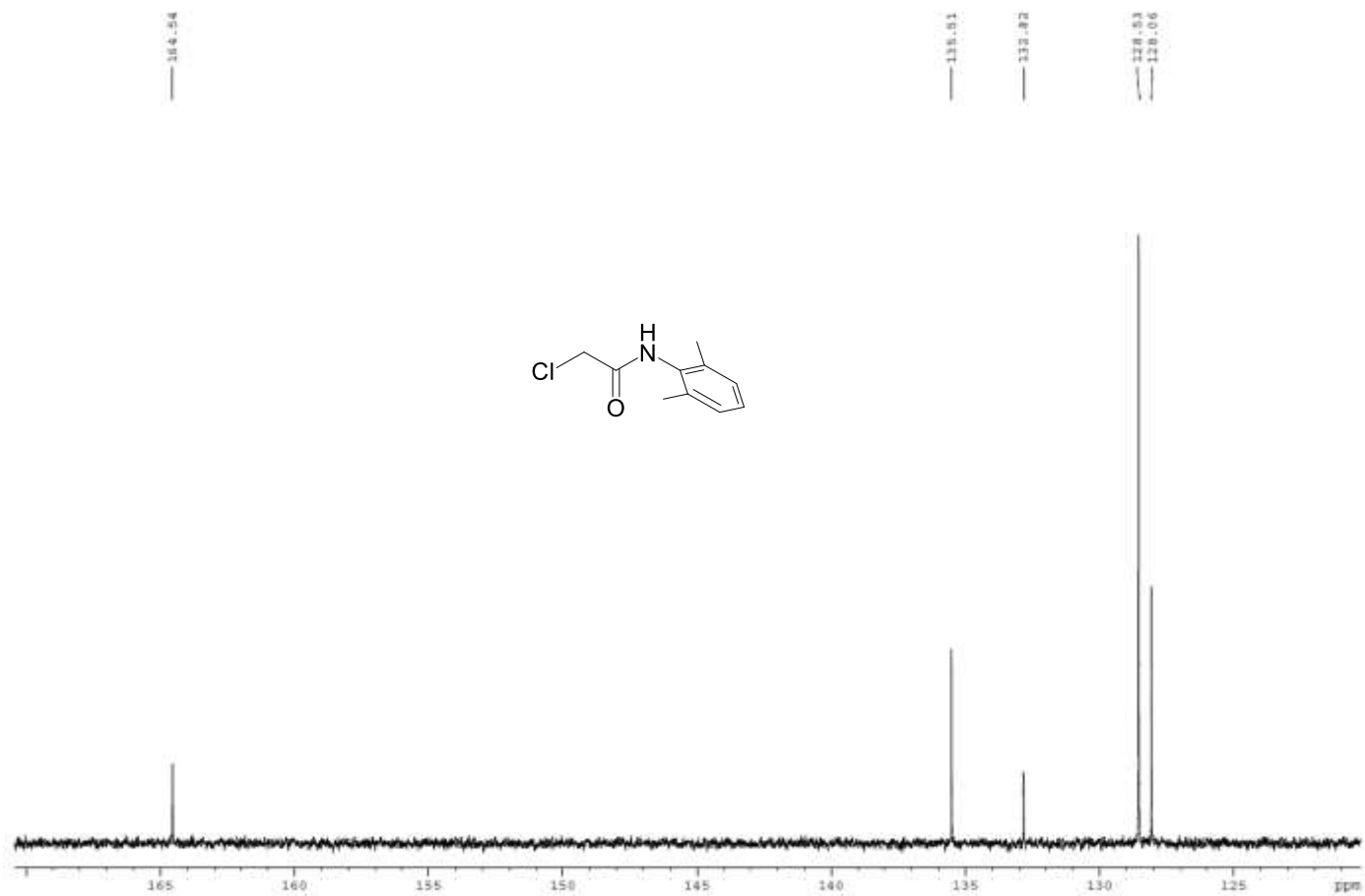


Figure S7.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of 2-chloro-*N*-(2,6-Me<sub>2</sub>-phenyl)acetamide in CDCl<sub>3</sub>.



PG-APP-09-209-1-13C



**Figure S8.** Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of 2-chloro-*N*-(2,6-Me<sub>2</sub>-phenyl)acetamide in CDCl<sub>3</sub>.

NAME PG-APP-08-75-1-1  
EXPNO 5  
PROCNO 1  
Date\_ 20170107  
Time 21.31  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2999091 sec  
RG 161  
DW 60.800 usec  
DE 6.50 usec  
TE 298.2 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.75 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SFO1 400.1324710 MHz  
SI 32768  
SF 400.1300101 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

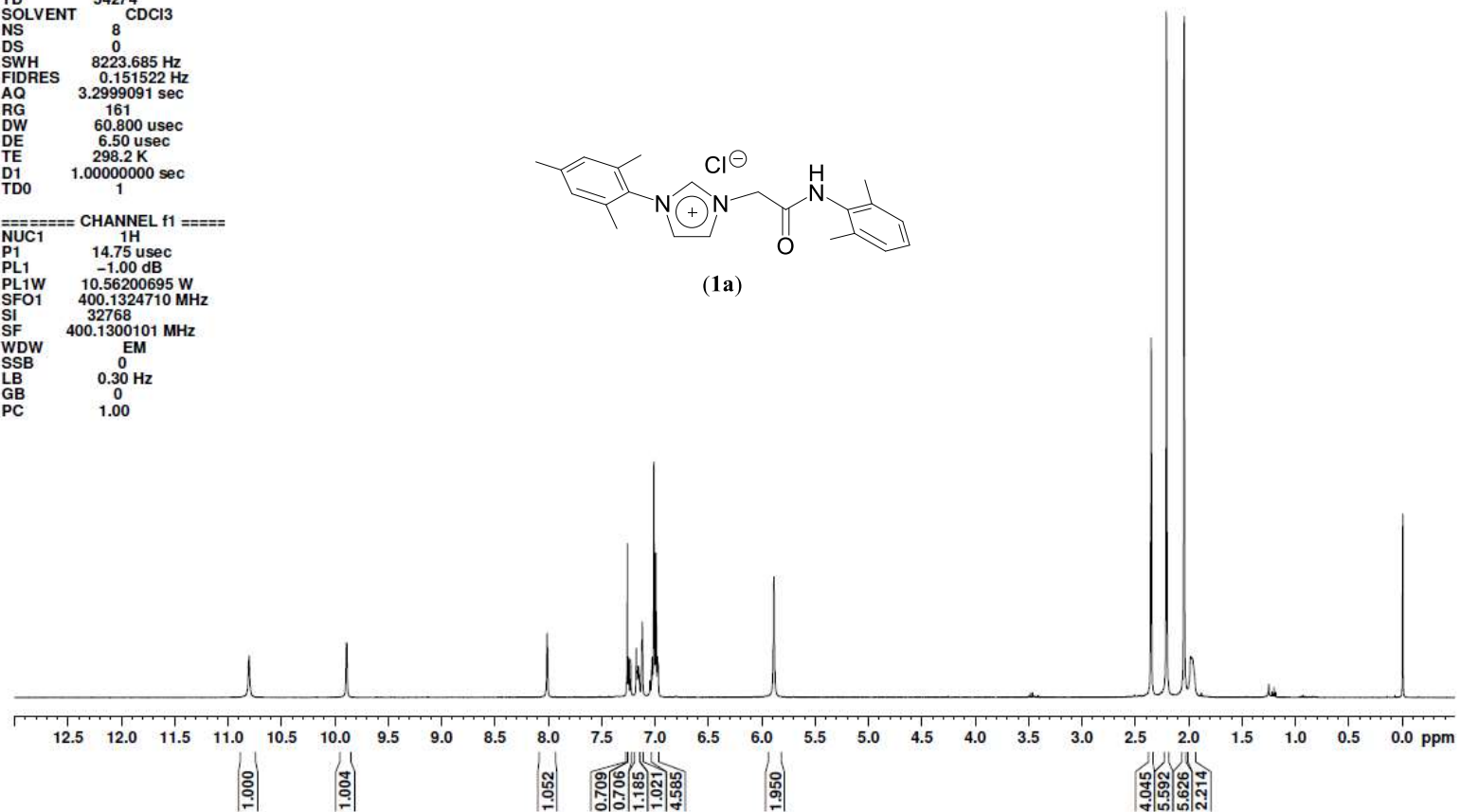
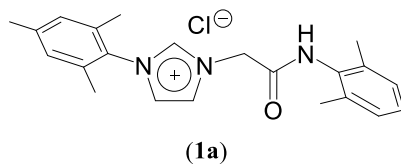
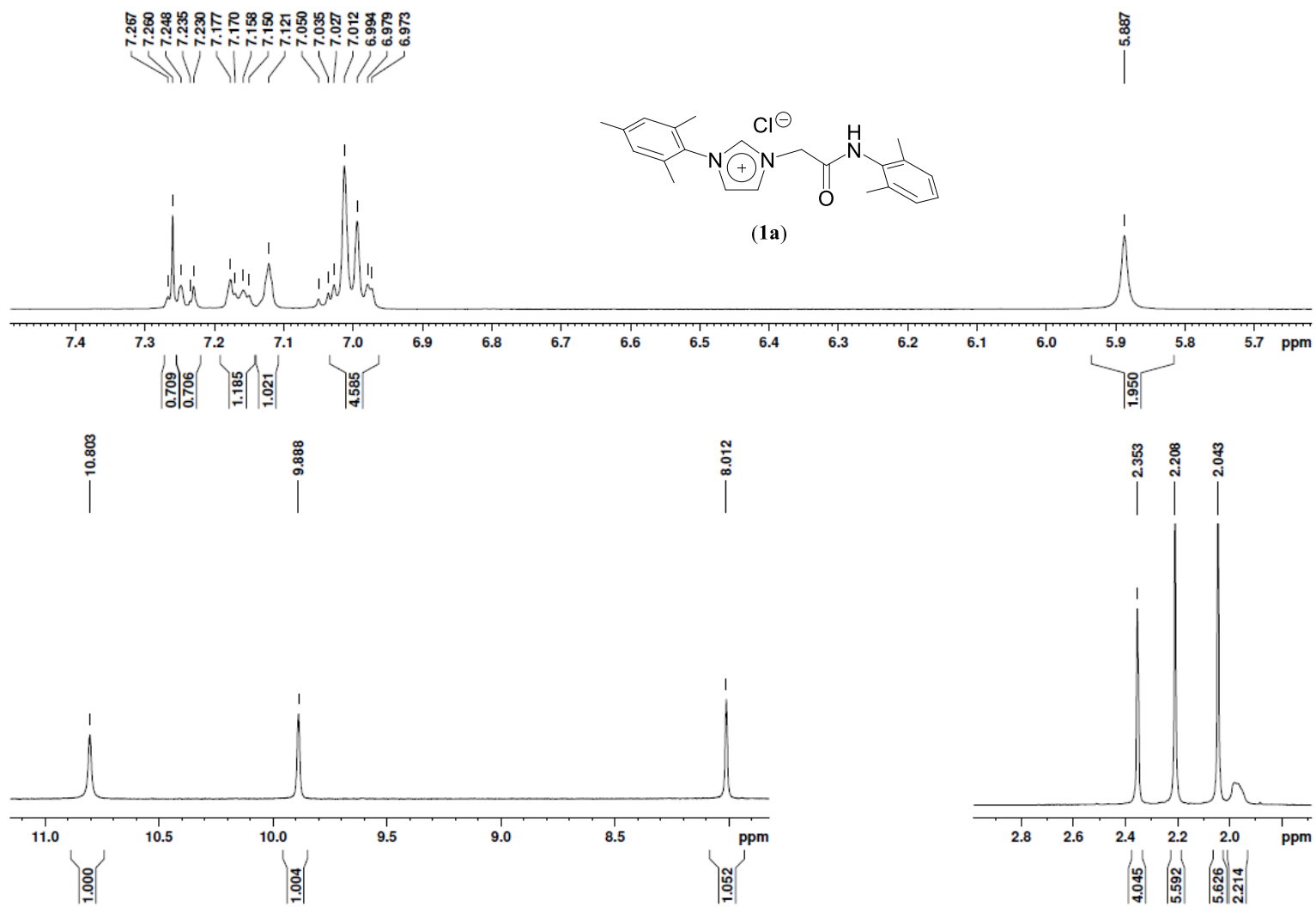


Figure S9.  $^1\text{H}$  NMR spectrum of **1a** in  $\text{CDCl}_3$ .



**Figure S10.** Expanded  $^1\text{H}$  NMR spectrum of **1a** in  $\text{CDCl}_3$ .

```

NAME      PG-APP-09-217-1-13C
EXPNO     9
PROCNO    1
Date_     20171213
Time      12.18
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         200
DS         0
SWH        26041.666 Hz
FIDRES     0.397364 Hz
AQ         1.2583412 sec
RG         2050
DW         19.200 usec
DE         6.50 usec
TE         296.2 K
D1         1.00000000 sec
D11        0.03000000 sec
TD0        1

```

```

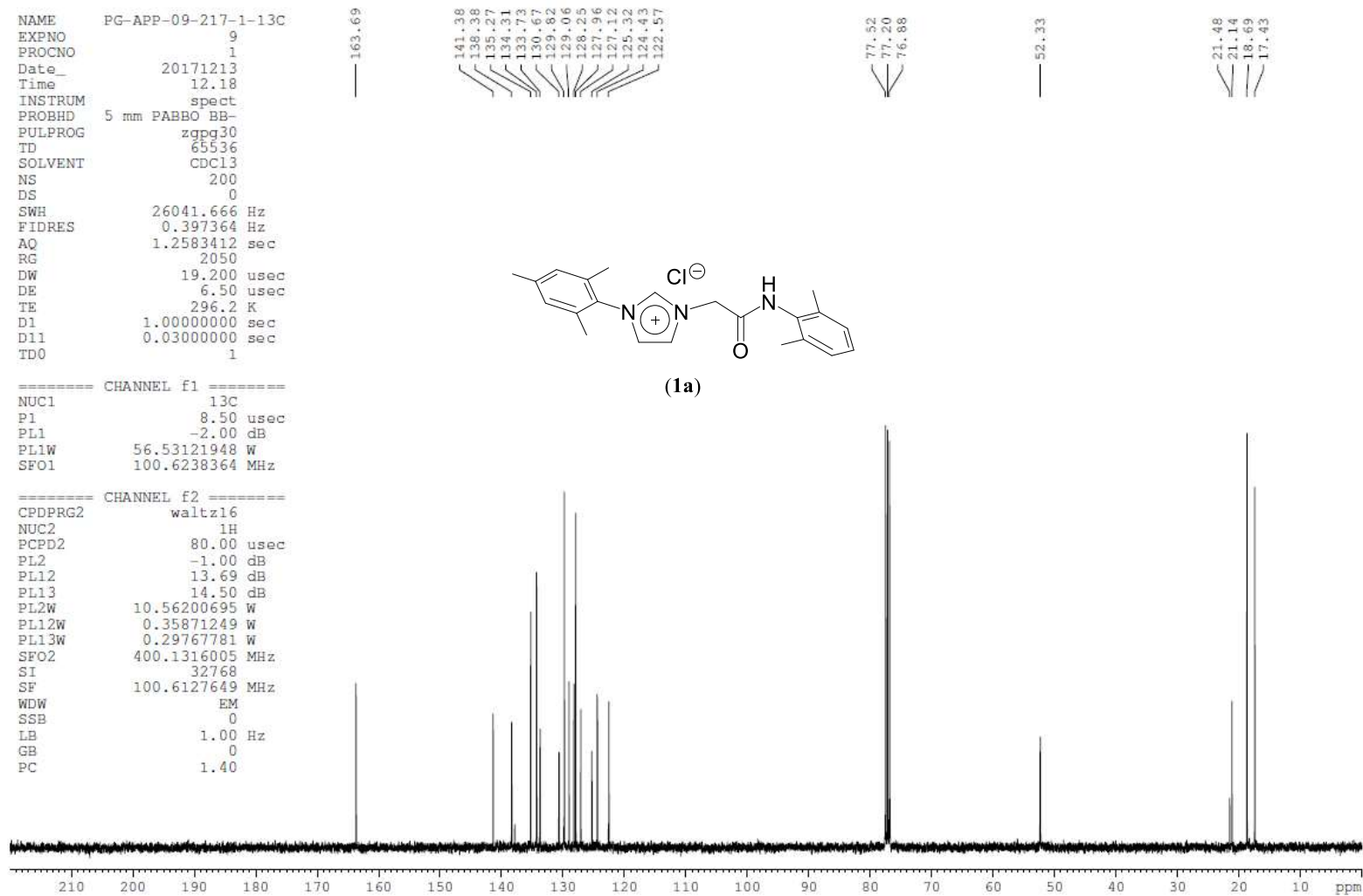
===== CHANNEL f1 =====
NUC1       13C
P1         8.50 usec
PL1        -2.00 dB
PL1W       56.53121948 W
SFO1       100.6238364 MHz

```

```

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         -1.00 dB
PL12        13.69 dB
PL13        14.50 dB
PL2W        10.56200695 W
PL12W       0.35871249 W
PL13W       0.29767781 W
SFO2        400.1316005 MHz
SI          32768
SF          100.6127649 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40

```



**Figure S11.** <sup>13</sup>C{<sup>1</sup>H} NMR spectrum of **1a** in CDCl<sub>3</sub>.

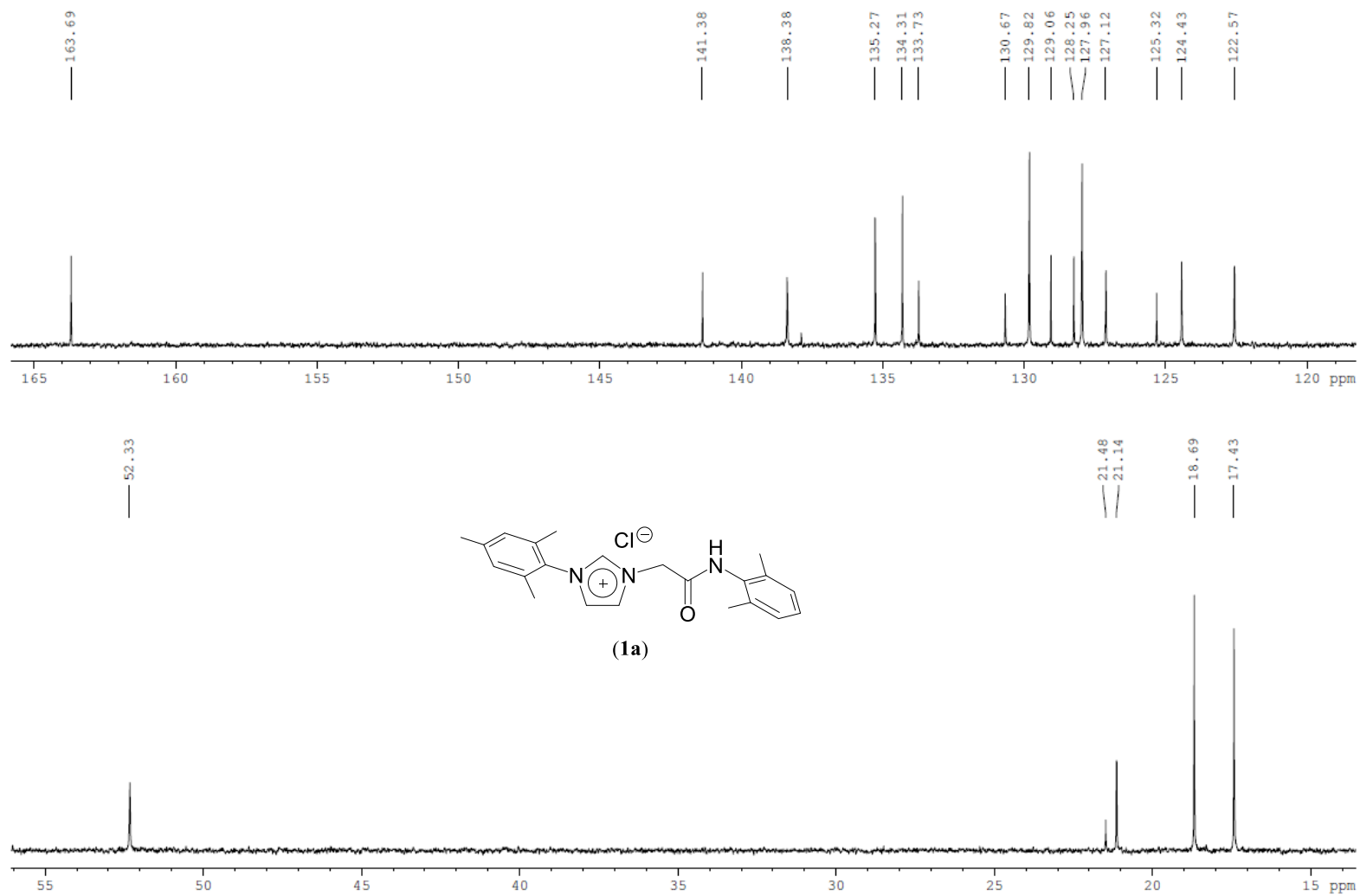
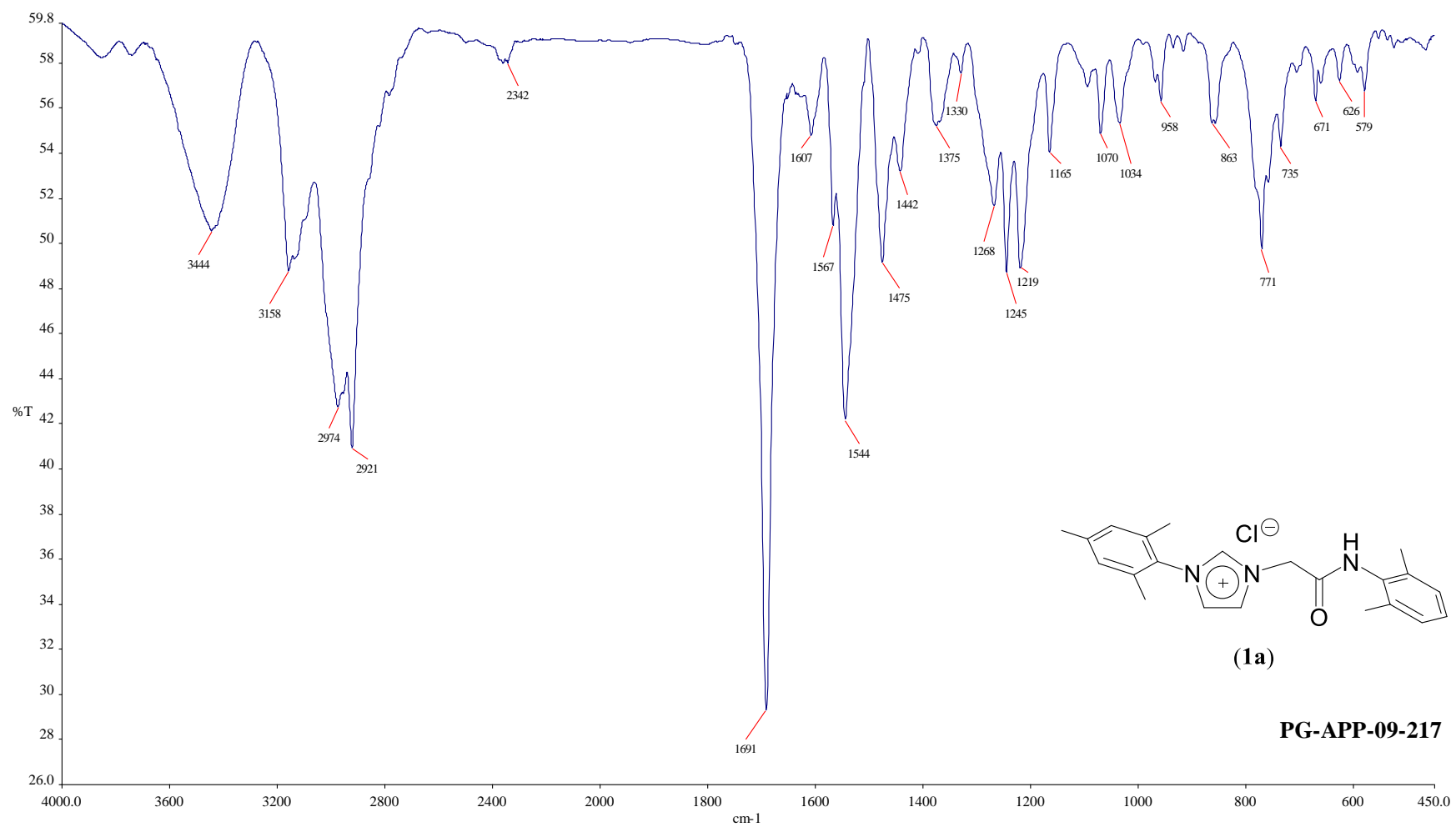


Figure S12. Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of **1a** in  $\text{CDCl}_3$ .



**Figure S13.** IR spectrum of **1a** in KBr.

**DEPARTMENT OF CHEMISTRY, I.I.T.(B)**

**Analysis Info**

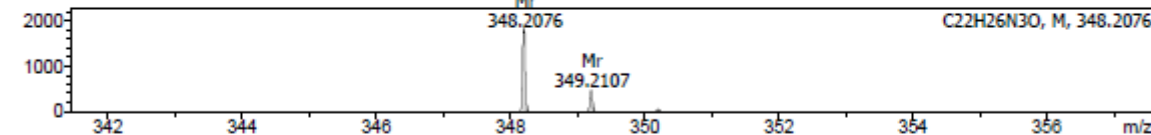
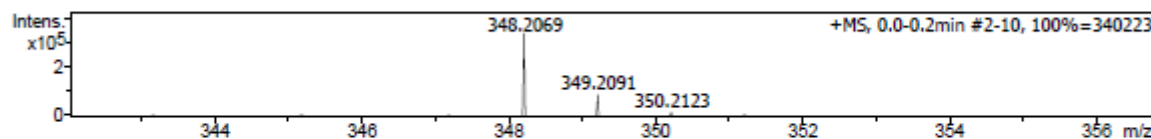
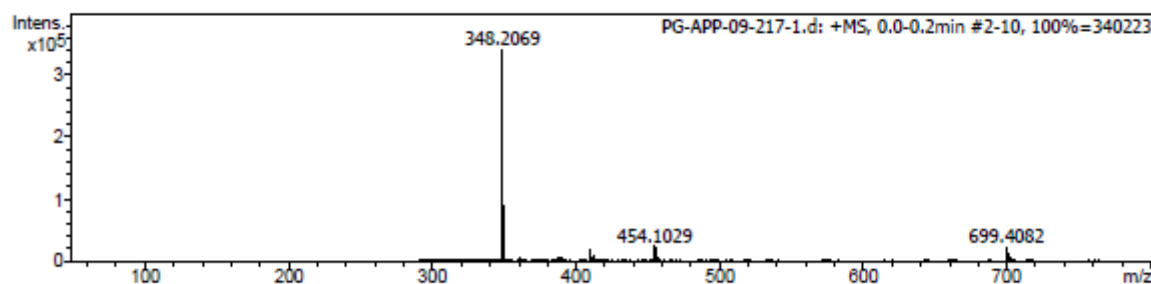
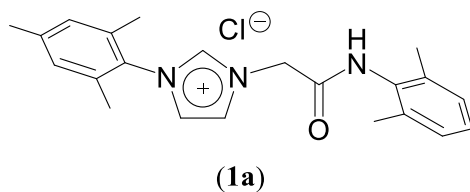
Analysis Name D:\Data\Dec 2017\PG-APP-09-217-1.d  
 Method Tune\_pos\_NAICSI-1500A.m  
 Sample Name PG-APP-09-217-1  
 Comment C22H26N3OCl

Acquisition Date 12/22/2017 8:53:52 AM

Operator PG APP IN  
 Instrument maXis impact 282001.00081

**Acquisition Parameter**

|             |         |                       |            |                  |           |
|-------------|---------|-----------------------|------------|------------------|-----------|
| Source Type | ESI     | Ion Polarity          | Positive   | Set Nebulizer    | 0.3 Bar   |
| Focus       | Active  | Set Capillary         | 3700 V     | Set Dry Heater   | 180 °C    |
| Scan Begin  | 50 m/z  | Set End Plate Offset  | -500 V     | Set Dry Gas      | 4.0 l/min |
| Scan End    | 800 m/z | Set Collision Cell RF | 1800.0 Vpp | Set Divert Valve | Source    |

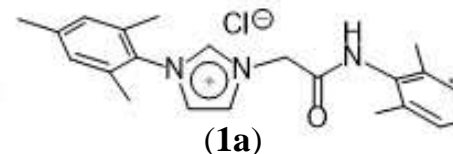


| Meas. m/z | # | Ion Formula | m/z      | err [ppm] | mSigma | # Sigma | Score  | rdb  | e <sup>-</sup> Conf | N-Rule |
|-----------|---|-------------|----------|-----------|--------|---------|--------|------|---------------------|--------|
| 348.2069  | 1 | C22H26N3O   | 348.2070 | 0.5       | 6.0    | 1       | 100.00 | 11.5 | even                | ok     |

**Figure S14.** High Resolution Mass Spectrometry (HRMS) data of **1a**.

## Eager 300 Report

Page: 1 Sample: PG-APP-09-217-2 (PG-APP-09-217-2)



Method Name : PGAPP10012018  
 Method File : D:\CHNS2018\PGAPP10012018.mth  
 Chromatogram : PG-APP-09-217-2  
 Operator ID : Prakash  
 Analysed : 02/19/2018 18:09  
 Sample ID : PG-APP-09-217-2 (# 30)  
 Analysis Type : UnkNown (Area)

Company Name : C.E. Instruments  
 Printed : 1/26/2019 03:08  
 Instrument N. : Instrument #1  
 Sample weight : 1.257

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.

| Element Name | %       | Ret.Time | Area    | BC | Area ratio | K factor    |
|--------------|---------|----------|---------|----|------------|-------------|
| 1            | 0.0000  | 2        | 18439   | FU |            | 0.0000      |
| Nitrogen     | 10.8331 | 41       | 151800  | FU | 16.614450  | .107234E+07 |
| Carbon       | 68.3967 | 63       | 2522079 | FU | 1.00000    | .262772E+07 |
| Hydrogen     | 6.7151  | 181      | 600547  | FU | 4.199636   | .660576E+07 |
| Totals       | 85.9449 |          | 3292865 |    |            |             |

Figure S15. Elemental analysis data of 1a.



PG-ST-01-181-01

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PROCNO 1

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Time\_ 7.20  
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PULPROG zg30  
TD 54274  
SOLVENT CDC13  
NS 6  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2998593 sec  
RG 161  
DW 60.800 usec  
DE 6.50 usec  
TE 297.5 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 14.75 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300095 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

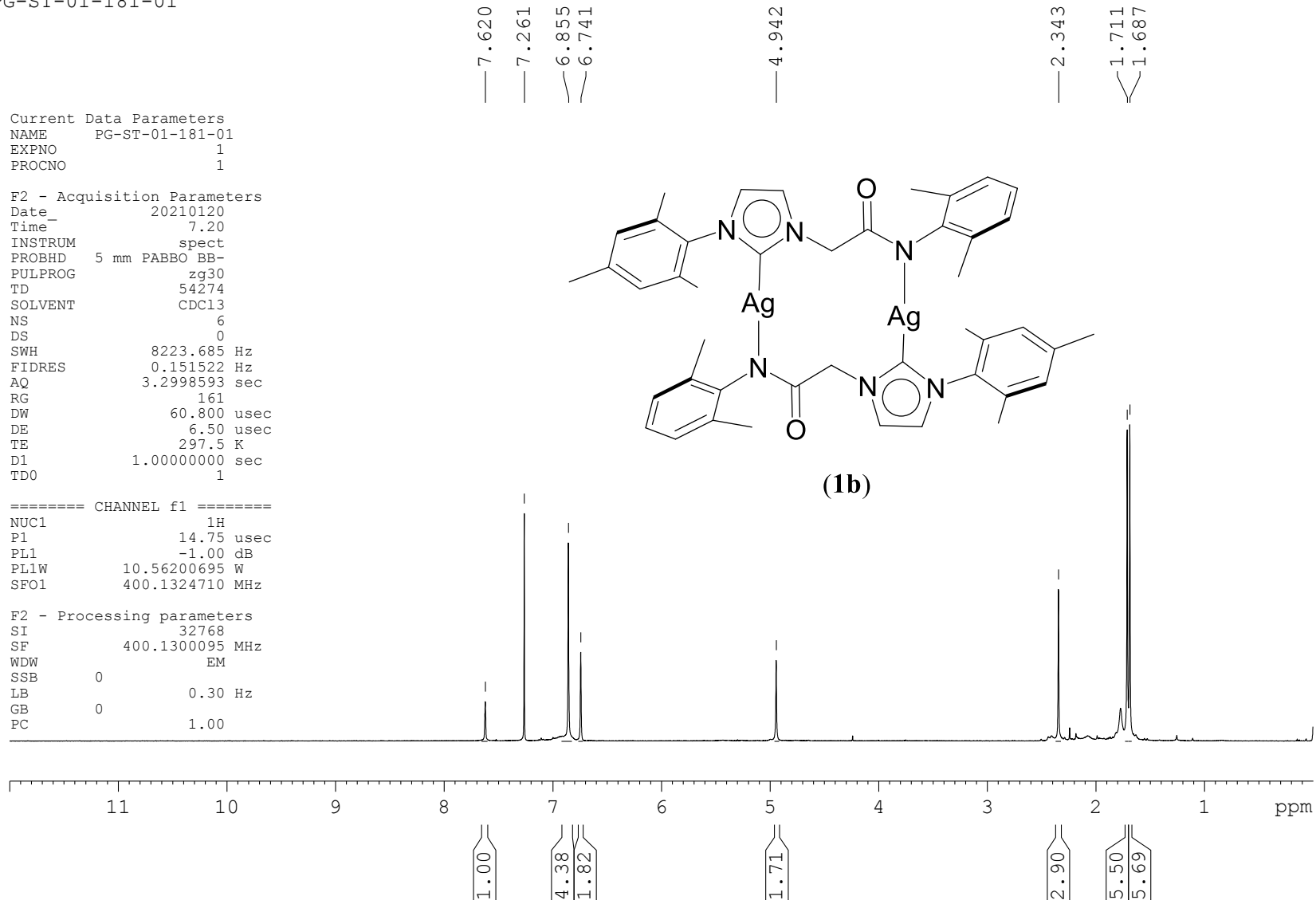
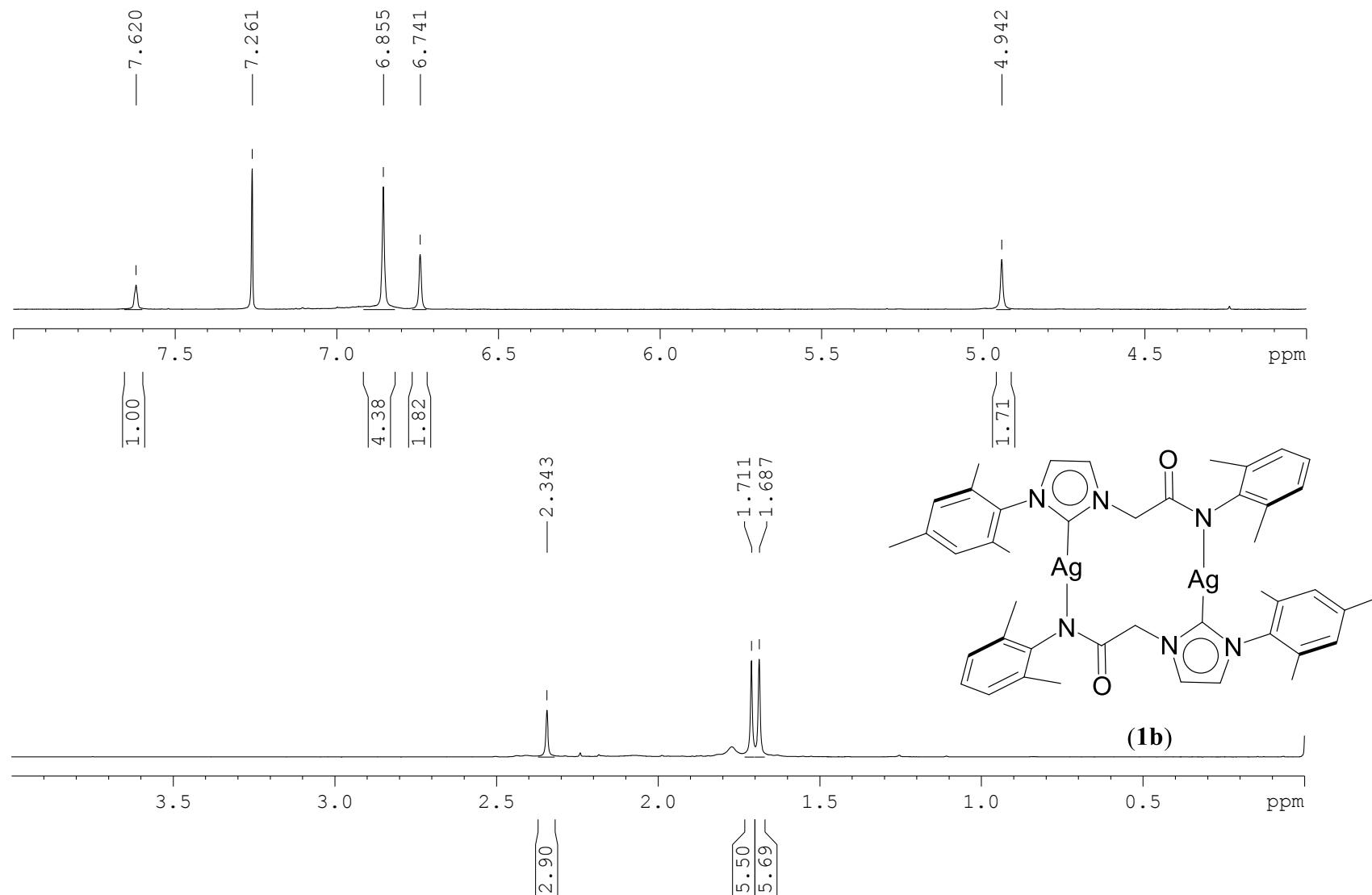


Figure S16.  $^1\text{H}$  NMR spectrum of **1b** in  $\text{CDCl}_3$ .

PG-ST-01-181-01



**Figure S17.** Expanded  $^1\text{H}$  NMR spectrum of **1b** in  $\text{CDCl}_3$ .

PG-APP-09-219-1-13C

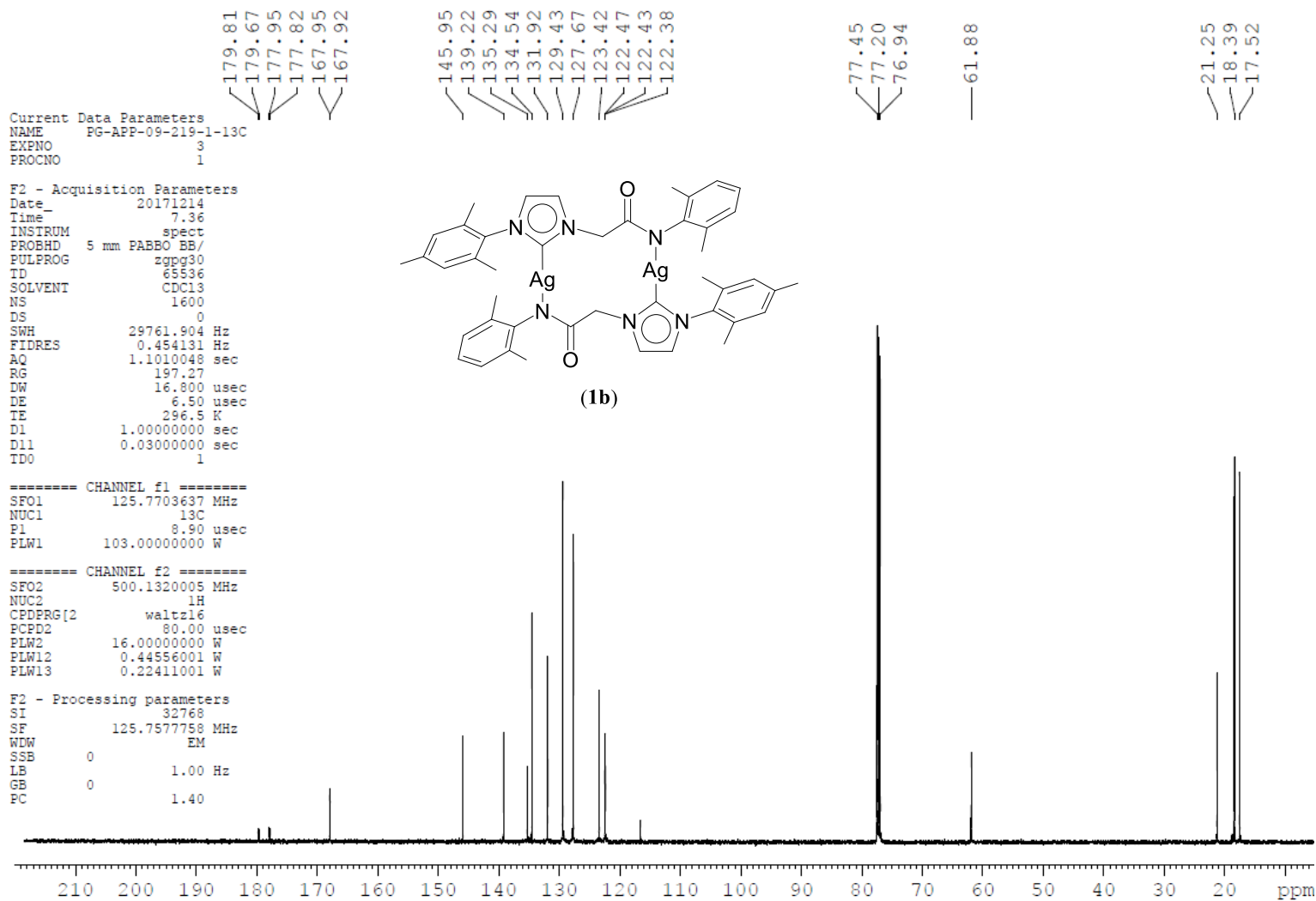
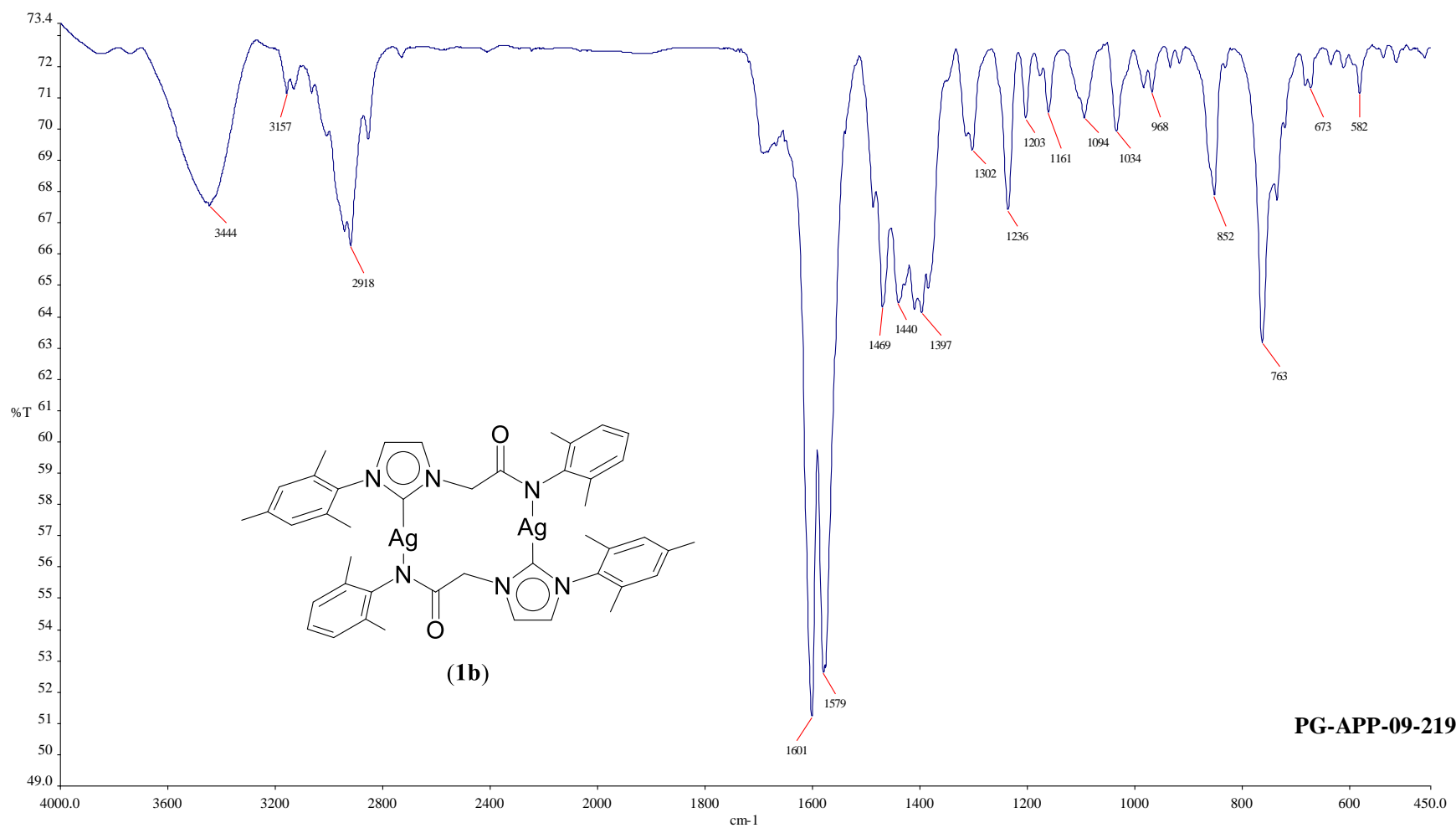


Figure S18.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of **1b** in  $\text{CDCl}_3$ .



PG-APP-09-219

Figure S19. IR spectrum of **1b** in KBr.

SP18022016  
varioMICRO CHNS  
serial number: 15154051

Graphic report

| No. | Weight [mg] | Name              | Method     | N Area | C Area | H Area | N [%] | C [%] | H [%] | Date       | Time  | Info |
|-----|-------------|-------------------|------------|--------|--------|--------|-------|-------|-------|------------|-------|------|
| 19  | 1.4030      | PG-ST-01-205-01-1 | 2mgChem80s | 8 047  | 23 159 | 7 958  | 9.23  | 58.24 | 5.165 | 21-09-2021 | 16:04 | Su   |

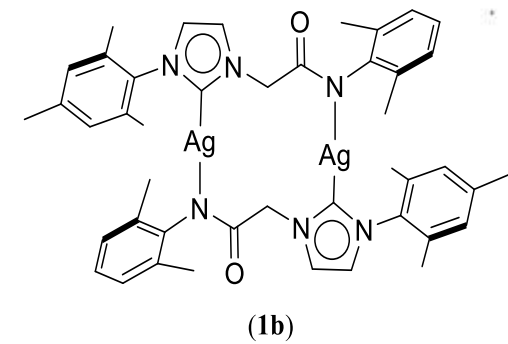
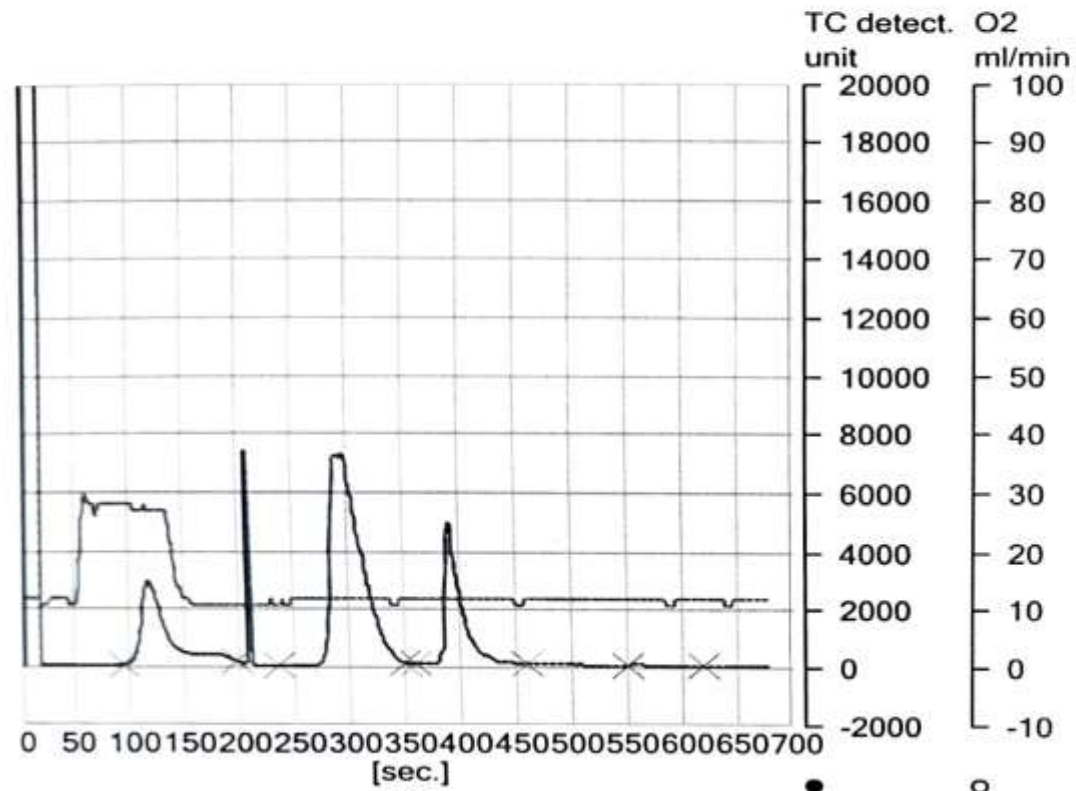


Figure S20. Elemental analysis data of **1b**.

PG-ST-01-150-01

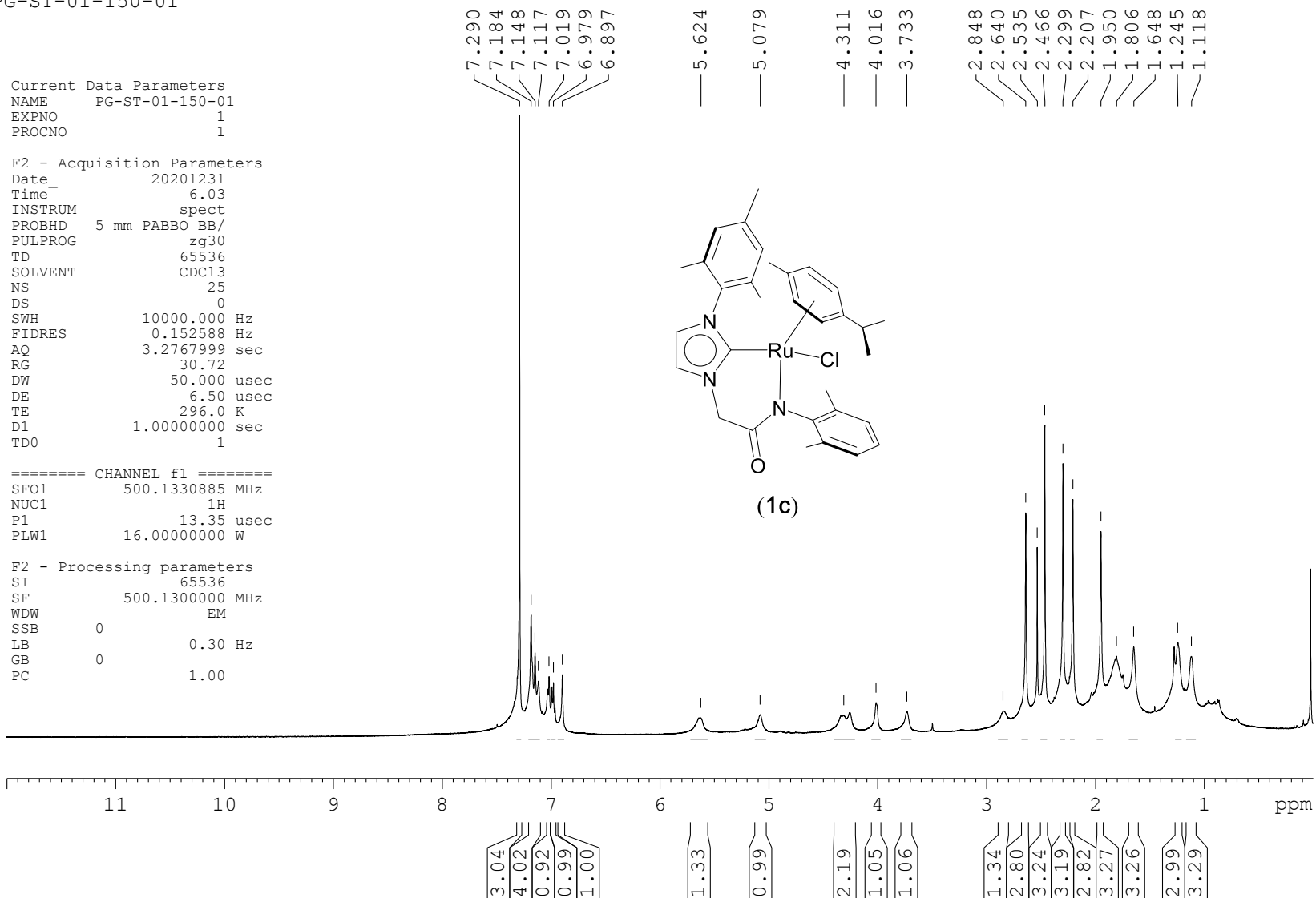
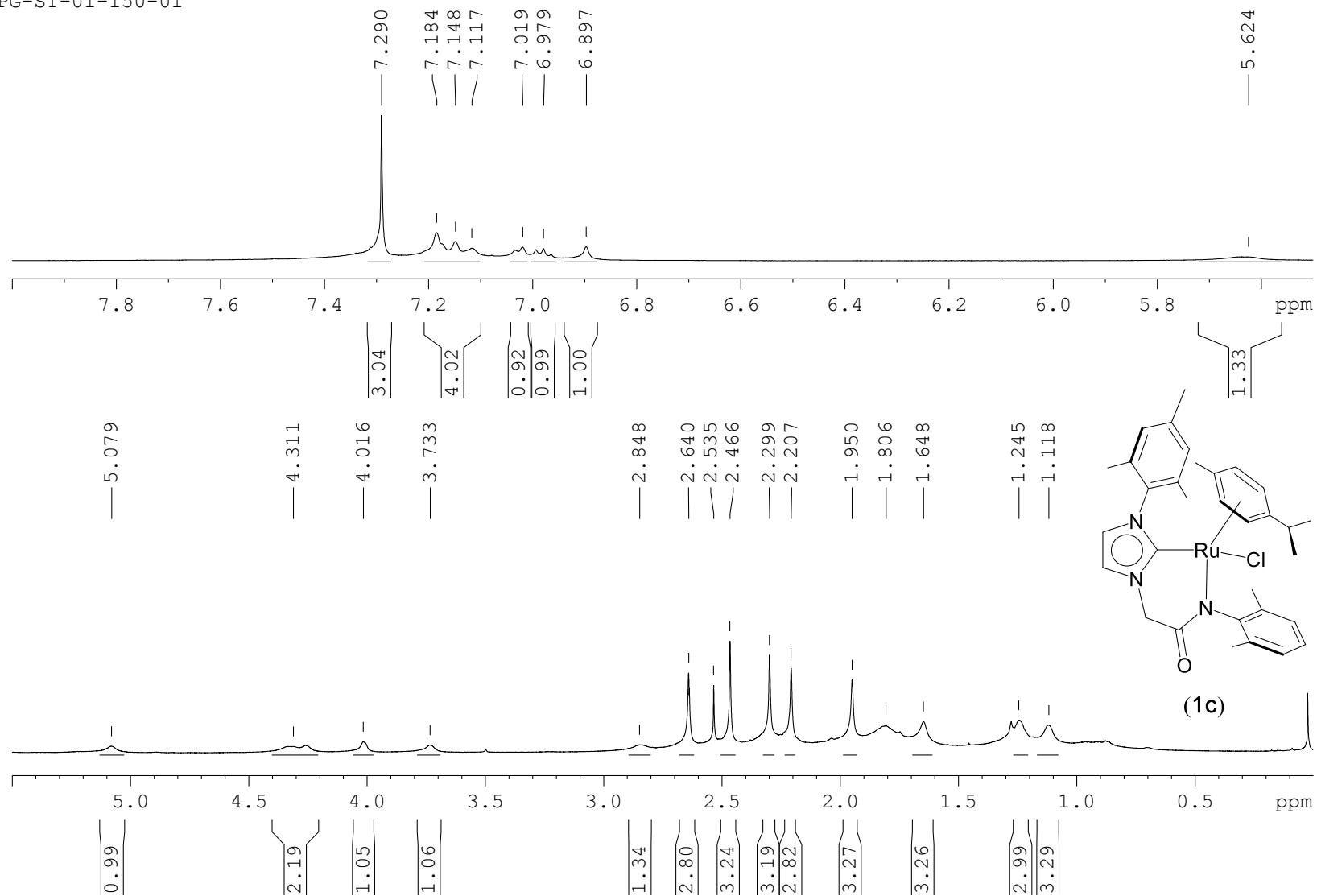


Figure S21. <sup>1</sup>H NMR spectrum of **1c** in CDCl<sub>3</sub>.

PG-ST-01-150-01



**Figure S22.** Expanded  $^1\text{H}$  NMR spectrum of **1c** in  $\text{CDCl}_3$ .

PG-APP-08-128-1-13C

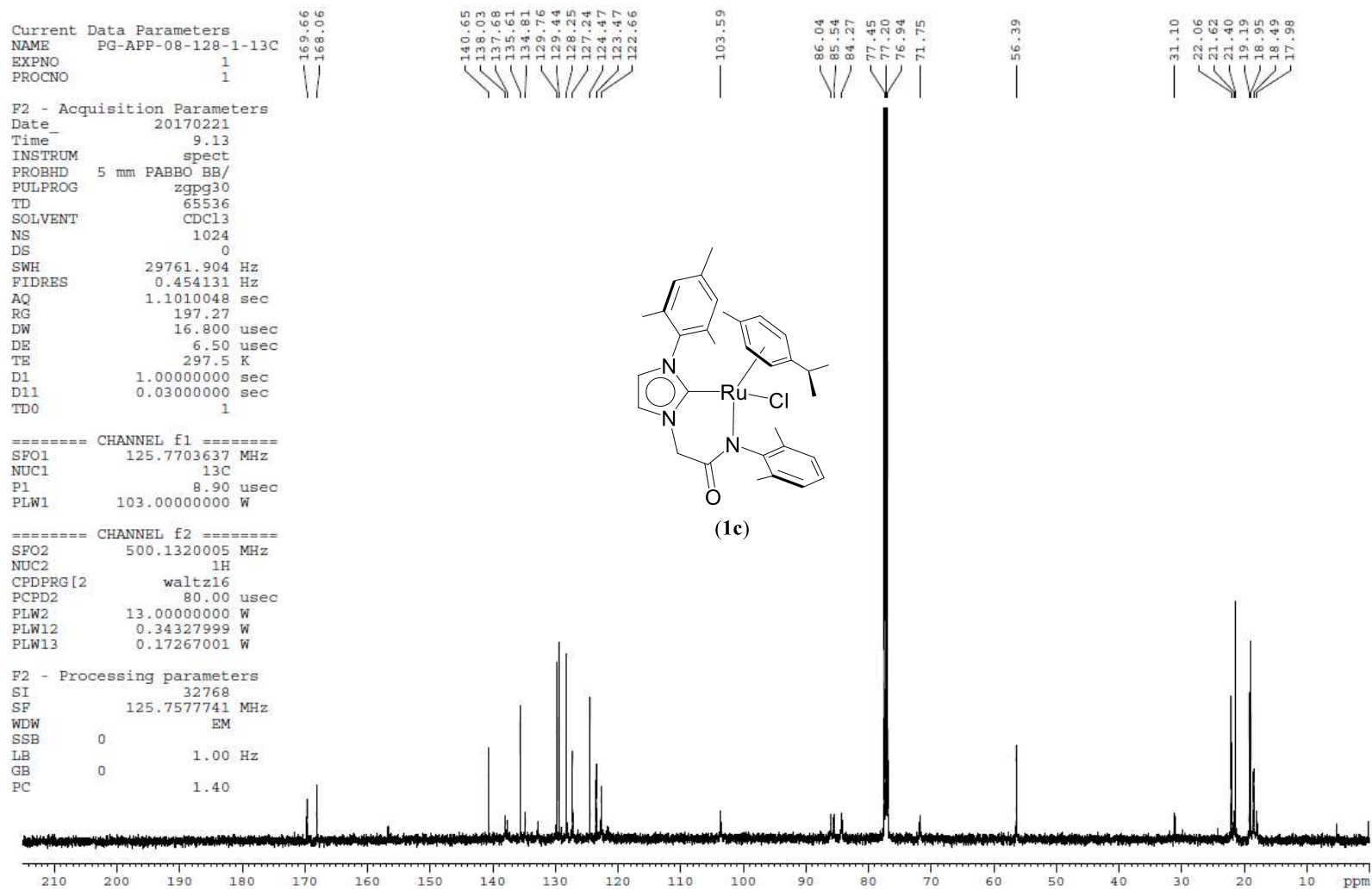
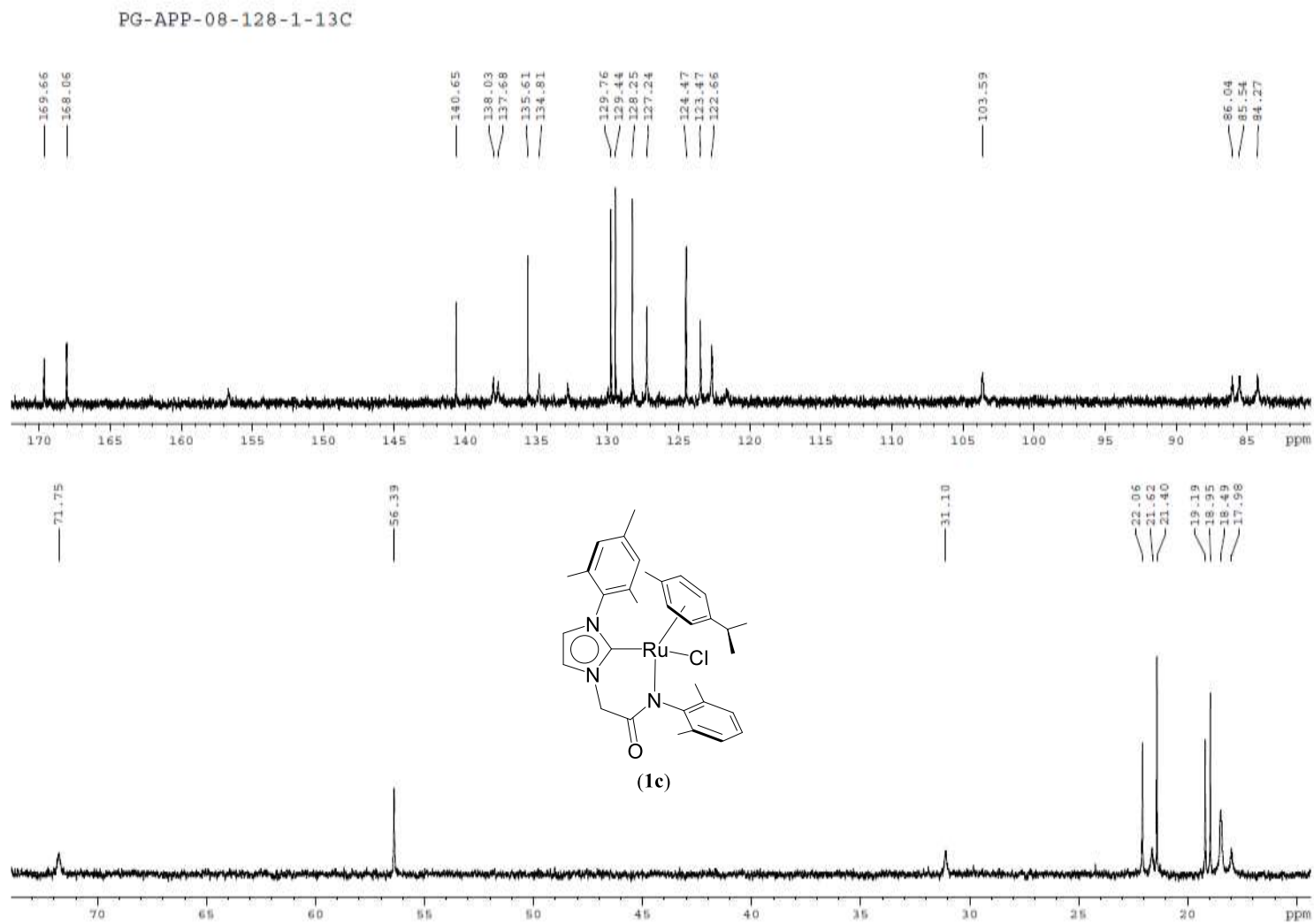
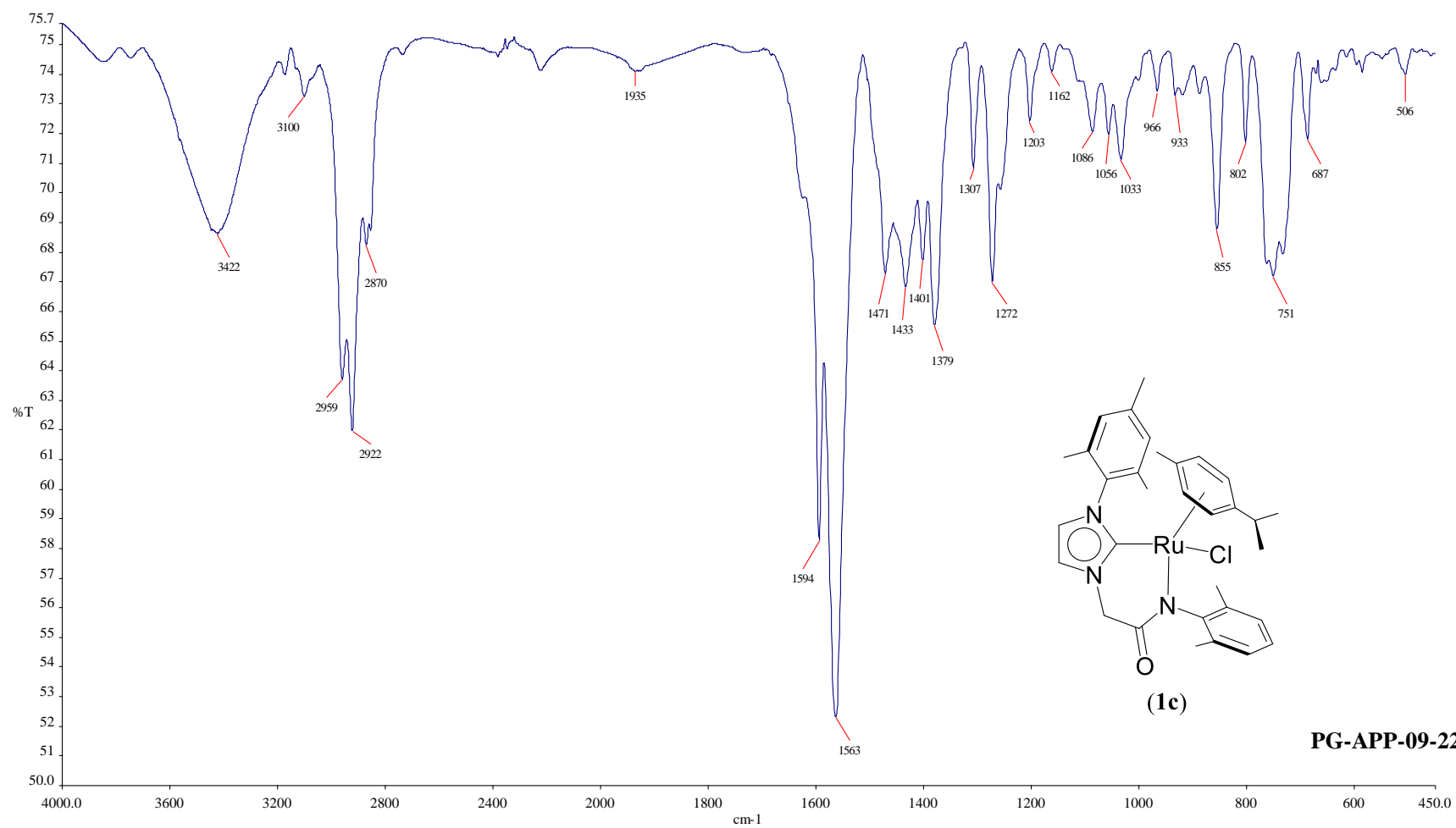


Figure S23.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of **1c** in  $\text{CDCl}_3$ .





**Figure S24.** Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of **1c** in  $\text{CDCl}_3$ .

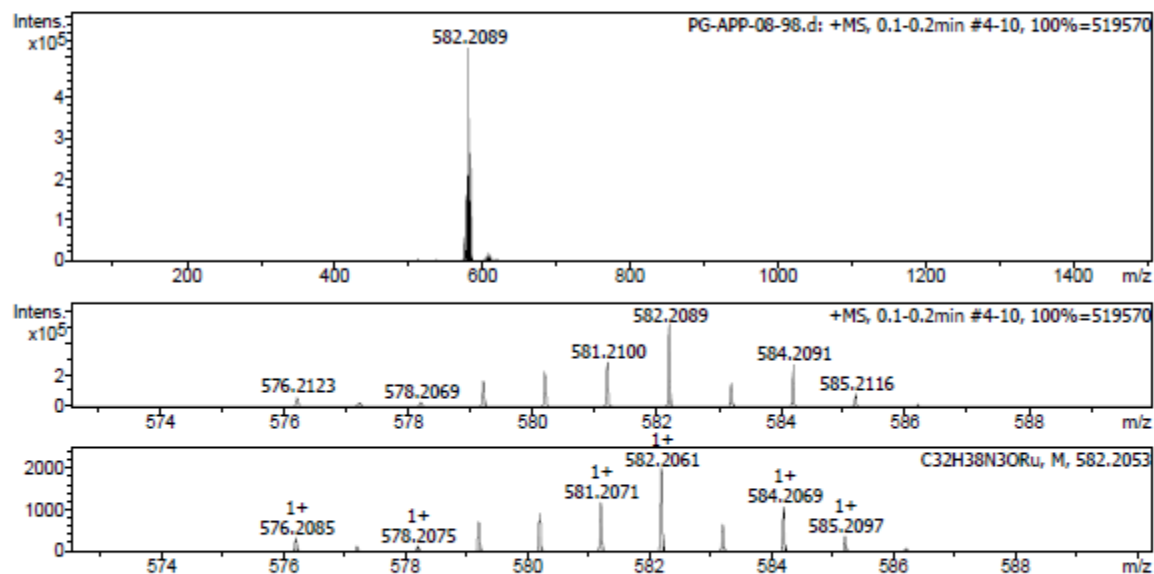
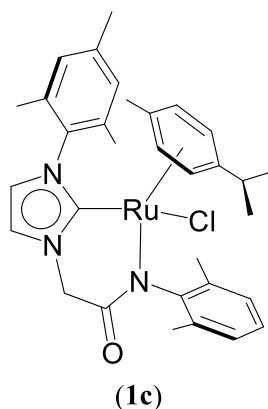


**Figure S25.** IR spectrum of **1c** in KBr.

**DEPARTMENT OF CHEMISTRY, I.I.T.(B)**

|                      |                                 |                                       |                           |
|----------------------|---------------------------------|---------------------------------------|---------------------------|
| <b>Analysis Info</b> |                                 | Acquisition Date 2/23/2017 3:59:06 PM |                           |
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| Method               | Tune_pos_NAF-1500.m             | Instrument                            | maXis impact 282001.00081 |
| Sample Name          | PG-APP-08-98                    |                                       |                           |
| Comment              | C32H38N3ORuCl                   |                                       |                           |

|                              |          |                       |            |                  |           |
|------------------------------|----------|-----------------------|------------|------------------|-----------|
| <b>Acquisition Parameter</b> |          |                       |            |                  |           |
| Source Type                  | ESI      | Ion Polarity          | Positive   | Set Nebulizer    | 0.5 Bar   |
| Focus                        | Active   | Set Capillary         | 3700 V     | Set Dry Heater   | 180 °C    |
| Scan Begin                   | 50 m/z   | Set End Plate Offset  | -500 V     | Set Dry Gas      | 4.0 l/min |
| Scan End                     | 1500 m/z | Set Collision Cell RF | 2100.0 Vpp | Set Divert Valve | Source    |



| Meas. m/z | # | Ion Formula | m/z      | err [ppm] | mSigma | # Sigma | Score  | rdb  | e <sup>-</sup> Conf | N-Rule |
|-----------|---|-------------|----------|-----------|--------|---------|--------|------|---------------------|--------|
| 582.2089  | 1 | C32H38N3ORu | 582.2062 | 4.6       | 30.4   | 1       | 100.00 | 16.0 | odd                 | -      |

**Figure S26.** High Resolution Mass Spectrometry (HRMS) data of **1c**.

Document: SP-29-01-2021 (varioMICRO) from: 30-01-2021 03:34:43

SP18022016  
varioMICRO CHNS  
serial number: 15154051

Graphic report

| No. | Weight [mg] | Name           | Method     | N Area | C Area | H Area | S Area | N [%] | C [%] | H [%] | S [%] | Date       | Time  |
|-----|-------------|----------------|------------|--------|--------|--------|--------|-------|-------|-------|-------|------------|-------|
| 21  | 1.4520      | PG-ST-01-158-1 | 2mgChem80s | 6 192  | 22 989 | 8 612  | 27     | 6.04  | 55.76 | 5.970 | 0.000 | 29-01-2021 | 17:14 |

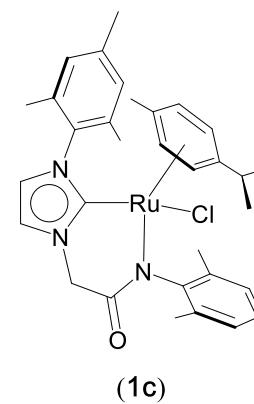
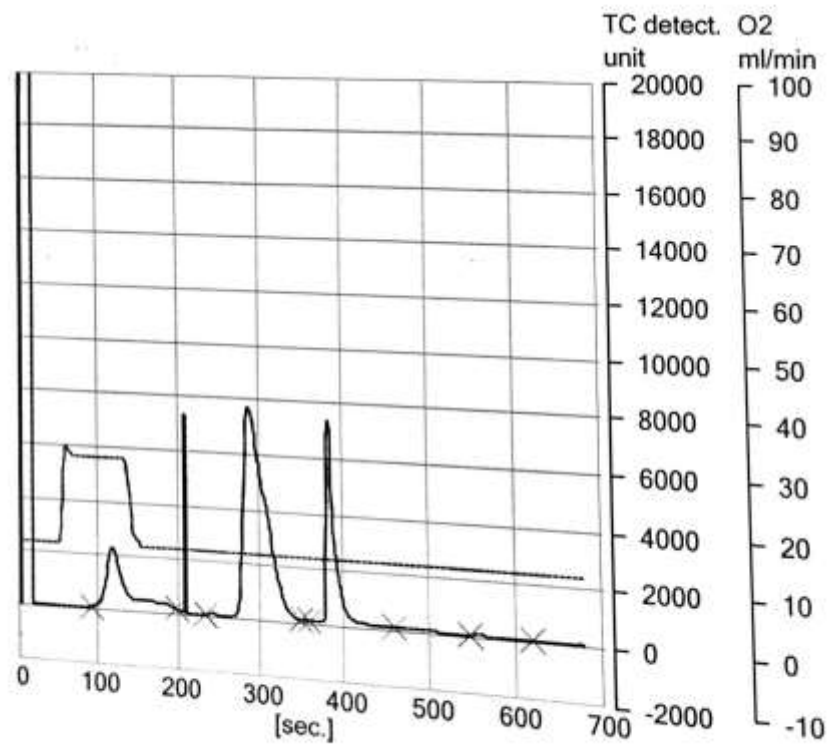
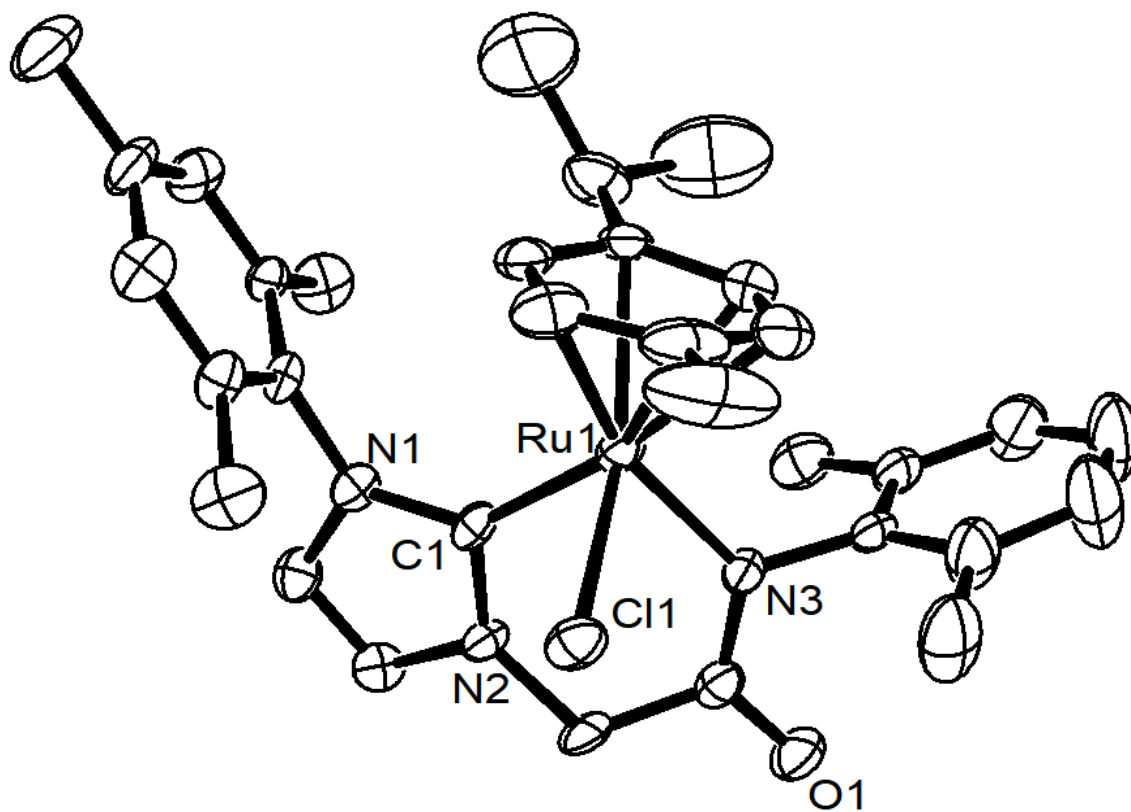


Figure S27. Elemental analysis data of 1c.



**Figure S28.** ORTEP of **1c** with thermal ellipsoids shown at the 50 % probability level. Hydrogen atoms were omitted for clarity. Selected bond lengths (Å) and angles (°): Ru(1)–C(1) 2.087(5), Ru(1)–Cl(1) 2.4299(14), Ru(1)–N(3) 2.153(4), C(1)–N(1) 1.367(6), C(1)–N(2) 1.366(6), C(14)–N(3) 1.330(7), C(1)–Ru(1)–Cl(1) 84.22(15), Cl(1)–Ru(1)–N(3) 87.64(12), N(3)–Ru(1)–C(1) 84.94(19), N(1)–C(1)–Ru(1) 134.6(4), N(2)–C(1)–Ru(1) 122.3(4), N(1)–C(1)–N(2) 103.0(4).

PG-APP-09-210-1-1H

Current Data Parameters  
NAME PG-APP-09-210-1-1H  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20171207  
Time 22.31  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 85.91  
DW 50.000 usec  
DE 6.50 usec  
TE 298.0 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO1 500.1330885 MHz  
NUC1 1H  
P1 13.35 usec  
PLW1 16.00000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1300090 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

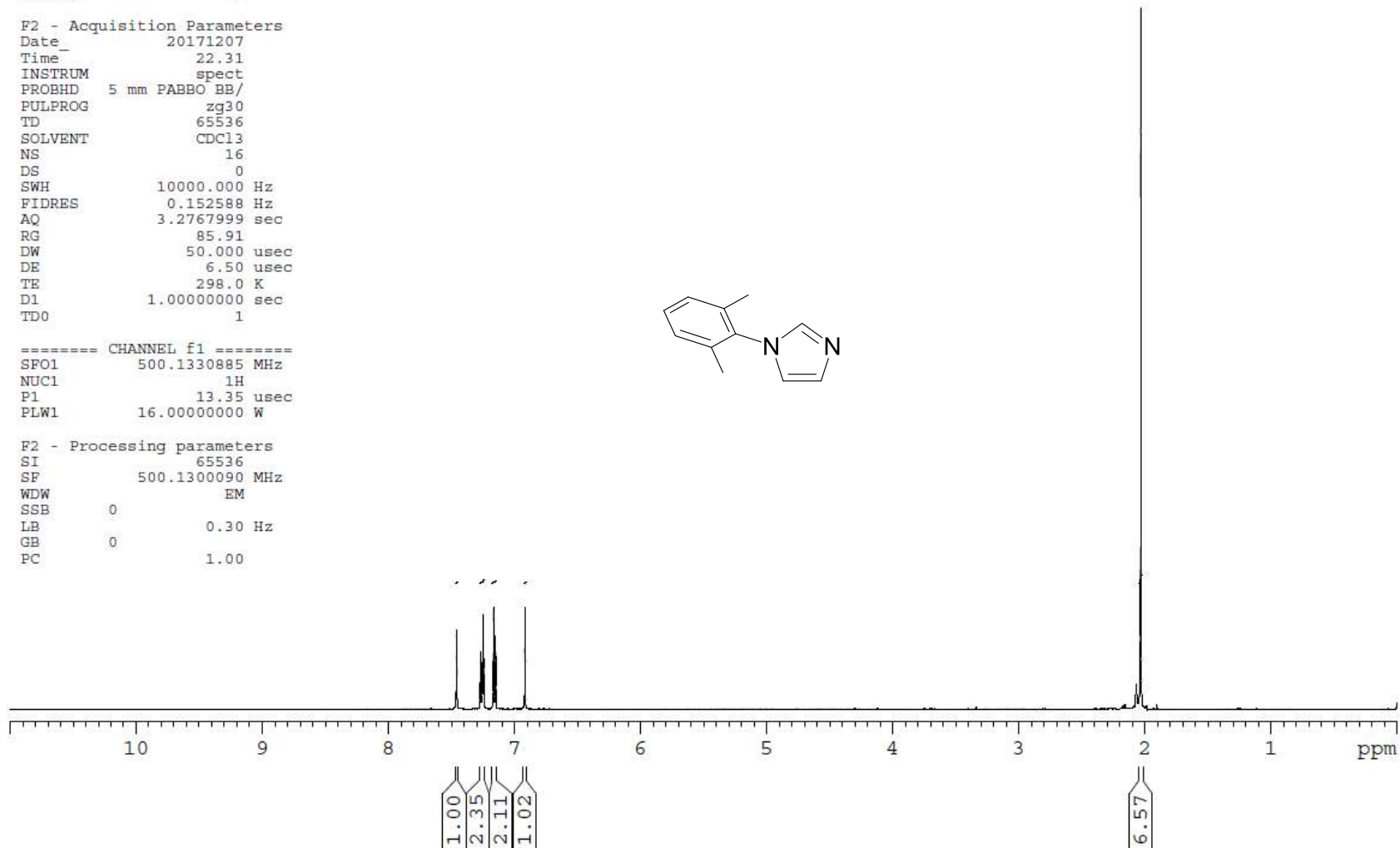
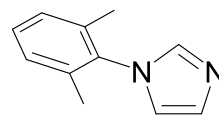
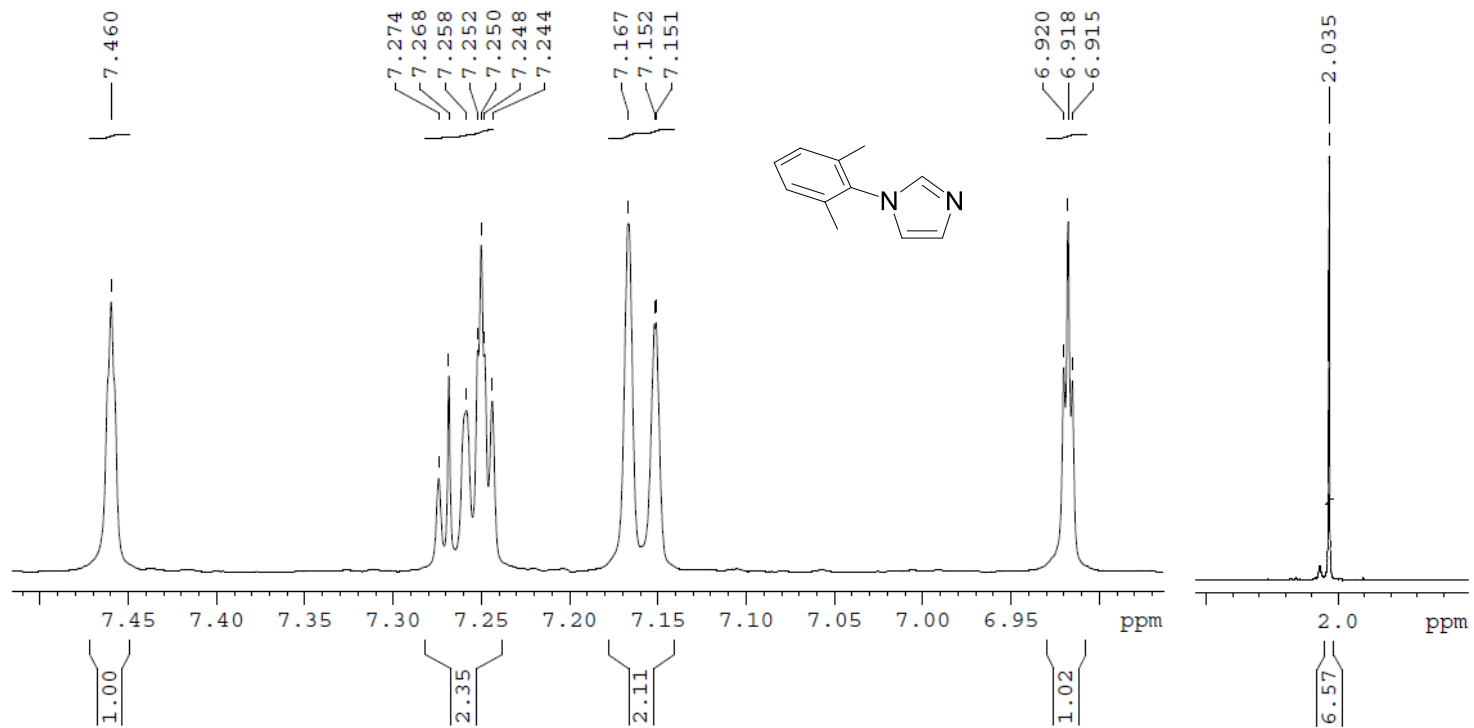


Figure S29. <sup>1</sup>H NMR spectrum of 1-(2,6-Me<sub>2</sub>-phenyl)imidazole in CDCl<sub>3</sub>.



**Figure S30.** Expanded <sup>1</sup>H NMR spectrum of 1-(2,6-Me<sub>2</sub>-phenyl)imidazole in CDCl<sub>3</sub>.

PG-APP-09-210-1-13C

Current Data Parameters  
NAME PG-APP-09-210-1-13C  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20171207  
Time\_ 22.33  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 150  
DS 0  
SWH 29761.904 Hz  
FIDRRS 0.454131 Hz  
AQ 1.1010048 sec  
RG 197.27  
DW 16.800 usec  
DE 6.50 usec  
TE 298.1 K  
D1 1.00000000 sec  
D11 0.03000000 sec  
TD0 1

----- CHANNEL f1 -----  
SFO1 125.7703637 MHz  
NUC1 13C  
P1 8.90 usec  
PLW1 103.00000000 W

----- CHANNEL F2 -----  
SFO2 500.1320005 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 80.00 usec  
PLW2 16.00000000 W  
PLW12 0.44556001 W  
PLW13 0.22411001 W

F2 - Processing parameters  
SI 32768  
SF 125.7577699 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

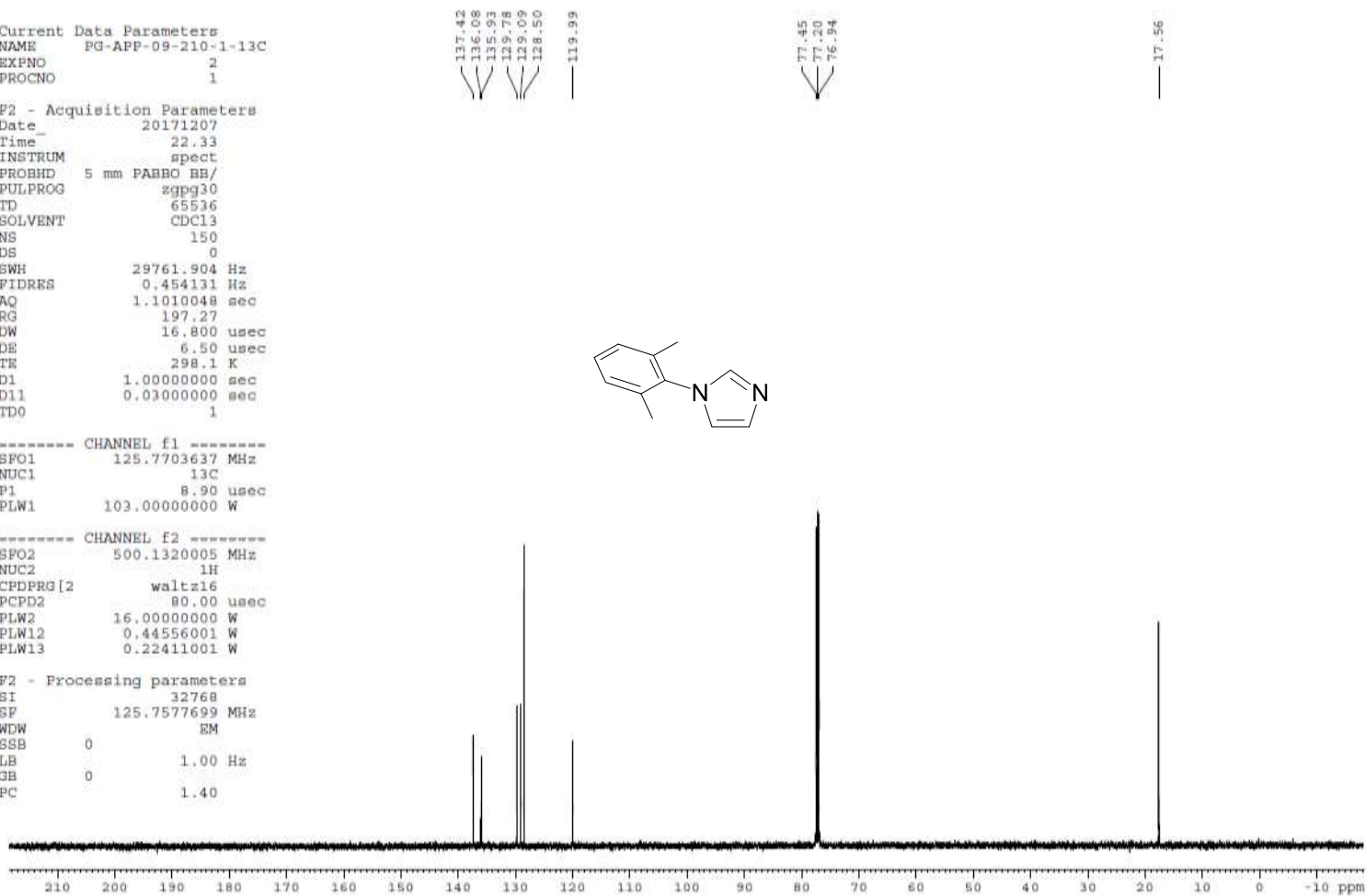
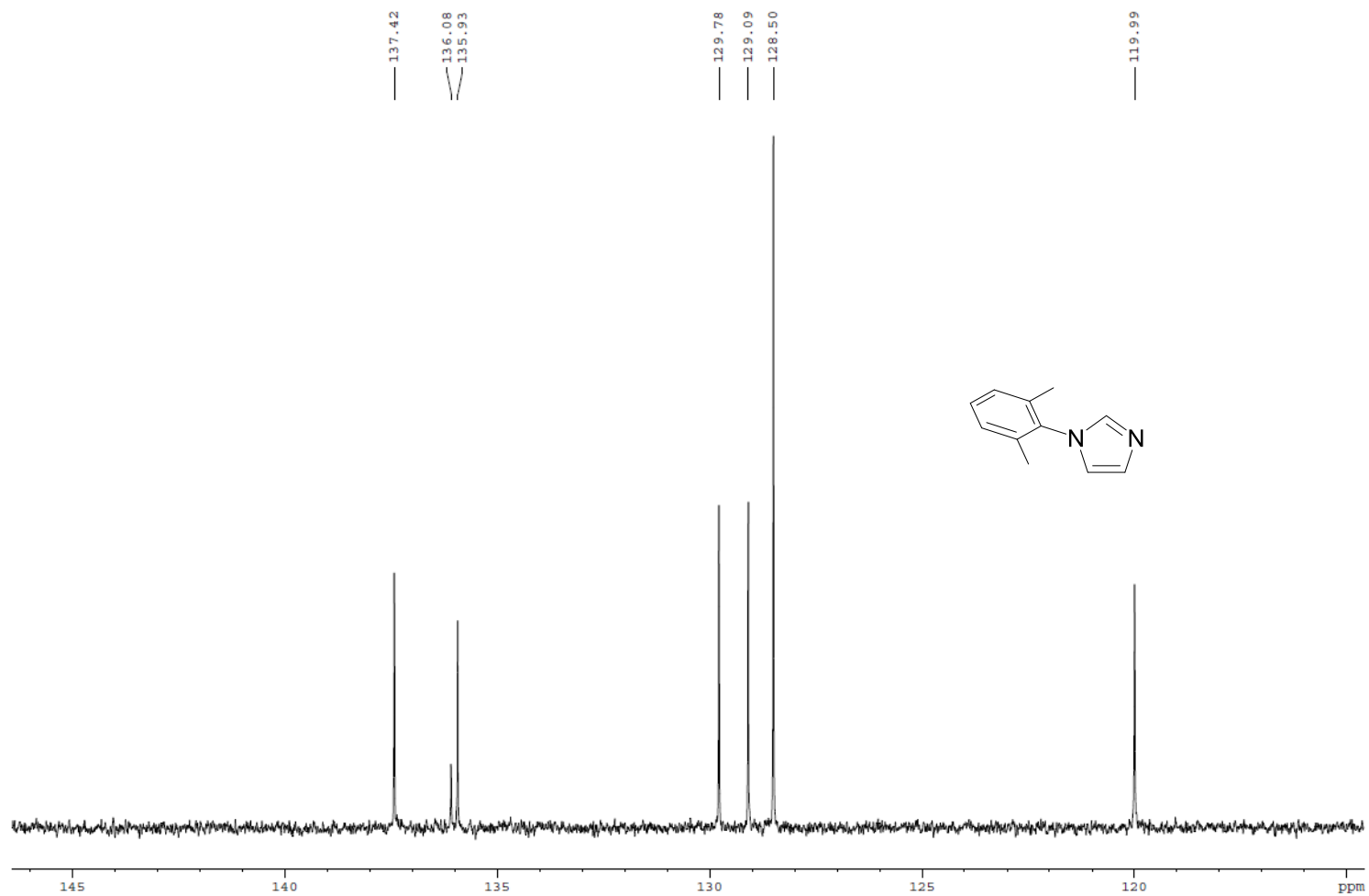


Figure S31.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of 1-(2,6-Me<sub>2</sub>-phenyl)imidazole in CDCl<sub>3</sub>.



PG-APP-09-210-1-13C



**Figure S32.** Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of 1-(2,6- $\text{Me}_2$ -phenyl)imidazole in  $\text{CDCl}_3$ .

NAME PG-APP-10-46-1-1  
 EXPNO 1  
 PROCNO 1  
 Date\_ 20180204  
 Time 8.32  
 INSTRUM spect  
 PROBHD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 54274  
 SOLVENT CDCl3  
 NS 16  
 DS 0  
 SWH 8223.685 Hz  
 FIDRES 0.151522 Hz  
 AQ 3.2999091 sec  
 RG 228  
 DW 60.800 usec  
 DE 6.50 usec  
 TE 296.6 K  
 D1 1.0000000 sec  
 TD0 1

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 14.75 usec  
 PL1 -1.00 dB  
 PL1W 10.56200695 W  
 SFO1 400.1324710 MHz  
 SI 32768  
 SF 400.1300101 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

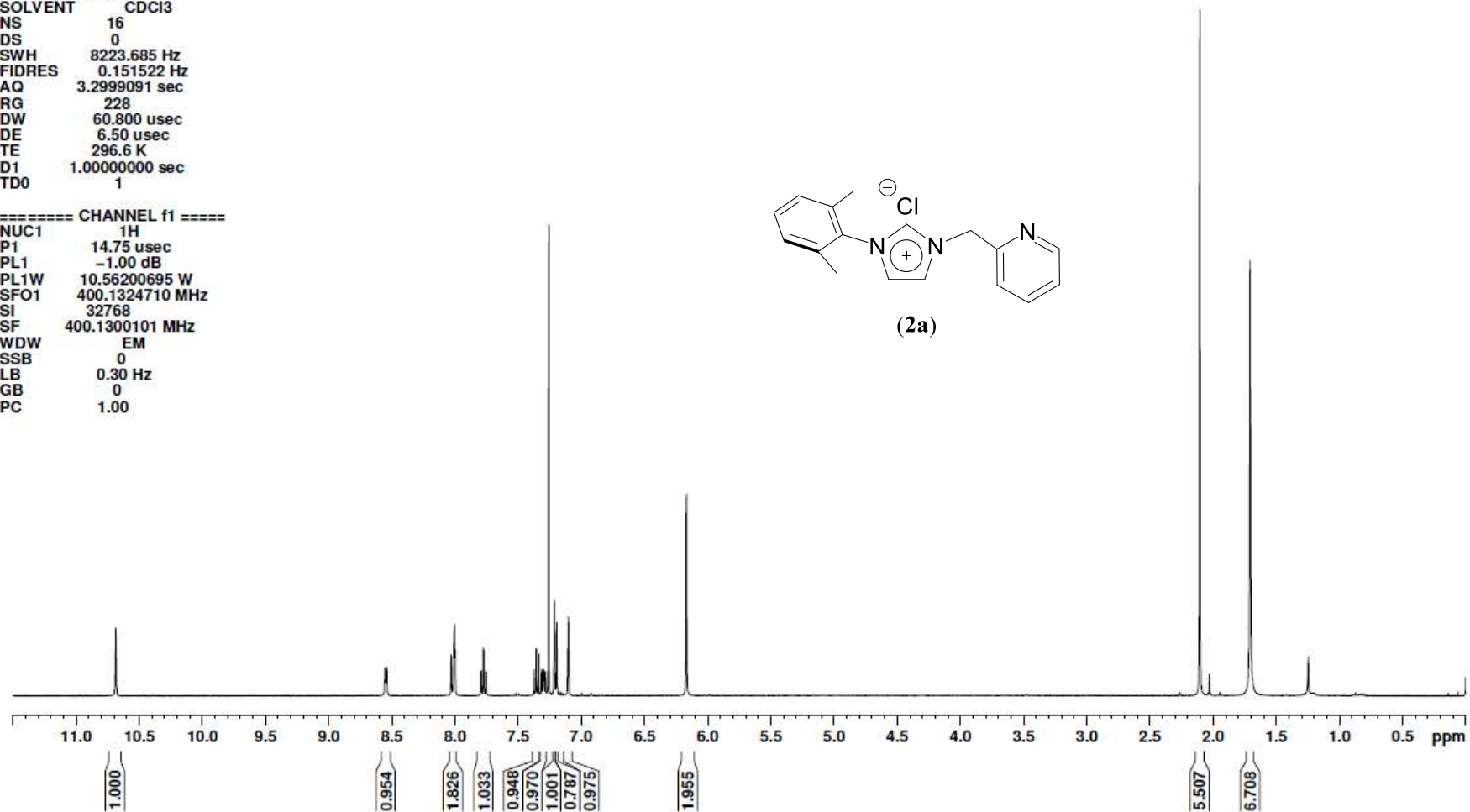
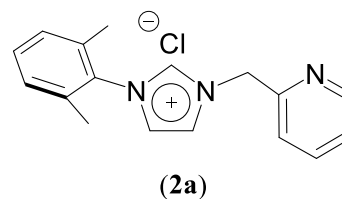


Figure S33. <sup>1</sup>H NMR spectrum of **2a** in CDCl<sub>3</sub>.

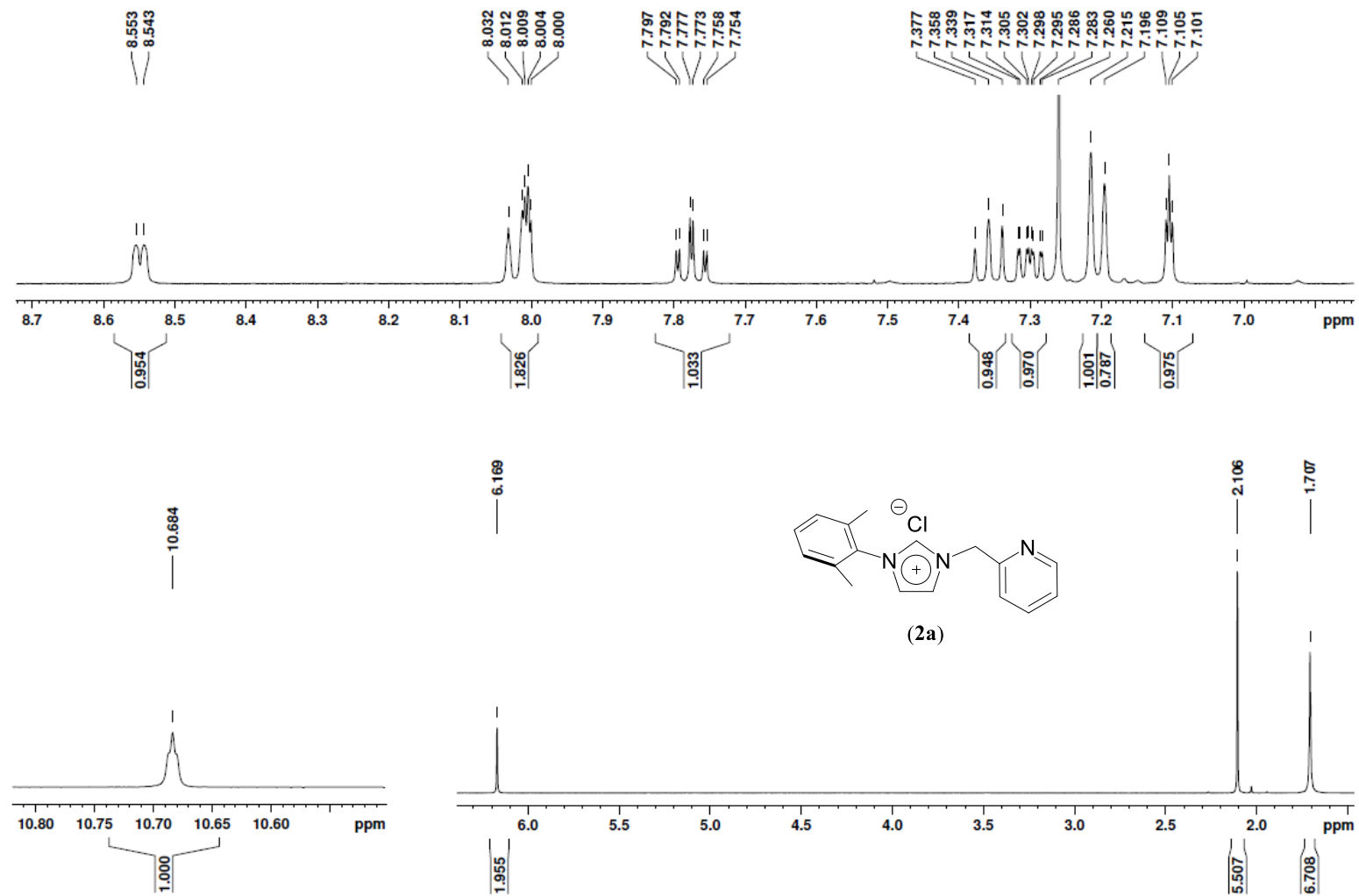


Figure S34. Expanded  $^1\text{H}$  NMR spectrum of **2a** in  $\text{CDCl}_3$ .

PG-APP-09-230-1-13C

NAME PG-APP-09-230-1-13C  
EXPNO 6  
PROCNO 1  
Date\_ 20180115  
Time 13.05  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zpgpg30  
TD 65536  
SOLVENT CDCl3  
NS 200  
DS 0  
SWH 26041.666 Hz  
FIDRES 0.397364 Hz  
AQ 1.2583412 sec  
RG 2050  
DW 19.200 usec  
DE 6.50 usec  
TE 298.0 K  
D1 1.00000000 sec  
D11 0.03000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 8.50 usec  
PL1 -2.00 dB  
PL1W 56.53121948 W  
SFO1 100.6238364 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -1.00 dB  
PL12 13.69 dB  
PL13 14.50 dB  
PL2W 10.56200695 W  
PL12W 0.35871249 W  
PL13W 0.29767781 W  
SFO2 400.1316005 MHz  
SI 32768  
SF 100.6127636 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

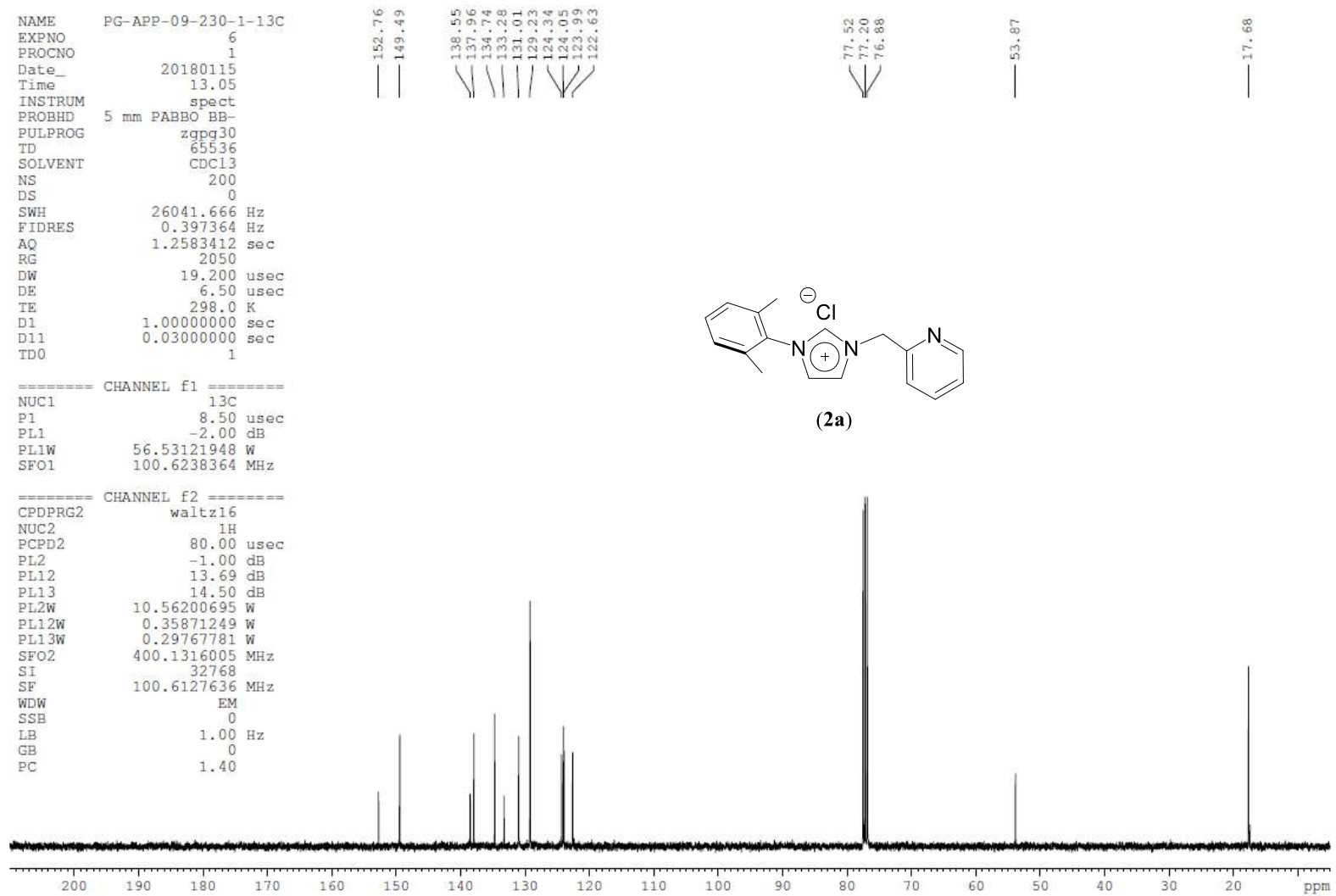
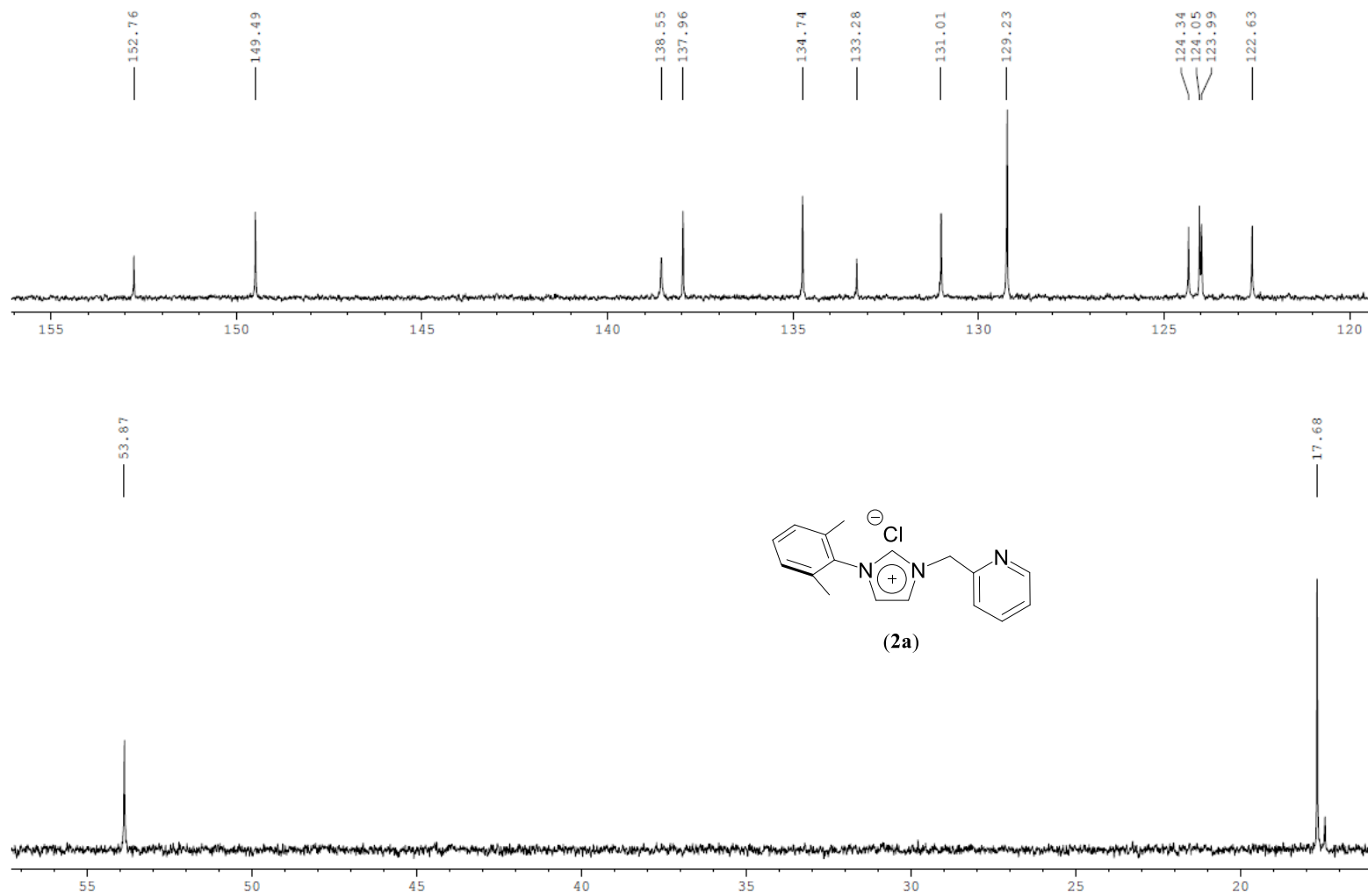
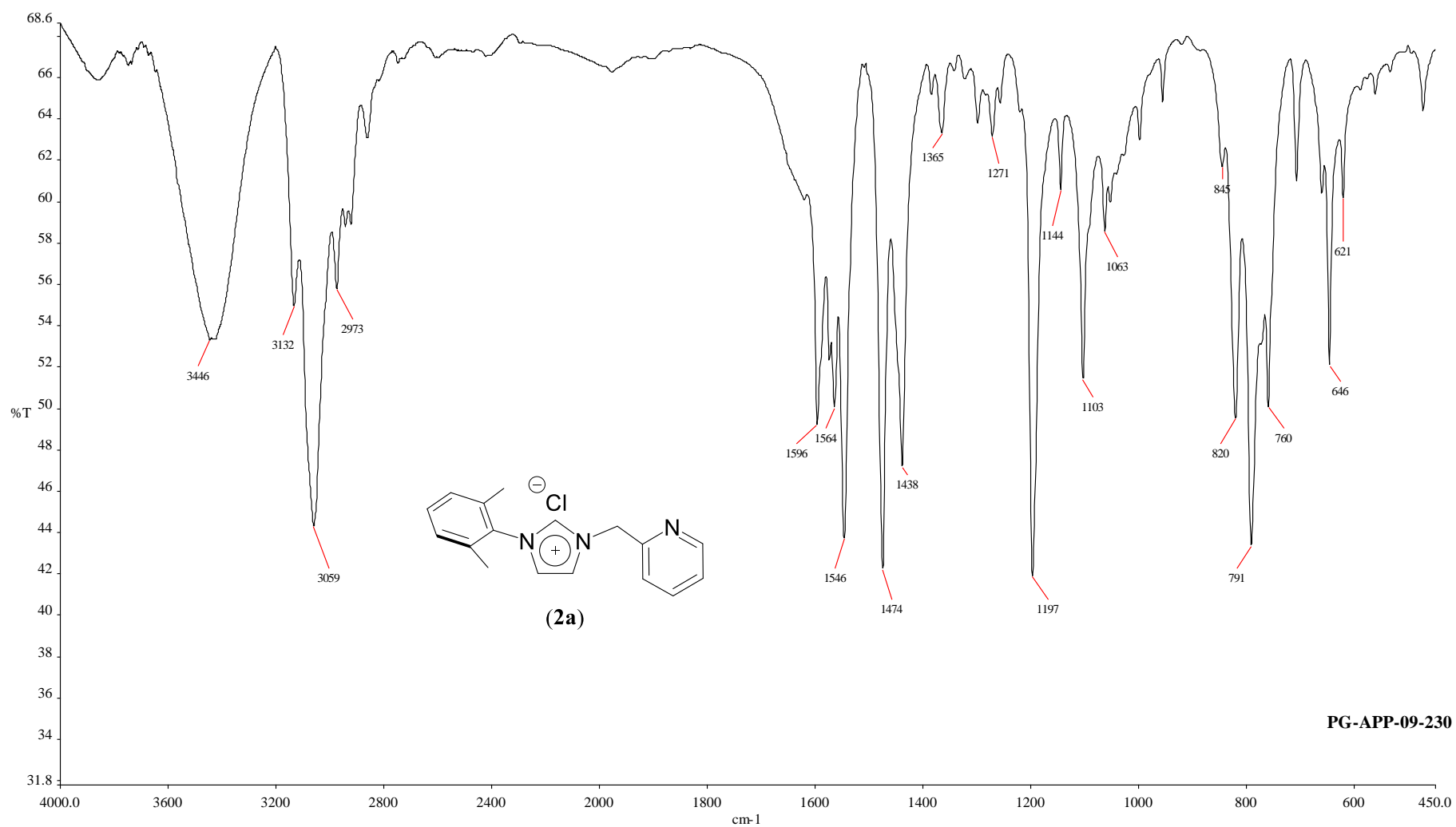


Figure S35.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of **2a** in  $\text{CDCl}_3$ .

PG-APP-09-230-1-13C



**Figure S36.** Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of **2a** in  $\text{CDCl}_3$ .



PG-APP-09-230

PG-APP-10-15

Figure S37. IR spectrum of **2a** in KBr.

**DEPARTMENT OF CHEMISTRY, I.I.T.(B)**

**Analysis Info**

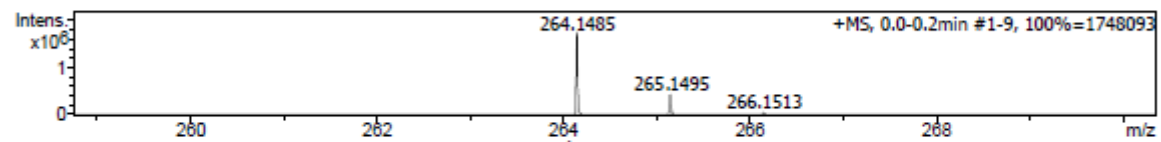
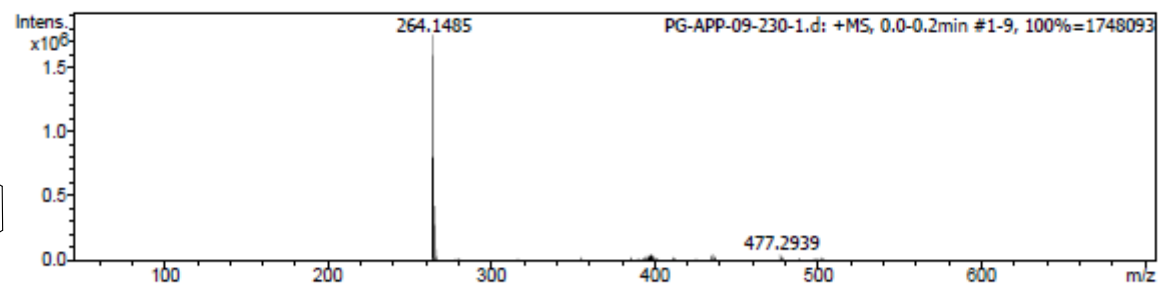
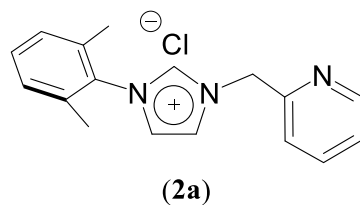
Analysis Name D:\Data\FEB-2018\PG-APP-09-230-1.d  
 Method Tune\_pos\_NAICSI-1000A.m  
 Sample Name PG-APP-09-230-1  
 Comment C17H18N3Cl

Acquisition Date 2/1/2018 1:01:52 PM

Operator PG APP IN  
 Instrument maXis impact 282001.00081

**Acquisition Parameter**

|             |         |                       |            |                  |           |
|-------------|---------|-----------------------|------------|------------------|-----------|
| Source Type | ESI     | Ion Polarity          | Positive   | Set Nebulizer    | 0.4 Bar   |
| Focus       | Active  | Set Capillary         | 3700 V     | Set Dry Heater   | 180 °C    |
| Scan Begin  | 50 m/z  | Set End Plate Offset  | -500 V     | Set Dry Gas      | 4.0 l/min |
| Scan End    | 500 m/z | Set Collision Cell RF | 1500.0 Vpp | Set Divert Valve | Source    |

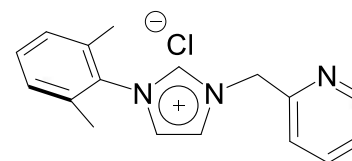


| Meas. m/z | # | Ion Formula | m/z      | err [ppm] | mSigma | # Sigma | Score  | rdb  | e <sup>-</sup> Conf | N-Rule |
|-----------|---|-------------|----------|-----------|--------|---------|--------|------|---------------------|--------|
| 264.1485  | 1 | C17H18N3    | 264.1495 | 3.8       | 24.5   | 1       | 100.00 | 10.5 | even                | ok     |

**Figure S38.** High Resolution Mass Spectrometry (HRMS) data of **2a**.

## Eager 300 Report

Page: 1 Sample: PG-APP-09-230-2 (PG-APP-09-230-2)



(2a)

Method Name : PGAPP29012018R  
 Method File : D:\CHNS2018\PGAPP29012018R.mth  
 Chromatogram : PG-APP-09-230-2  
 Operator ID : Prakash Company Name : C.E. Instruments  
 Analysed : 01/29/2018 14:46 Printed : 2/22/2018 18:23  
 Sample ID : PG-APP-09-230-2 (# 16) Instrument N. : Instrument #1  
 Analysis Type : UnkNown (Area) Sample weight : .987

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.

| Element Name | %       | Ret.Time | Area    | BC | Area ratio | K factor    |
|--------------|---------|----------|---------|----|------------|-------------|
| 1            | 0.0000  | 2        | 21525   | FU |            | 0.0000      |
| 2            | 0.0000  | 6        | 115783  | FU |            | 0.0000      |
| Nitrogen     | 13.7310 | 41       | 138189  | RS | 12.522860  | .101966E+07 |
| Carbon       | 67.6290 | 64       | 1730515 | RS | 1.000000   | .259254E+07 |
| Hydrogen     | 5.8837  | 181      | 370805  | RS | 4.666914   | .638521E+07 |
| Totals       | 87.2437 |          | 2376817 |    |            |             |

Figure S39. Elemental analysis data of 2a.



PG-ST-01-90-01-1H

Current Data Parameters  
NAME PG-ST-01-90-01-1H  
EXPNO 5  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20201126  
Time\_ 7.15  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 98.91  
DW 50.000 usec  
DE 6.50 usec  
TE 299.2 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 500.1330885 MHz  
NUC1 1H  
P1 13.35 usec  
PLW1 16.00000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1300000 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

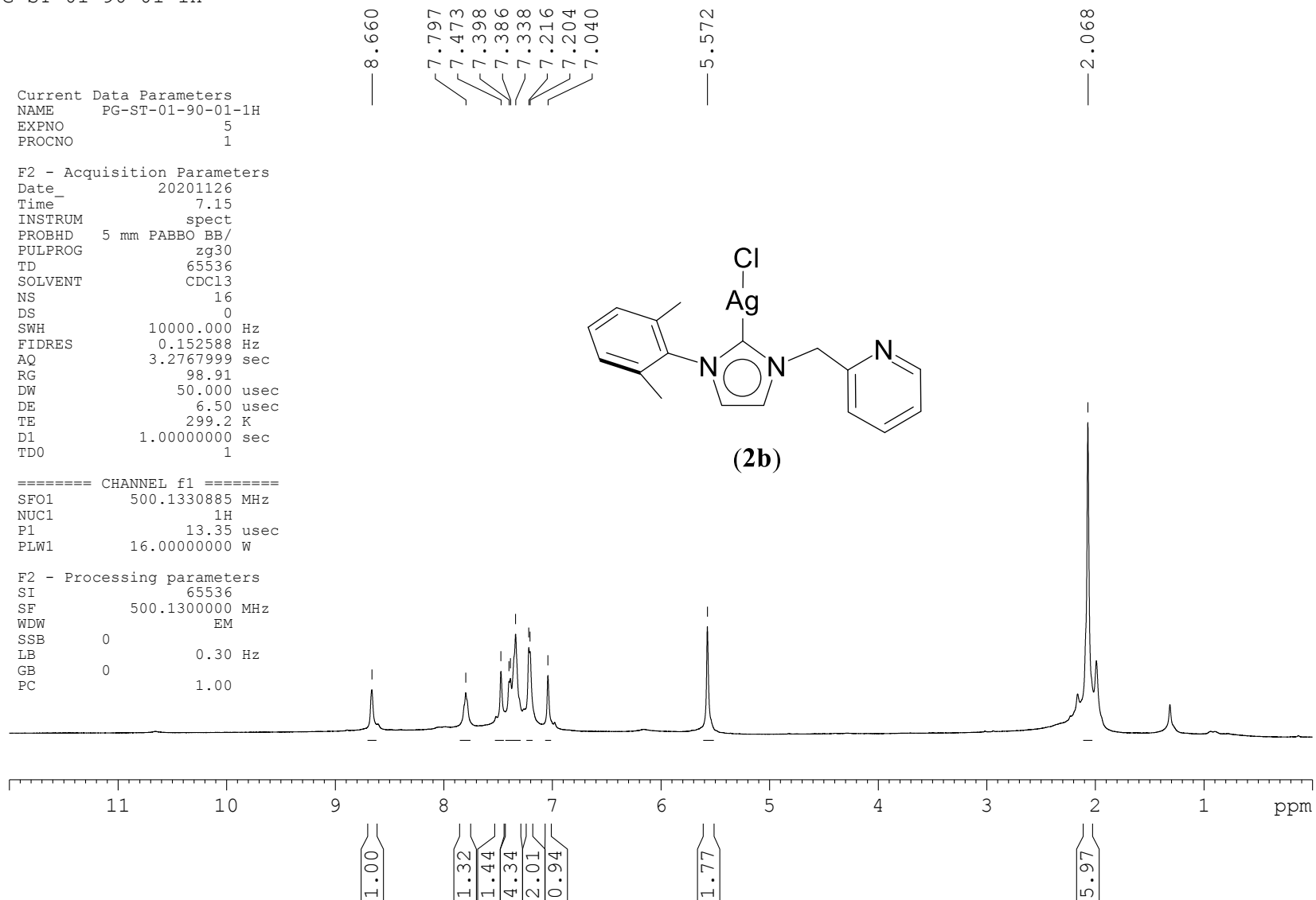
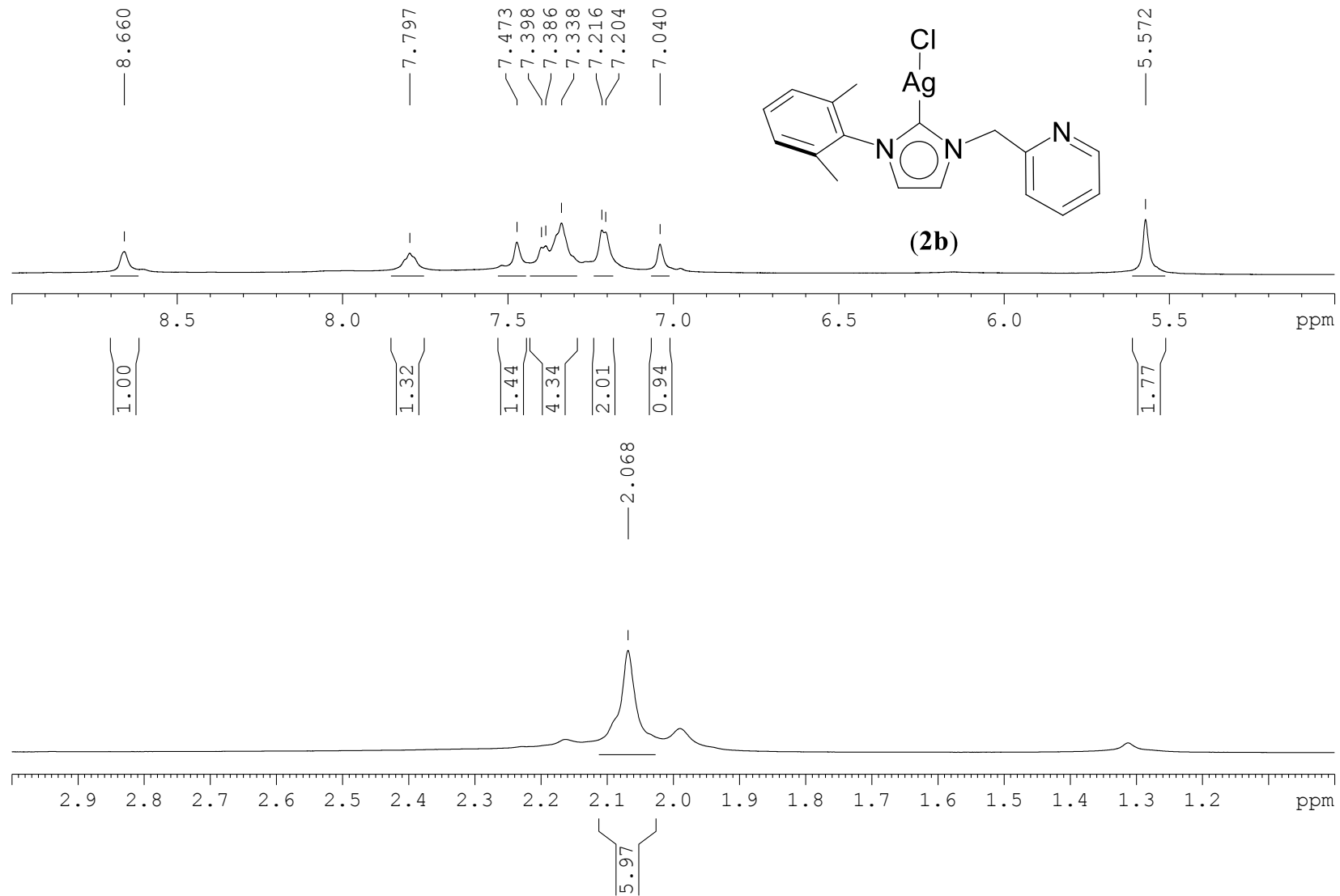


Figure S40. <sup>1</sup>H NMR spectrum of **2b** in CDCl<sub>3</sub>.

PG-ST-01-90-01-1H



**Figure S41.** Expanded <sup>1</sup>H NMR spectrum of **2b** in CDCl<sub>3</sub>.

PG-APP-10-15-1-13C

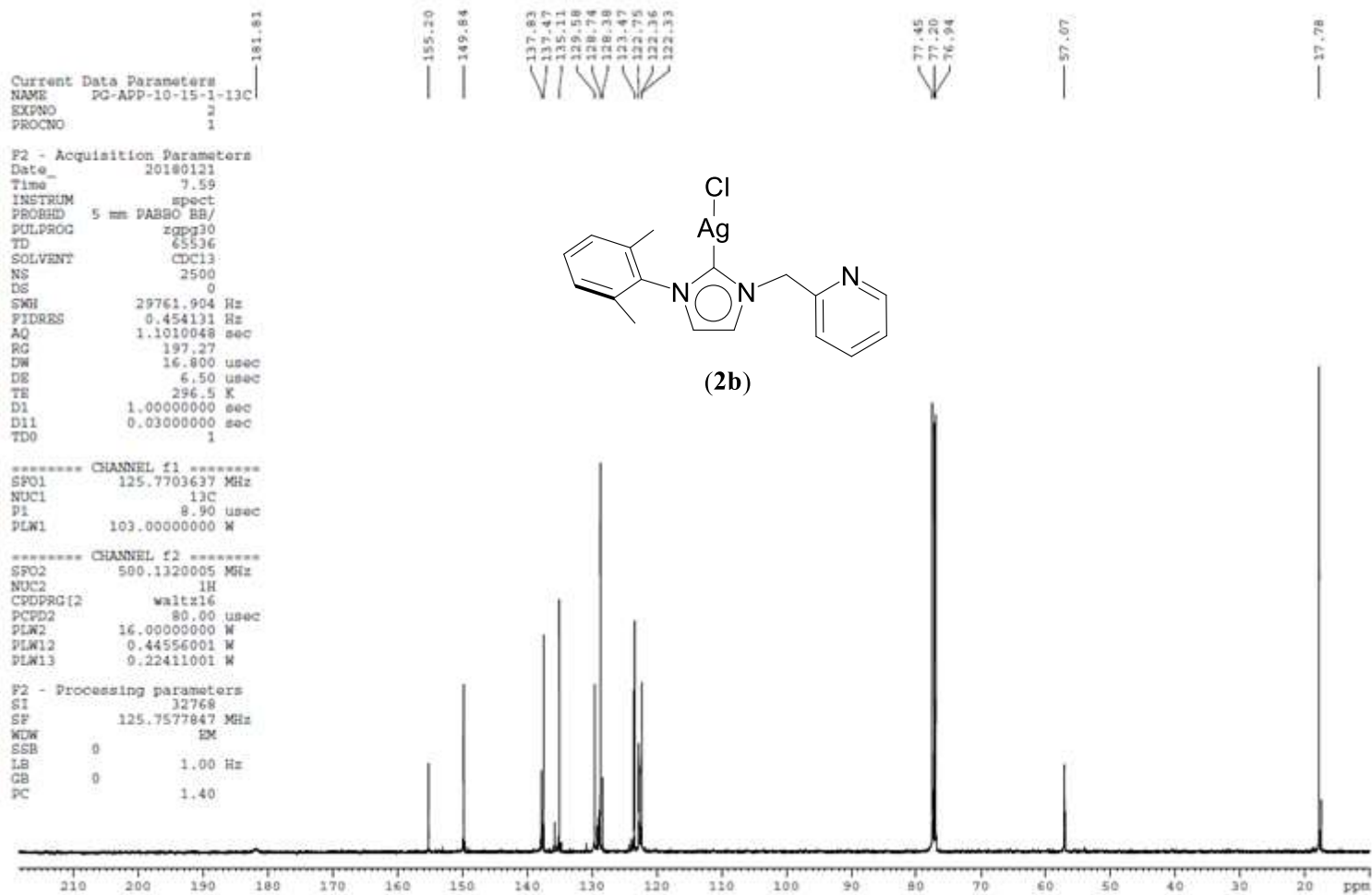
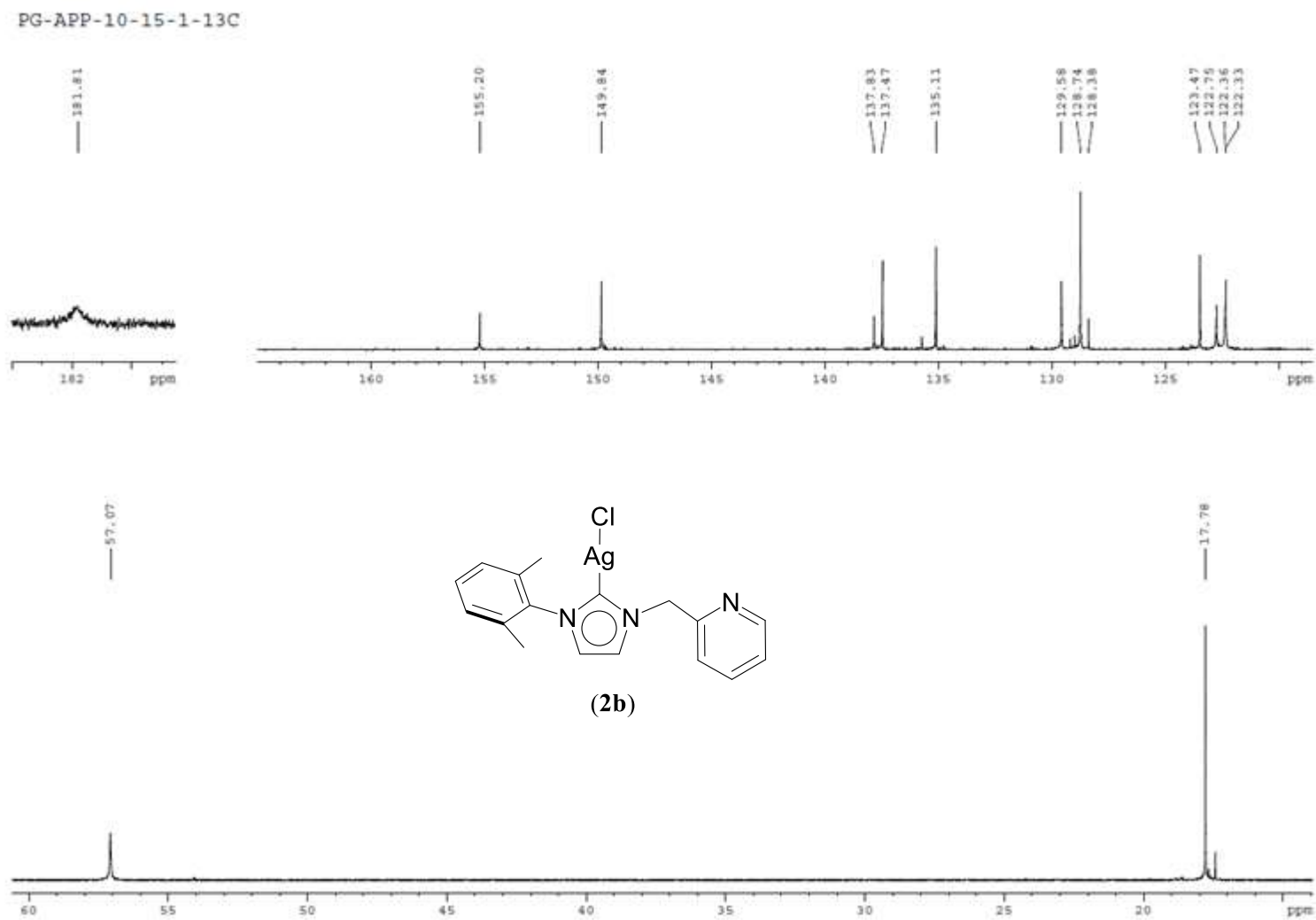
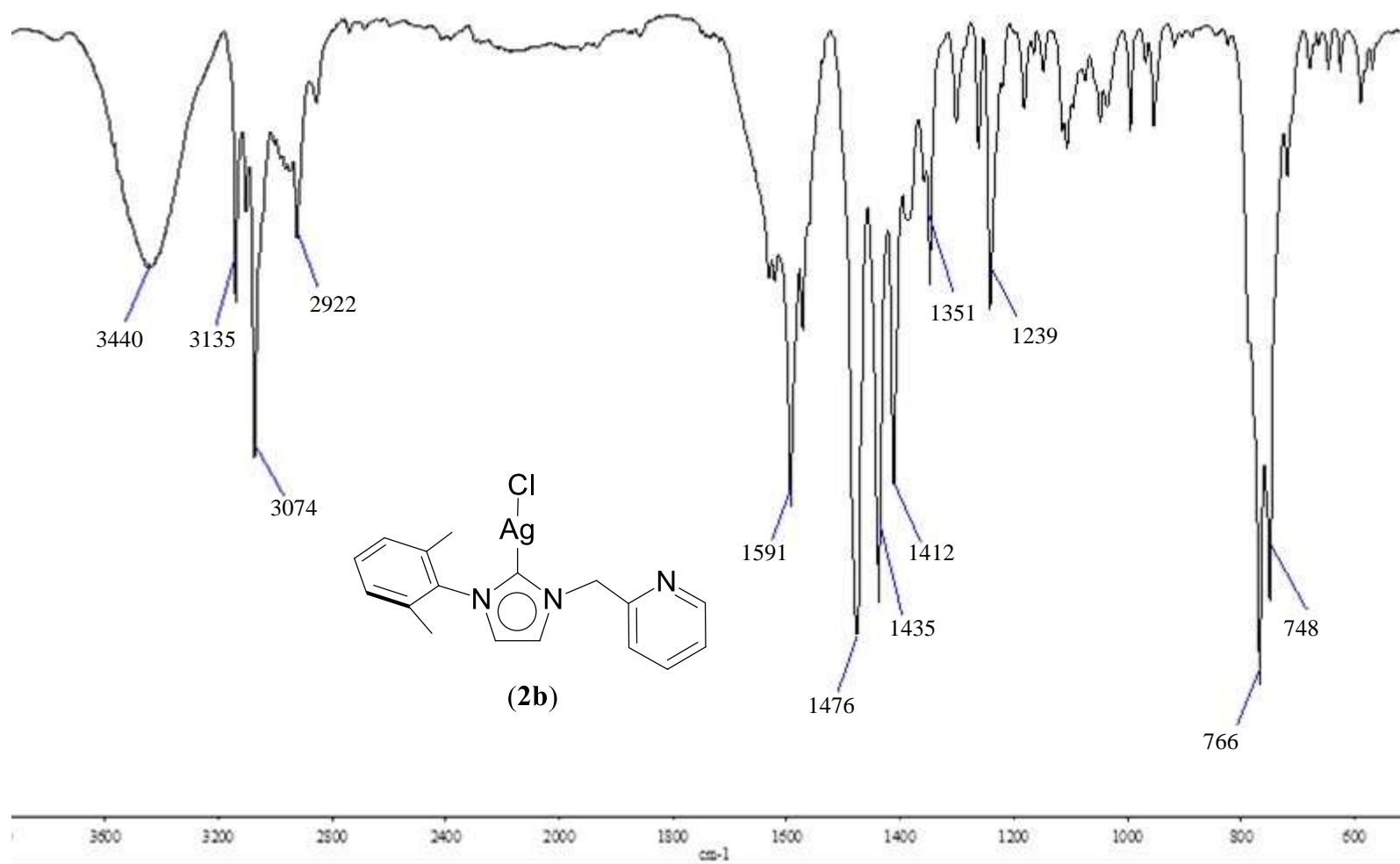


Figure S42.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of **2b** in  $\text{CDCl}_3$ .



**Figure S43.** Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of **2b** in  $\text{CDCl}_3$ .



**Figure S44.** IR spectrum of **2b** in KBr.

DEPARTMENT OF CHEMISTRY, I.I.T.(B)

Analysis Info  
 Analysis Name D:\Data\FEB-21\PG-ST-01-113-03-5.d Acquisition Date 2/8/2021 7:18:06 PM  
 Method NaICSI\_pos\_1000.m Operator SSK IN  
 Sample Name PG-ST-01-113-03-5 Instrument maXis impact 282001.00081  
 Comment C17H17N3AgCl

Acquisition Parameter  
 Source Type ESI Ion Polarity Positive Set Nebulizer 0.3 Bar  
 Focus Not active Set Capillary 3700 V Set Dry Heater 180 °C  
 Scan Begin 50 m/z Set End Plate Offset -500 V Set Dry Gas 4.0 l/min  
 Scan End 1000 m/z Set Charging Voltage 2000 V Set Divert Valve Source  
 Set Corona 0 nA Set APCl Heater 0 °C

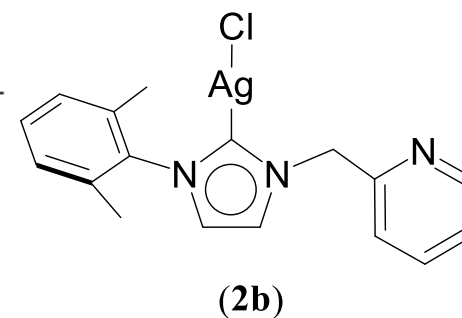
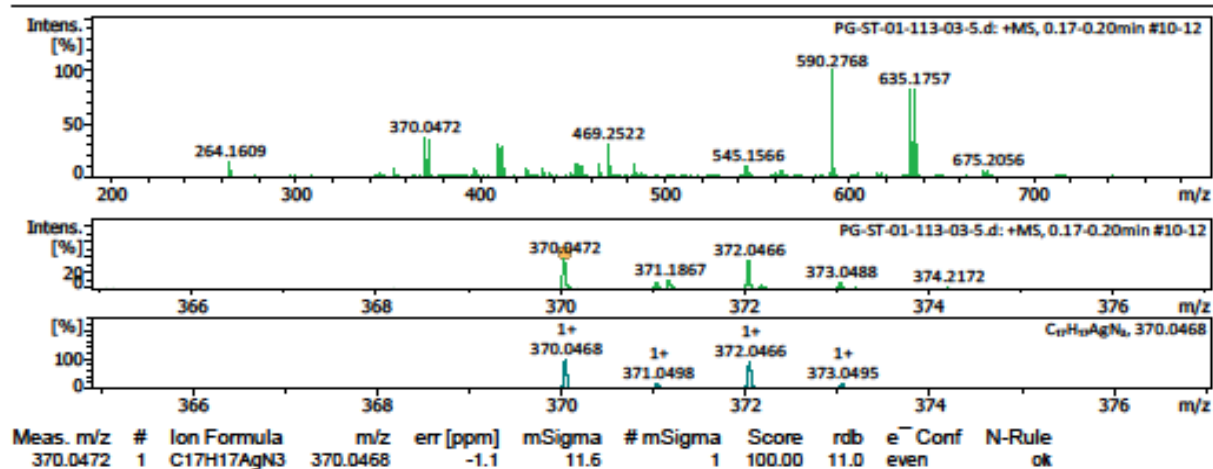


Figure S45. High Resolution Mass Spectrometry (HRMS) data of **2b**.

SP18022016  
varioMICRO CHNS  
serial number: 15154051

Graphic report

| No. | Weight [mg] | Name          | Method     | N Area | C Area | H Area | N [%] | C [%] | H [%] | Date       | Time  | Info |
|-----|-------------|---------------|------------|--------|--------|--------|-------|-------|-------|------------|-------|------|
| 23  | 1.5330      | PG-ST-01-90-1 | 2mgChem80s | 9 261  | 22 077 | 7 355  | 10.61 | 50.42 | 4.293 | 21-09-2021 | 16:50 | Su   |

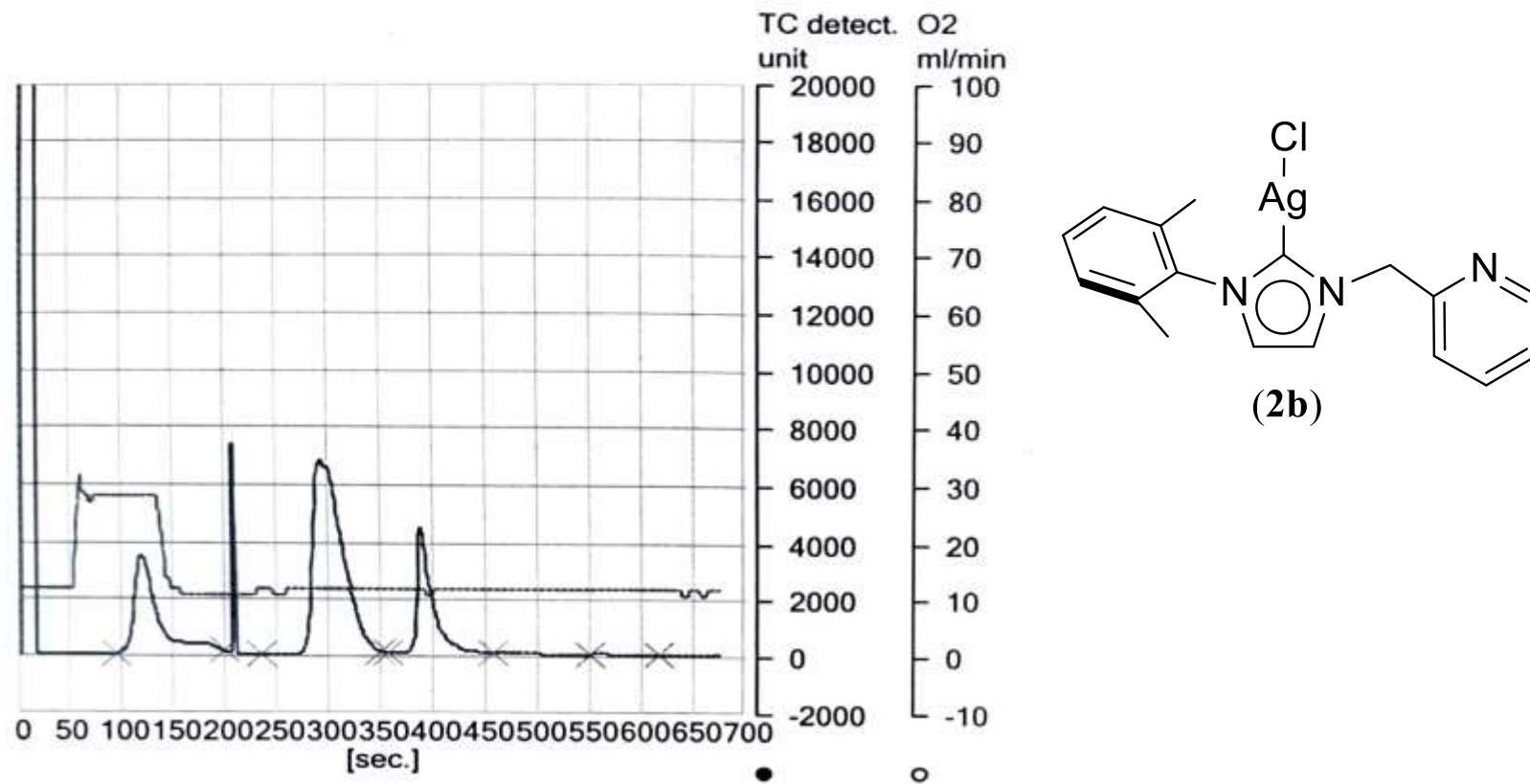
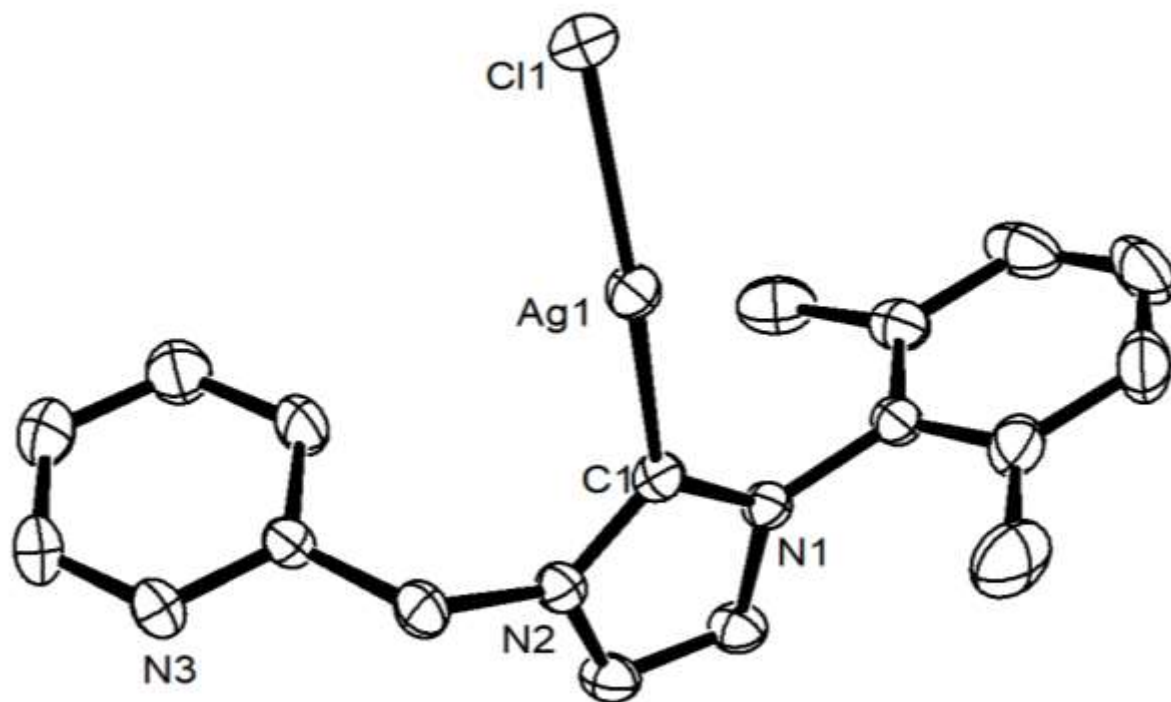


Figure S46. Elemental analysis data of 2b.



**Figure S47.** ORTEP of **2b** with thermal ellipsoids shown at the 50 % probability level. Hydrogen atoms were omitted for clarity. Selected bond lengths (Å) and angles (°): Ag(1)–C(1) 2.0813(17), Ag(1)–Cl(1) 2.3684(5), C(1)–N(1) 1.352(2), C(1)–N(2) 1.357(2), C(1)–Ag(1)–Cl(1) 168.09(5), N(1)–C(1)–N(2) 104.05(15), N(1)–C(1)–Ag(1) 129.13(13), N(2)–C(1)–Ag(1) 126.74(13).



PG-ST-01-145-02-1H

Current Data Parameters  
NAME PG-ST-01-145-02-1H  
EXPNO 5  
PROCNO 1

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Date\_ 20201229  
Time\_ 3.44  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 13  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2998593 sec  
RG 161  
DW 60.800 usec  
DE 6.50 usec  
TE 295.6 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 14.75 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300095 MHz  
WDW EM  
SSB 0  
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GB 0  
PC 1.00

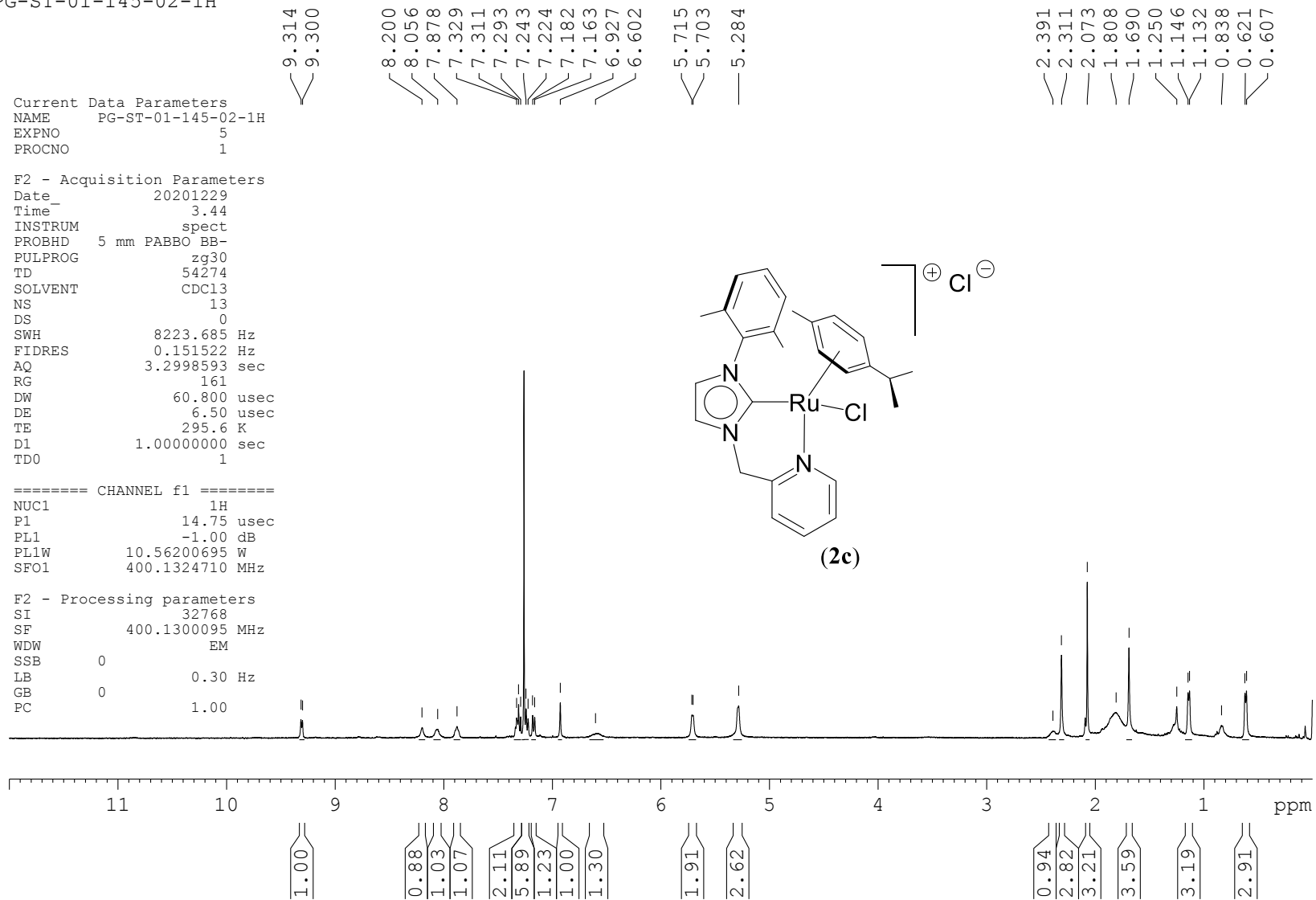
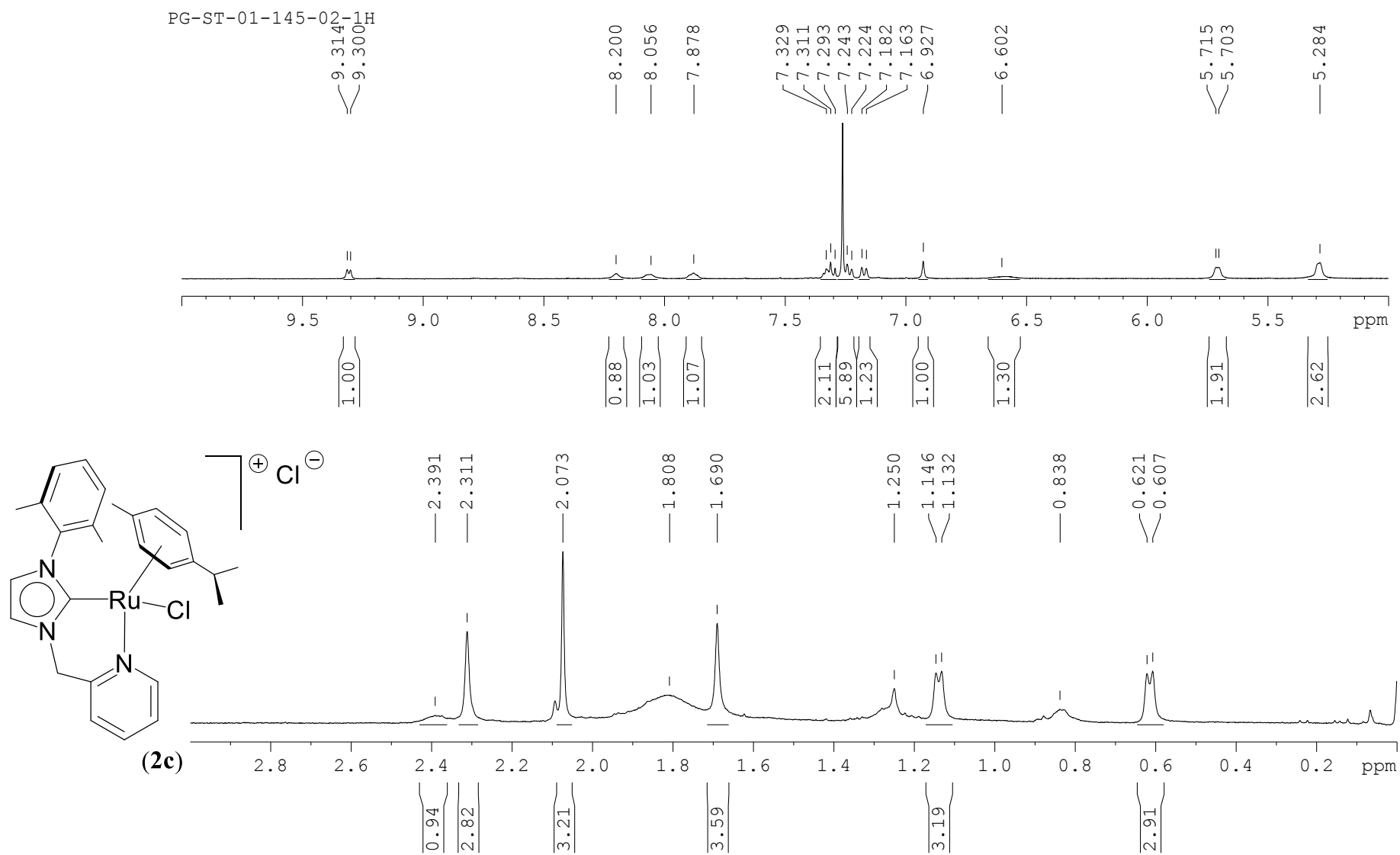


Figure S48. <sup>1</sup>H NMR spectrum of **2c** in CDCl<sub>3</sub>.



**Figure S49.** Expanded <sup>1</sup>H NMR spectrum of **2c** in CDCl<sub>3</sub>.

PG-APP-10-53-1-13C

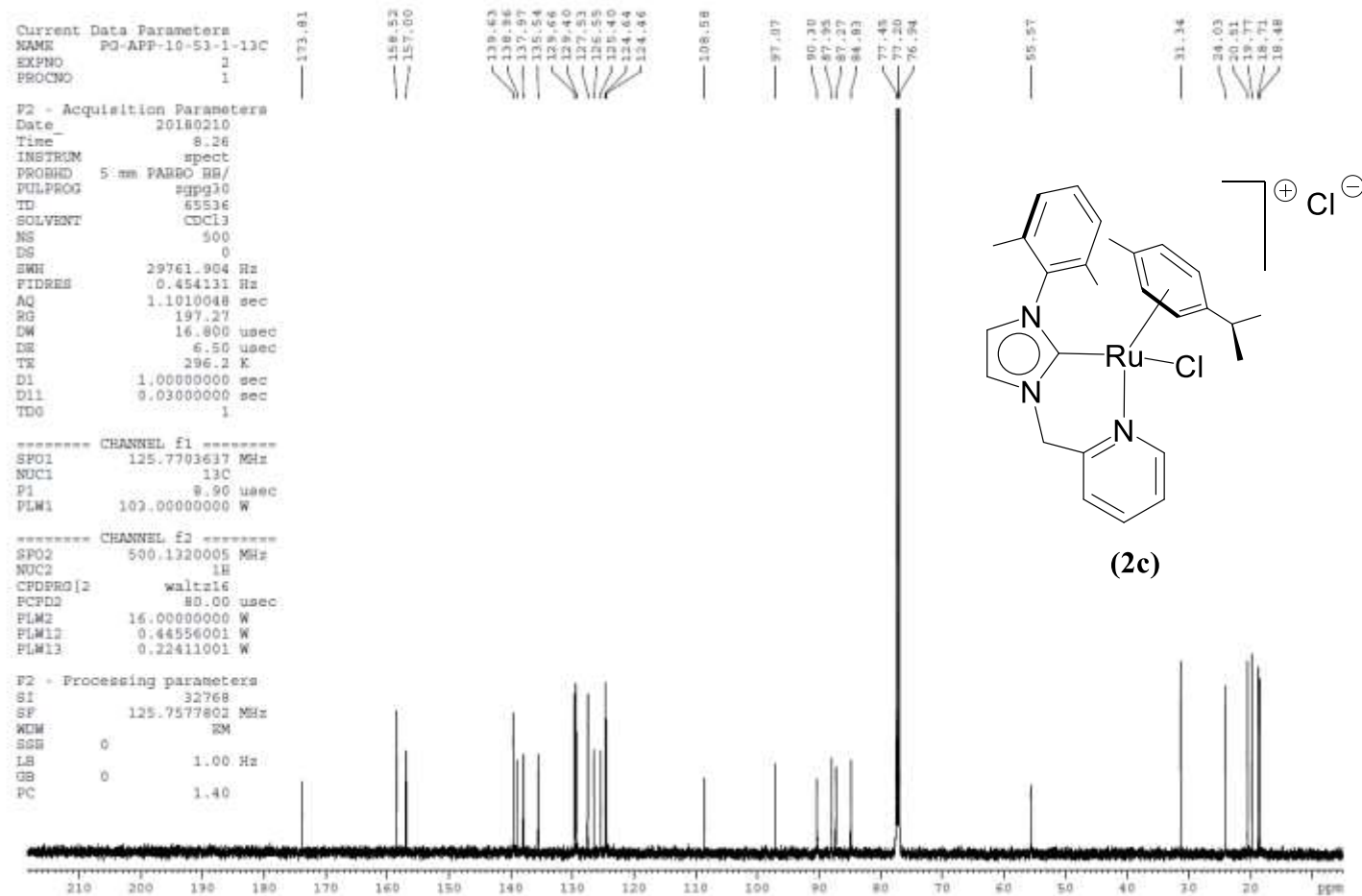
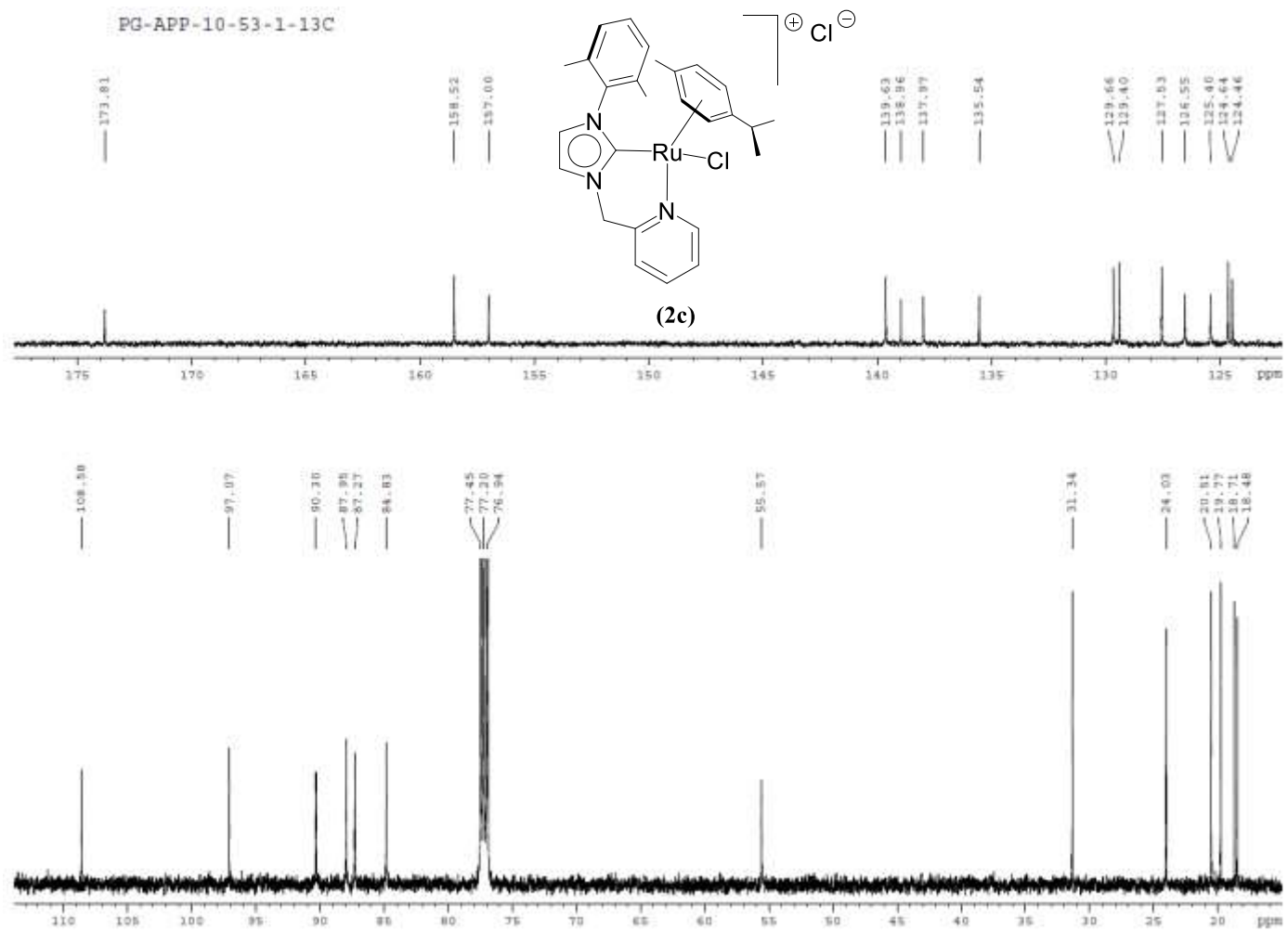
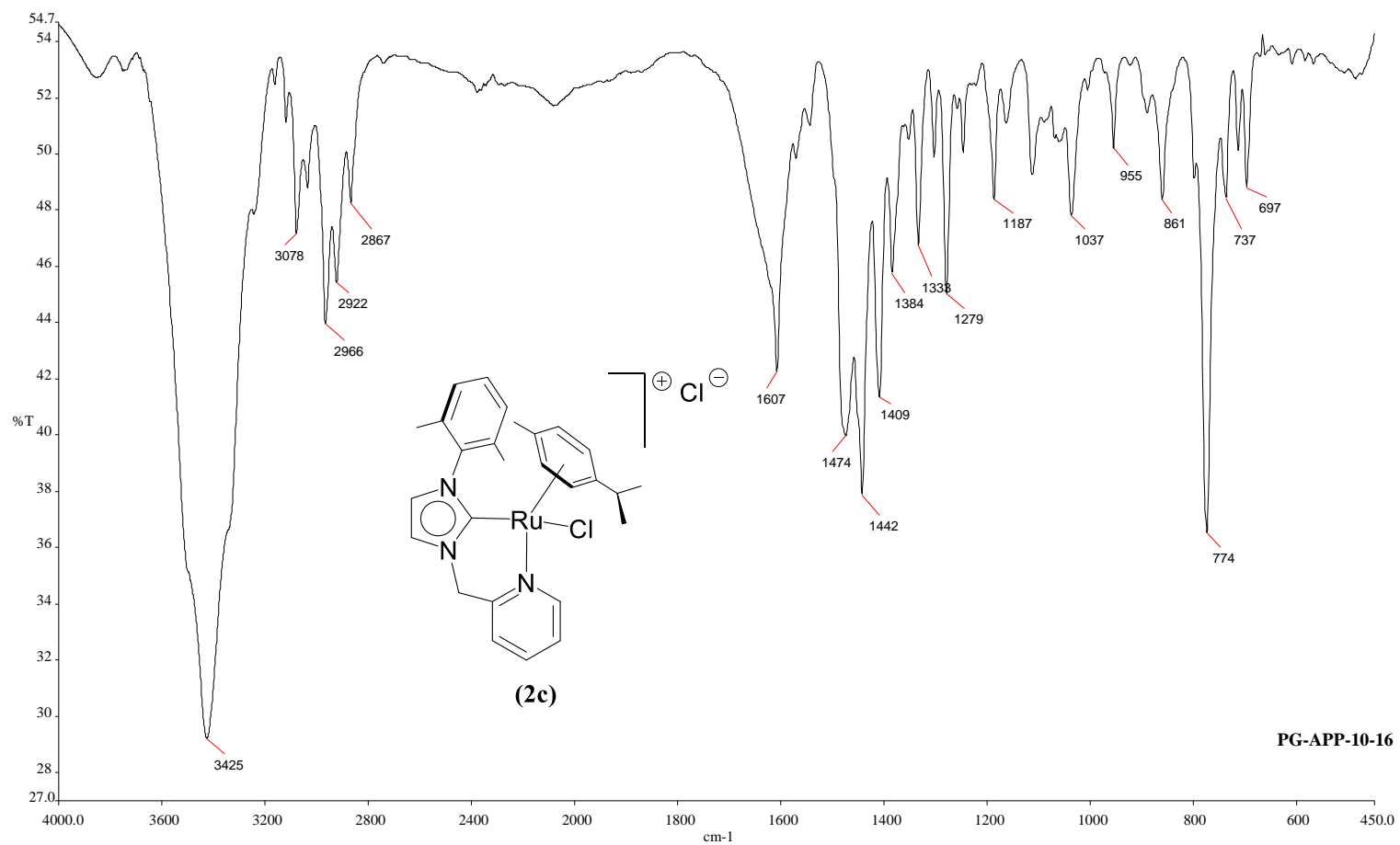


Figure S50.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of **2c** in  $\text{CDCl}_3$ .



**Figure S51.** Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of **2c** in  $\text{CDCl}_3$ .



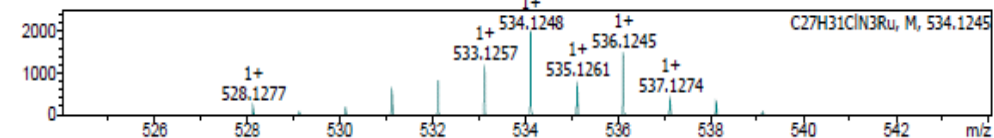
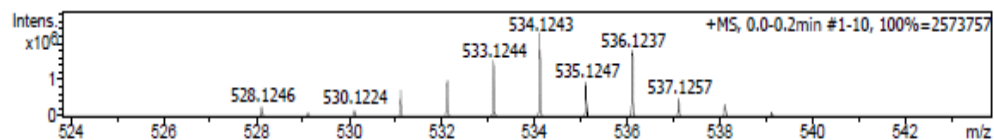
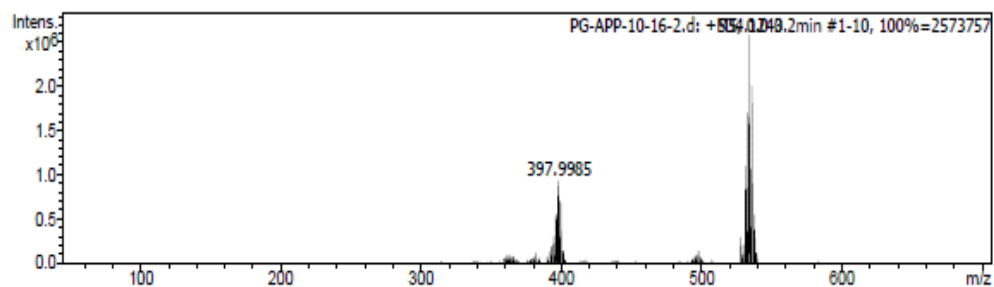
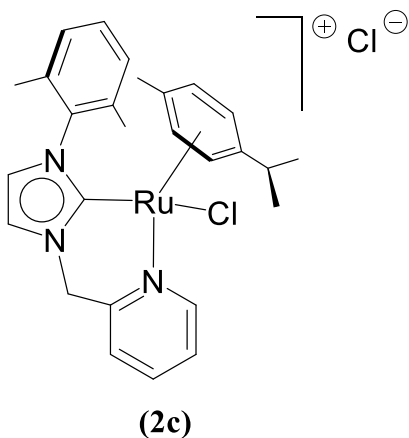
PG-APP-10-16

Figure S52. IR spectrum of **2c** in KBr.

DEPARTMENT OF CHEMISTRY, I.I.T.(B)

**Analysis Info**  
 Analysis Name D:\Data\FEB-2018\PG-APP-10-16-2.d  
 Method Tune\_pos\_NAICSI-1000A.m  
 Sample Name PG-APP-10-16-2  
 Comment C27H31N3RuCl2  
 Acquisition Date 2/1/2018 12:38:42 PM  
 Operator PG APP IN  
 Instrument maXis impact 282001.00081

**Acquisition Parameter**  
 Source Type ESI Ion Polarity Positive Set Nebulizer 0.4 Bar  
 Focus Active Set Capillary 3700 V Set Dry Heater 180 °C  
 Scan Begin 50 m/z Set End Plate Offset -500 V Set Dry Gas 4.0 l/min  
 Scan End 1000 m/z Set Collision Cell RF 1500.0 Vpp Set Divert Valve Source



| Meas. m/z | # | Ion Formula  | m/z      | err [ppm] | mSigma | # Sigma | Score  | rdb  | e <sup>-</sup> Conf | N-Rule |
|-----------|---|--------------|----------|-----------|--------|---------|--------|------|---------------------|--------|
| 534.1243  | 1 | C27H31ClN3Ru | 534.1249 | 1.0       | 21.1   | 1       | 100.00 | 14.0 | odd                 | -      |

Figure S53. High Resolution Mass Spectrometry (HRMS) data of 2c.

| No. | Weight [mg] | Name              | Method     | N Area | C Area | H Area | N [%] | C [%] | H [%] | Date       | Time  | Info |
|-----|-------------|-------------------|------------|--------|--------|--------|-------|-------|-------|------------|-------|------|
| 36  | 0.7350      | PG-ST-01-172-02-1 | 2mgChem80s | 4 855  | 12 078 | 4 862  | 7.04  | 57.55 | 5.129 | 21-09-2021 | 19:59 | Su   |

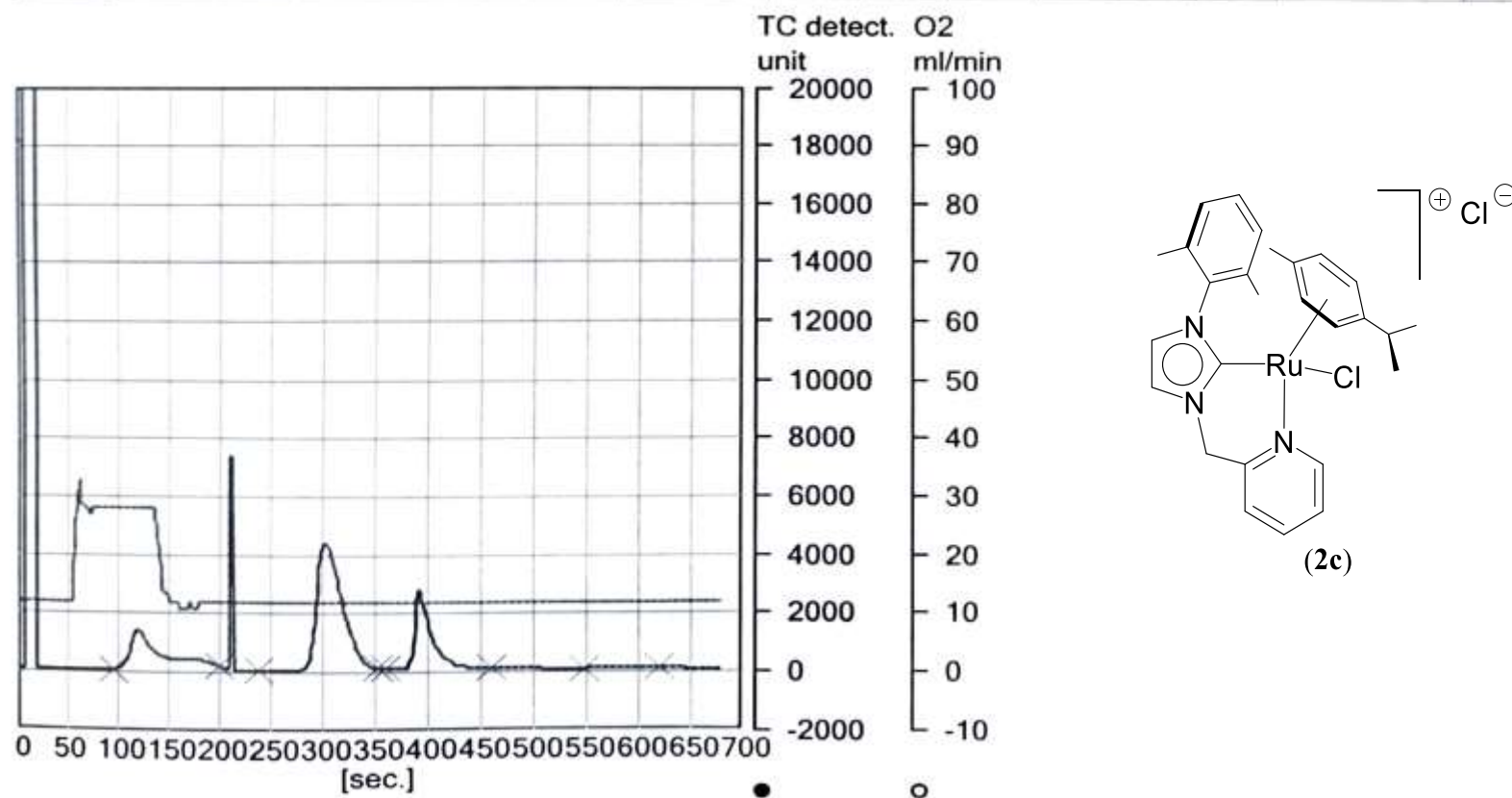
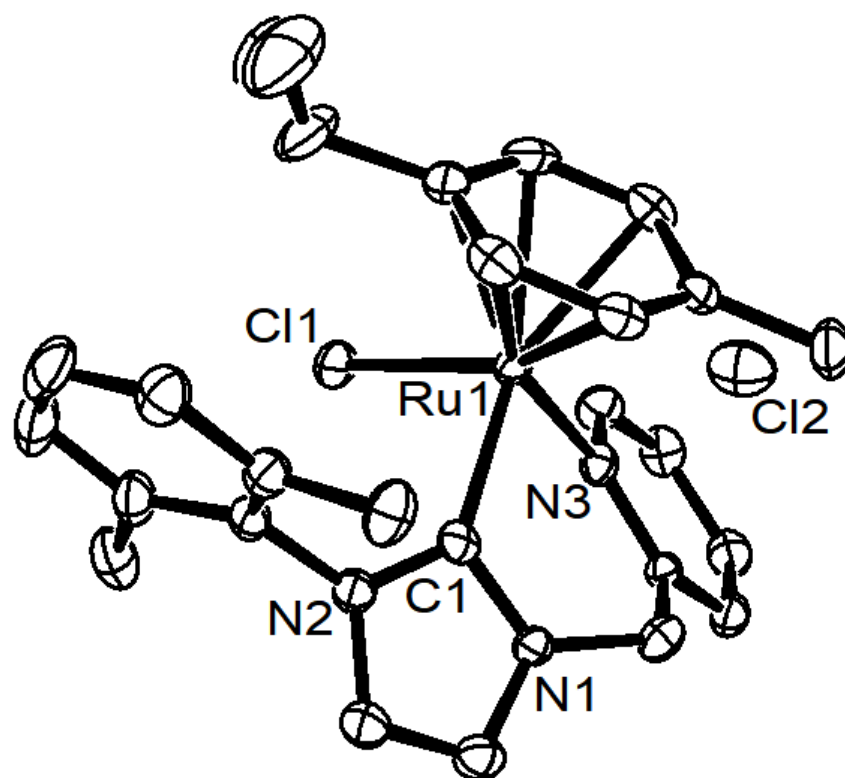


Figure S54. Elemental analysis data of 2c.



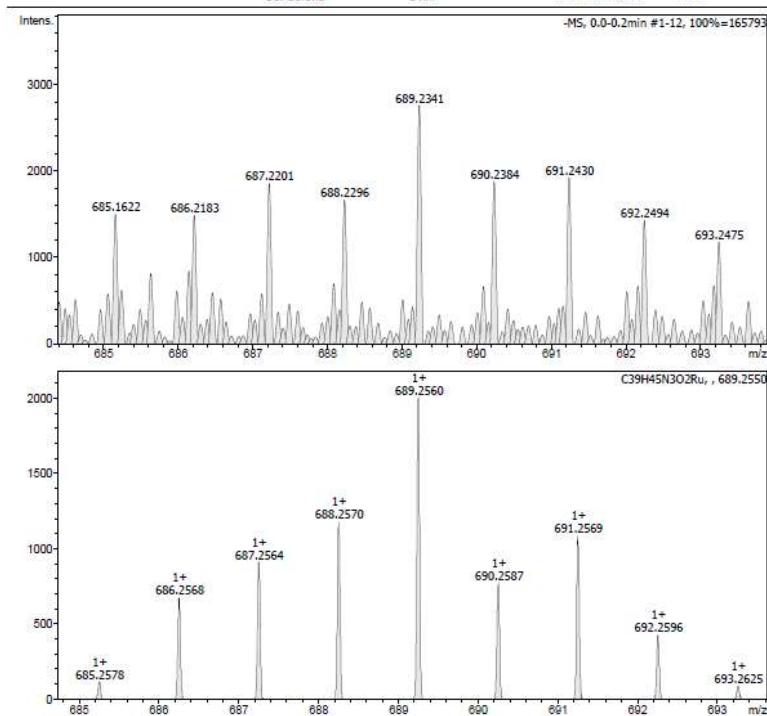
**Figure S55.** ORTEP of **2c** with thermal ellipsoids shown at the 50 % probability level. Hydrogen atoms and co-crystallized water molecules were omitted for clarity. Selected bond lengths (Å) and angles (°): Ru(1)–C(1) 2.052(4), Ru(1)–N(3) 2.110(3), Ru(1)–Cl(1) 2.3936(9), C(1)–N(1) 1.359(5), C(1)–N(2) 1.358(5), C(1)–Ru(1)–Cl(1) 89.28(10), C(1)–Ru(1)–N(3) 85.17(13), N(3)–Ru(1)–Cl(1) 84.85(8), N(2)–C(1)–Ru(1) 134.7(3), N(1)–C(1)–Ru(1) 121.1(3), N(1)–C(1)–N(2) 104.2(3).



DEPARTMENT OF CHEMISTRY, I.I.T.(B)

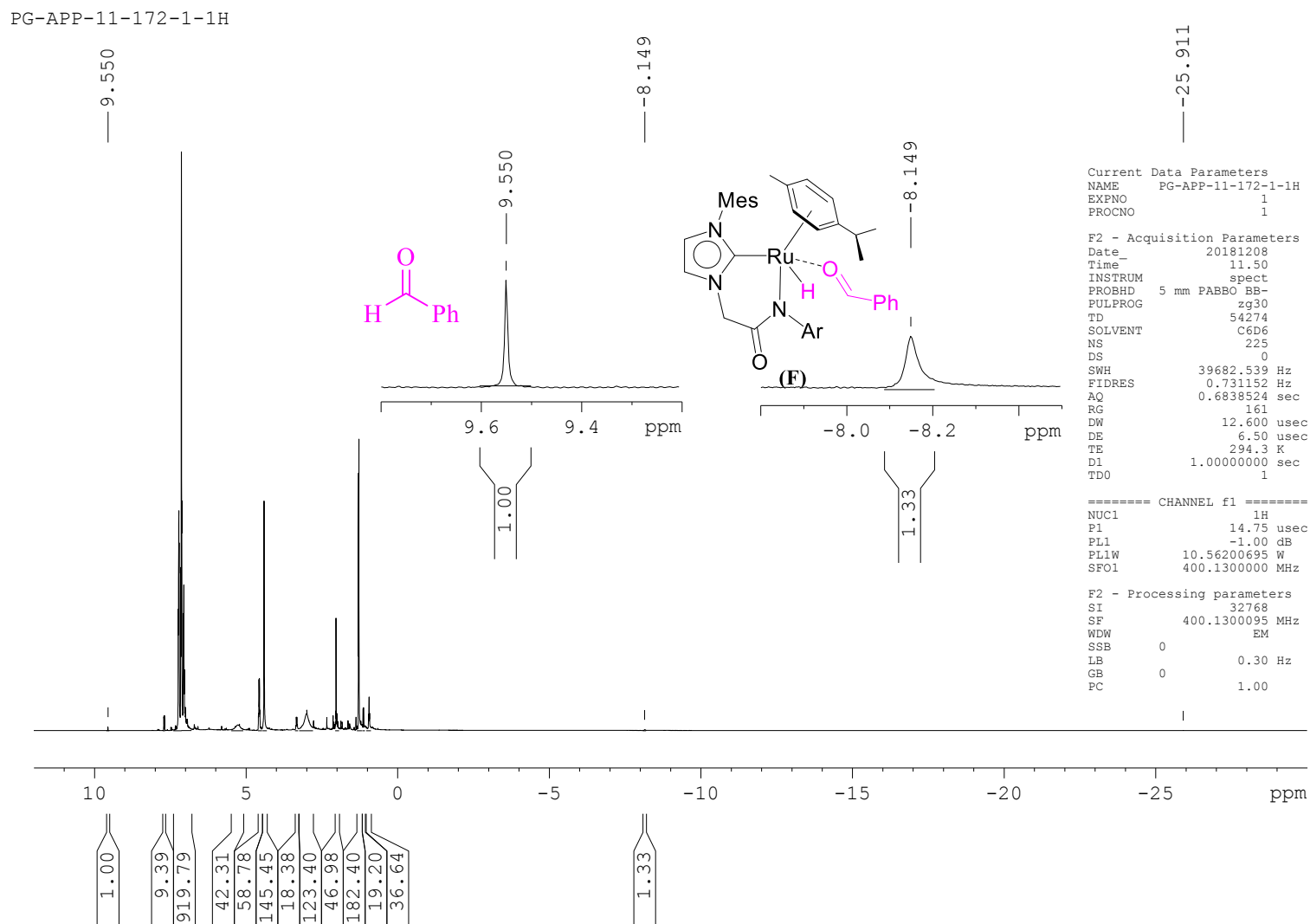
|                      |                                      |                  |                       |  |
|----------------------|--------------------------------------|------------------|-----------------------|--|
| <b>Analysis Info</b> |                                      | Acquisition Date | 12/13/2018 9:35:20 AM |  |
| Analysis Name        | D:\Data\DEC 2018\PG-APP-11-176-2-1.d | Operator         | PG APP IN             |  |
| Method               | Tune_neg_Standard_NAF-1000NEW.m      | Instrument       | maXis impact          |  |
| Sample Name          | PG-APP-11-176-2-1                    |                  | 282001.00081          |  |
| Comment              | C32H38N3ORuCl                        |                  |                       |  |

|                              |          |                      |          |                  |           |
|------------------------------|----------|----------------------|----------|------------------|-----------|
| <b>Acquisition Parameter</b> |          |                      |          |                  |           |
| Source Type                  | ESI      | Ion Polarity         | Negative | Set Nebulizer    | 0.3 Bar   |
| Focus                        | Active   | Set Capillary        | 3500 V   | Set Dry Heater   | 180 °C    |
| Scan Begin                   | 50 m/z   | Set End Plate Offset | -500 V   | Set Dry Gas      | 4.0 l/min |
| Scan End                     | 1000 m/z | Set Charging Voltage | 2000 V   | Set Divert Valve | Source    |
|                              |          | Set Corona           | 0 nA     | Set APCI Heater  | 0 °C      |

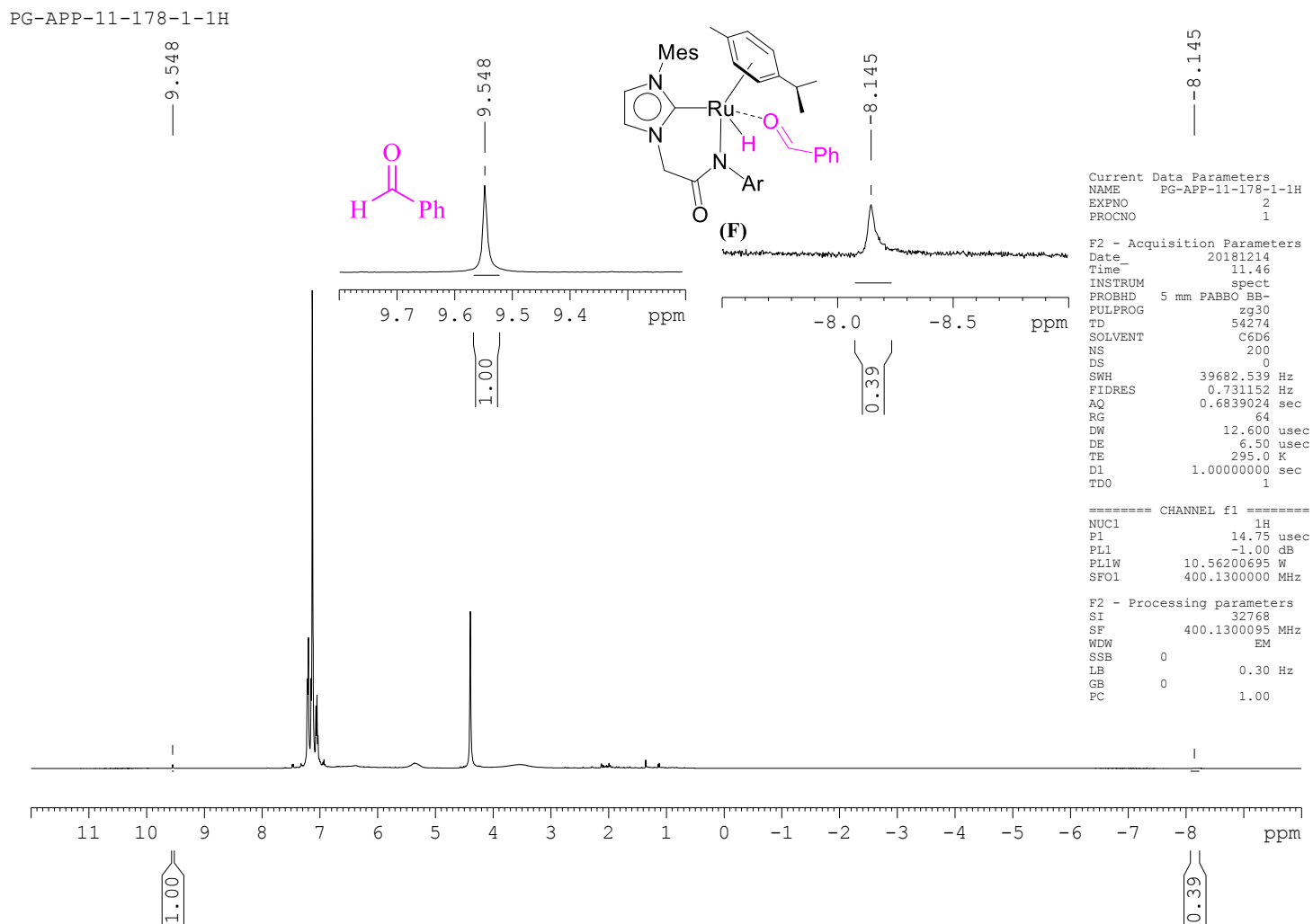


PG-APP-11-176-2-1.d  
 Bruker Compass DataAnalysis 4.1 printed: 4/25/2019 5:51:02 PM by: PG APP IN Page 1 of 1

**Figure S56.** ESI-MS data of the benzaldehyde bound Ru-H specie (**F**) detected in the reaction mixture of 1:1:1 ratio of benzyl alcohol:1-phenylethan-1-ol:NaO*i*Pr 0.1 mmol, 1 mol % of (**1c**), 2.0 mL of toluene at 110 °C for 1 hour.

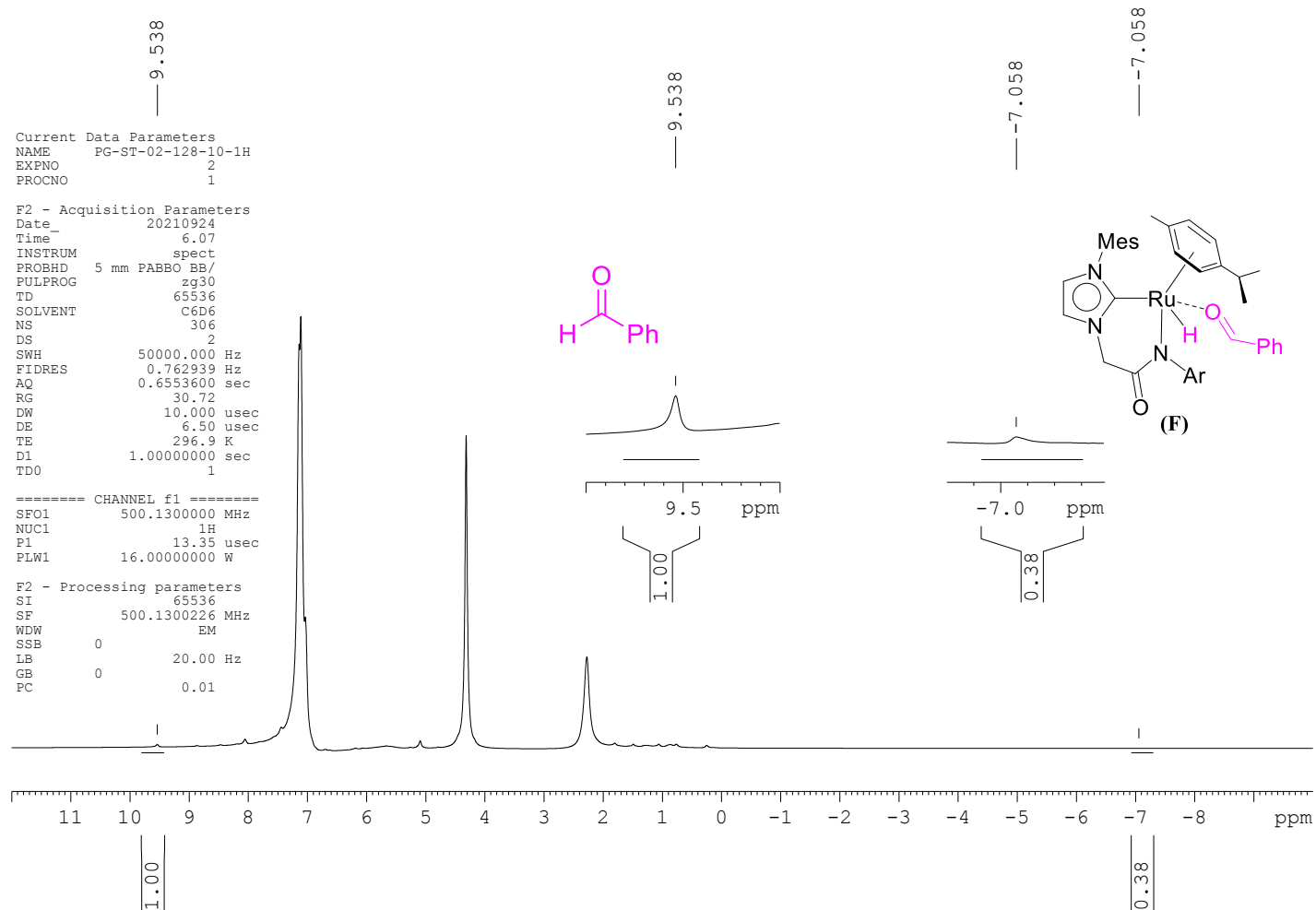


**Figure S57.**  $^1\text{H}$  NMR spectrum of the ruthenium hydride specie (**F**) ( $-8.15$  ppm) detected in the reaction mixture of 1:1:1 ratio of benzyl alcohol:1-phenylethanol:KOH 0.1 mmol, 1 mol % of (**1c**), 0.5 mL of  $\text{C}_6\text{D}_6$  at  $90^\circ\text{C}$  for 30 mins.



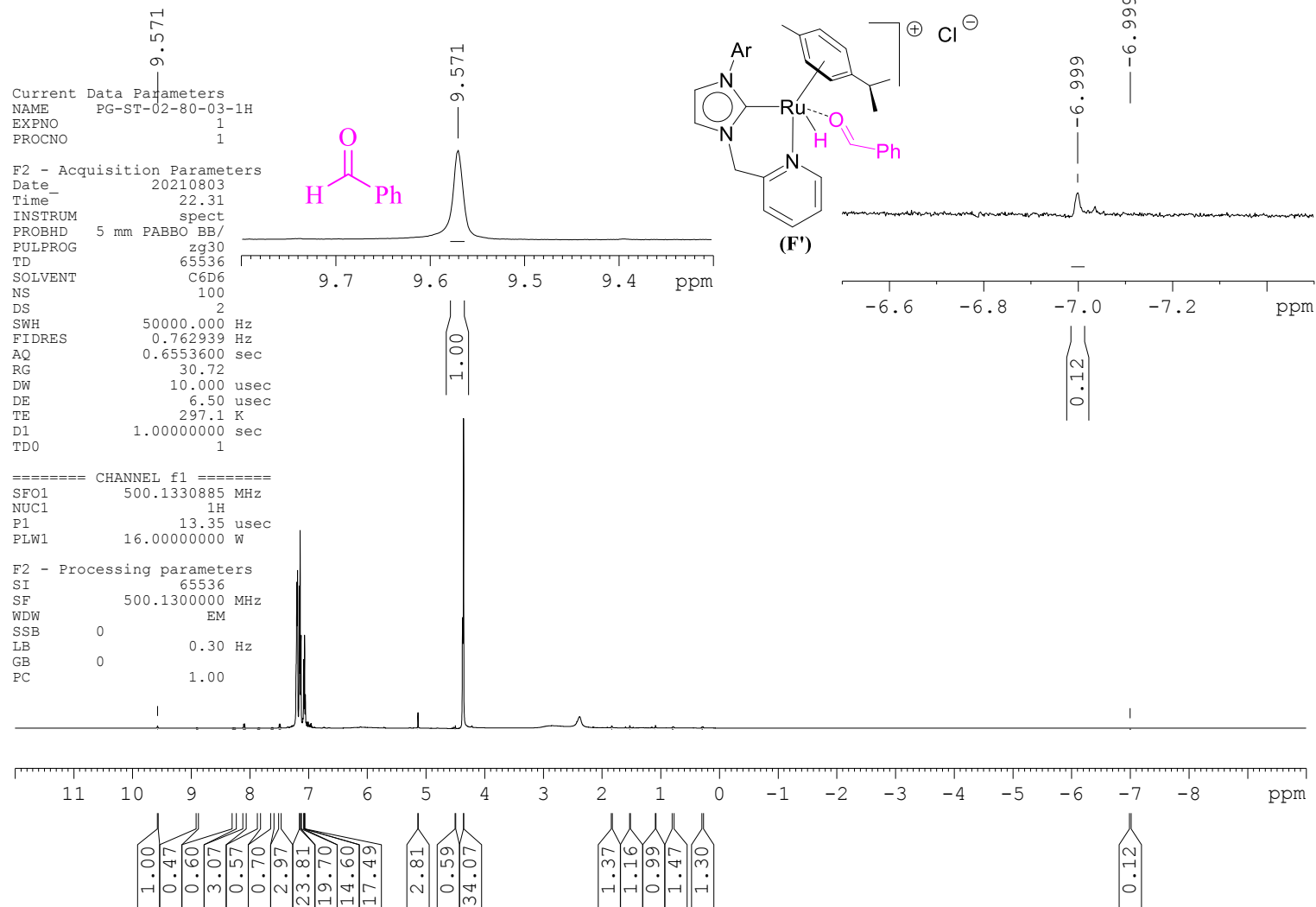
**Figure S58.**  $^1\text{H}$  NMR spectrum of the ruthenium hydride specie (**F**) ( $-8.15$  ppm) detected in the reaction mixture of 1:1 ratio of benzyl alcohol:KOH 0.1 mmol, 1 mol % of (**1c**), 0.5 mL of  $\text{C}_6\text{D}_6$  at  $90^\circ\text{C}$  for 30 mins.

PG-ST-02-128-10-1H



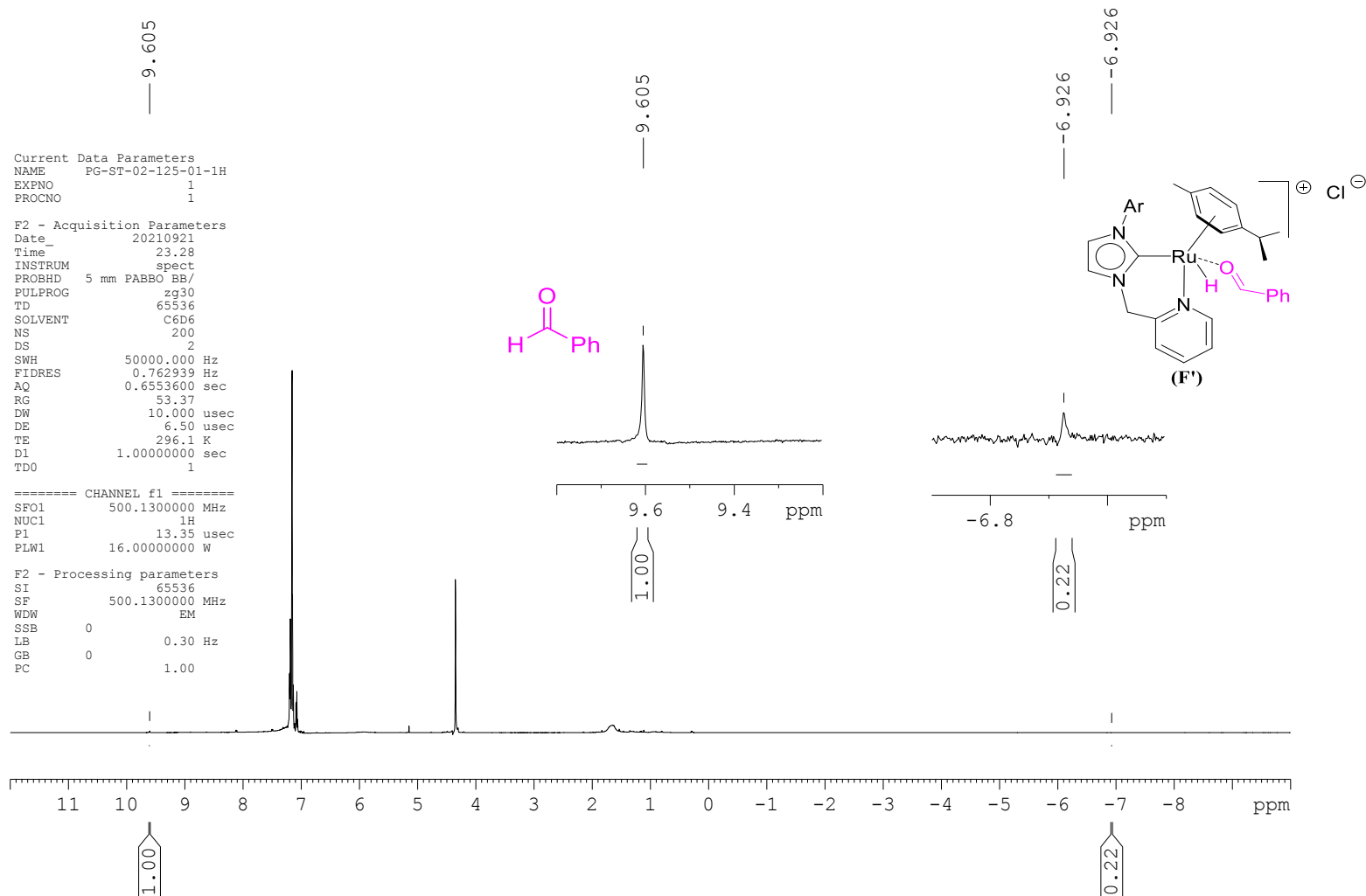
**Figure S59.**  $^1\text{H}$  NMR spectrum of the ruthenium hydride specie (**F**) ( $-7.06$  ppm) detected in the reaction mixture of 1:1 ratio of benzyl alcohol:NaO-*i*-Pr 0.1 mmol, 1 mol % of (**1c**), 0.5 mL of  $\text{C}_6\text{D}_6$  at  $90^\circ\text{C}$  for 30 mins.

PG-ST-02-80-03-1H



**Figure S60.**  $^1\text{H}$  NMR spectrum of the ruthenium hydride specie (**F'**) ( $-6.99$  ppm) detected in the reaction mixture of 1:1 ratio of benzyl alcohol: KOH 0.1 mmol, 1 mol % of (**2c**), 0.5 mL of  $\text{C}_6\text{D}_6$  at  $90^\circ\text{C}$  for 30 mins.

PG-ST-02-125-01-1H



**Figure S61.**  $^1\text{H}$  NMR spectrum of the ruthenium hydride specie (**F'**) ( $-6.93$  ppm) detected in the reaction mixture of 1:1 ratio of benzyl alcohol:NaO-*i*-Pr 0.1 mmol, 1 mol % of (**2c**), 0.5 mL of  $\text{C}_6\text{D}_6$  at  $90^\circ\text{C}$  for 30 mins.

PG-APP-11-149-2-1H

NAME PG-APP-11-149-2-1H  
EXPNO 1  
PROCNO 1  
Date\_ 20181117  
Time\_ 8.50  
INSTRUM spect  
PROBHD 5 mm PASBO BB-  
PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2999091 sec  
RG 80.6  
DW 60.800 usec  
DE 6.50 usec  
TE 296.9 K  
D1 1.00000000 sec  
TD0 1

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
NUC1 1H  
P1 14.75 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SF01 400.1324710 MHz  
SI 32768  
SF 400.1300095 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

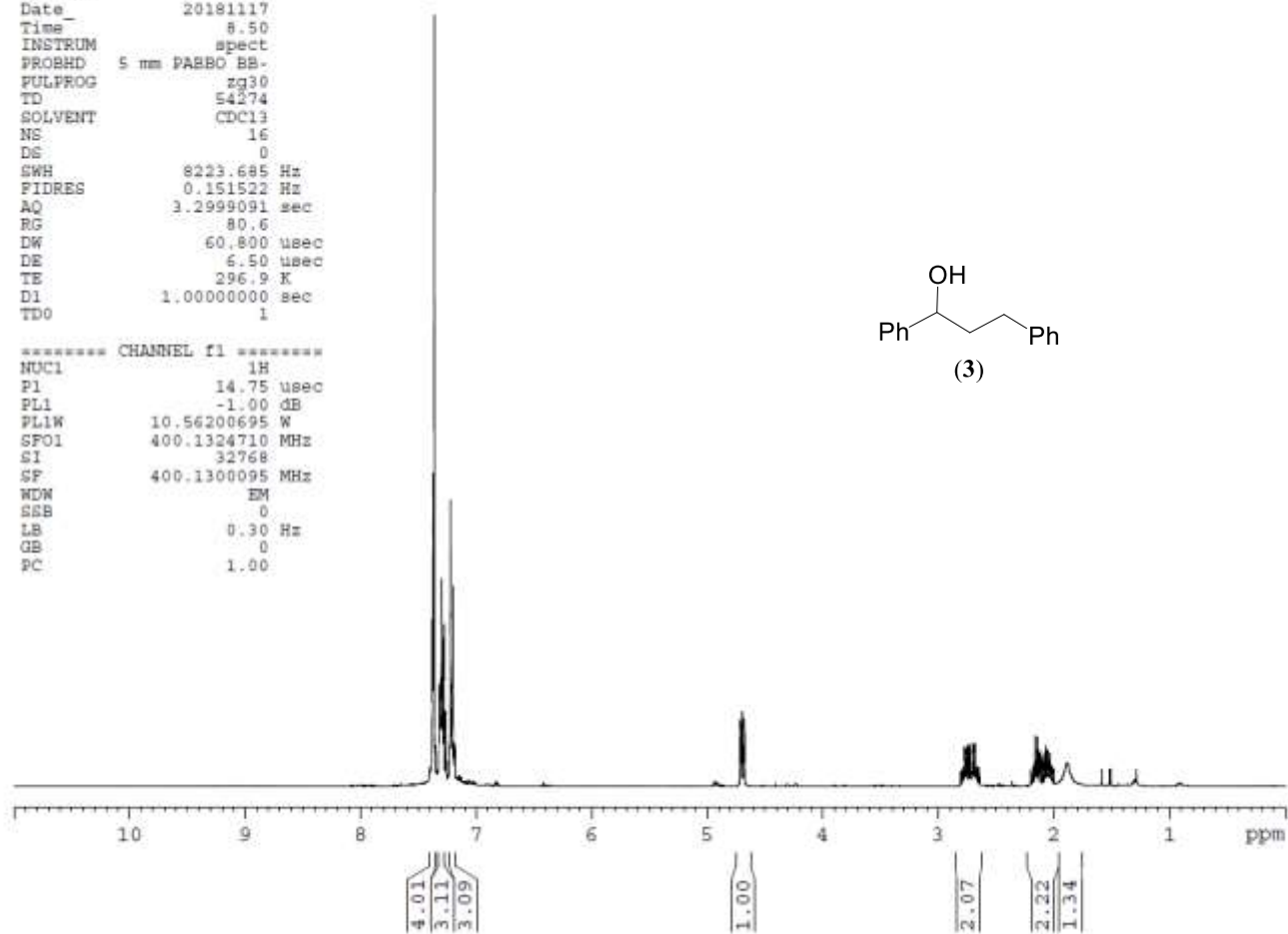


Figure S62. <sup>1</sup>H NMR spectrum of (3) in CDCl<sub>3</sub>.

PG-APP-11-149-2-1H

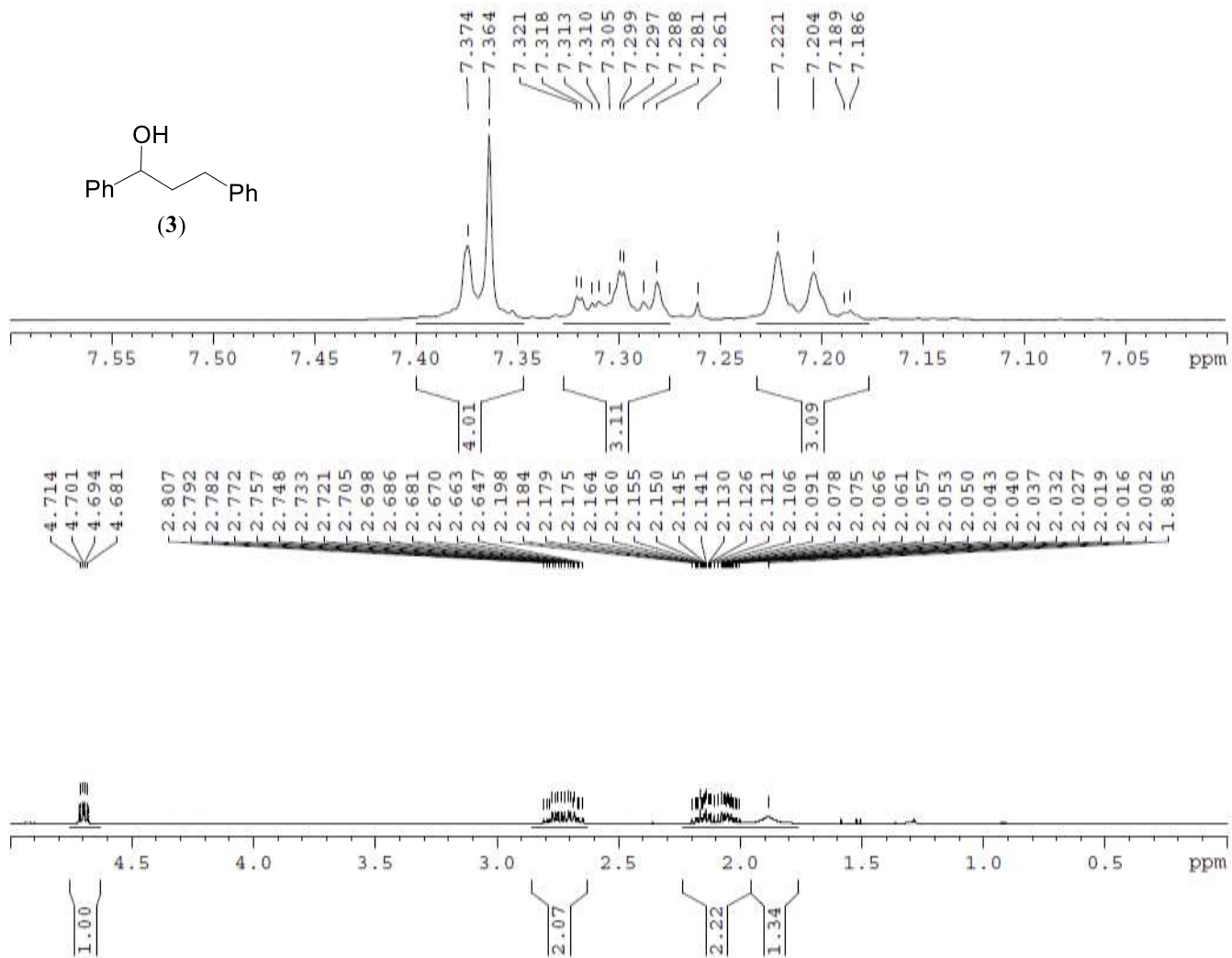


Figure S63. Expanded  $^1\text{H}$  NMR spectrum of (3) in  $\text{CDCl}_3$ .



PG-APP-11-149-2-13C

NAME PG-APP-11-149-2-13C  
EXPNO 2  
PROCNO 1  
Date\_ 20181117  
Time 8.54  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 150  
DS 0  
SWH 26041.666 Hz  
FIDRRS 0.397364 Hz  
AQ 1.2583412 sec  
RG 1030  
DW 19.200 usec  
DE 6.50 usec  
TE 297.7 K  
D1 1.00000000 sec  
D11 0.03000000 sec  
TD0 1

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
NUC1 13C  
P1 8.50 usec  
PL1 -2.00 dB  
PL1W 56.53121948 W  
SFO1 100.6238364 MHz

\*\*\*\*\* CHANNEL f2 \*\*\*\*\*  
CPDPRQ2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -1.00 dB  
PL12 13.69 dB  
PL13 14.50 dB  
PL2W 10.56200695 W  
PL12W 0.35871249 W  
PL13W 0.29767781 W  
SFO2 400.1316005 MHz  
SI 32768  
SF 100.6127555 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

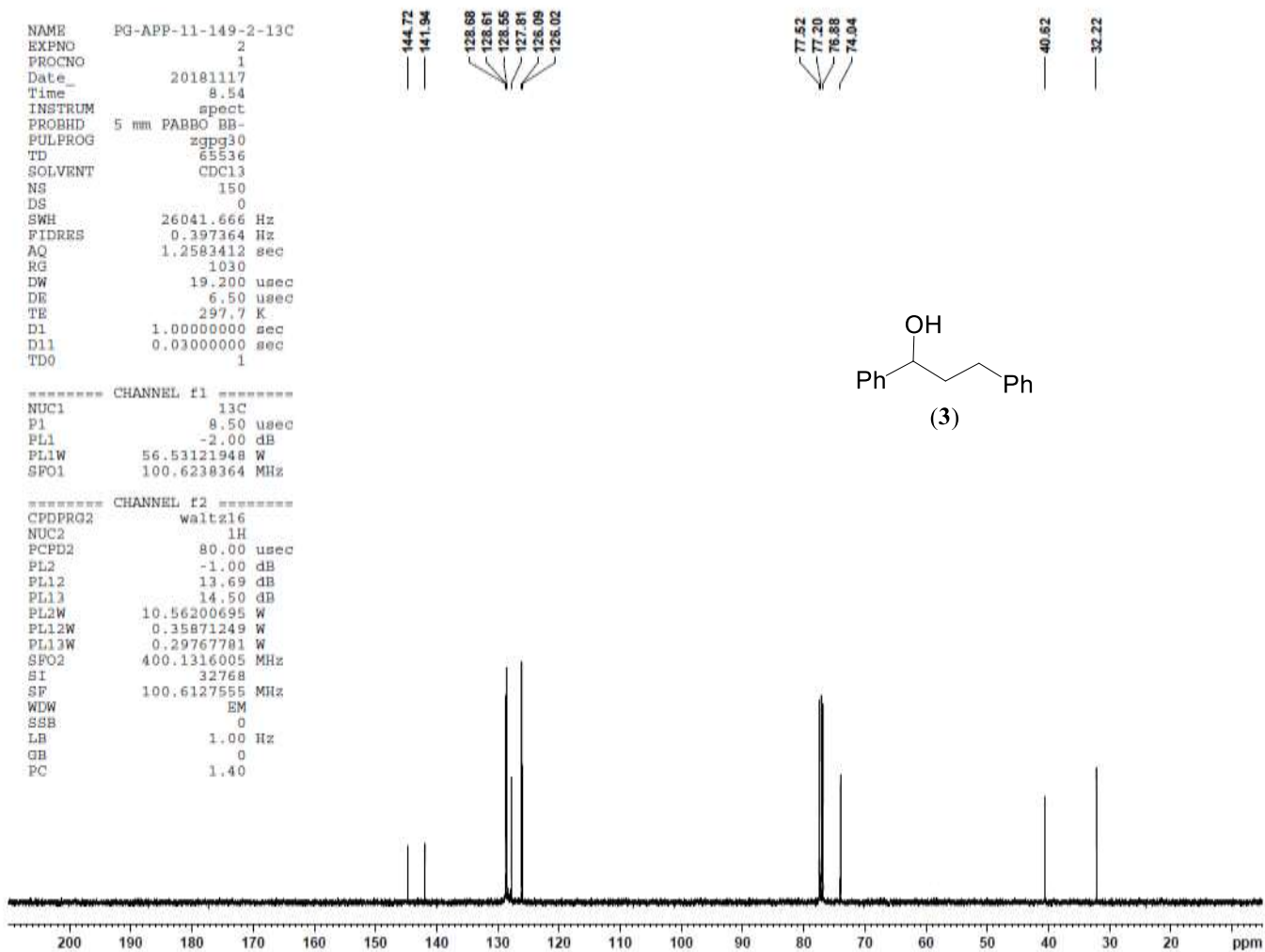
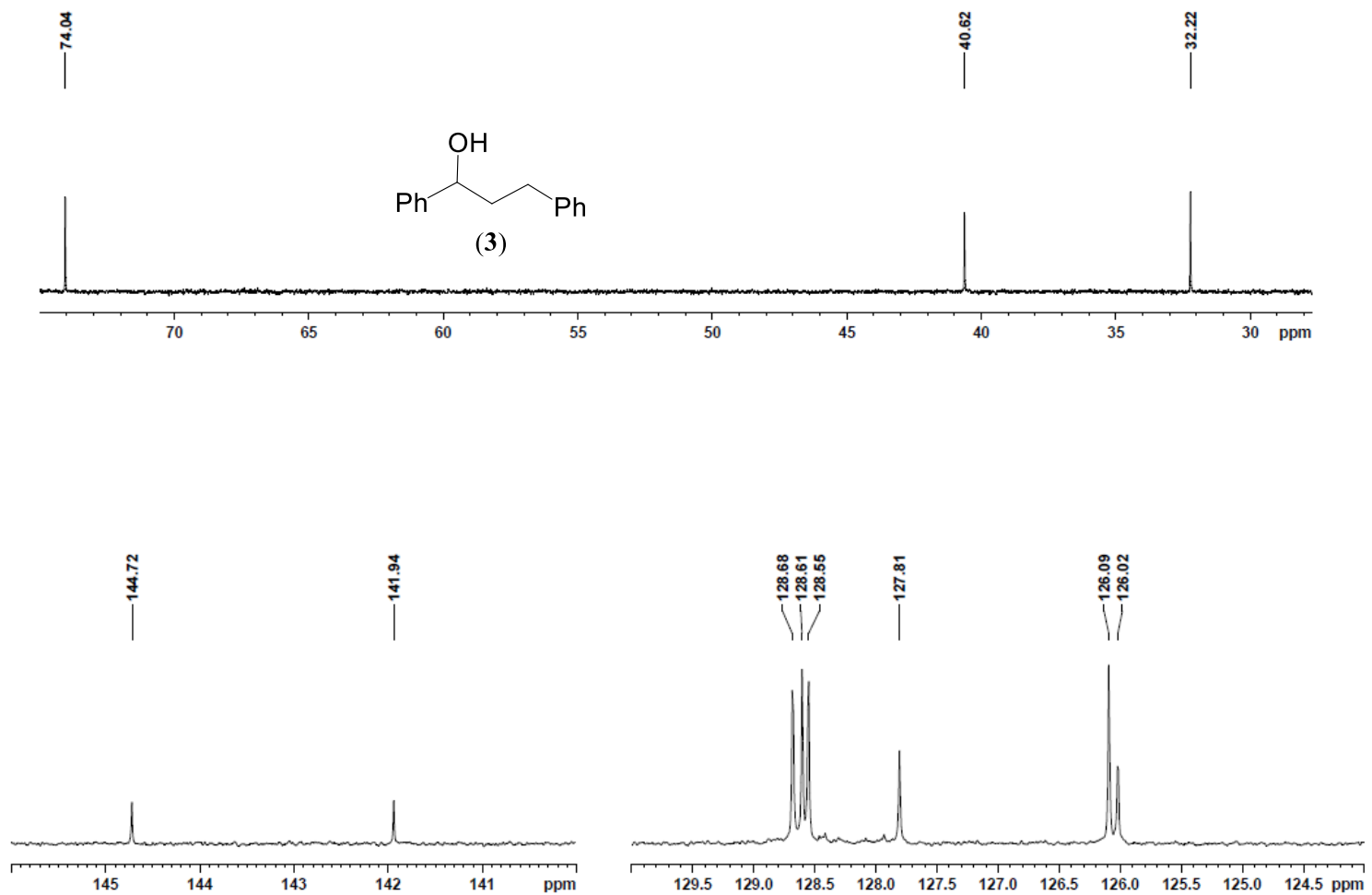


Figure S64.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (3) in  $\text{CDCl}_3$ .



**Figure S65.** Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (3) in  $\text{CDCl}_3$ .

File : F:\GCMSDATA2018\NOV 2018\PG-APP-11-149-2.D  
Operator : APP  
Acquired : 17 Nov 2018 00:50 using AcqMethod COMMONMETHOD\_2018.M  
Instrument : GCMS  
Sample Name: PG-APP-11-149-2  
Misc Info :  
Vial Number: 2

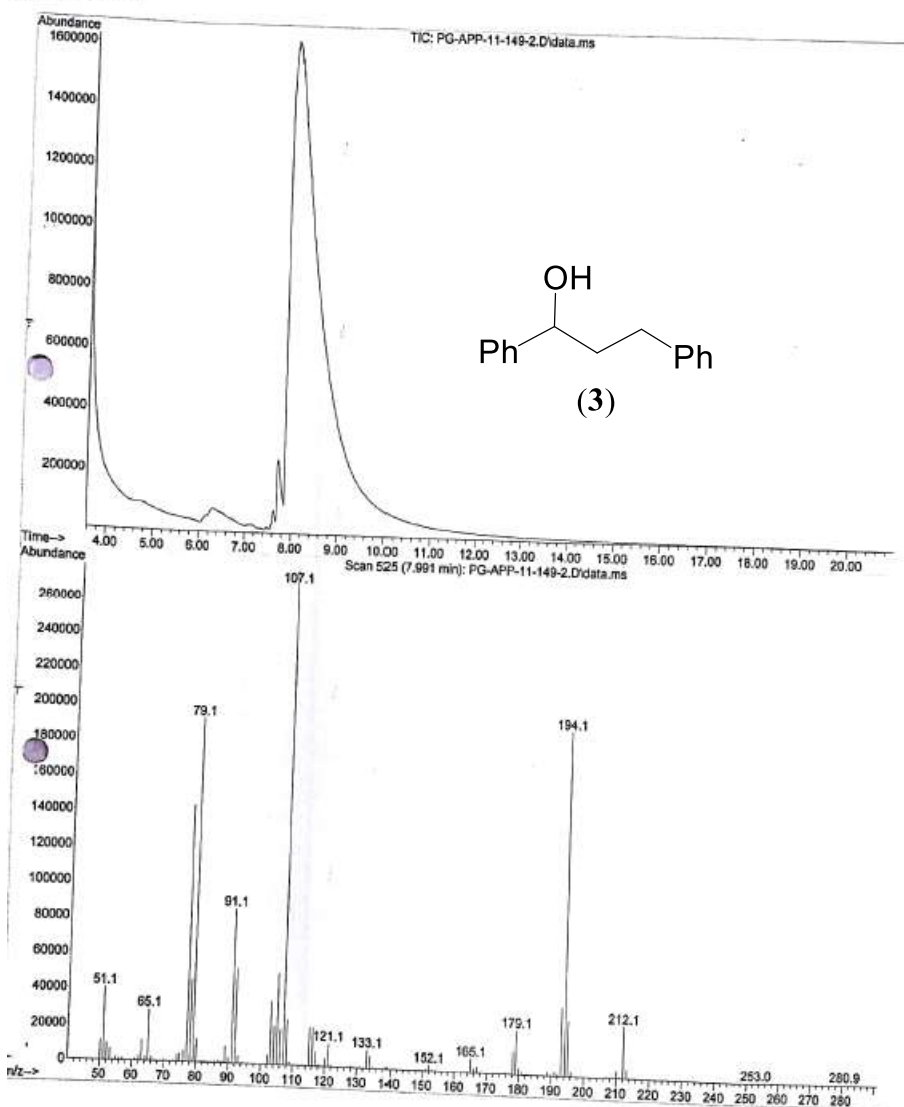
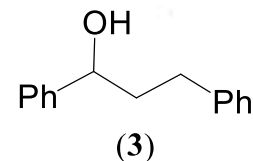


Figure S66. GCMS trace in EtOAc of (3) showing the  $M^+$  peak at  $m/z$  212.

### Eager 300 Report

Page: 1    Sample: PG-APP-11-150-2-2 (PG-APP-11-150-2-2)



```

Method Name   : PGAPP21112018
Method File   : D:\CHNS2018\PGAPP21112018.mth
Chromatogram  : PG-APP-11-150-2-2
Operator ID   : Prakash
Analysed      : 11/21/2018 22:27
Sample ID     : PG-APP-11-150-2-2 (# 28)

Company Name  : C.E. Instruments
Printed       : 11/23/2018 11:01
Instrument N. : Instrument #1
Sample weight : 1.125
    
```

Analysis Type : UnkNown (Area)

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.

| Element Name | %       | Ret.Time | Area    | BC | Area ratio | K factor    |
|--------------|---------|----------|---------|----|------------|-------------|
| 1            | 0.0000  | 6        | 137871  | RS |            | 0.0000      |
| Carbon       | 84.1693 | 62       | 2509913 | RS | 1.000000   | .265065E+07 |
| Hydrogen     | 7.4285  | 184      | 532988  | RS | 4.709136   | .637770E+07 |
| Totals       | 91.5978 |          | 3180772 |    |            |             |

Figure S67. Elemental analysis data of (3).

PG-APP-11-150-1-1H

Current Data Parameters  
NAME PG-APP-11-150-1-1H  
EXPNO 7  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20181129  
Time\_ 18.46  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 30.72  
DW 50.000 usec  
DE 6.50 usec  
TE 297.6 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 500.1330885 MHz  
NUC1 1H  
P1 13.35 usec  
PLW1 16.00000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1300000 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

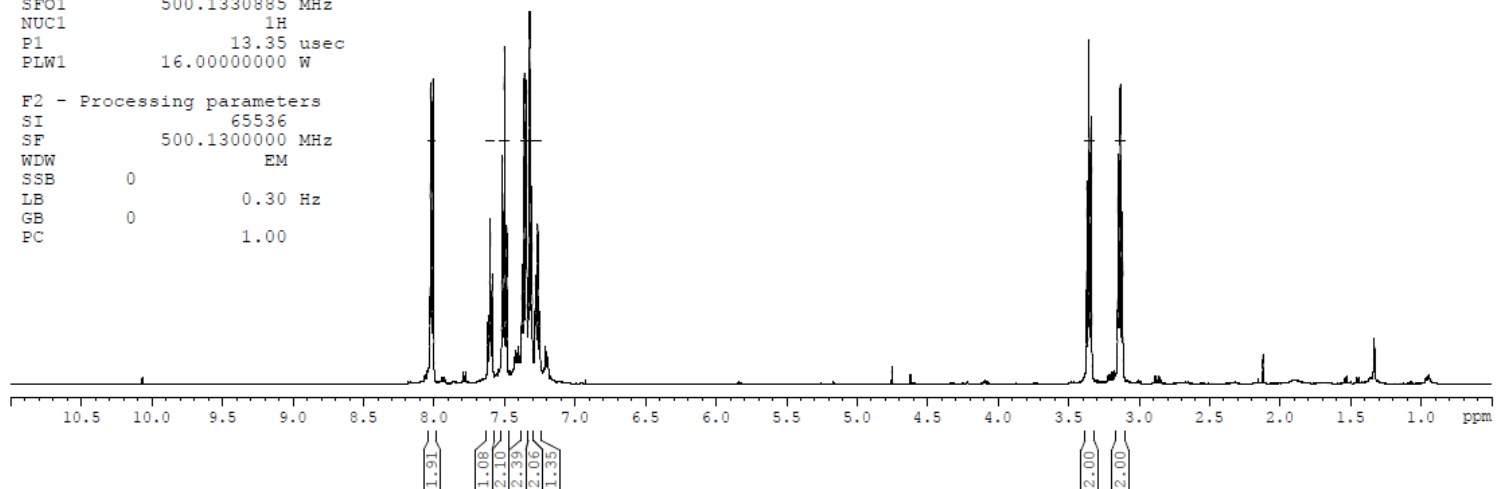
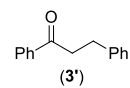
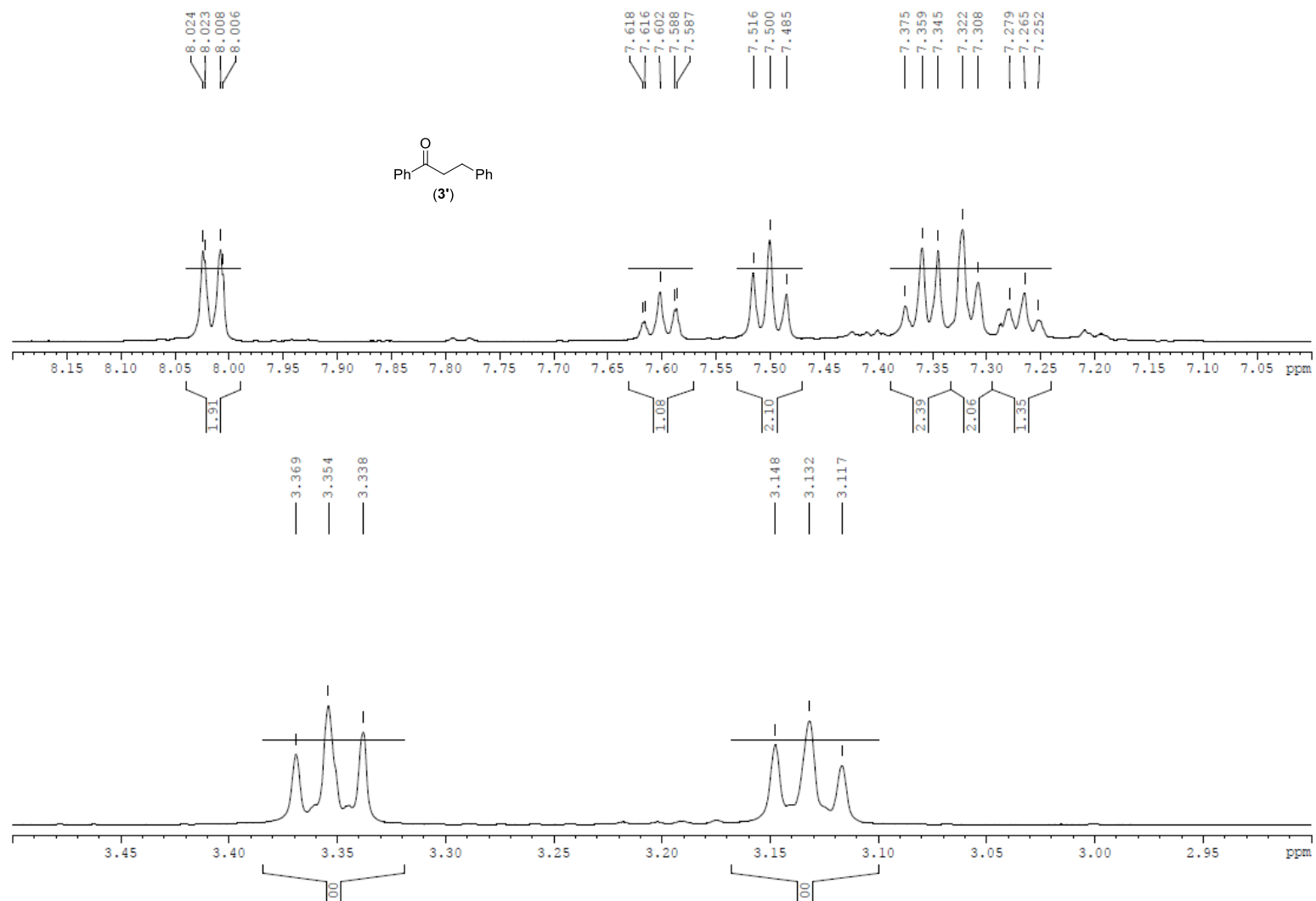


Figure S68. <sup>1</sup>H NMR spectrum of (3') in CDCl<sub>3</sub>.

PG-APP-11-150-1-1H



**Figure S69.** Expanded <sup>1</sup>H NMR spectrum of (3') in CDCl<sub>3</sub>.

PG-APP-11-150-1-13C

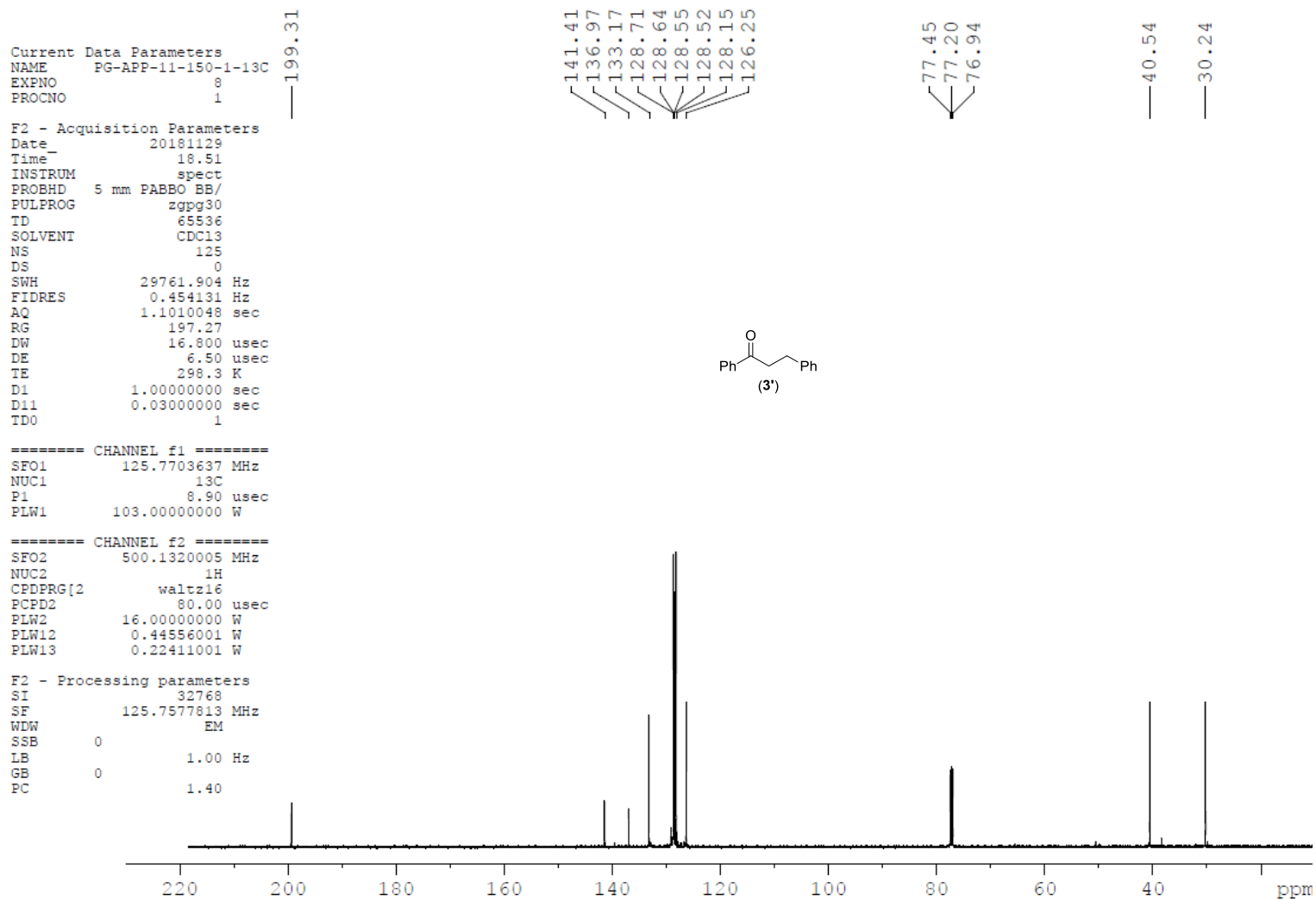
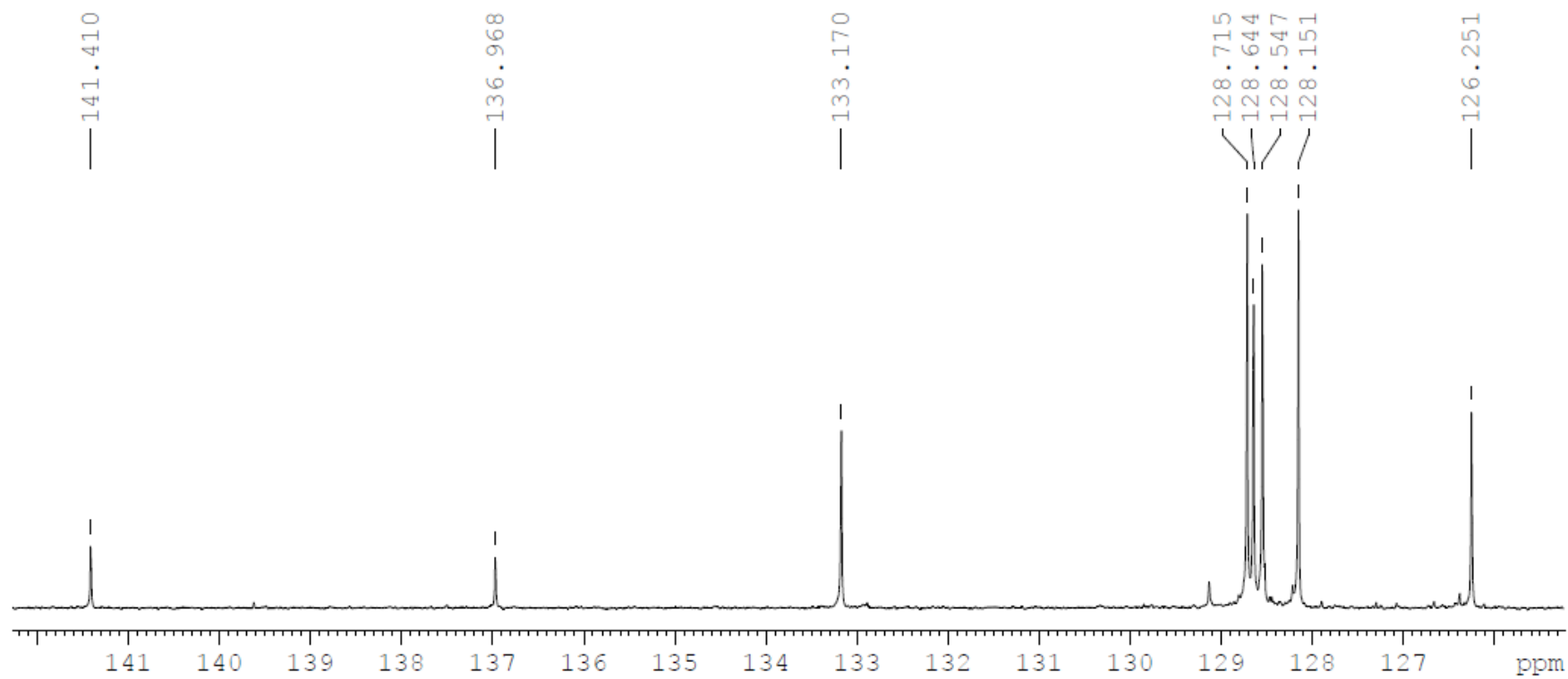
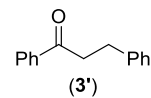


Figure S70.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (3') in  $\text{CDCl}_3$ .

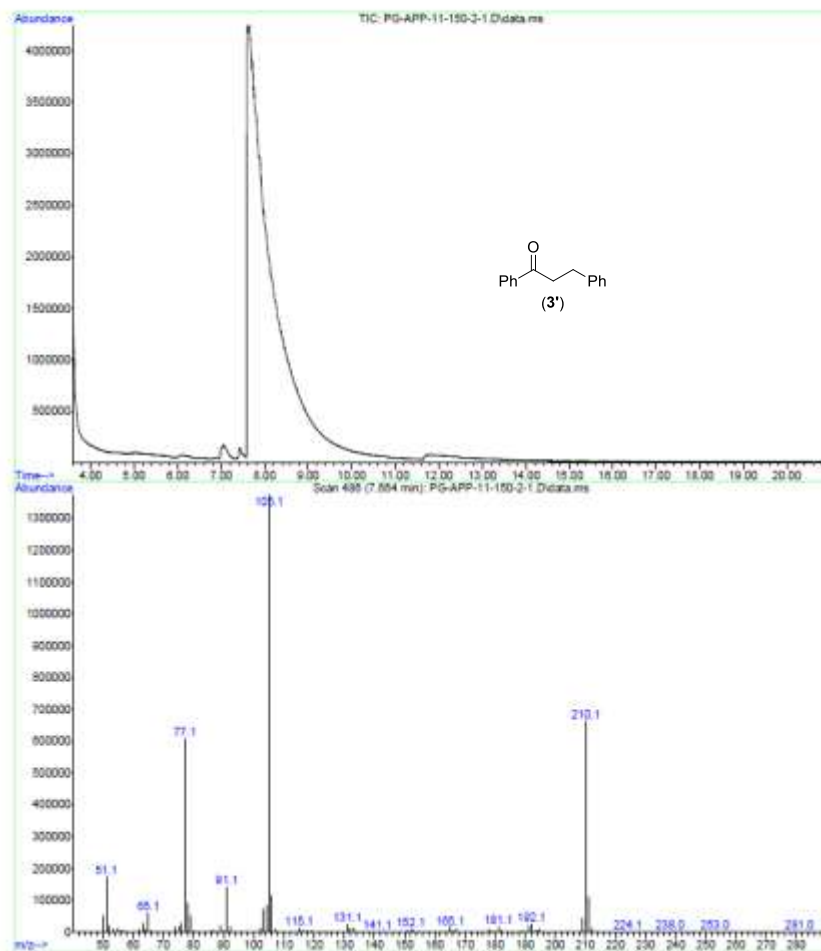
PG-APP-11-150-1-13C



**Figure S71.** Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (3') in  $\text{CDCl}_3$ .



File : F:\GCMSDATA\2018\NOV 2018\PG-APP-11-190-2-1.D  
Operator : ASF  
Acquired : 13 Nov 2018 11:47 using AcqMethod GCMS\METHOD\_2018.H  
Instrument : GCMS  
Sample Name : PG-APP-11-190-2-1  
Misc Info :  
Vial Number: 1



**Figure S72.** GCMS trace in EtOAc of (3') showing the  $M^+$  peak at  $m/z$  210.

PG-APP-12-13-1-1H

NAME PG-APP-12-13-1-1H  
EXPNO 1  
PROCNO 1  
Date\_ 20190529  
Time\_ 8.18  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2999091 sec  
RG 64  
DN 60.800 usec  
DE 6.50 usec  
TE 296.1 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.75 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SFO1 400.1324710 MHz  
SI 32768  
SF 400.1300095 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

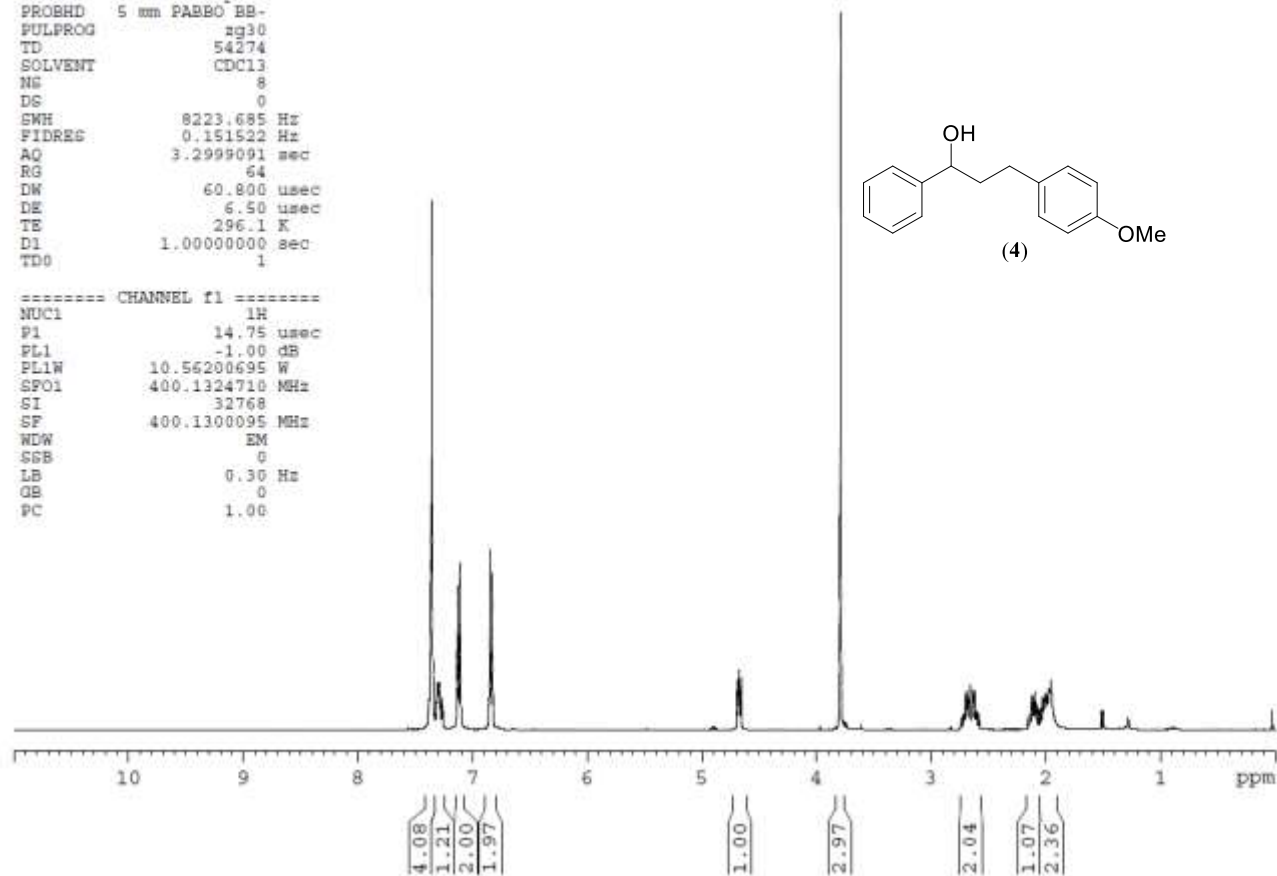
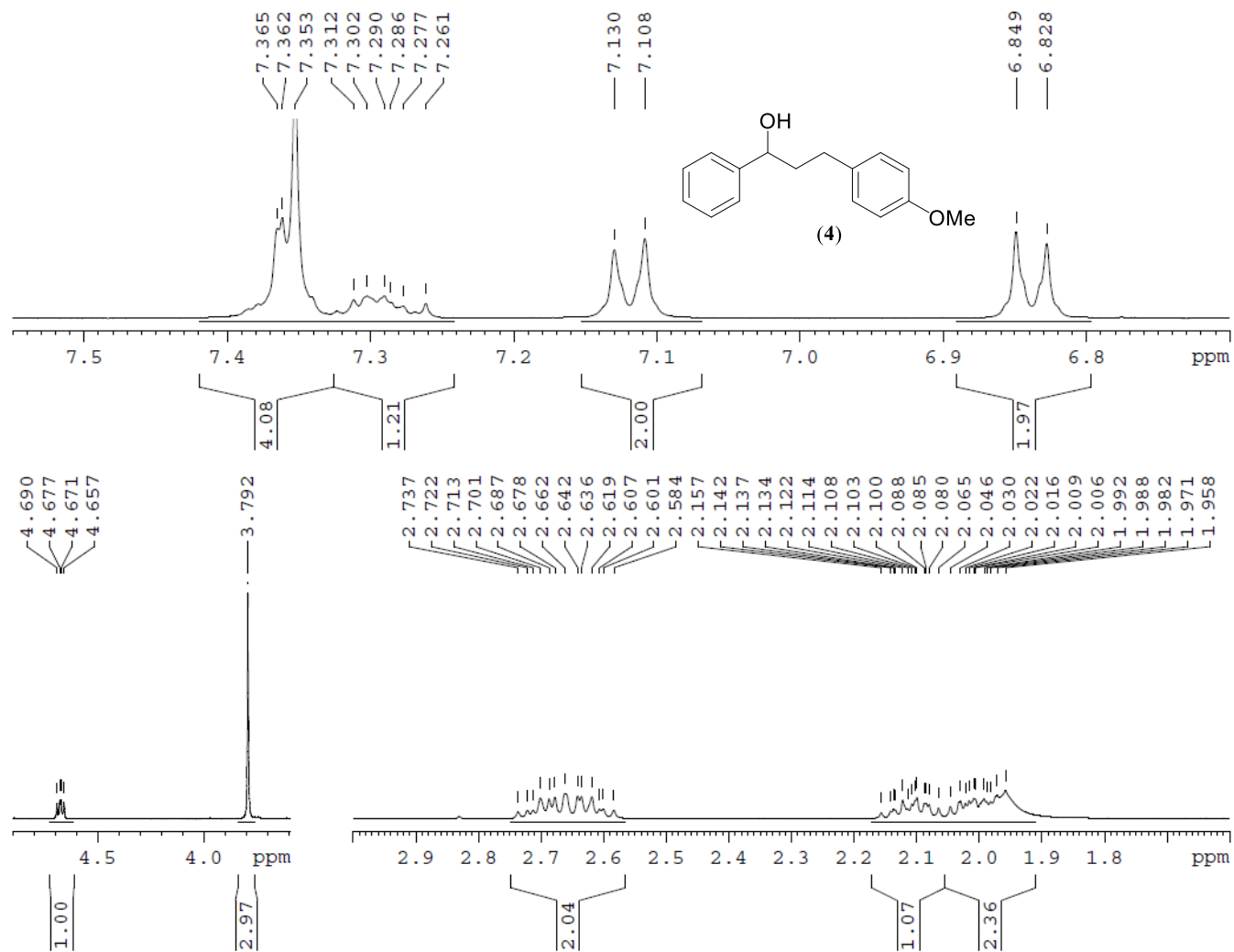


Figure S73. <sup>1</sup>H NMR spectrum of (4) in CDCl<sub>3</sub>.

PG-APP-12-13-1-1H



**Figure S74.** Expanded  $^1\text{H}$  NMR spectrum of (4) in  $\text{CDCl}_3$ .

PG-APP-12-13-1-13C

NAME PG-APP-12-13-1-13C  
EXPNO 2  
PROCNO 1  
Date\_ 20190529  
Time 8.18  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 75  
DS 0  
SWH 26041.666 Hz  
FIDRES 0.397364 Hz  
AQ 1.2583412 sec  
RG 1030  
DW 19.200 usec  
DE 6.50 usec  
TE 296.2 K  
D1 1.00000000 sec  
D11 0.03000000 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 8.50 usec  
PL1 -2.00 dB  
PL1W 56.53121948 W  
SFO1 100.6238364 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -1.00 dB  
PL12 13.69 dB  
PL13 14.50 dB  
PL2W 10.56200695 W  
PL12W 0.35871249 W  
PL13W 0.29767781 W  
SFO2 400.1316005 MHz  
SI 32768  
SF 100.6127567 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

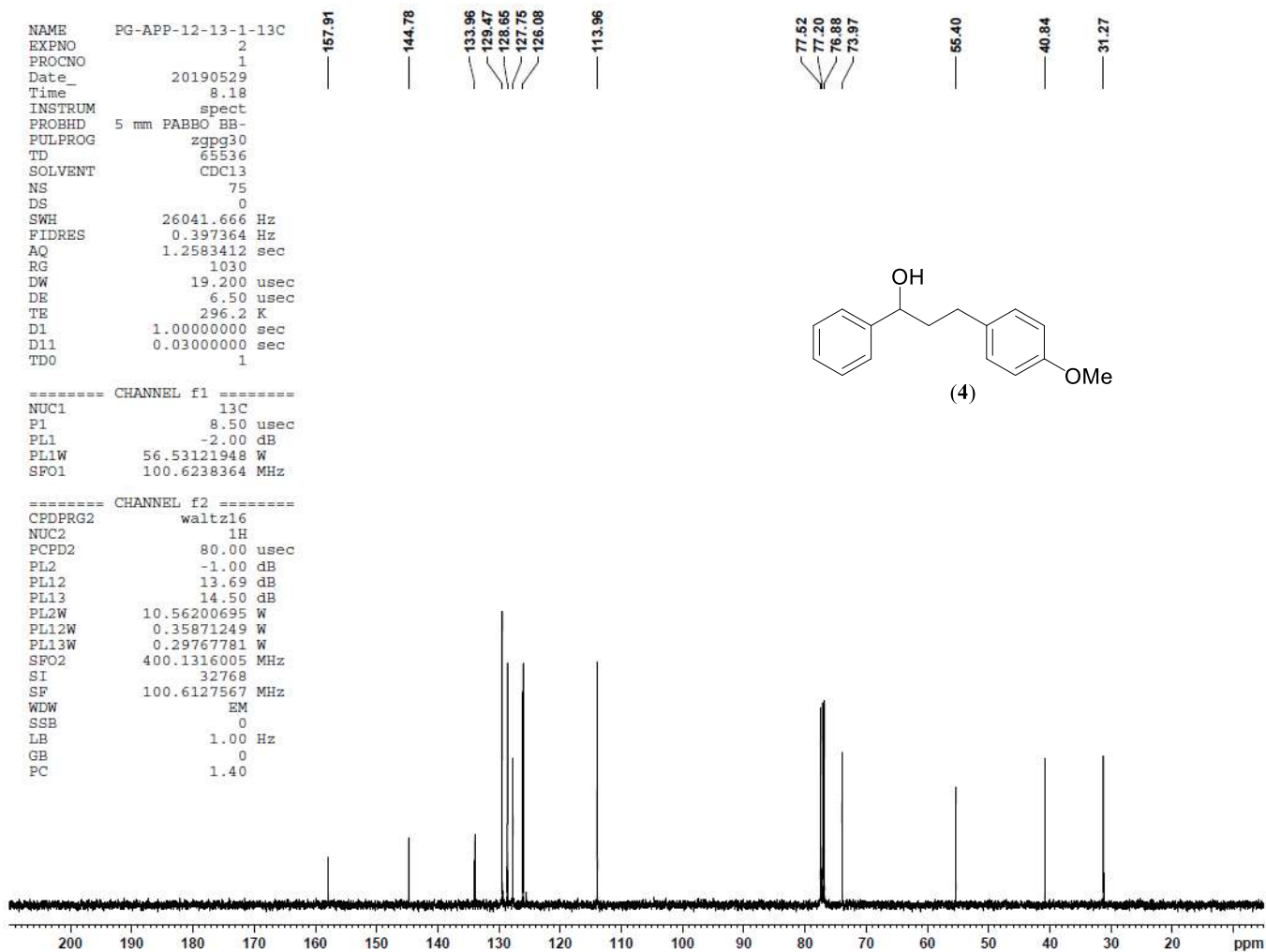
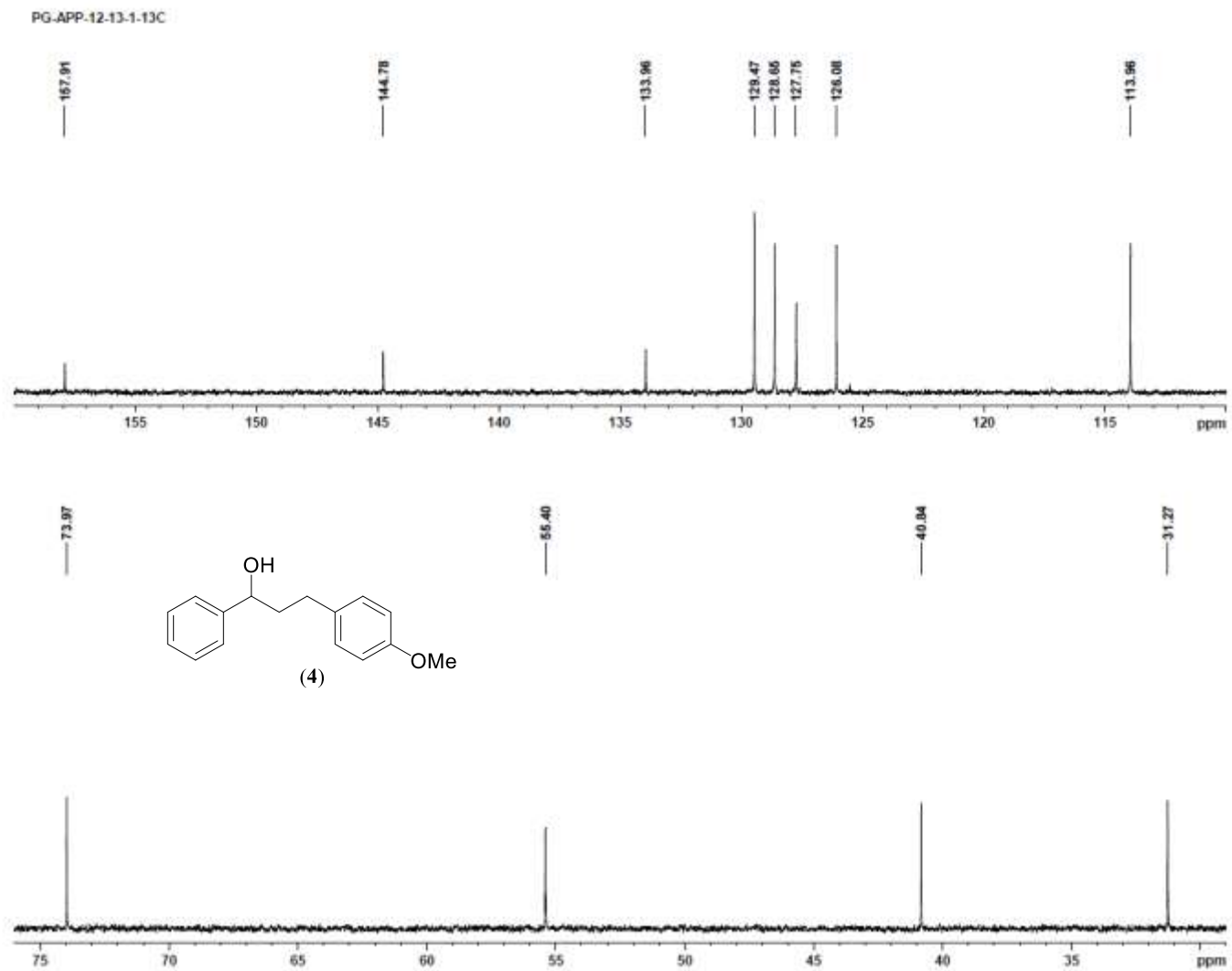
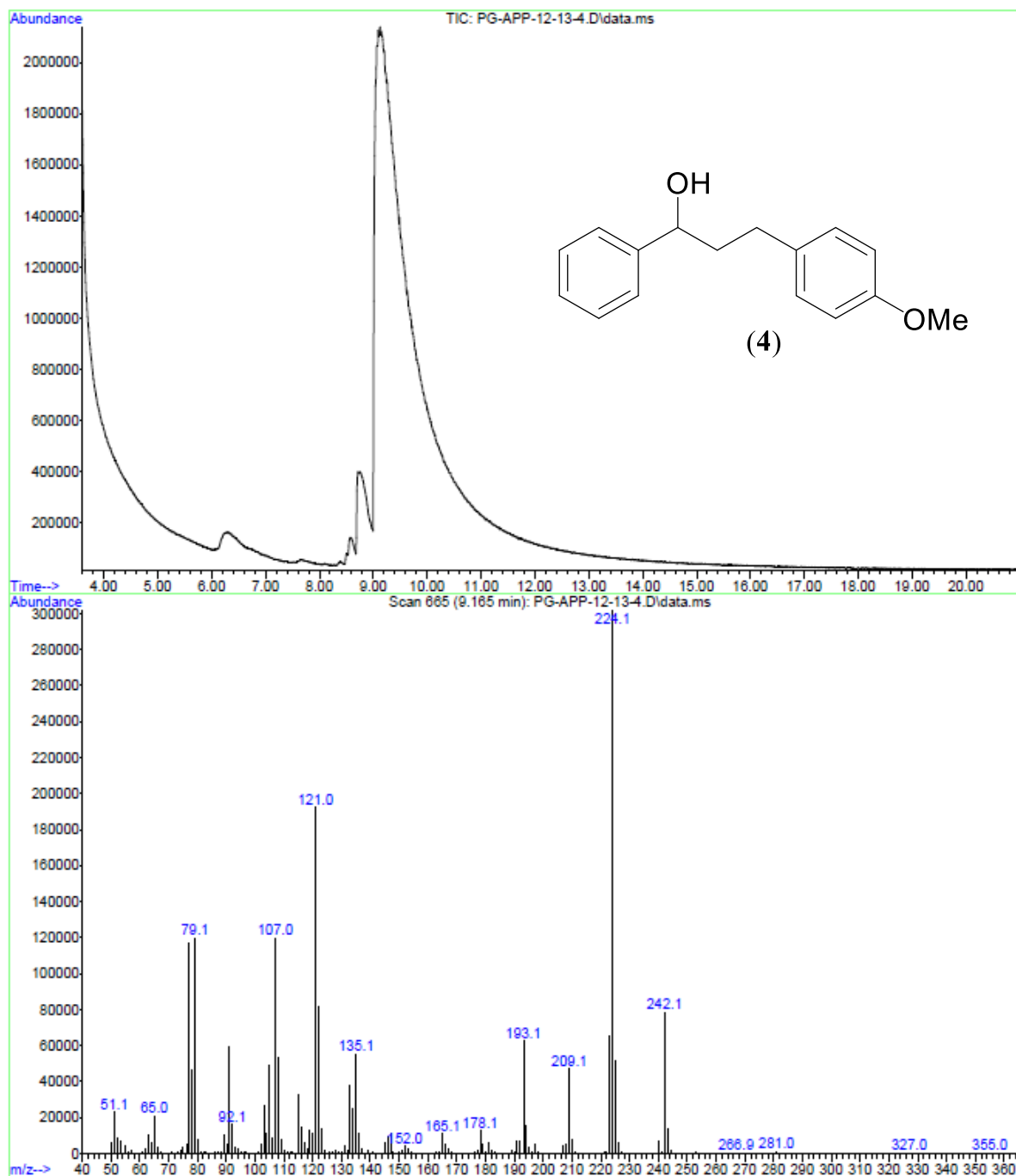


Figure S75.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (4) in  $\text{CDCl}_3$ .



**Figure S76.** Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (4) in  $\text{CDCl}_3$ .

File : F:\GCMSDATA2019\May 2019\PG-APP-12-13-4.D  
Operator : PG  
Acquired : 28 May 2019 17:55 using AcqMethod COMMONMETHOD-2018.M  
Instrument : GCMS  
Sample Name: PG-APP-12-13-4  
Misc Info :  
Vial Number: 3



**Figure S77.** GCMS trace in EtOAc of (4) showing the  $M^+$  peak at  $m/z$  242.

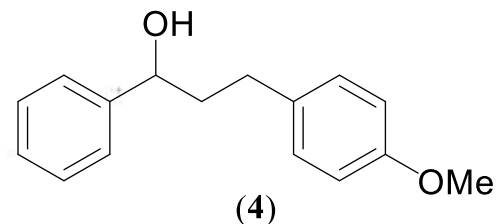
## Eager 300 Report

Page: 1    Sample: PG-APP-12-13-4 (PG-APP-12-13-4)

```

Method Name      : PGAPP300519
Method File     : D:\CHNS2019\PGAPP300519.mth
Chromatogram    : PG-APP-12-13-4
Operator ID     : Prakash
Analysed        : 05/30/2019  14:30
Sample ID       : PG-APP-12-13-4 (# 19)
Analysis Type   : UnkNown (Area)

Company Name    : C.E. Instruments
Printed         : 5/30/2019  22:59
Instrument N.   : Instrument #1
Sample weight   : .789
    
```



Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.

| Element Name | %       | Ret. Time | Area    | BC | Area ratio | K factor    |
|--------------|---------|-----------|---------|----|------------|-------------|
| 1            | 0.0000  | 6         | 103472  | RS |            | 0.0000      |
| Carbon       | 78.9676 | 64        | 1664418 | RS | 1.000000   | .267139E+07 |
| Hydrogen     | 7.0210  | 184       | 378670  | RS | 4.395432   | .683575E+07 |
| Totals       | 85.9886 |           | 2146560 |    |            |             |

Figure S78. Elemental analysis data of (4).

PG-APP-11-208-2-1H

Current Data Parameters  
NAME PG-APP-11-208-2-1H  
EXPNO 13  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20190314  
Time\_ 22.24  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 157.24  
DW 50.000 usec  
DE 6.50 usec  
TE 297.1 K  
D1 1.00000000 sec  
TD0 1

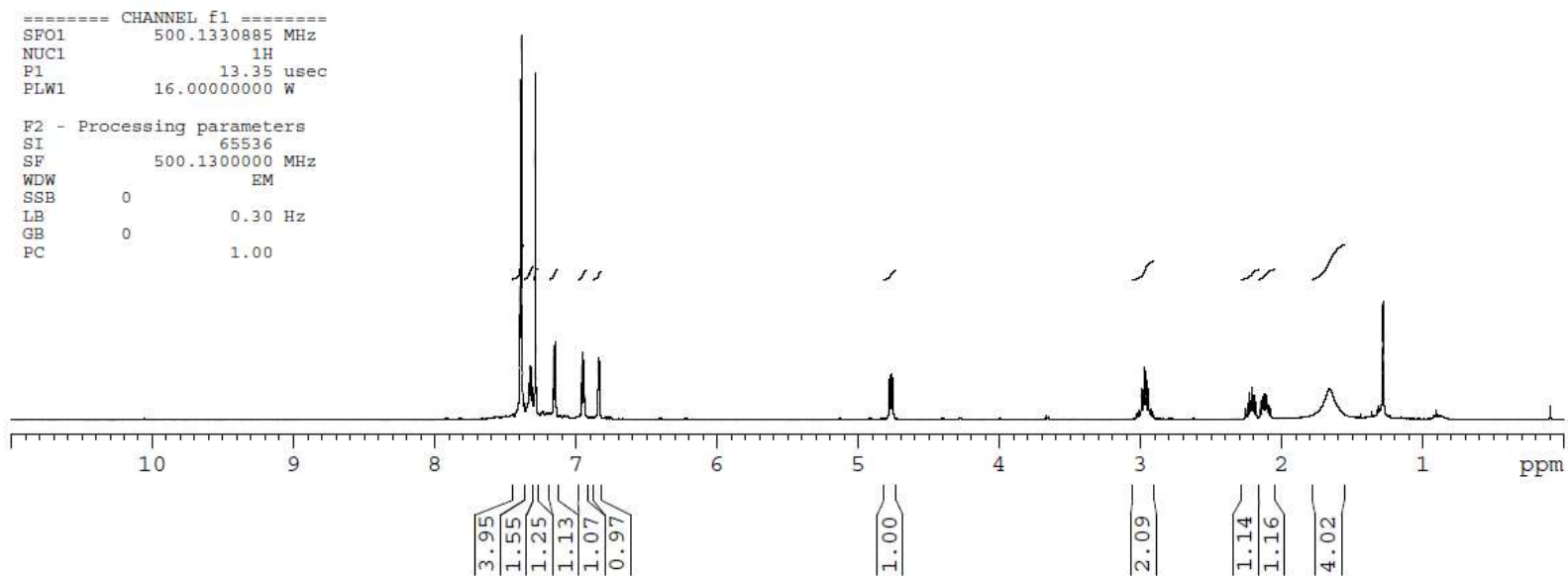
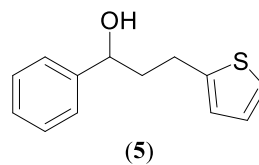
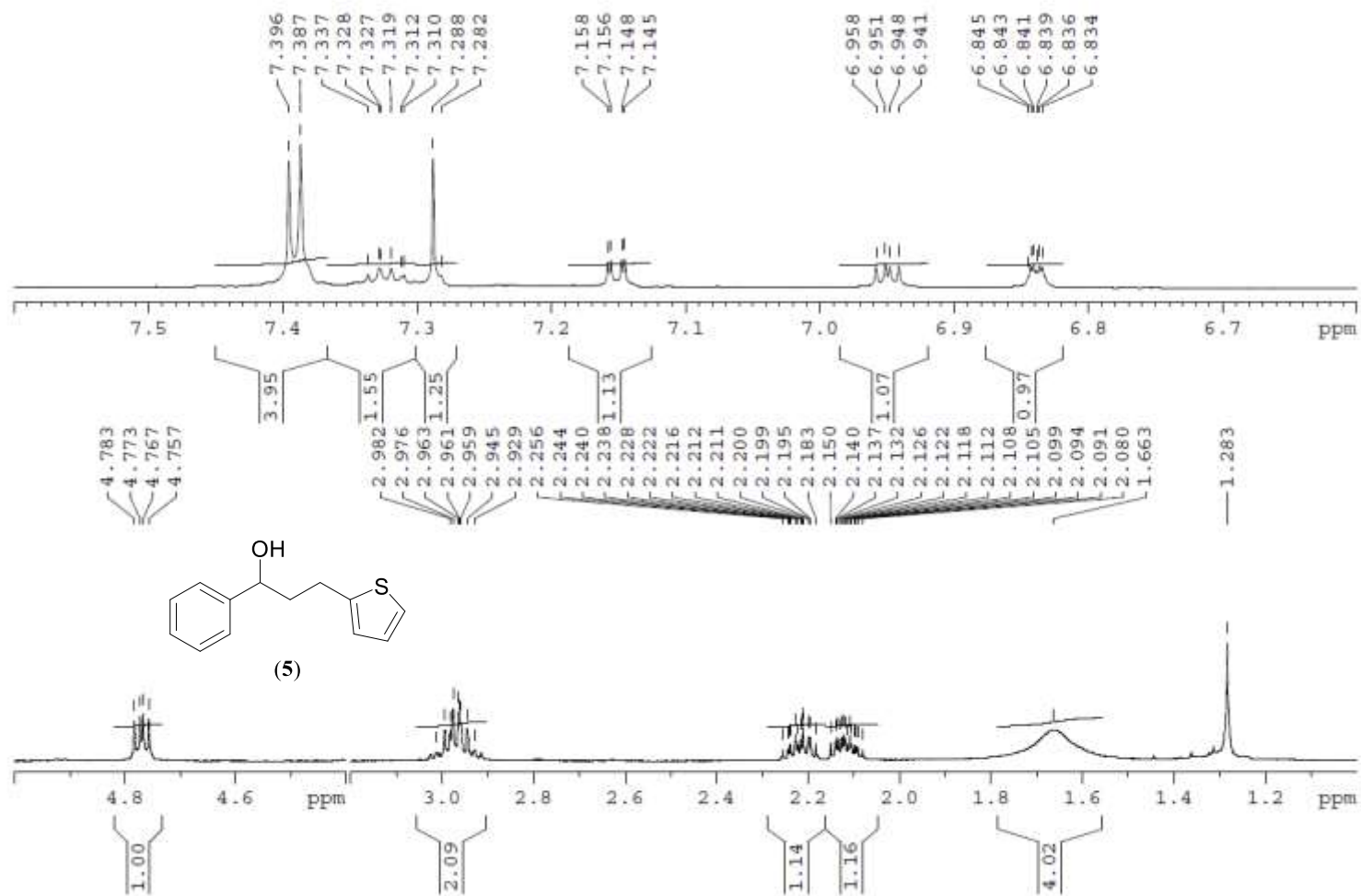


Figure S79. <sup>1</sup>H NMR spectrum of (5) in CDCl<sub>3</sub>.



PG-APP-11-208-2-1H



**Figure S80.** Expanded  $^1\text{H}$  NMR spectrum of (5) in  $\text{CDCl}_3$ .

PG-APP-11-208-2-13C

Current Data Parameters  
NAME PG-APP-11-208-2-13C  
EXPNO 14  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20190314  
Time\_ 22.26  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 250  
DS 0  
SWH 29761.904 Hz  
FIDRES 0.454131 Hz  
AQ 1.1010048 sec  
RG 197.27  
DW 16.800 usec  
DE 6.50 usec  
TE 297.7 K  
D1 1.00000000 sec  
D11 0.03000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO1 125.7703637 MHz  
NUC1 13C  
P1 8.90 usec  
PLW1 103.00000000 W

===== CHANNEL f2 =====  
SFO2 500.1320005 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 80.00 usec  
PLW2 16.00000000 W  
PLW12 0.44556001 W  
PLW13 0.22411001 W

F2 - Processing parameters  
SI 32768  
SF 125.7577673 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

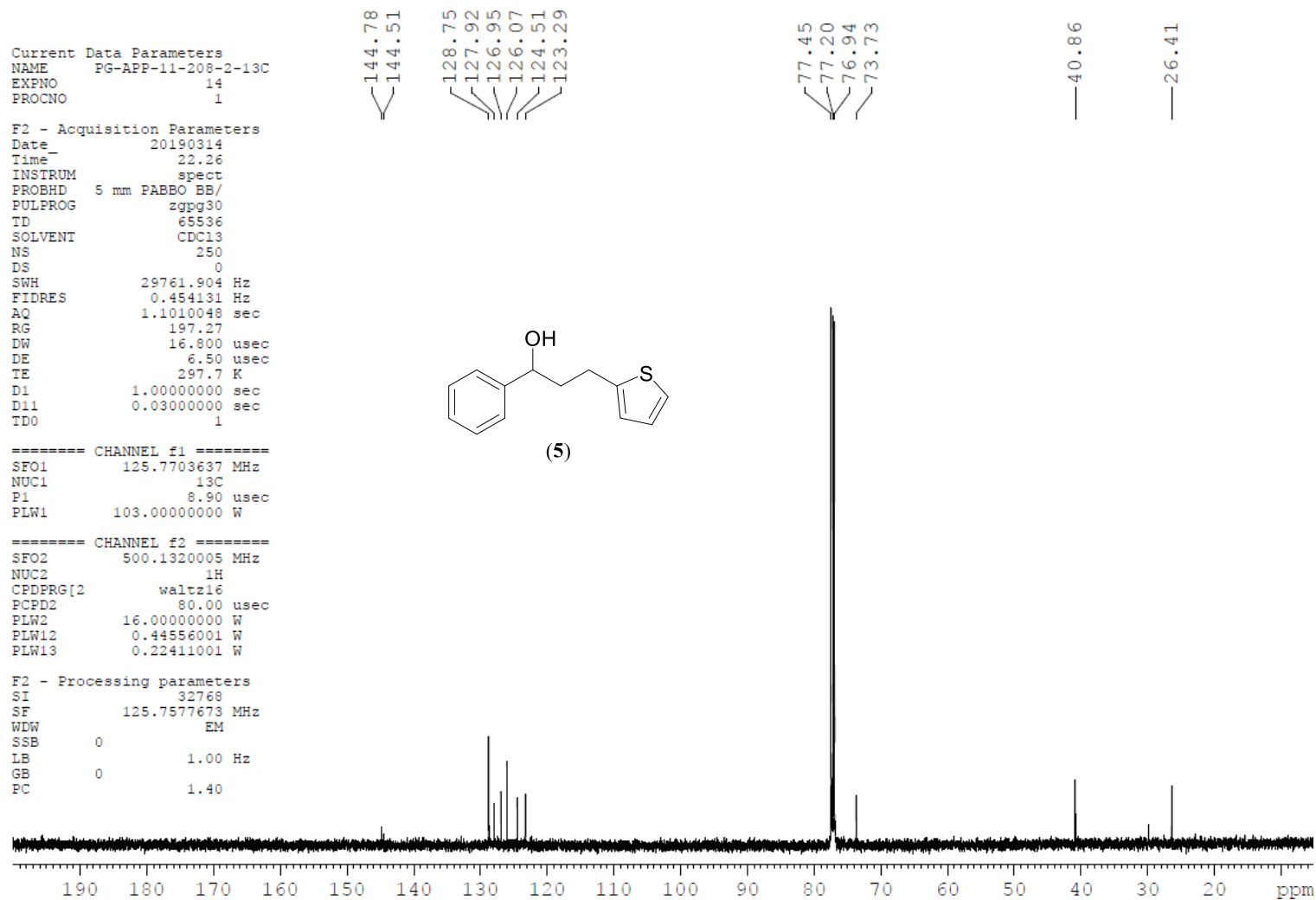


Figure S81.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (5) in  $\text{CDCl}_3$ .

File :F:\GCMS-DATA-2021\APRIL 2021\PG-SRD-4-61-1R.D  
Operator : JS  
Acquired : 18 Apr 2021 14:23 using AcqMethod COMMONMETHOD-2020.M  
Instrument : GCMS  
Sample Name: PG-SRD-4-61-1R  
Misc Info :  
Vial Number: 3

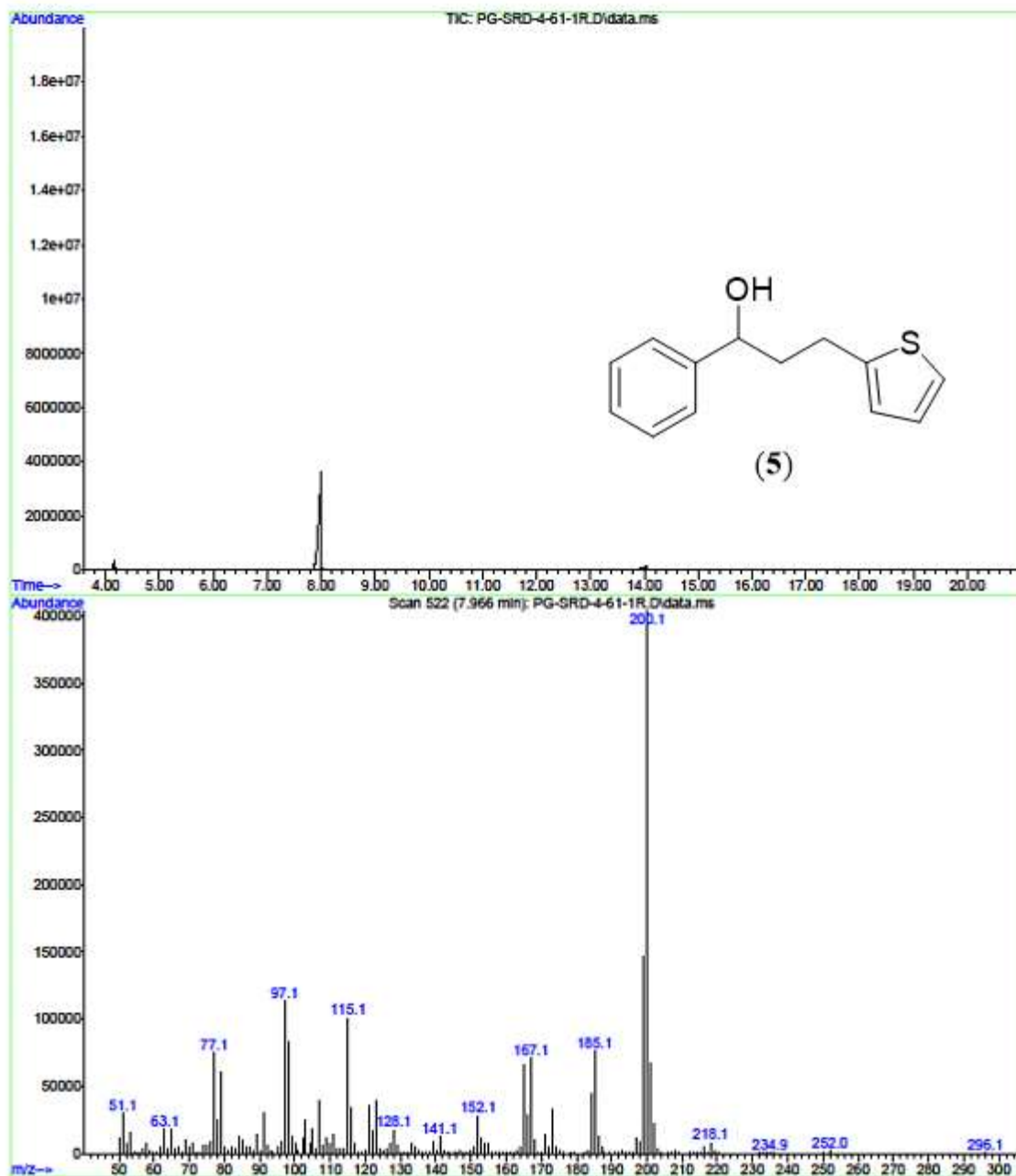


Figure S82. GCMS trace in EtOAc of (5) showing the  $M^+$  peak at  $m/z$  218.

Document: SP-13-04-2021 (varioMICRO) from: --- (modified)

SP18022016  
varioMICRO CHNS  
serial number: 15154051

Graphic report

| No. | Weight [mg] | Name          | Method     | N Area | C Area | H Area | S Area | N (%) | C (%) | H (%) | S (%)  | Date       | Time  |
|-----|-------------|---------------|------------|--------|--------|--------|--------|-------|-------|-------|--------|------------|-------|
| 16  | 1.3380      | PG-SRD-4-61-1 | 2mgChem80s | 2530   | 27127  | 8053   | 2152   | 0.00  | 71.76 | 6.488 | 14.336 | 13-04-2021 | 12:57 |

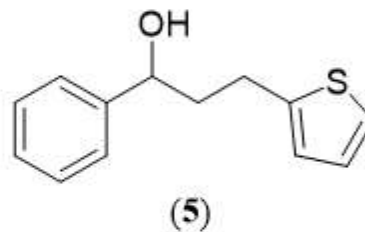
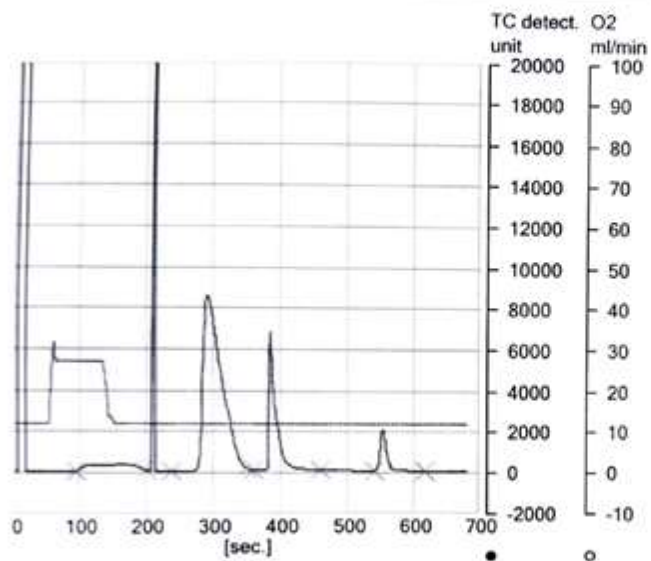


Figure S83. Elemental analysis data of (5).

PG-APP-11-210-1-1H

Current Data Parameters  
NAME PG-APP-11-210-1-1H  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20190314  
Time 21.58  
INSTRUM spect  
PROBHD 5 mm PASPO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 177.33  
DW 50.000 usec  
DE 6.50 usec  
TE 297.2 K  
D1 1.00000000 sec  
TDO 1

----- CHANNEL f1 -----  
SFO1 500.1330885 MHz  
NUC1 1H  
P1 13.35 usec  
PLW1 16.00000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1306479 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

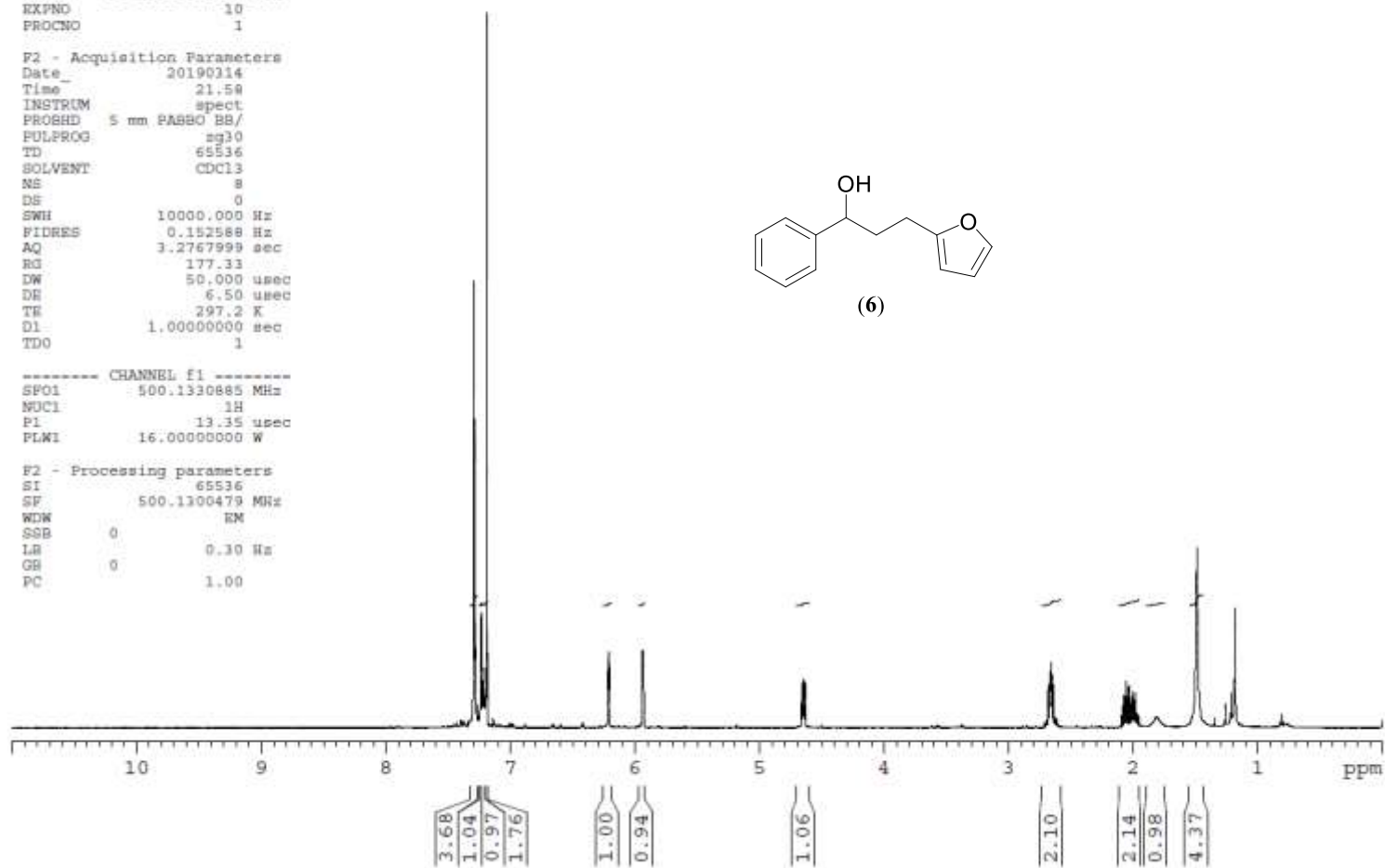


Figure S84. <sup>1</sup>H NMR spectrum of (6) in CDCl<sub>3</sub>.

PG-APP-11-210-1-1H

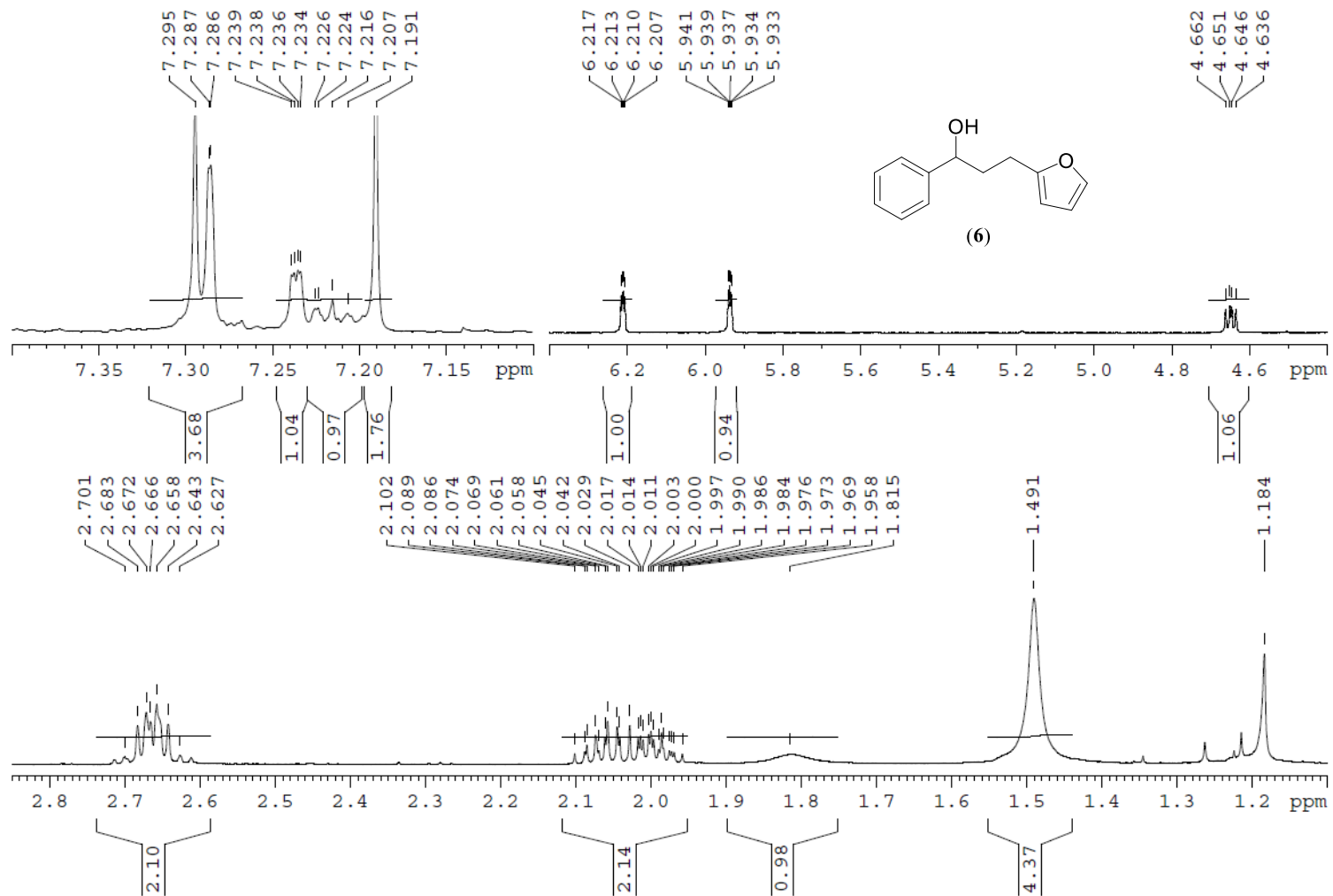


Figure S85. Expanded  $^1\text{H}$  NMR spectrum of (6) in  $\text{CDCl}_3$ .

PG-APP-11-210-1-13C

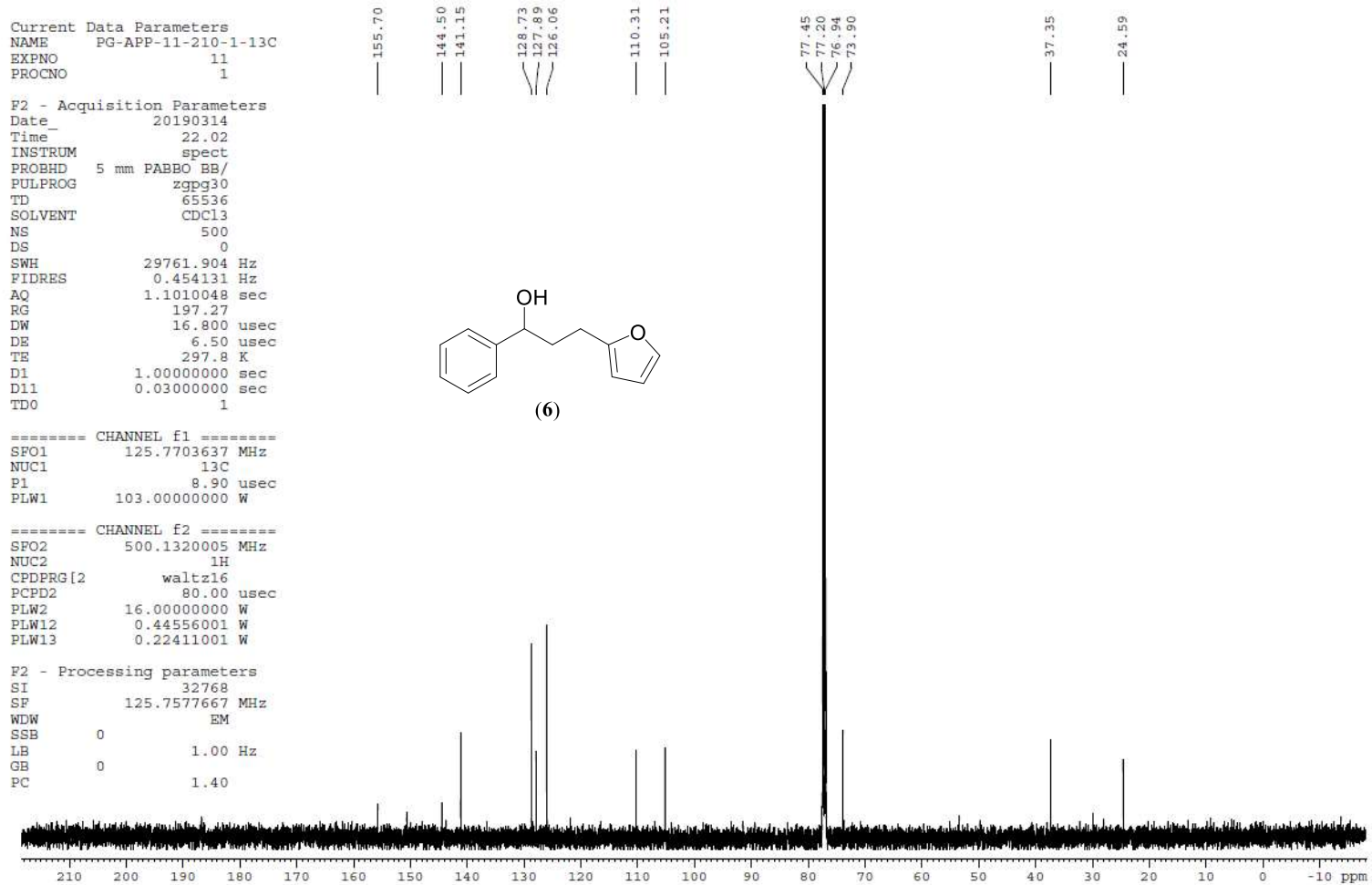
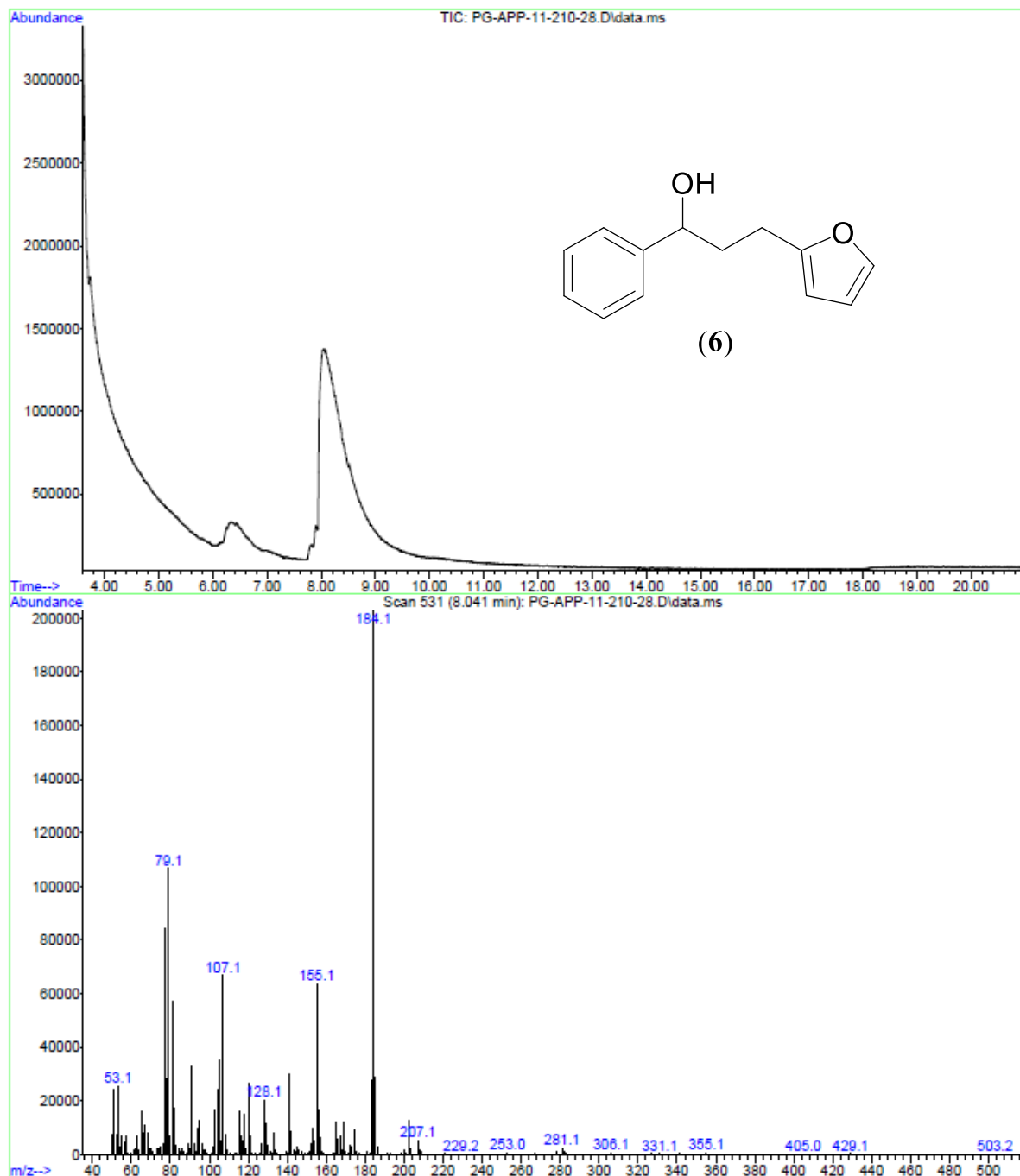


Figure S86.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (6) in  $\text{CDCl}_3$ .

File : F:\GCMSDATA2019\MAR 2019\PG-APP-11-210-28.D  
Operator : APP  
Acquired : 13 Mar 2019 23:00 using AcqMethod COMMONMETHOD\_2018.M  
Instrument : GCMS  
Sample Name: PG-APP-11-210-28  
Misc Info :  
Vial Number: 1

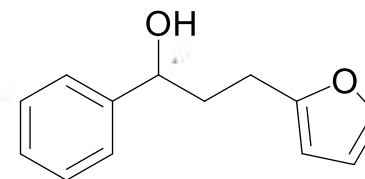


**Figure S87.** GCMS trace in EtOAc of (6) showing the  $M^+$  peak at  $m/z$  202.



# Eager 300 Report

Page: 1 Sample: PG-APP-11-210-5 (PG-APP-11-210-5)



(6)

Method Name : PGAPP300519  
 Method File : D:\CHNS2019\PGAPP300519.mth  
 Chromatogram : PG-APP-11-210-5  
 Operator ID : Prakash  
 Analysed : 05/30/2019 17:32  
 Sample ID : PG-APP-11-210-5 (# 35)  
 Analysis Type : UnkNown (Area)

Company Name : C.E. Instruments  
 Printed : 5/30/2019 23:00  
 Instrument N. : Instrument #1  
 Sample weight : 1.462

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.

| Element Name | %       | Ret. Time | Area    | BC | Area ratio | K factor    |
|--------------|---------|-----------|---------|----|------------|-------------|
| 1            | 0.0000  | 2         | 21547   | FU |            |             |
| 2            | 0.0000  | 6         | 123910  | FU |            | 0.0000      |
| Carbon       | 77.7111 | 63        | 3035056 | RS | 1.000000   | .267139E+07 |
| Hydrogen     | 7.3492  | 187       | 734473  | RS | 4.132291   | .683575E+07 |
| Totals       | 85.0603 |           | 3914986 |    |            |             |

Figure S88. Elemental analysis data of (6).

PG-APP-11-209-1-1H

```
NAME      PG-APP-11-209-1-1H
EXPNO     1
PROCNO    1
Date_     20190313
Time      8.18
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD        54274
SOLVENT   CDCl3
NS        8
DS        0
SWH       8223.685 Hz
FIDRES    0.151522 Hz
AQ        3.2999091 sec
RG        57
DW        60.800 usec
DE        6.50 usec
TE        296.7 K
D1        1.00000000 sec
TDO       1
```

```
***** CHANNEL f1 *****
NUC1      1H
P1        14.75 usec
PL1       -1.00 dB
PL1W      10.56200695 W
SFO1      400.1324710 MHz
SI        32768
SF        400.1300435 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
```

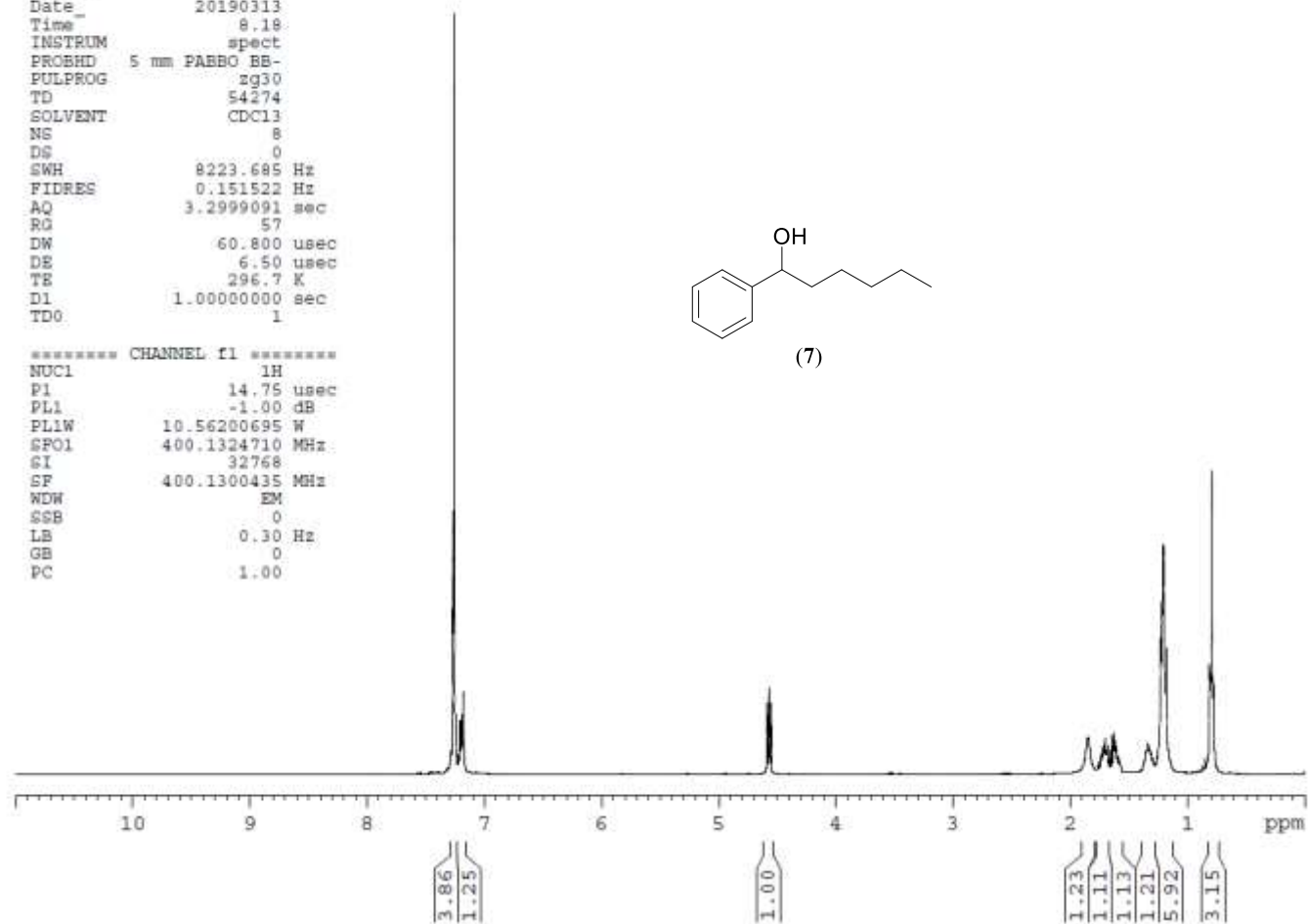
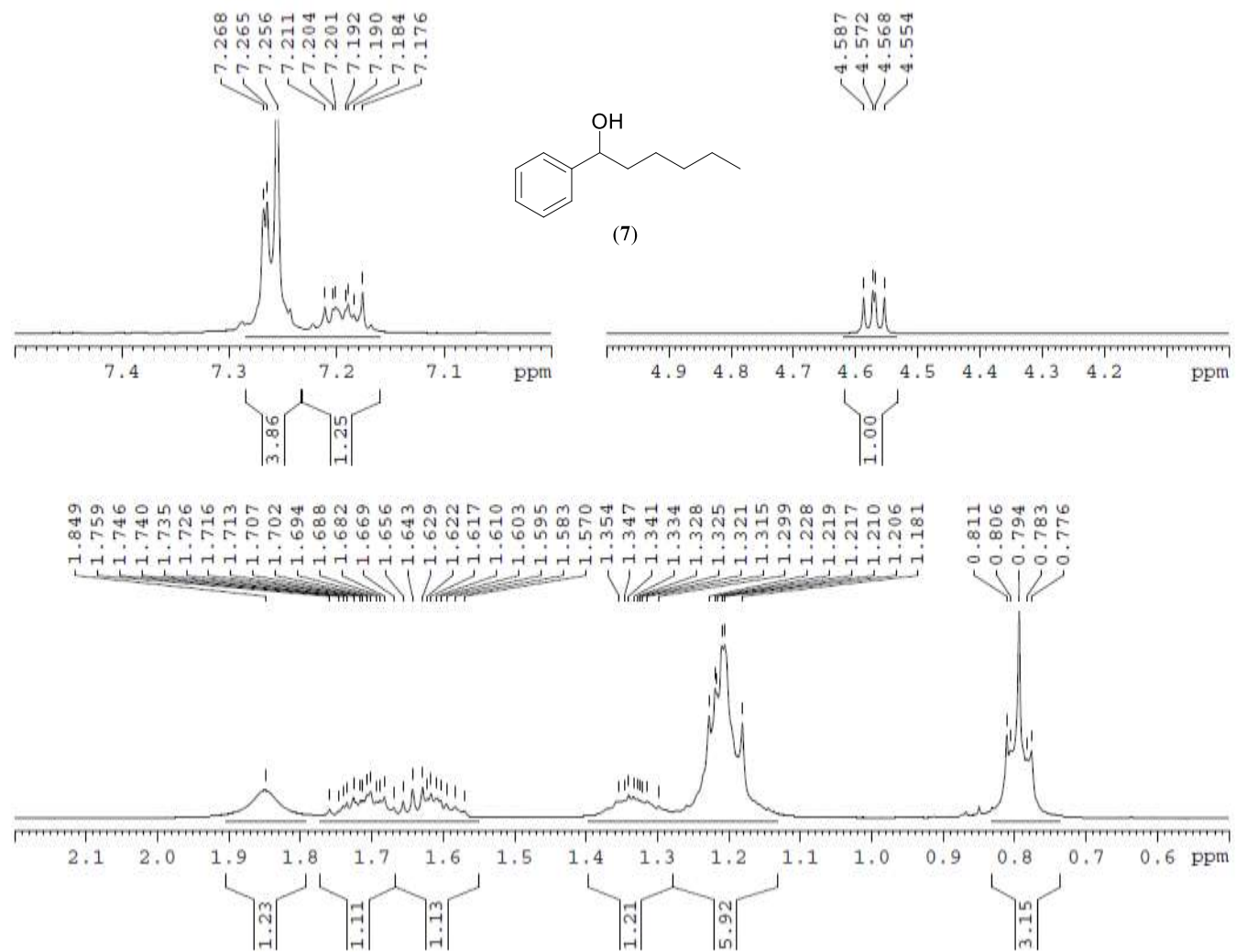


Figure S89. <sup>1</sup>H NMR spectrum of (7) in CDCl<sub>3</sub>.

PG-APP-11-209-1-1H



**Figure S90.** Expanded  $^1\text{H}$  NMR spectrum of (7) in  $\text{CDCl}_3$ .

PG-APP-11-209-1-13C

NAME PG-APP-11-209-1-13C  
EXPNO 2  
PROCNO 1  
Date\_ 20190313  
Time 8.18  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 100  
DS 0  
SWH 26041.666 Hz  
FIDRES 0.397364 Hz  
AQ 1.2583412 sec  
RG 1030  
DW 19.200 usec  
DE 6.50 usec  
TE 296.7 K  
D1 1.00000000 sec  
D11 0.03000000 sec  
TDO 1

----- CHANNEL f1 -----  
NUC1 13C  
P1 8.50 usec  
PL1 -2.00 dB  
PL1W 56.53121948 W  
SFO1 100.6238364 MHz

----- CHANNEL f2 -----  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -1.00 dB  
PL12 13.69 dB  
PL13 14.50 dB  
PL2W 10.56200695 W  
PL12W 0.35871249 W  
PL13W 0.29767781 W  
SFO2 400.1316005 MHz  
SI 32768  
SF 100.6127548 MHz  
WDW EM  
SSR 0  
LB 1.00 Hz  
GB 0  
PC 1.40

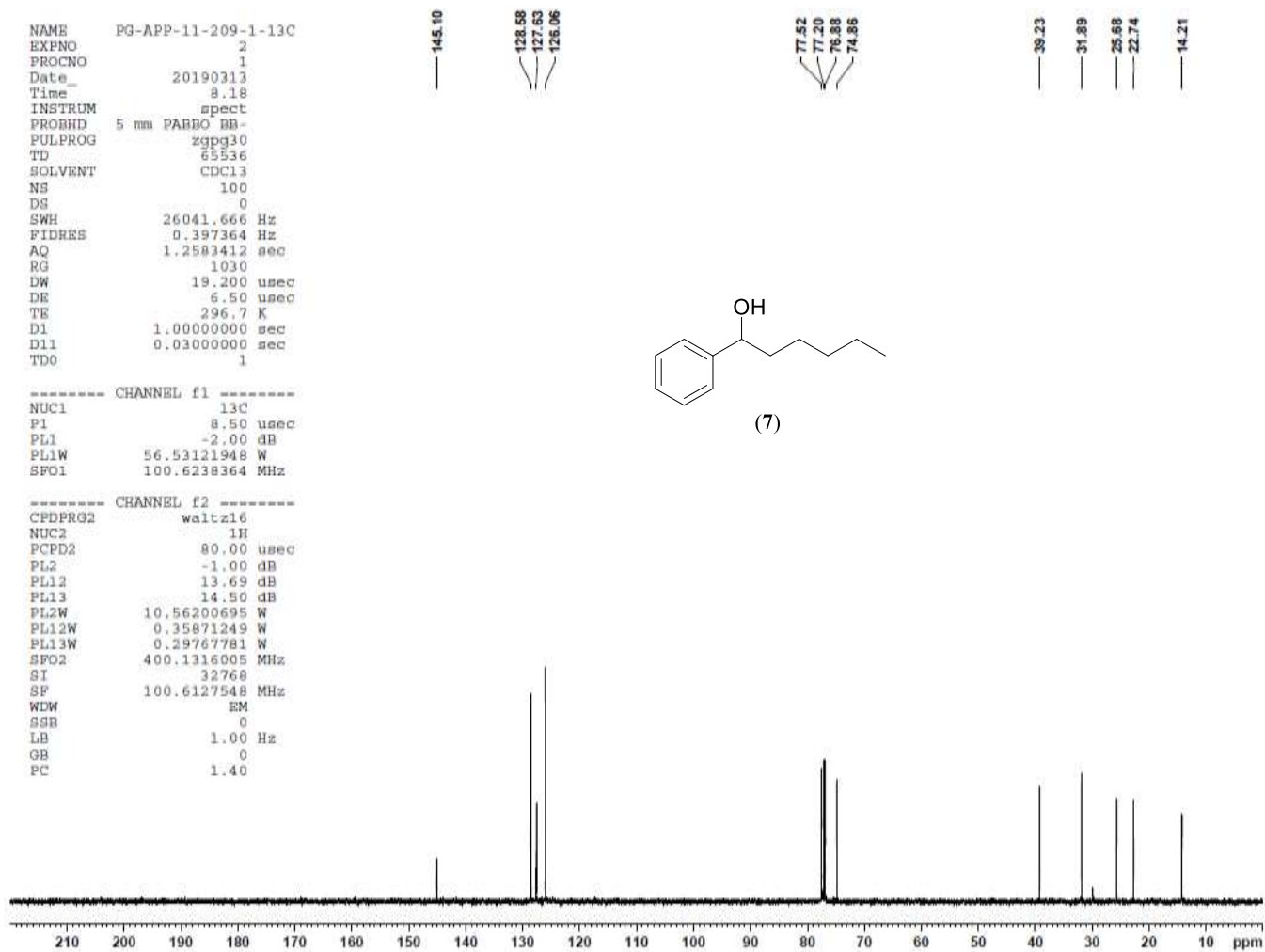
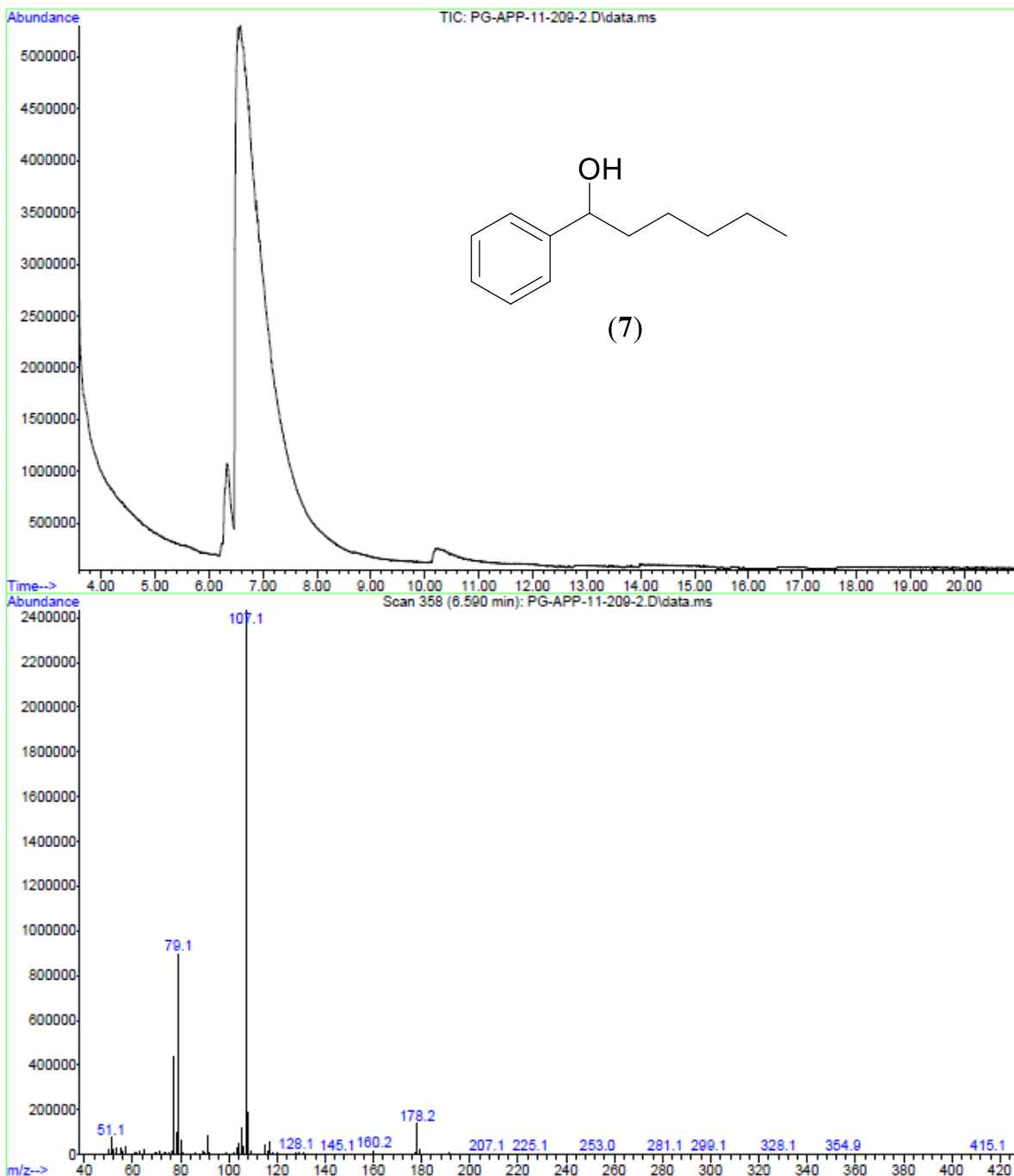


Figure S91.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (7) in  $\text{CDCl}_3$ .

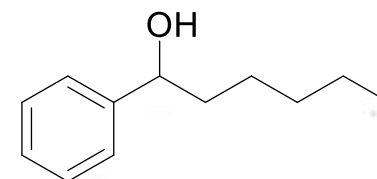
File : F:\GCMSDATA2019\MAR 2019\PG-APP-11-209-2.D  
Operator : pg  
Acquired : 11 Mar 2019 21:53 using AcqMethod COMMONMETHOD\_2018.M  
Instrument : GCMS  
Sample Name: PG-APP-11-209-2  
Misc Info :  
Vial Number: 1



**Figure S92.** GCMS trace in EtOAc of (7) showing the  $M^+$  peak at  $m/z$  178.

# Eager 300 Report

Page: 1 Sample: PG-APP-11-209-2 (PG-APP-11-209-2)



(7)

Method Name : PGAPP060619  
Method File : D:\CHNS2019\PGAPP060619.mth  
Chromatogram : PG-APP-11-209-2  
Operator ID : Prakash  
Analysed : 06/06/2019 15:18  
Sample ID : PG-APP-11-209-2 (# 16)  
Analysis Type : UnkNown (Area)  
Company Name : C.E. Instruments  
Printed : 6/7/2019 08:27  
Instrument N. : Instrument #1  
Sample weight : .979

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.

| Element Name | %       | Ret.Time | Area    | BC | Area ratio | K factor    |
|--------------|---------|----------|---------|----|------------|-------------|
| 1            | 0.0000  | 2        | 141541  | RS |            | 0.0000      |
| Nitrogen     | 6.7411  | 48       | 193184  | FU | 10.811430  | .103544E+07 |
| Carbon       | 80.0470 | 64       | 2088598 | FU | 1.000000   | .265769E+07 |
| Hydrogen     | 10.0655 | 184      | 646869  | RS | 3.228780   | .656444E+07 |
| Totals       | 96.8536 |          | 3070192 |    |            |             |

Figure S93. Elemental analysis data of (7).

PG-APP-11-230-1-1H

Current Data Parameters  
NAME PG-APP-11-230-1-1H  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20190416  
Time 8.42  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 106.54  
DW 50.000 usec  
DE 6.50 usec  
TE 298.2 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
SFO1 500.1330885 MHz  
NUC1 1H  
P1 13.35 usec  
PLW1 16.0000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1300000 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

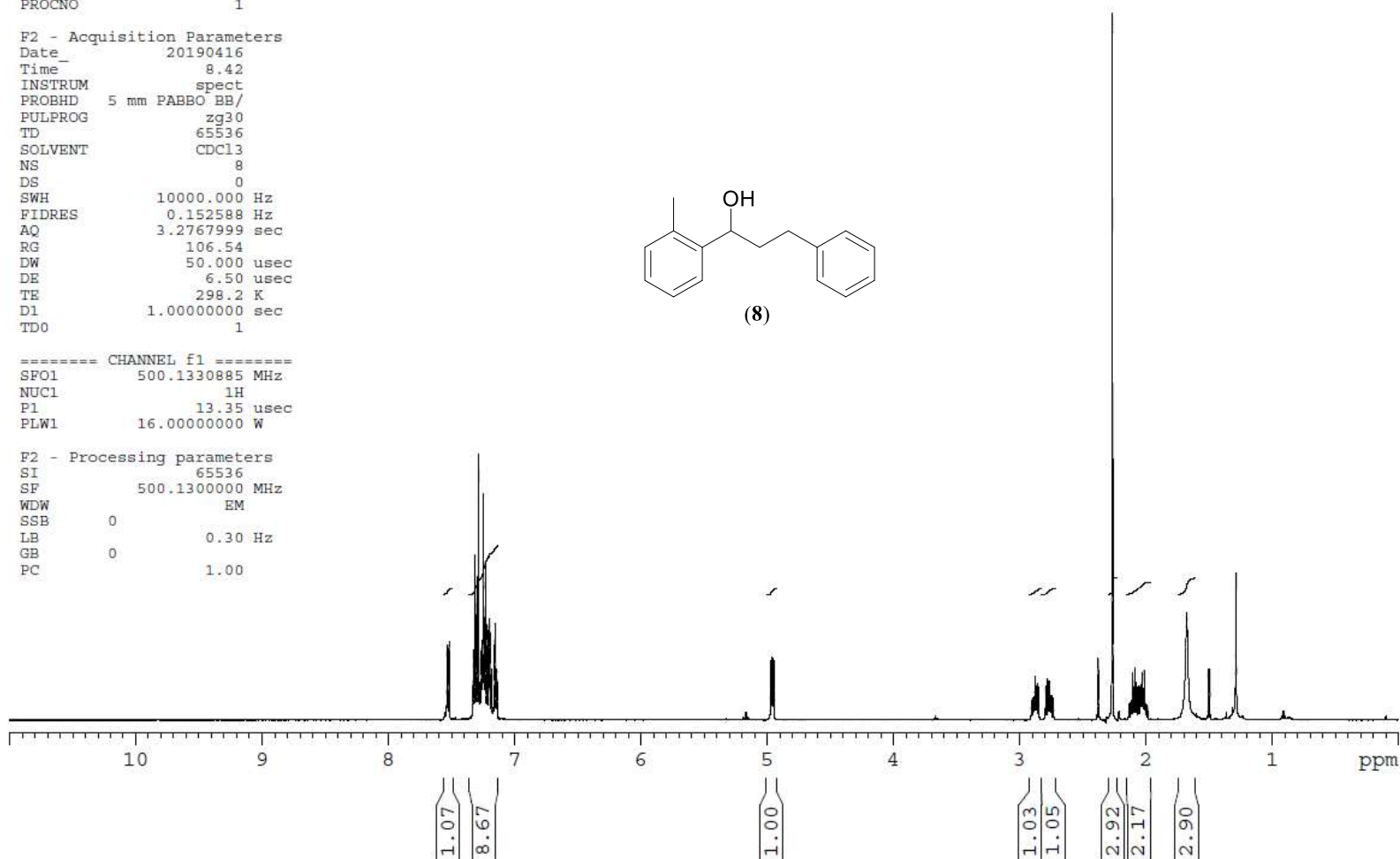
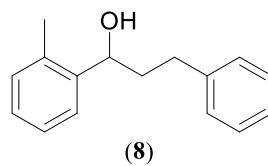
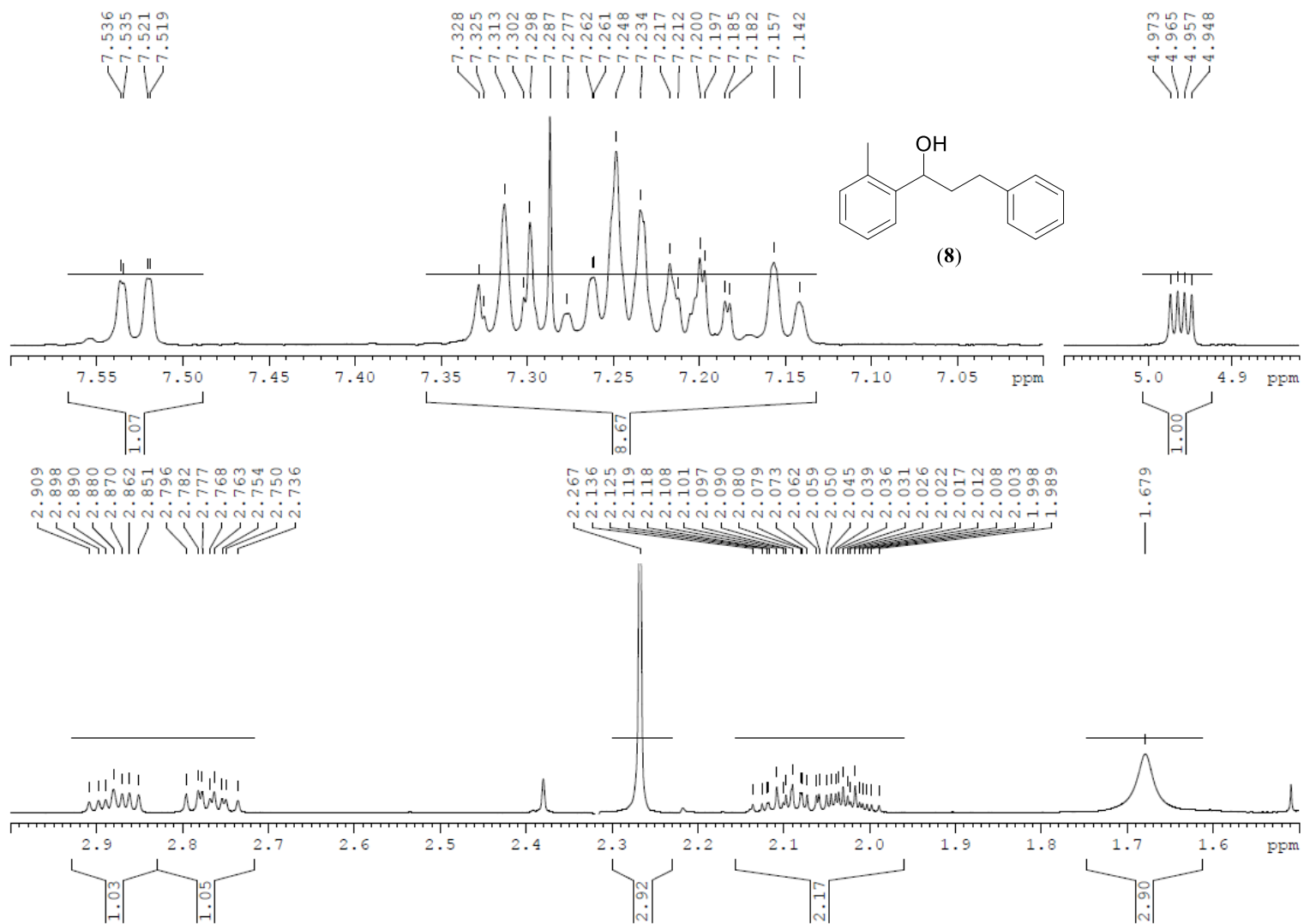


Figure S94.  $^1\text{H}$  NMR spectrum of (8) in  $\text{CDCl}_3$ .

PG-APP-11-230-1-1H



**Figure S95.** Expanded  $^1\text{H}$  NMR spectrum of (8) in  $\text{CDCl}_3$ .



PG-APP-11-230-1-13C

Current Data Parameters  
NAME PG-APP-11-230-1-13C  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20190416  
Time\_ 8.43  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 160  
DS 0  
SWH 29761.904 Hz  
FIDRES 0.454131 Hz  
AQ 1.1010048 sec  
RG 197.27  
DW 16.800 usec  
DE 6.50 usec  
TE 298.4 K  
D1 1.00000000 sec  
D11 0.03000000 sec  
TDO 1

===== CHANNEL f1 =====  
SFO1 125.7703637 MHz  
NUC1 13C  
P1 8.90 usec  
PLW1 103.00000000 W

===== CHANNEL f2 =====  
SFO2 500.1320005 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 80.00 usec  
PLW2 16.00000000 W  
PLW12 0.44556001 W  
PLW13 0.22411001 W

F2 - Processing parameters  
SI 32768  
SF 125.7577681 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

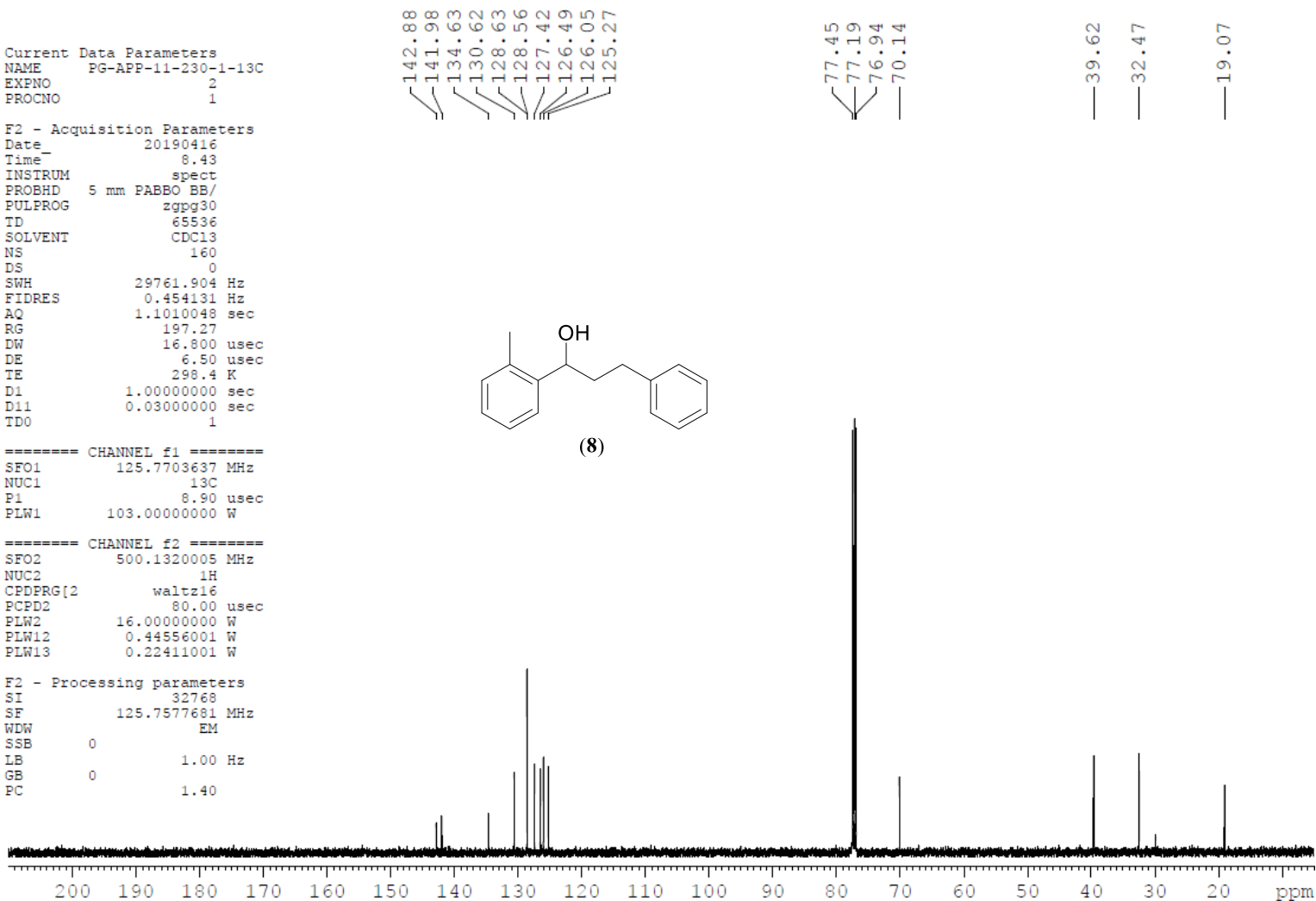


Figure S96.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (8) in  $\text{CDCl}_3$ .

PG-APP-11-230-1-13C

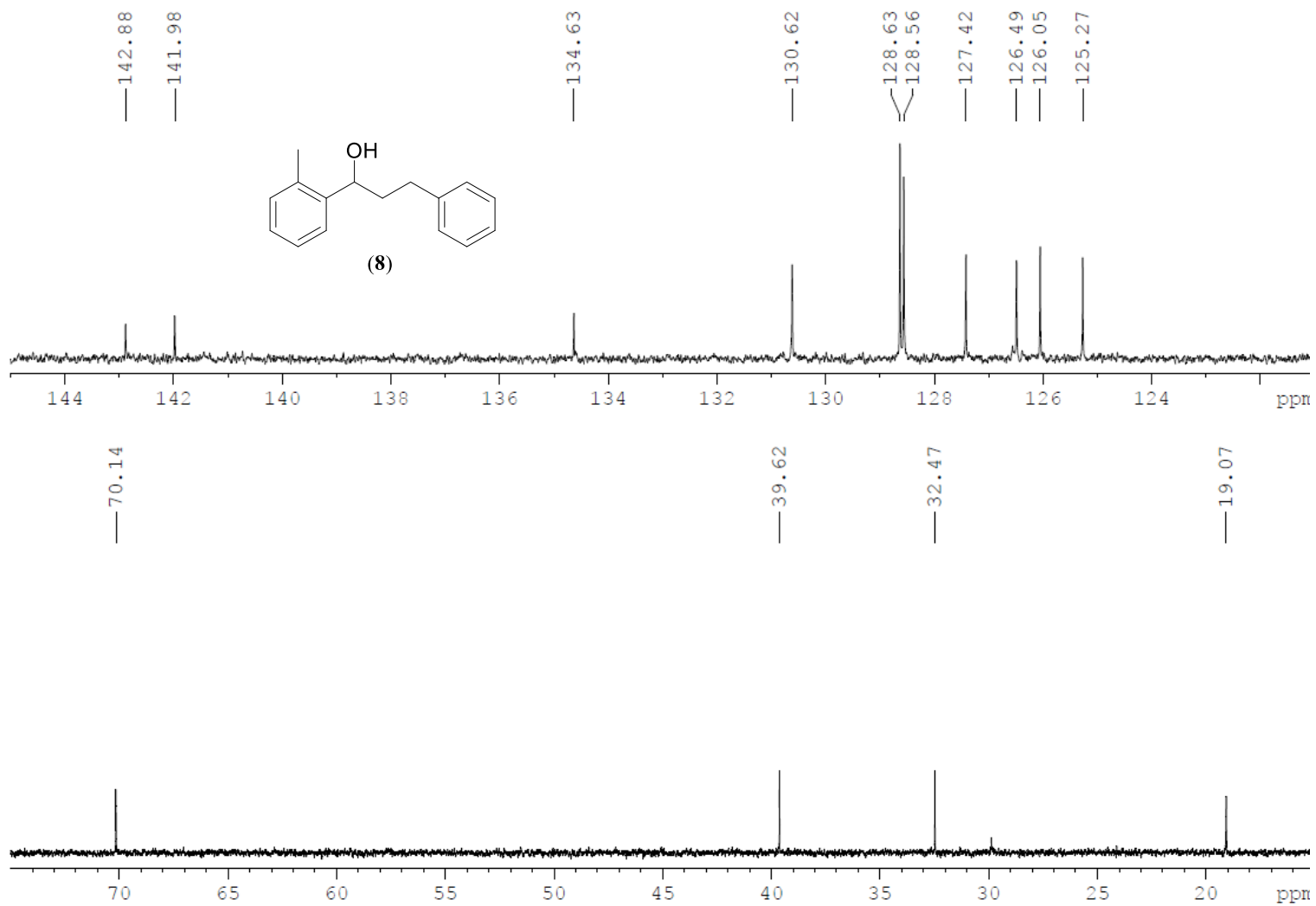


Figure S97. Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (8) in  $\text{CDCl}_3$ .

File : F:\GCMSDATA2019\APR2019\PG-APP-11-230-29.D  
Operator : APP  
Acquired : 15 Apr 2019 20:34 using AcqMethod COMMONMETHOD\_2018.M  
Instrument : GCMS  
Sample Name: PG-APP-11-230-29  
Misc Info :  
Vial Number: 5

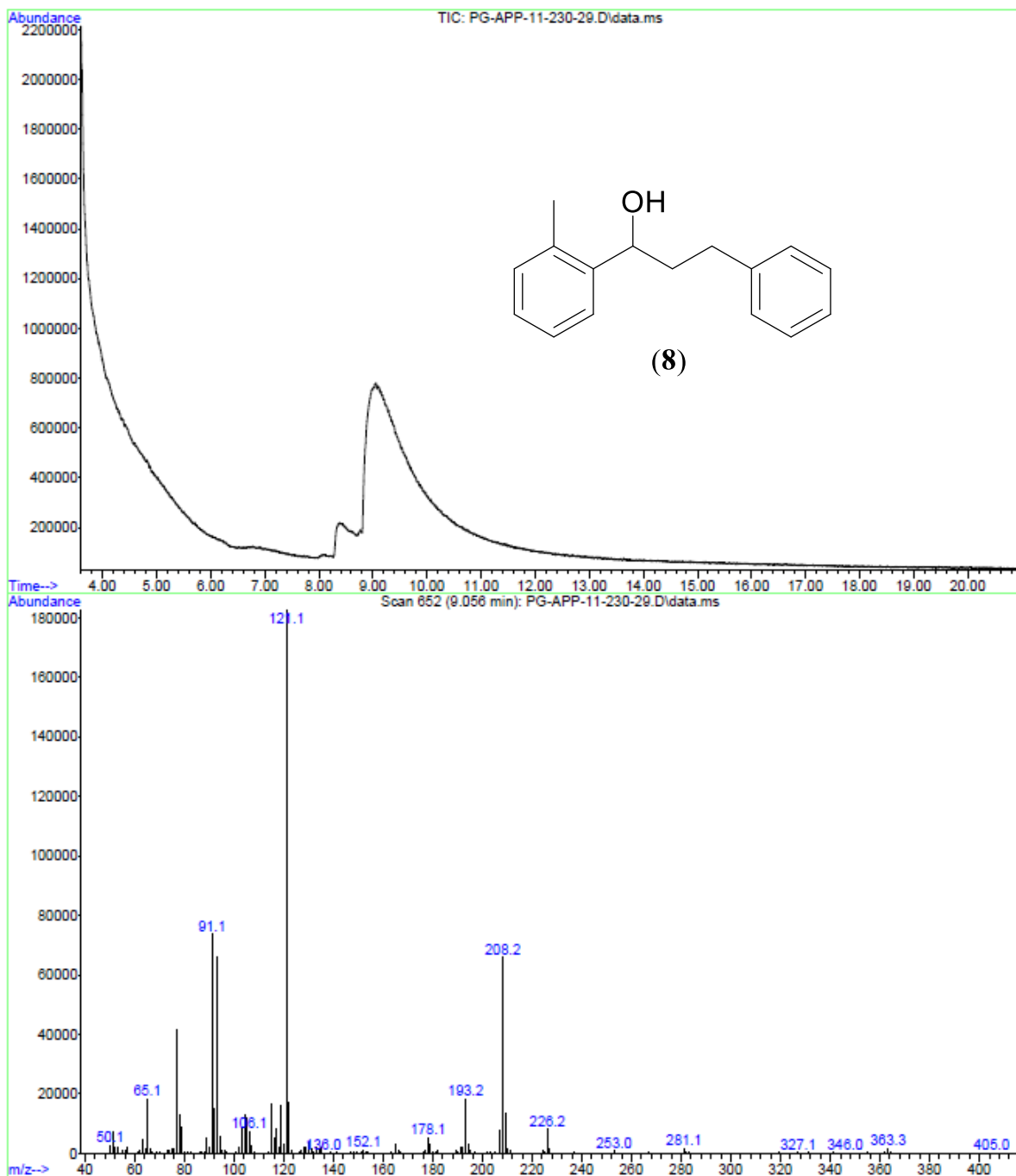
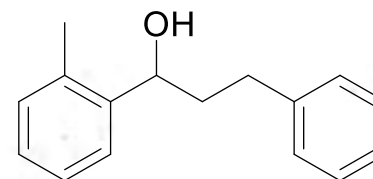


Figure S98. GCMS trace in EtOAc of (8) showing the  $M^+$  peak at  $m/z$  226.

## Eager 300 Report

Page: 1    Sample: PG-APP-11-230-1 (PG-APP-11-230-1)



(8)

```

Method Name      : PGAPP240519
Method File     : D:\CHNS2019\PGAPP240519.mth
Chromatogram    : PG-APP-11-230-1
Operator ID     : Prakash
Analysed        : 05/24/2019 18:48
Sample ID       : PG-APP-11-230-1 (# 34)
Analysis Type   : UnkNown (Area)

Company Name    : C.E. Instruments
Printed         : 5/25/2019 00:21
Instrument N.   : Instrument #1
Sample weight   : 1.244
    
```

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.

| Element Name | %       | Ret.Time | Area    | BC | Area ratio | K factor    |
|--------------|---------|----------|---------|----|------------|-------------|
| 1            | 0.0000  | 2        | 135010  | RS |            | 0.0000      |
| Carbon       | 84.7228 | 62       | 2809745 | RS | 1.000000   | .266592E+07 |
| Hydrogen     | 8.2142  | 187      | 610397  | RS | 4.603144   | .597347E+07 |
| Totals       | 92.9370 |          | 3555152 |    |            |             |

Figure S99. Elemental analysis data of (8).

PG-APP-11-233-3-1H

Current Data Parameters  
NAME PG-APP-11-233-3-1H  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20190418  
Time 8.35  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 134.65  
DW 50.000 usec  
DE 6.50 usec  
TE 300.3 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 500.1330885 MHz  
NUC1 1H  
P1 13.35 usec  
PLW1 16.00000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1300000 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

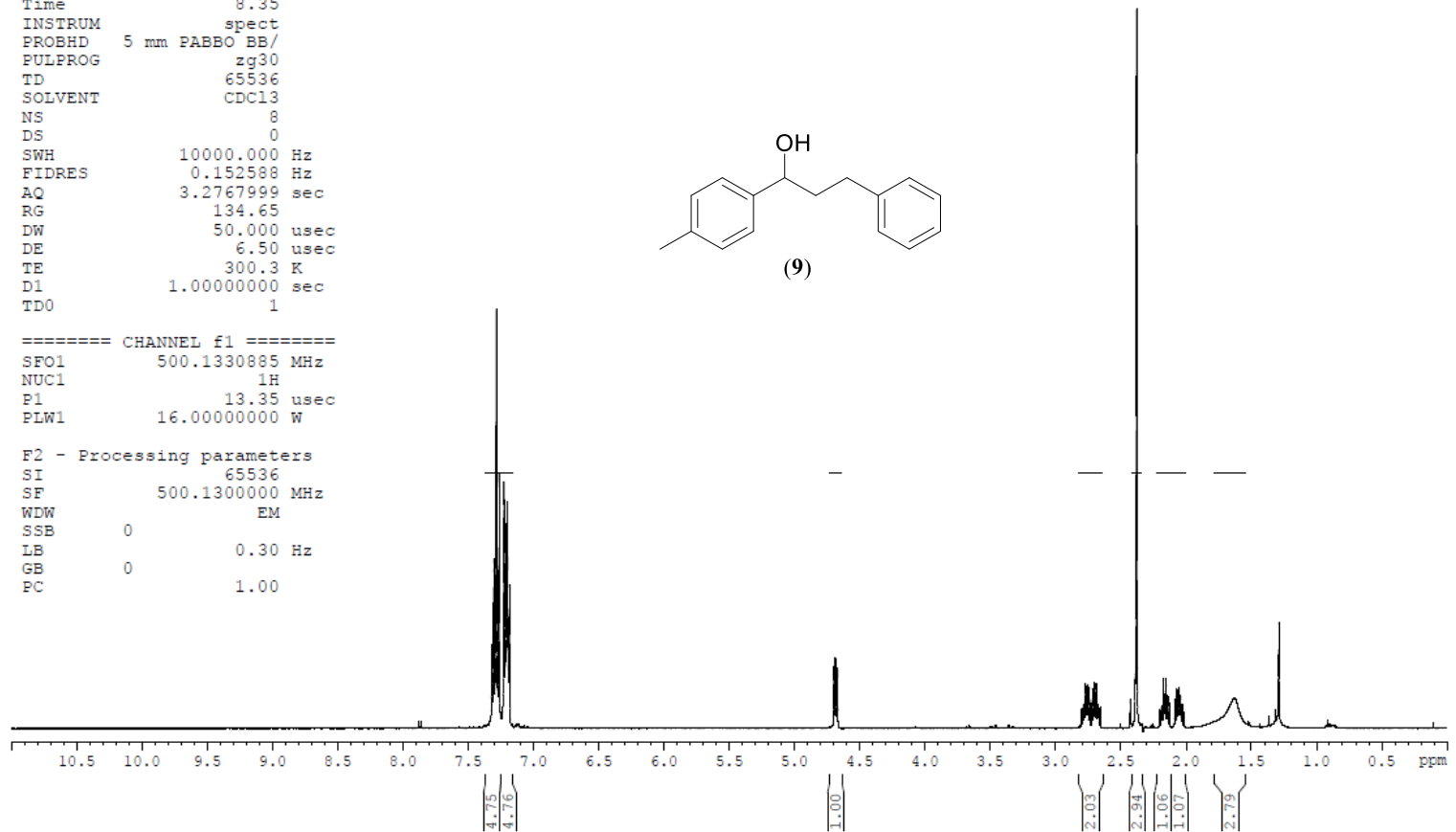


Figure S100. <sup>1</sup>H NMR spectrum of (9) in CDCl<sub>3</sub>.

PG-APP-11-233-3-1H

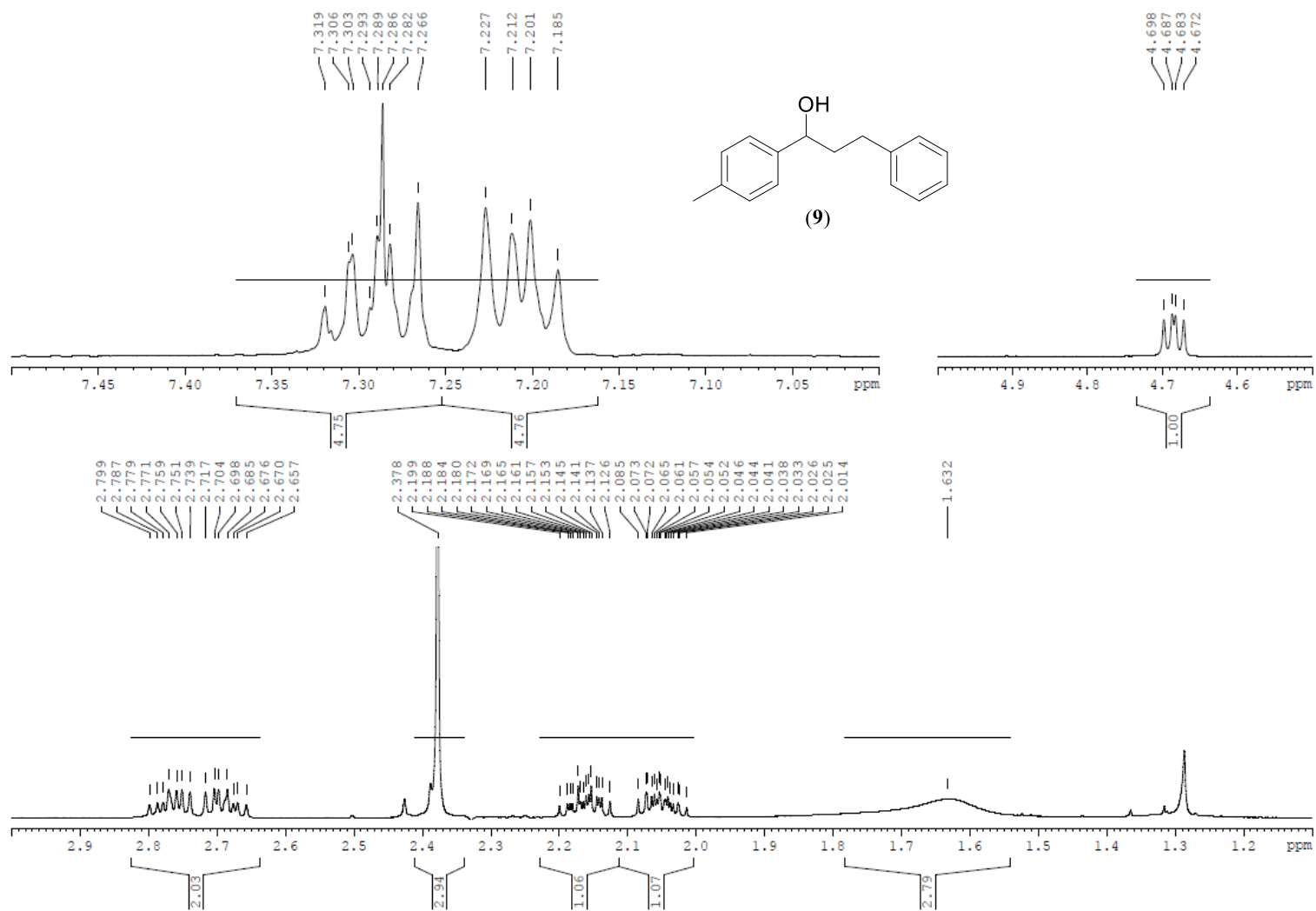


Figure S101. Expanded  $^1\text{H}$  NMR spectrum of (9) in  $\text{CDCl}_3$ .

PG-ARP-11-233-3-13C

NAME PG-ARP-11-233-3-13C  
EXPNO 11  
PROCNO 1  
Date\_ 20190418  
Time 8.49  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 882  
DS 0  
SWH 26041.666 Hz  
FIDRES 0.397364 Hz  
AQ 1.2583412 sec  
RG 2050  
DW 19.200 usec  
DE 6.50 usec  
TE 298.0 K  
D1 1.00000000 sec  
D11 0.03000000 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 8.50 usec  
PL1 -2.00 dB  
PL1W 56.53121948 W  
SFO1 100.6238364 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -1.00 dB  
PL12 13.69 dB  
PL13 14.50 dB  
PL2W 10.56200695 W  
PL12W 0.35871249 W  
PL13W 0.29767781 W  
SFO2 400.1316005 MHz  
SI 32768  
SF 100.6127506 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

142.03  
141.76  
137.53  
129.38  
128.63  
128.66  
126.09  
126.01

77.52  
77.20  
76.88  
73.94

40.55

32.29

21.30

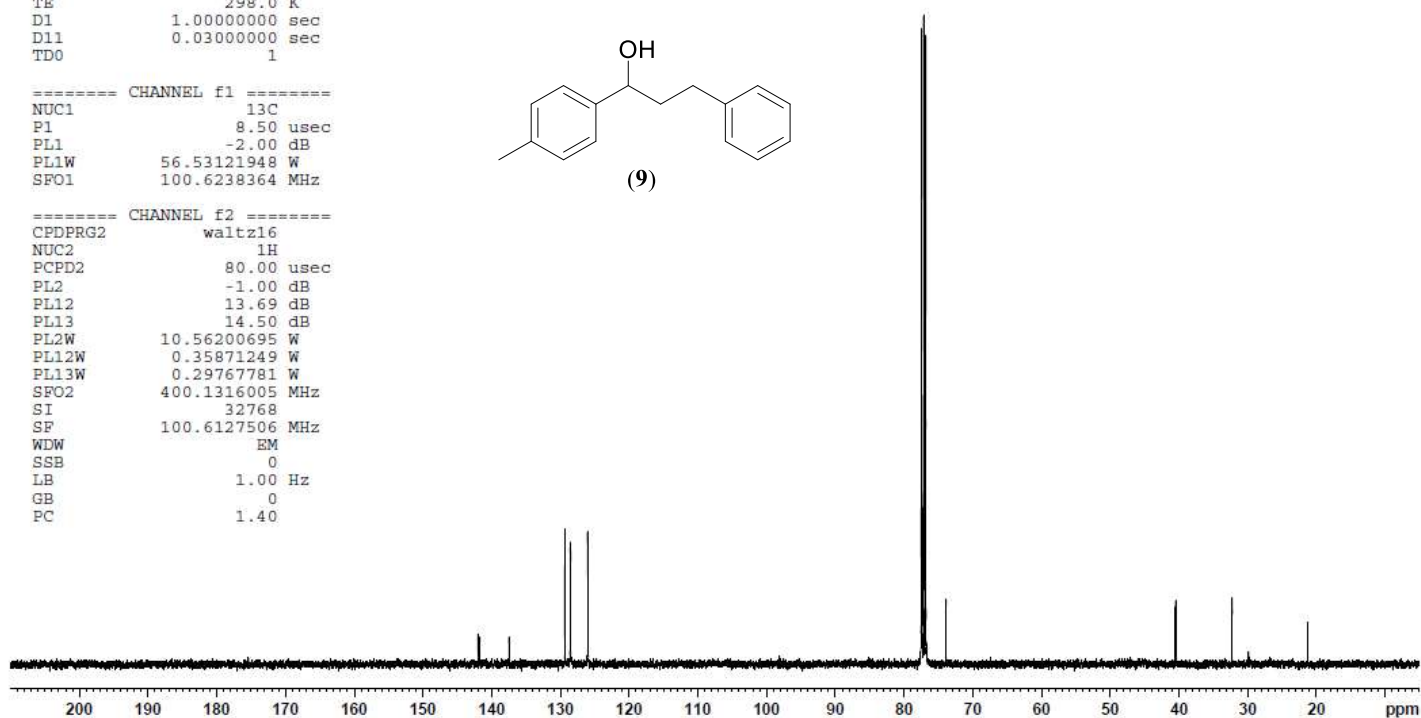
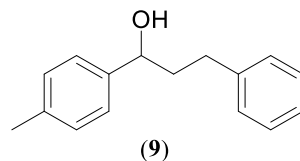
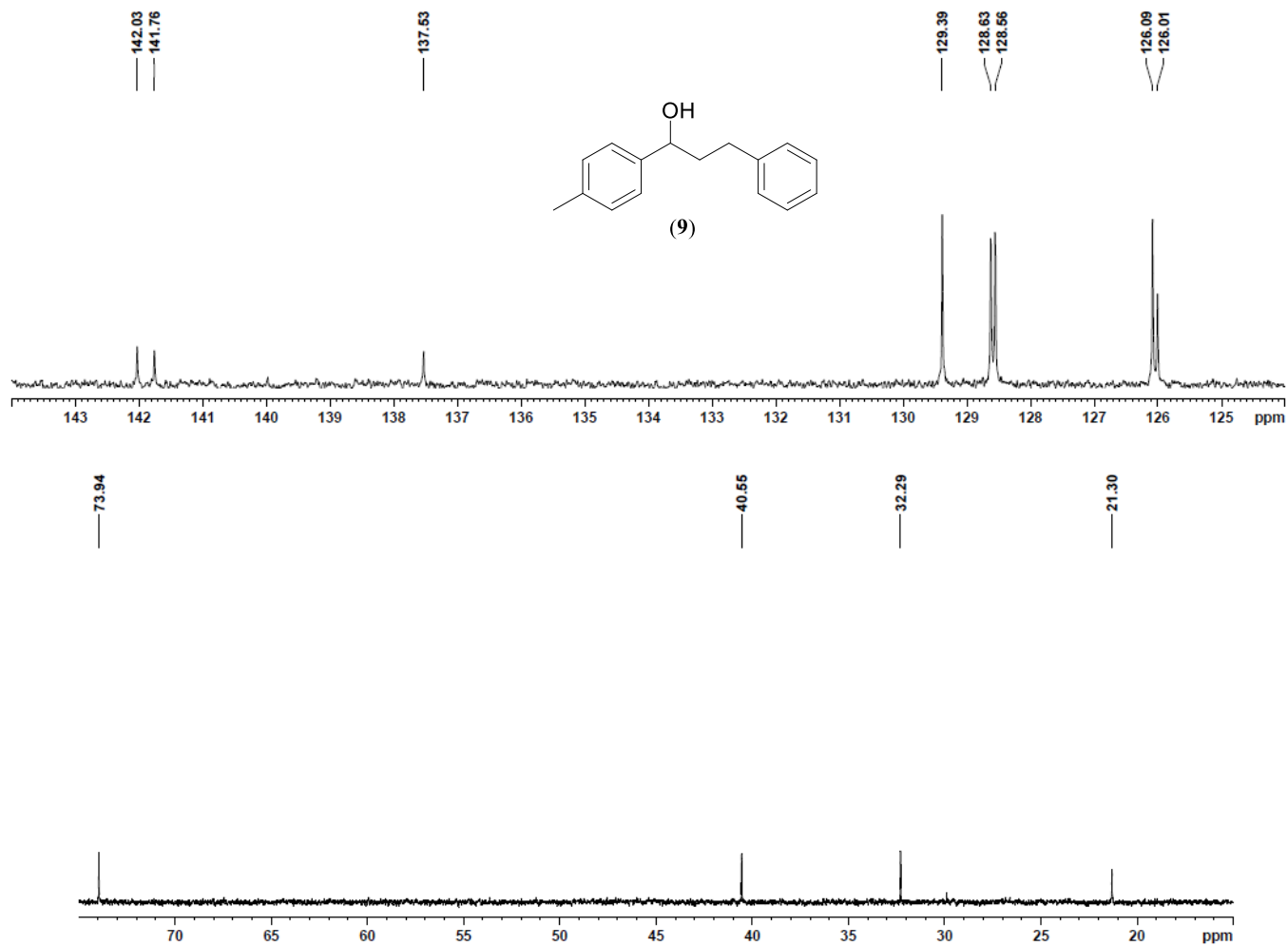


Figure S102.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (9) in  $\text{CDCl}_3$ .

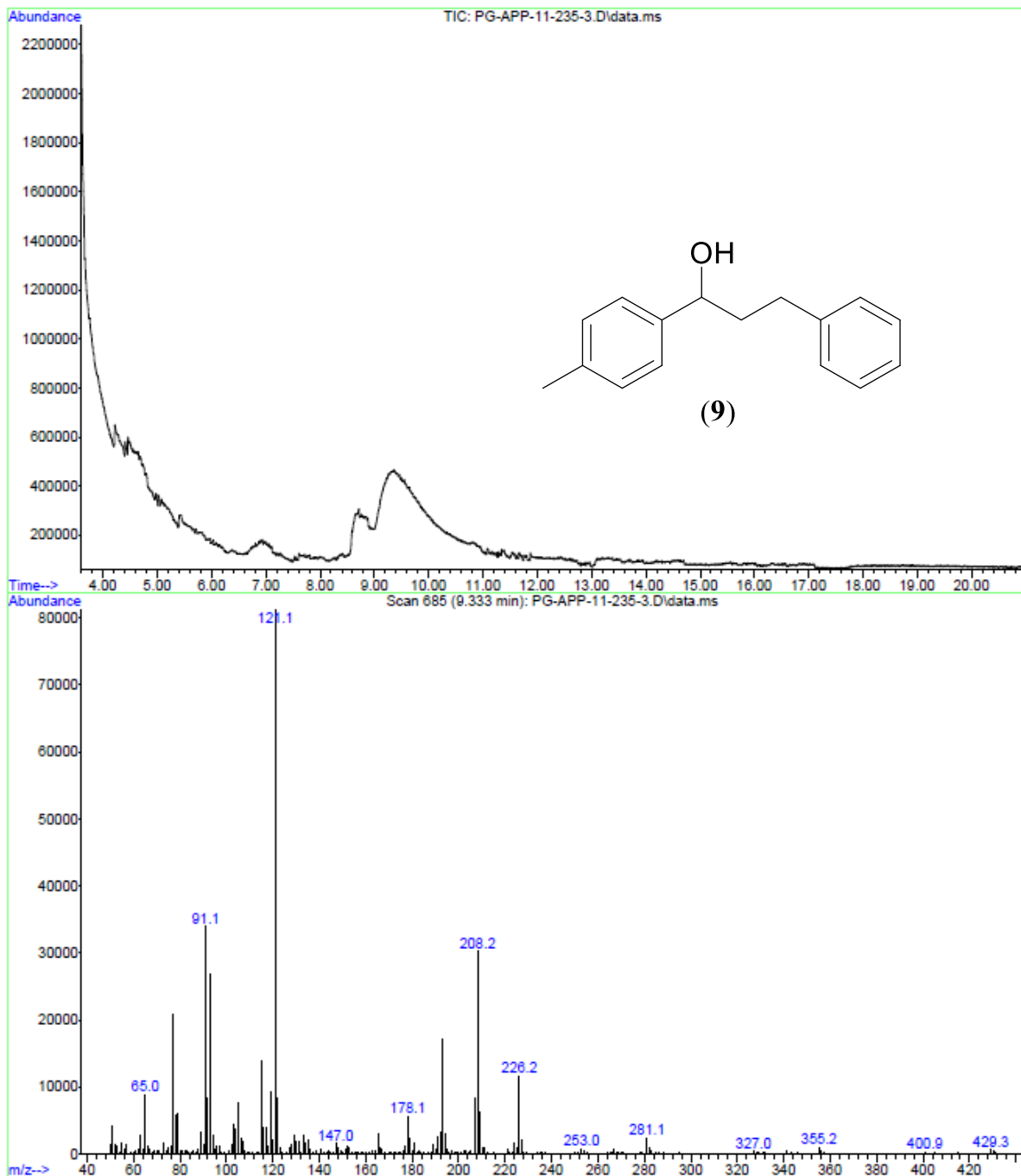
PG-ARP-11-233-3-13C



**Figure S103.** Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (9) in  $\text{CDCl}_3$ .



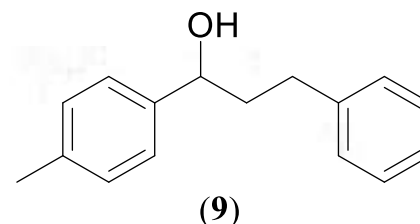
File : F:\GCMSDATA2019\APR2019\PG-APP-11-235-3.D  
Operator : APP  
Acquired : 17 Apr 2019 16:43 using AcqMethod COMMONMETHOD\_2018.M  
Instrument : GCMS  
Sample Name: PG-APP-11-235-3  
Misc Info :  
Vial Number: 1



**Figure S104.** GCMS trace in EtOAc of (9) showing the  $M^+$  peak at  $m/z$  226.

## Eager 300 Report

Page: 1 Sample: PG-APP-11-233-1 (PG-APP-11-233-1)



```

Method Name      : PGAPP240519
Method File     : D:\CHNS2019\PGAPP240519.mth
Chromatogram    : PG-APP-11-233-1
Operator ID     : Prakash
Analysed        : 05/24/2019 19:18
Sample ID       : PG-APP-11-233-1 (# 37)
Analysis Type   : UnkNown (Area)

Company Name    : C.E. Instruments
Printed         : 5/25/2019 00:22
Instrument N.   : Instrument #1
Sample weight   : .876
    
```

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.

| Element Name | %       | Ret.Time | Area    | BC | Area ratio | K factor    |
|--------------|---------|----------|---------|----|------------|-------------|
| 1            | 0.0000  | 2        | 134852  | RS |            | 0.0000      |
| Carbon       | 84.8281 | 63       | 1981027 | RS | 1.000000   | .266592E+07 |
| Hydrogen     | 7.8253  | 184      | 409480  | RS | 4.837908   | .597347E+07 |
| Totals       | 92.6534 |          | 2525359 |    |            |             |

Figure S105. Elemental analysis data of (9).

PG-APP-11-238-1-1H

NAME PG-APP-11-238-1-1H  
EXPNO 1  
PROCNO 1  
Date\_ 20190423  
Time 8.22  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2999091 sec  
RG 161  
DW 60.800 usec  
DE 6.50 usec  
TE 297.8 K  
D1 1.00000000 sec  
TD0 1

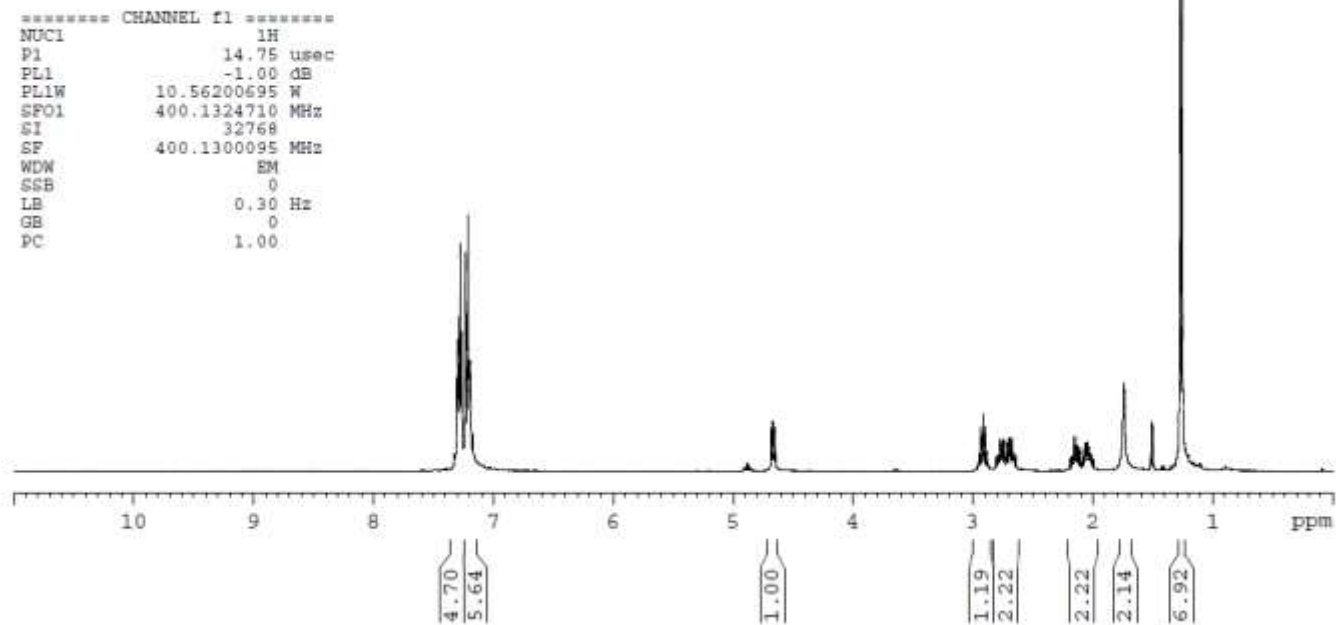
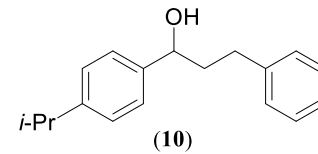
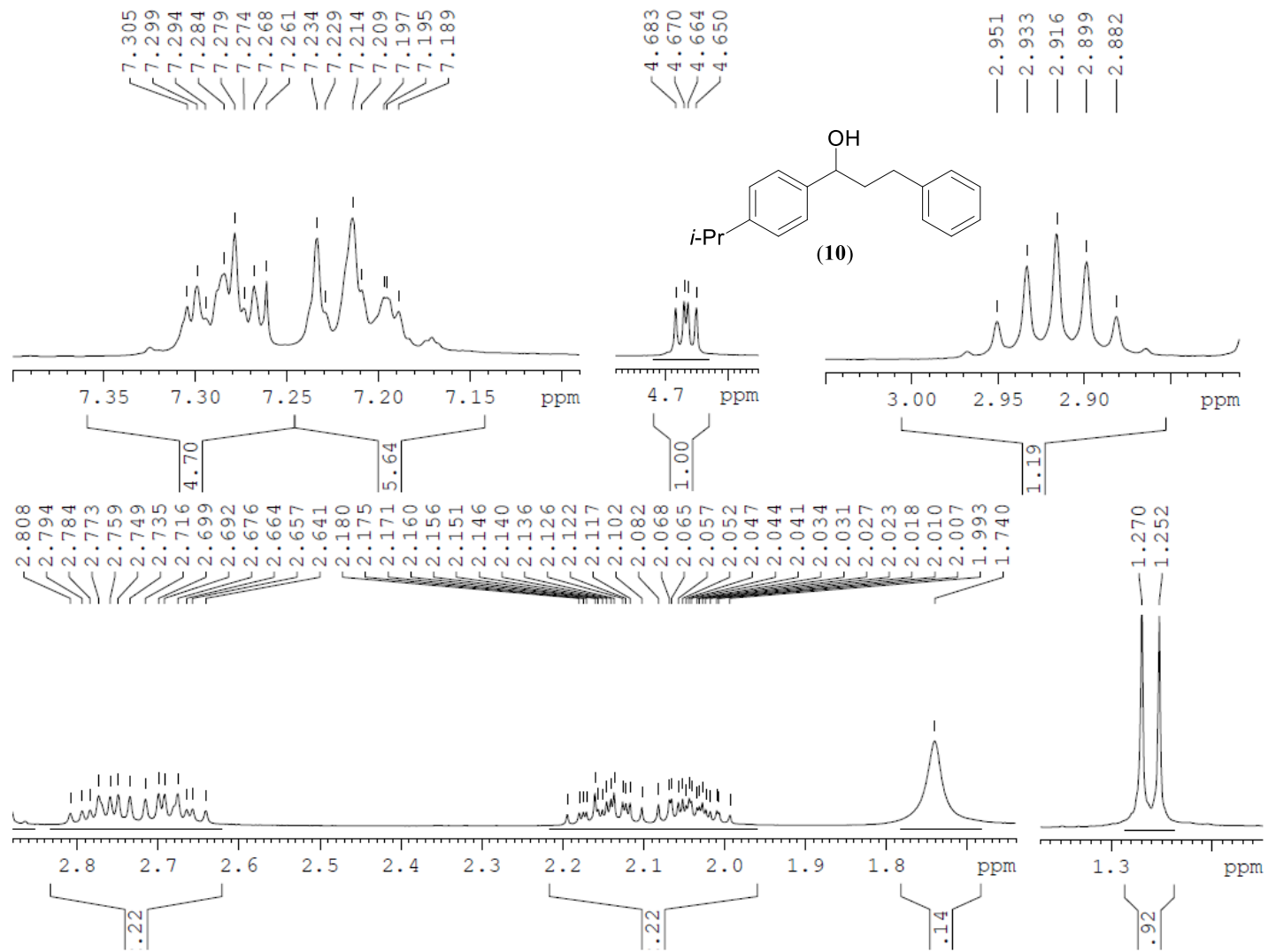


Figure S106.  $^1\text{H}$  NMR spectrum of (10) in  $\text{CDCl}_3$ .



**Figure S107.** Expanded  $^1\text{H}$  NMR spectrum of (10) in  $\text{CDCl}_3$ .

PG-APP-11-238-1-13C

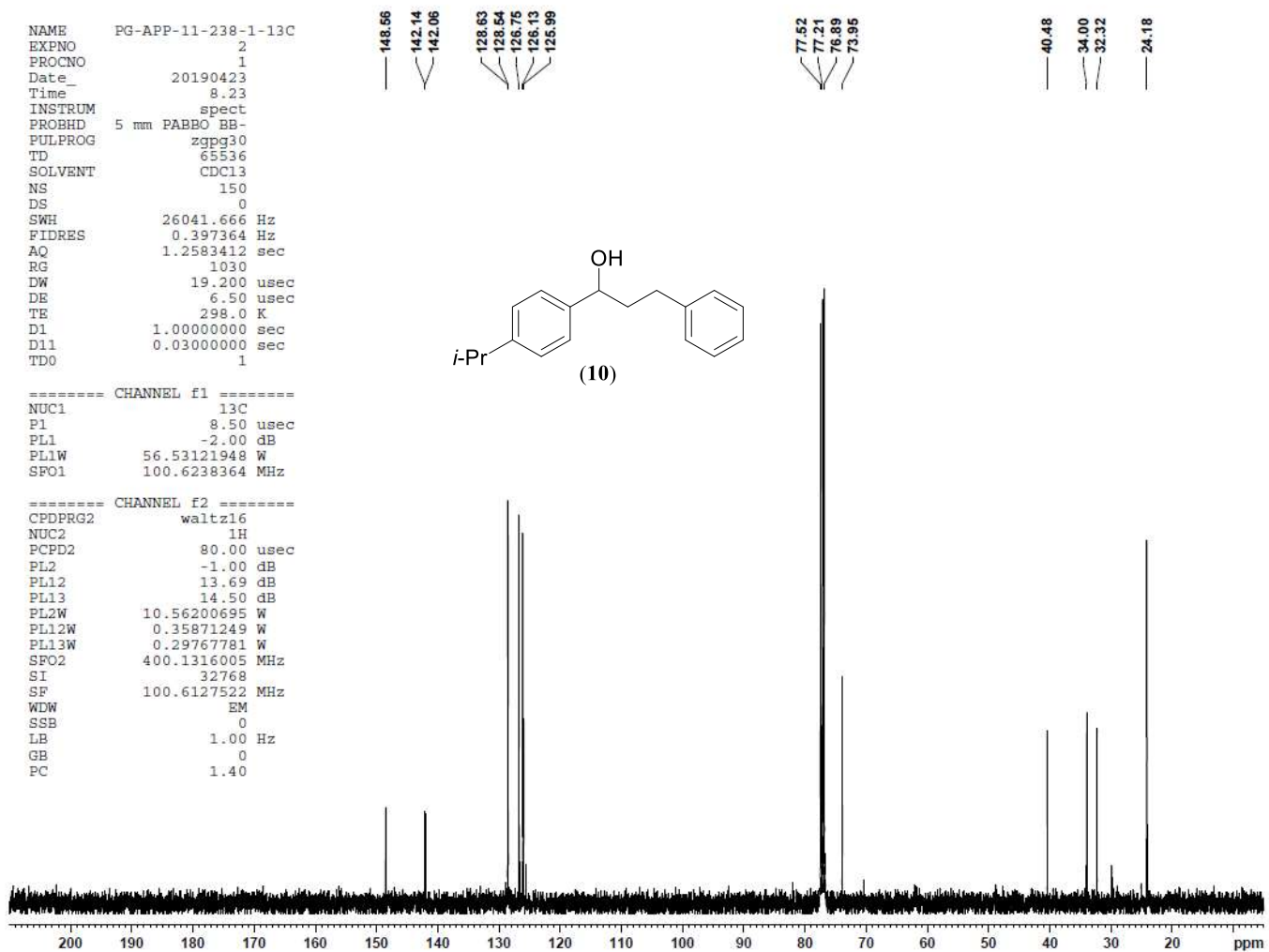
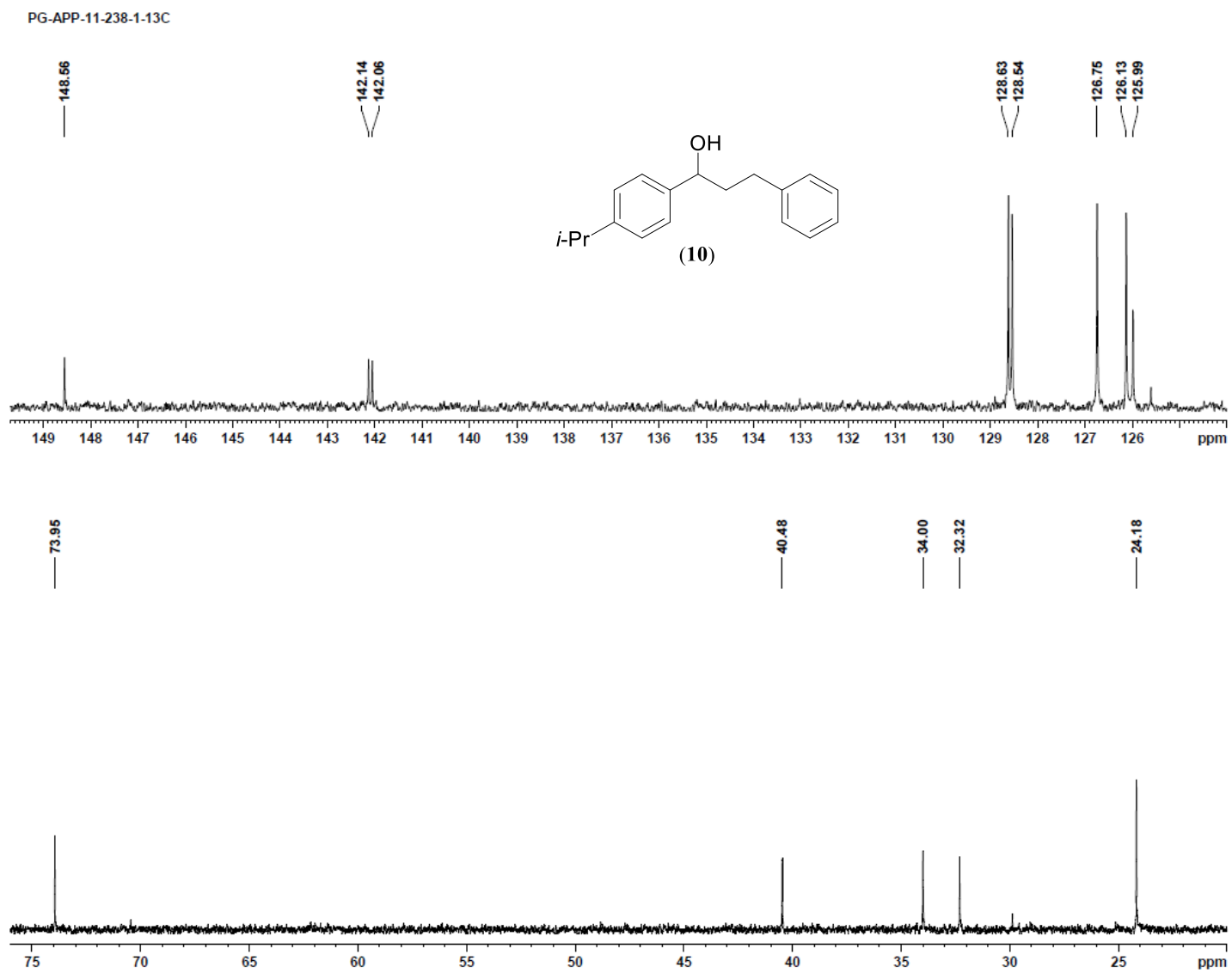
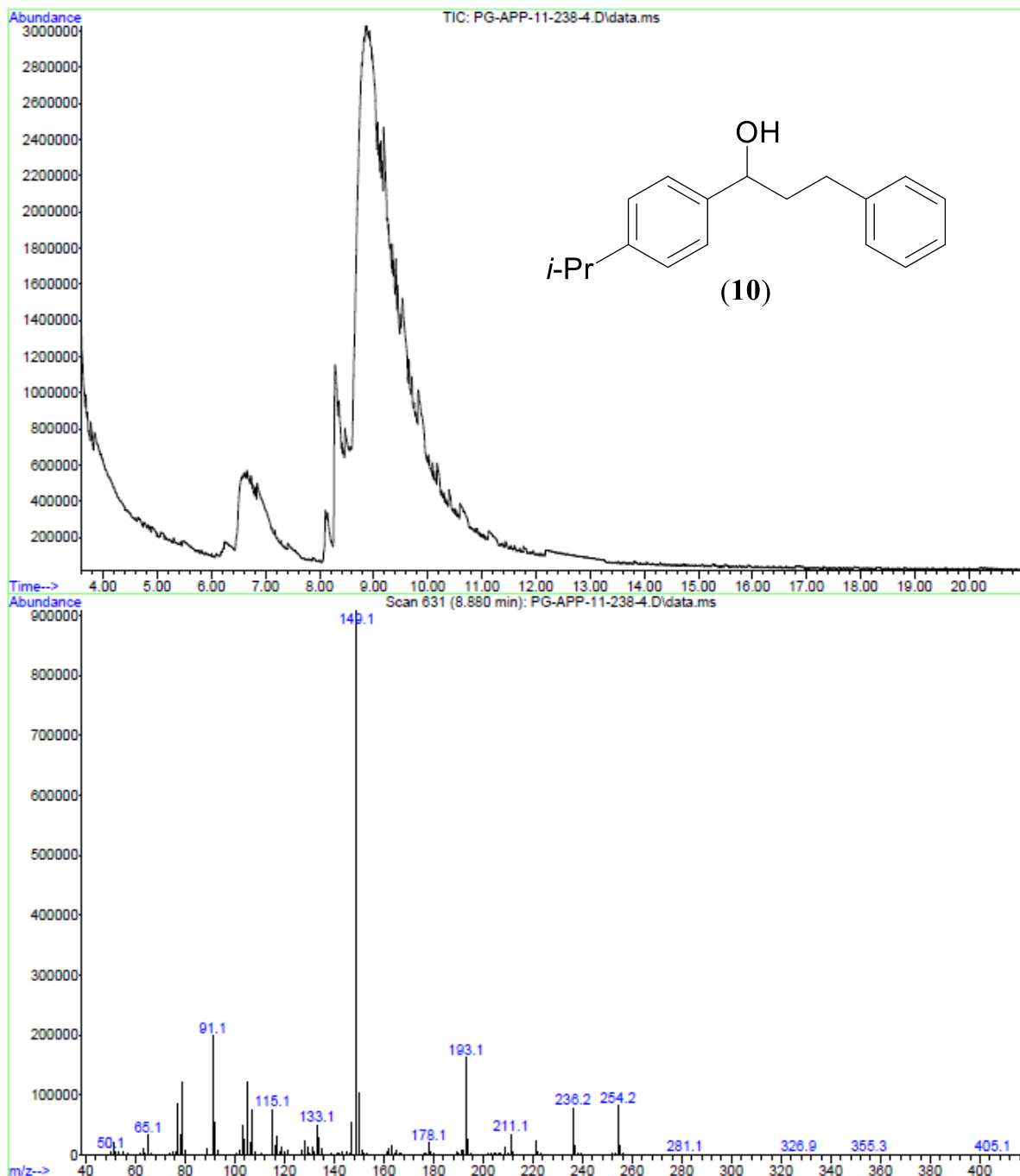


Figure S108.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (10) in  $\text{CDCl}_3$ .



**Figure S109.** Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (10) in  $\text{CDCl}_3$ .

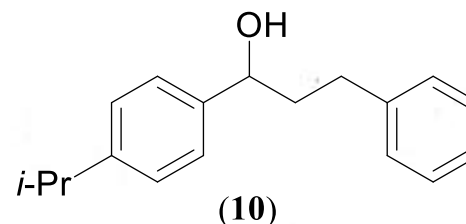
File : F:\GCMSDATA2019\APR2019\PG-APP-11-238-4.D  
Operator : APP  
Acquired : 22 Apr 2019 20:09 using AcqMethod COMMONMETHOD\_2016.M  
Instrument : GCMS  
Sample Name: PG-APP-11-238-4  
Misc Info :  
Vial Number: 2



**Figure S110.** GCMS trace in EtOAc of (10) showing the  $M^+$  peak at  $m/z$  254.

## Eager 300 Report

Page: 1 Sample: PG-APP-11-238-1 (PG-APP-11-238-1)



```

Method Name      : PGAPP240519
Method File     : D:\CHNS2019\PGAPP240519.mth
Chromatogram    : PG-APP-11-238-1
Operator ID     : Prakash
Analysed        : 05/24/2019 19:08
Sample ID       : PG-APP-11-238-1 (# 36)
Analysis Type   : UnkNown (Area)

Company Name    : C.E. Instruments
Printed         : 5/25/2019 00:22
Instrument N.   : Instrument #1
Sample weight   : 1.008
    
```

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.

| Element Name | %       | Ret.Time | Area    | BC | Area ratio | K factor    |
|--------------|---------|----------|---------|----|------------|-------------|
| 1            | 0.0000  | 2        | 134675  | RS |            | 0.0000      |
| Carbon       | 85.0741 | 63       | 2286149 | RS | 1.000000   | .266592E+07 |
| Hydrogen     | 8.1537  | 184      | 490957  | RS | 4.656515   | .597347E+07 |
| Totals       | 93.2279 |          | 2911781 |    |            |             |

Figure S111. Elemental analysis data of (10).



PG-APP-12-11-2-1H

NAME PG-APP-12-11-2-1H  
EXPNO 1  
PROCNO 1  
Date\_ 20190521  
Time 8.18  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2999091 sec  
RG 57  
DW 60.800 usec  
DE 6.50 usec  
TE 295.8 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.75 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SFO1 400.1324710 MHz  
SI 32768  
SF 400.1300095 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

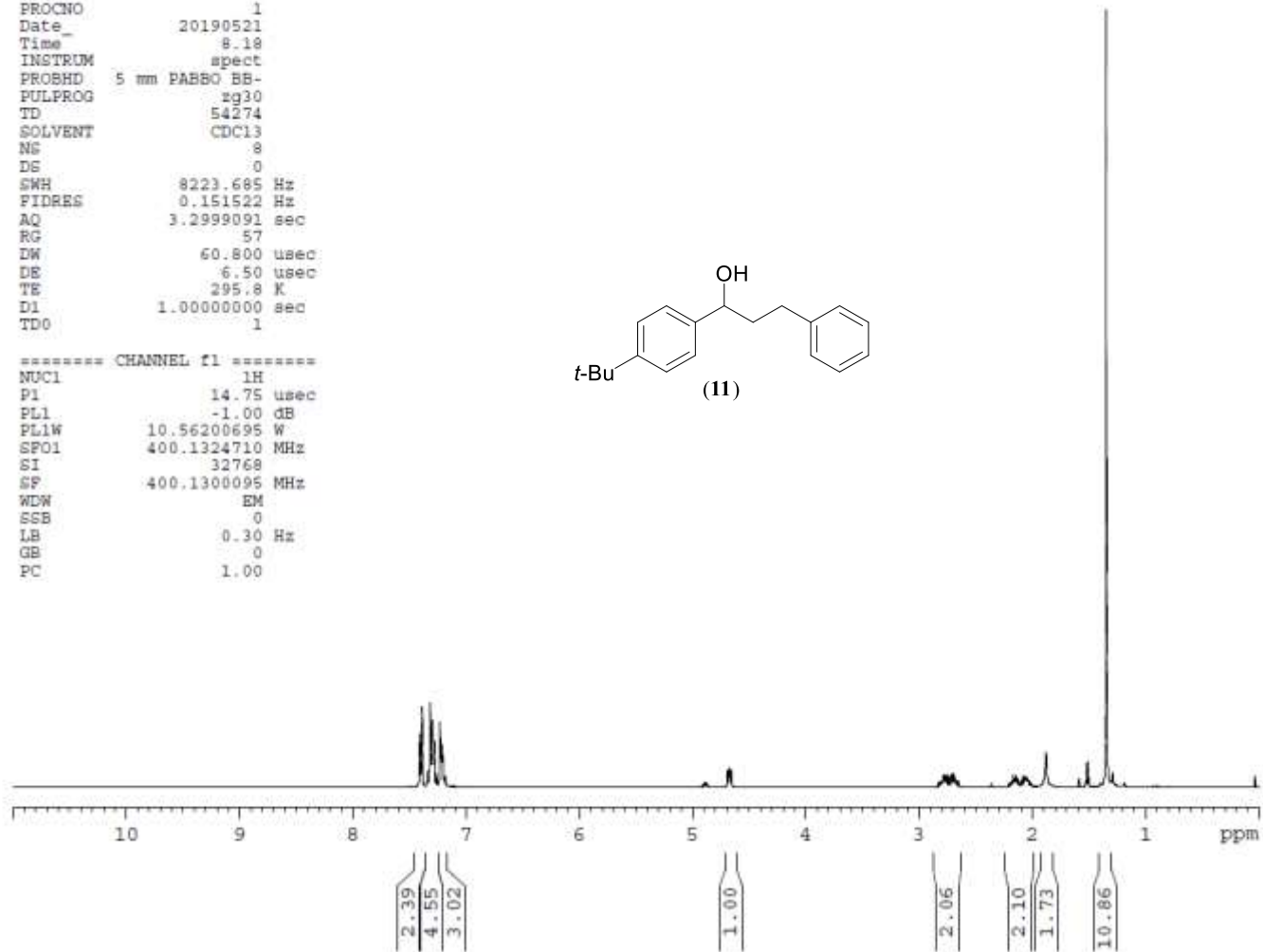
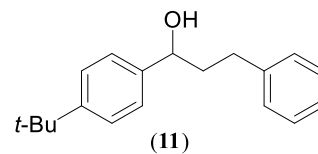
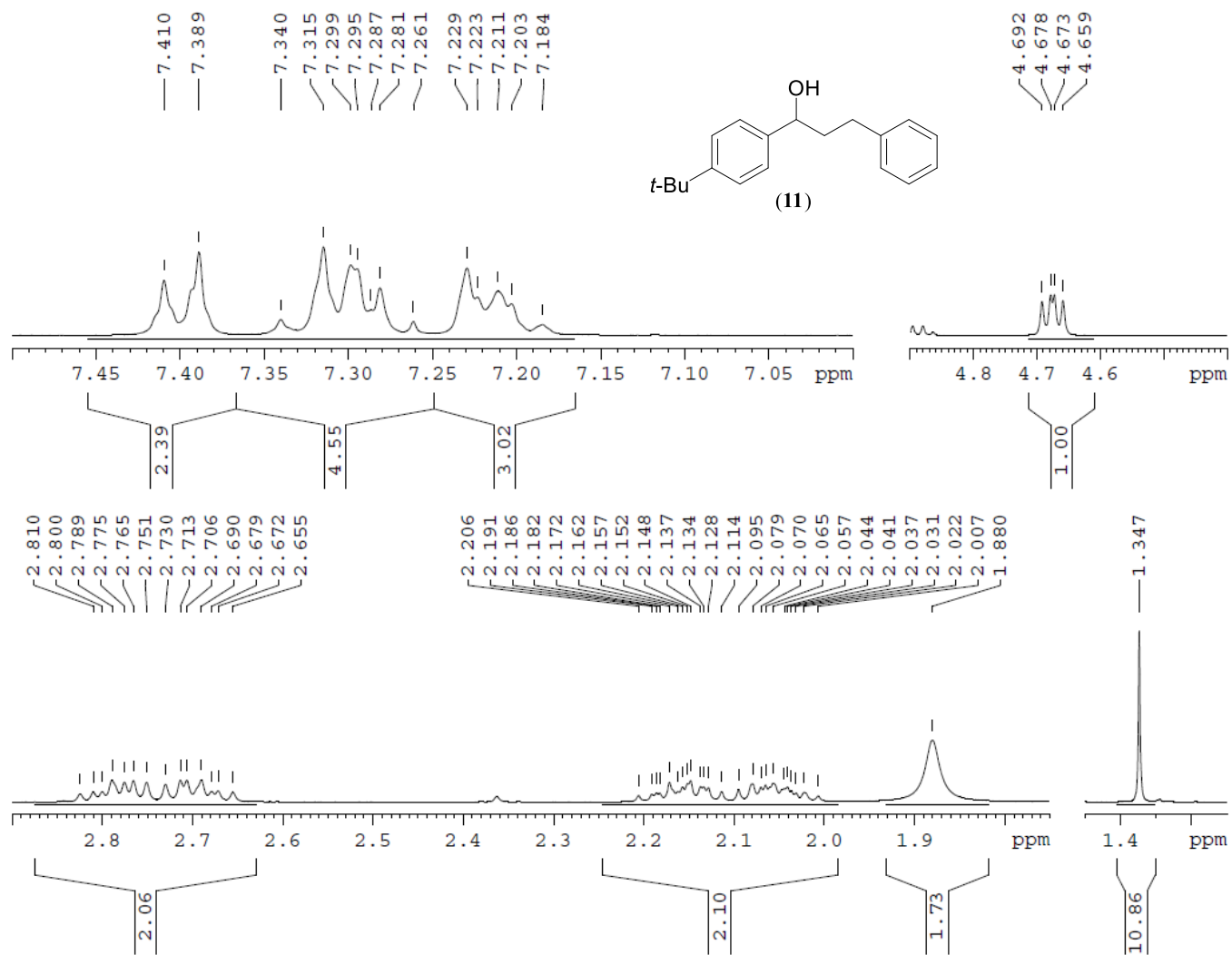


Figure S112.  $^1\text{H}$  NMR spectrum of (11) in  $\text{CDCl}_3$ .



**Figure S113.** Expanded  $^1\text{H}$  NMR spectrum of (11) in  $\text{CDCl}_3$ .

PG-APP-12-11-2-13C

NAME PG-APP-12-11-2-13C  
EXPNO 2  
PROCNO 1  
Date\_ 20190521  
Time 8.20  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 100  
DS 0  
SWH 26041.666 Hz  
FIDRES 0.397364 Hz  
AQ 1.2583412 sec  
RG 1030  
DW 19.200 usec  
DE 6.50 usec  
TE 296.3 K  
D1 1.00000000 sec  
D11 0.03000000 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 8.50 usec  
PL1 -2.00 dB  
PL1W 56.53121948 W  
SFO1 100.6238364 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -1.00 dB  
PL12 13.69 dB  
PL13 14.50 dB  
PL2W 10.56200695 W  
PL12W 0.35871249 W  
PL13W 0.29767781 W  
SFO2 400.1316005 MHz  
SI 32768  
SF 100.6127571 MHz  
WDB EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

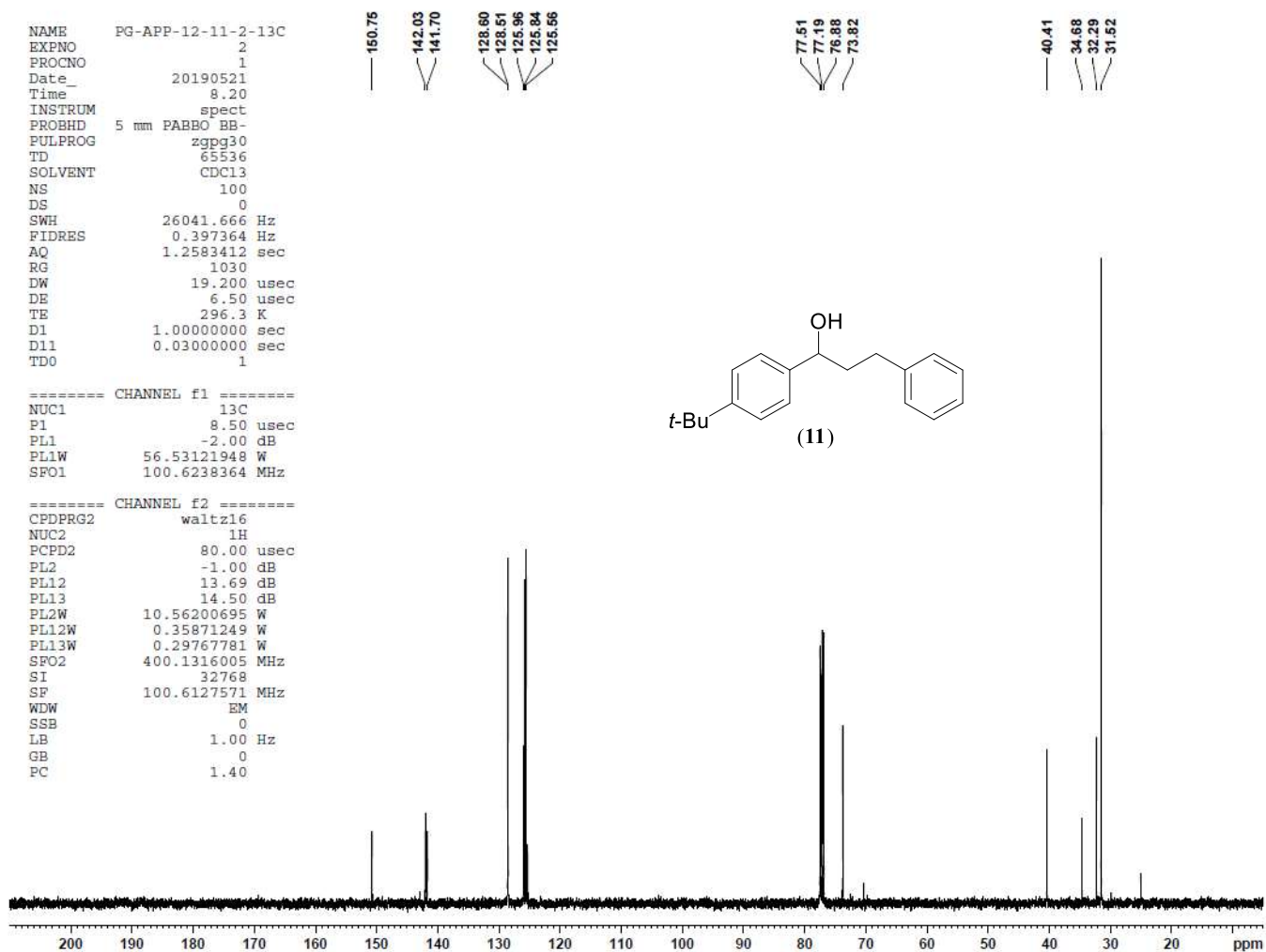


Figure S114.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (11) in  $\text{CDCl}_3$ .

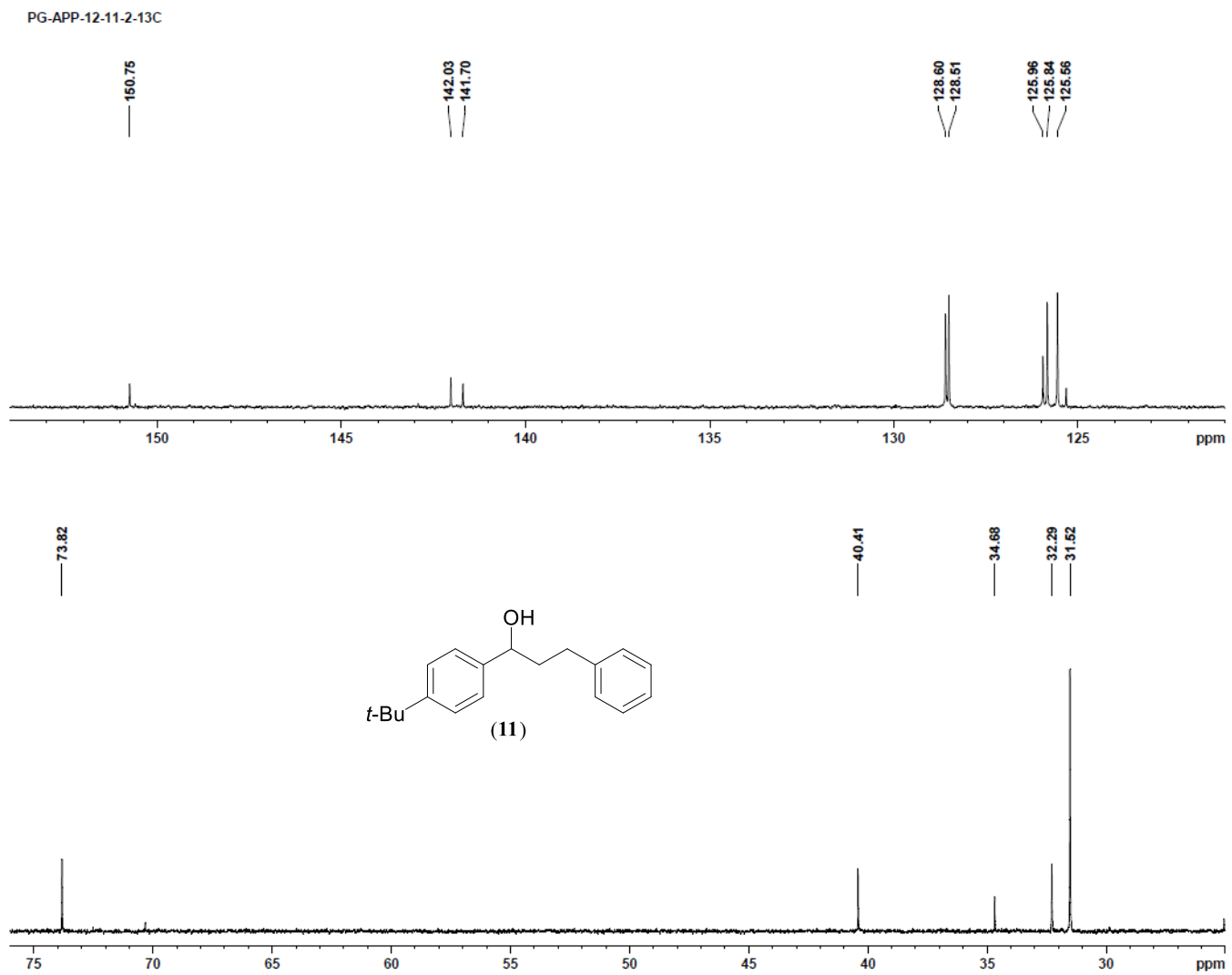


Figure S115. Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (11) in  $\text{CDCl}_3$ .

File : F:\GCMSDATA2019\May 2019\PG-APP-12-11-31.D  
Operator : APP  
Acquired : 20 May 2019 22:57 using AcqMethod COMMONMETHOD-2018.M  
Instrument : GCMS  
Sample Name: PG-APP-12-11-31  
Misc Info :  
Vial Number: 1

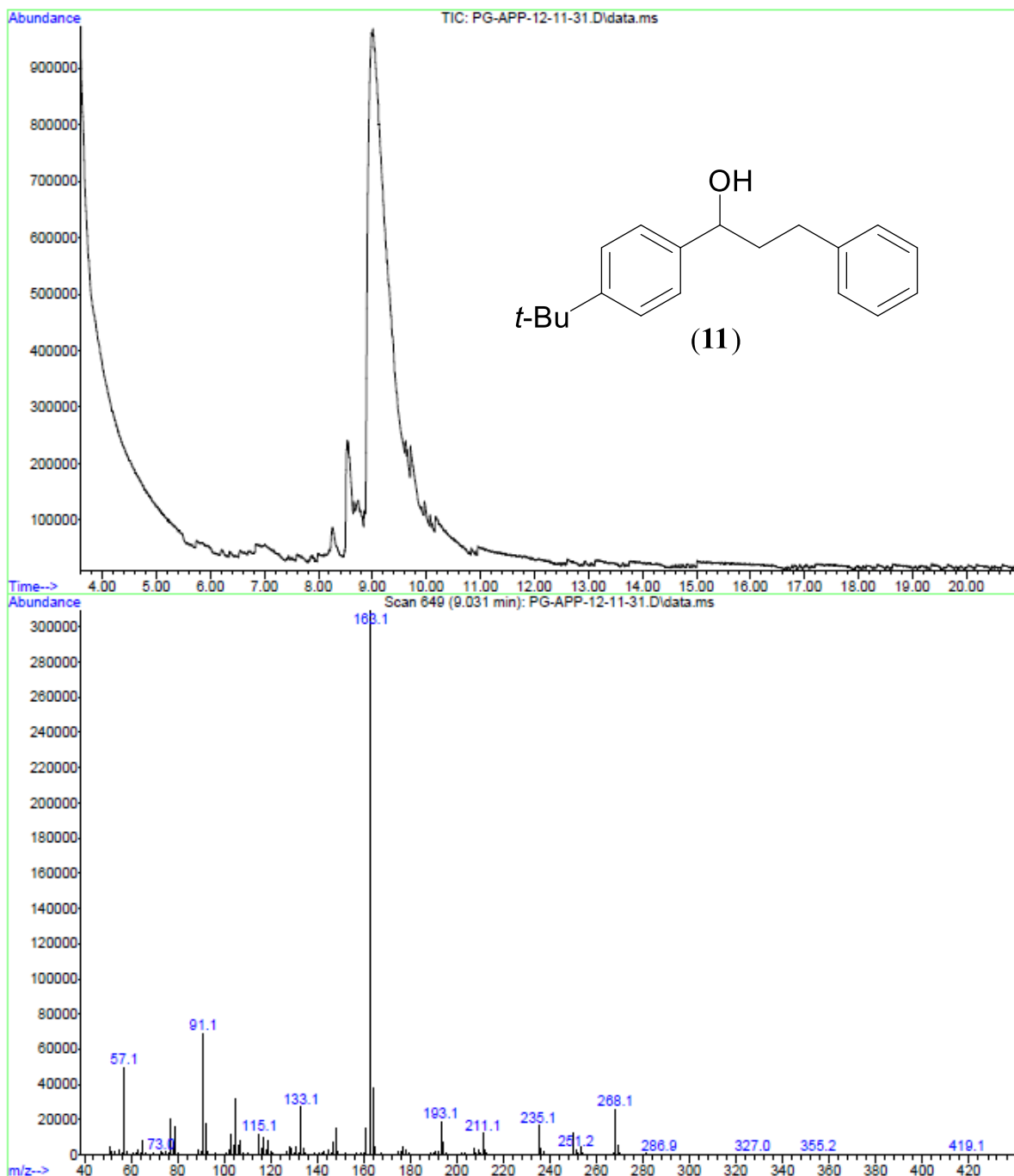
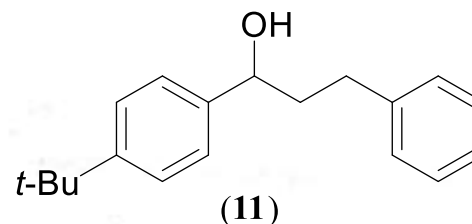


Figure S116. GCMS trace in EtOAc of (11) showing the  $M^+$  peak at  $m/z$  268.

## Eager 300 Report

Page: 1 Sample: PG-APP-12-11-5 (PG-APP-12-11-5)



```

Method Name      : PGAPP240519
Method File     : D:\CHNS2019\PGAPP240519.mth
Chromatogram    : PG-APP-12-11-5
Operator ID     : Prakash
Analysed        : 05/24/2019 20:37
Sample ID       : PG-APP-12-11-5 (# 44)
Analysis Type   : UnkNown (Area)

Company Name    : C.E. Instruments
Printed         : 5/25/2019 00:22
Instrument N.   : Instrument #1
Sample weight   : .759
    
```

Calib. method : using 'K Factors'

!!! Warning missing one or more peaks.

| Element Name | %       | Ret.Time | Area    | BC | Area ratio | K factor    |
|--------------|---------|----------|---------|----|------------|-------------|
| 1            | 0.0000  | 6        | 60133   | RS |            | 0.0000      |
| Carbon       | 84.3312 | 63       | 1706383 | RS | 1.000000   | .266592E+07 |
| Hydrogen     | 8.2963  | 184      | 376143  | RS | 4.536527   | .597347E+07 |
| Totals       | 92.6275 |          | 2142659 |    |            |             |

Figure S117. Elemental analysis data of (11).

PG-APP-12-03-2-1H

NAME PG-APP-12-03-2-1H  
EXPNO 1  
PROCNO 1  
Date\_ 20190501  
Time\_ 8.35  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2999091 sec  
RG 161  
DW 60.800 usec  
DE 6.50 usec  
TE 297.0 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.75 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SFO1 400.1324710 MHz  
SI 32768  
SF 400.1300095 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

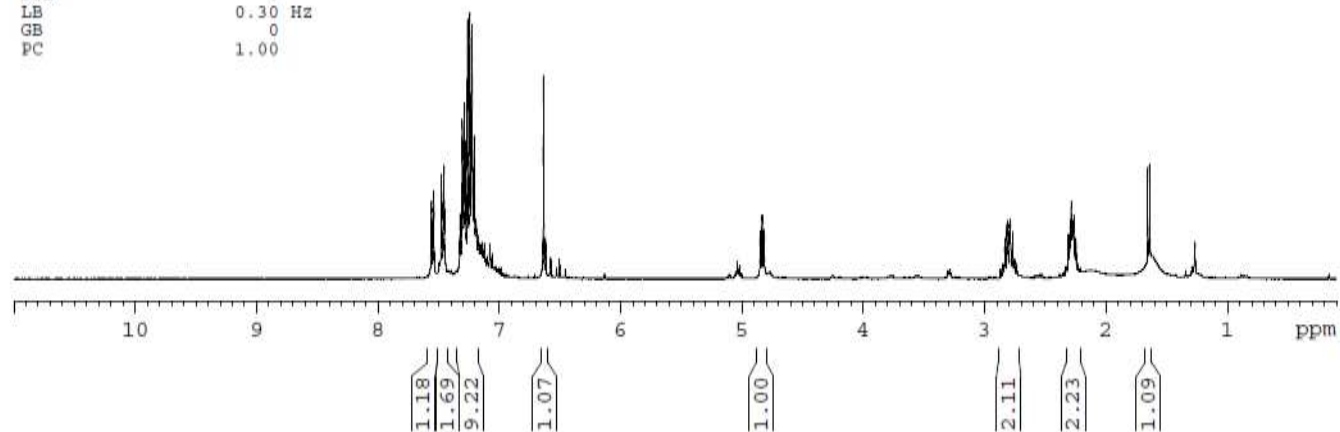
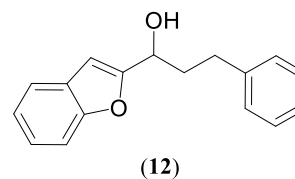
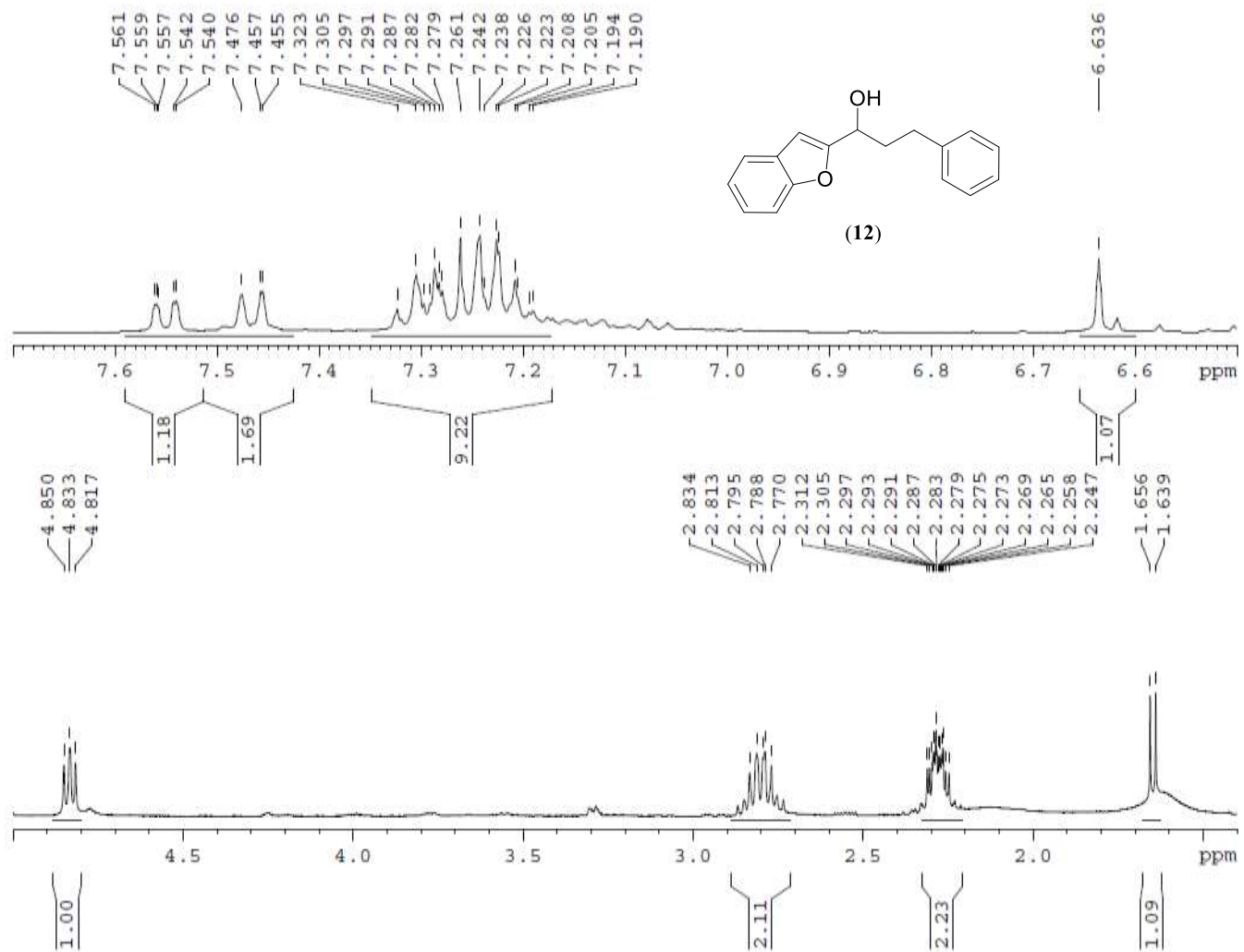


Figure S118. <sup>1</sup>H NMR spectrum of (12) in CDCl<sub>3</sub>.

PG-APP-12-03-2-1H



**Figure S119.** Expanded  $^1\text{H}$  NMR spectrum of (12) in  $\text{CDCl}_3$ .



PG-APP-12-10-1-13C

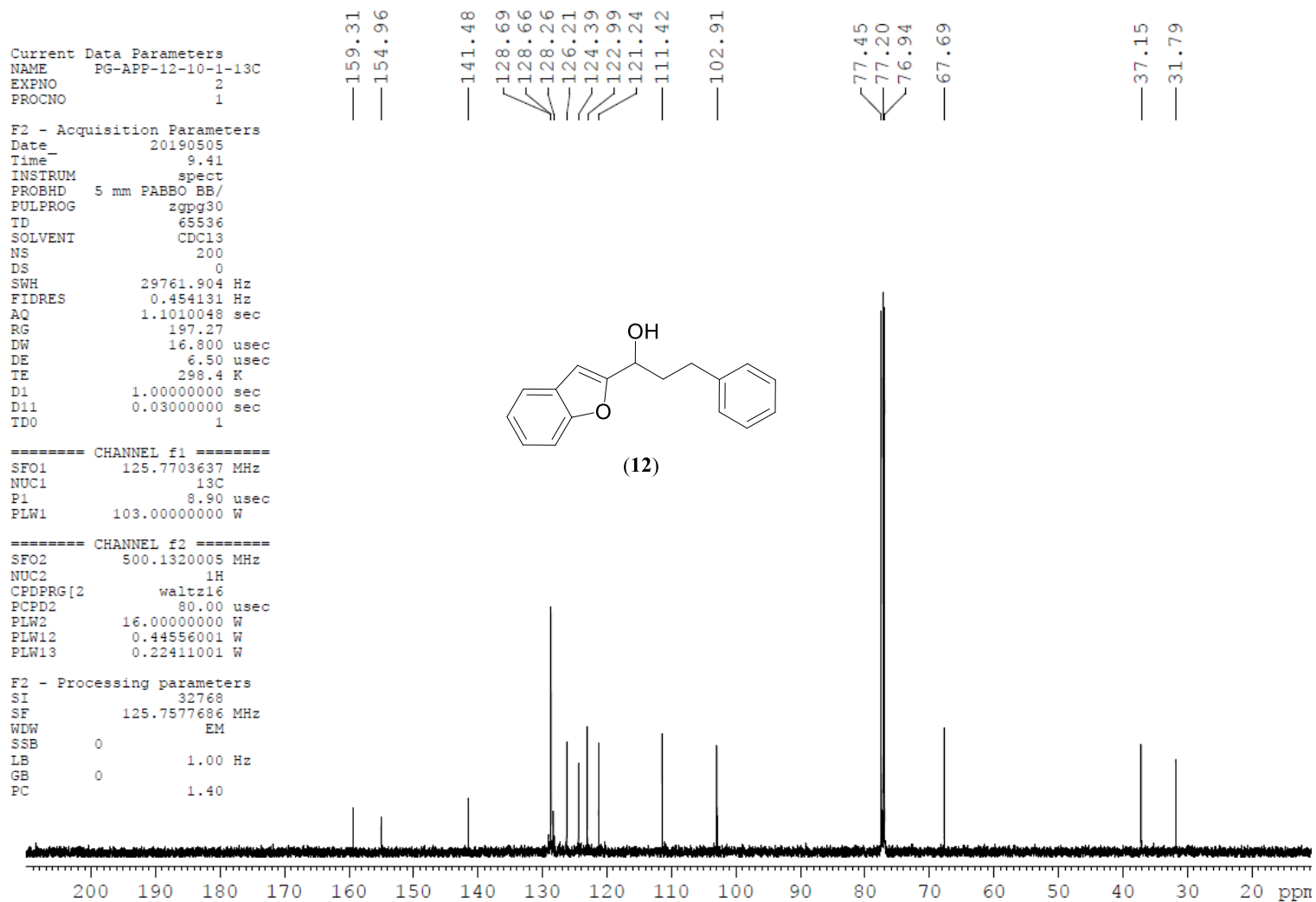
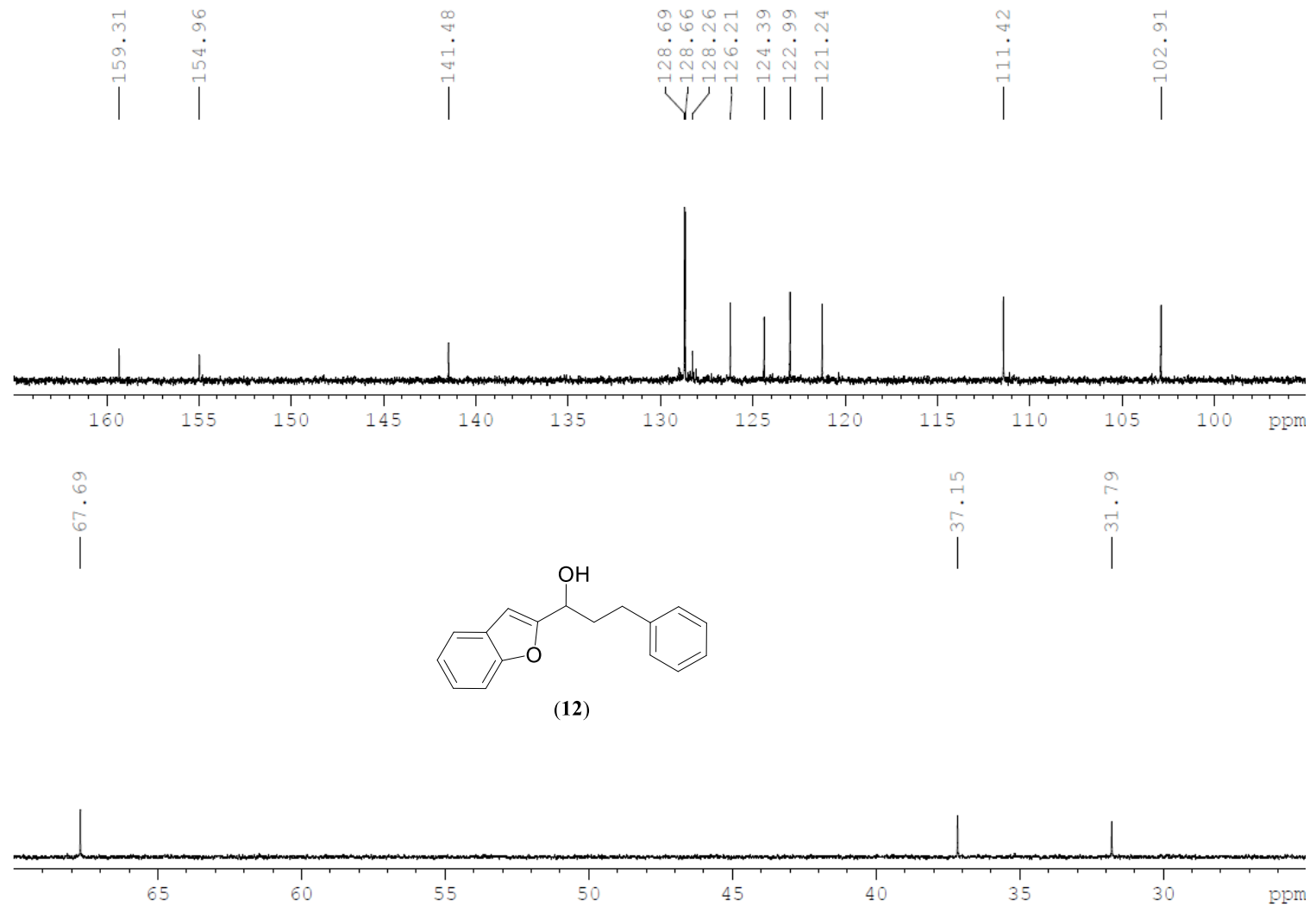


Figure S120.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (12) in  $\text{CDCl}_3$ .

PG-APP-12-10-1-13C



**Figure S121.** Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (12) in  $\text{CDCl}_3$ .

File : F:\GCMSDATA2019\May 2019\PG-APP-12-10-2.D  
Operator : APP  
Acquired : 5 May 2019 11:26 using AcqMethod COMMONMETHOD\_2018.M  
Instrument : GCMS  
Sample Name: PG-APP-12-10-2  
Misc Info :  
Vial Number: 3

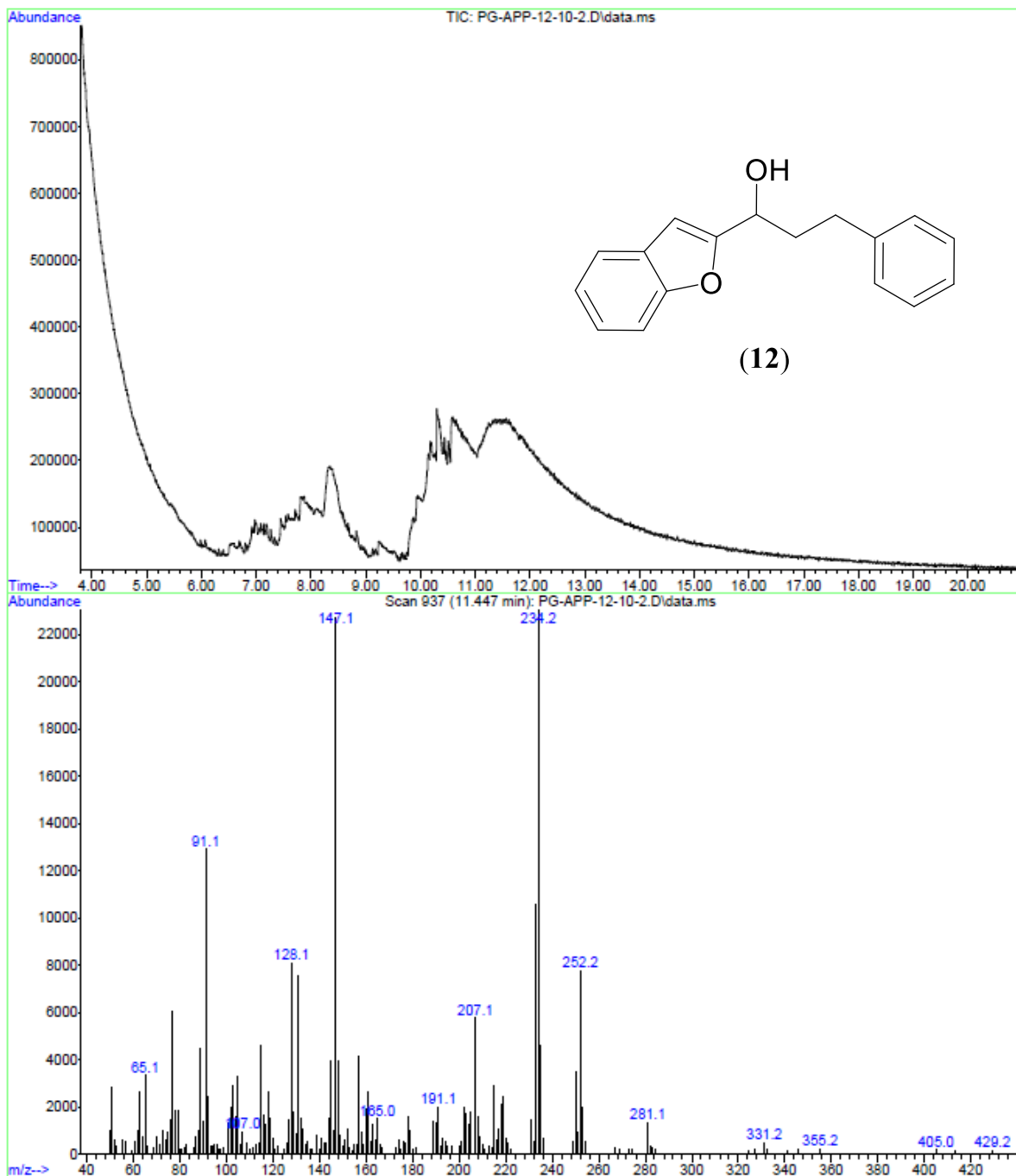
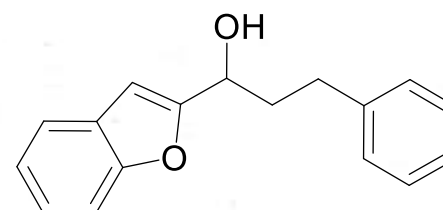


Figure S122. GCMS trace in EtOAc of (12) showing the  $M^+$  peak at  $m/z$  252.

## Eager 300 Report

Page: 1    Sample: PG-APP-12-03-1 (PG-APP-12-03-1)



(12)

```

Method Name      : PGAPP300519
Method File     : D:\CHNS2019\PGAPP300519.mth
Chromatogram    : PG-APP-12-03-1
Operator ID     : Prakash
Analysed        : 05/30/2019 17:22
Sample ID       : PG-APP-12-03-1 (# 34)
Analysis Type   : UnkNown (Area)

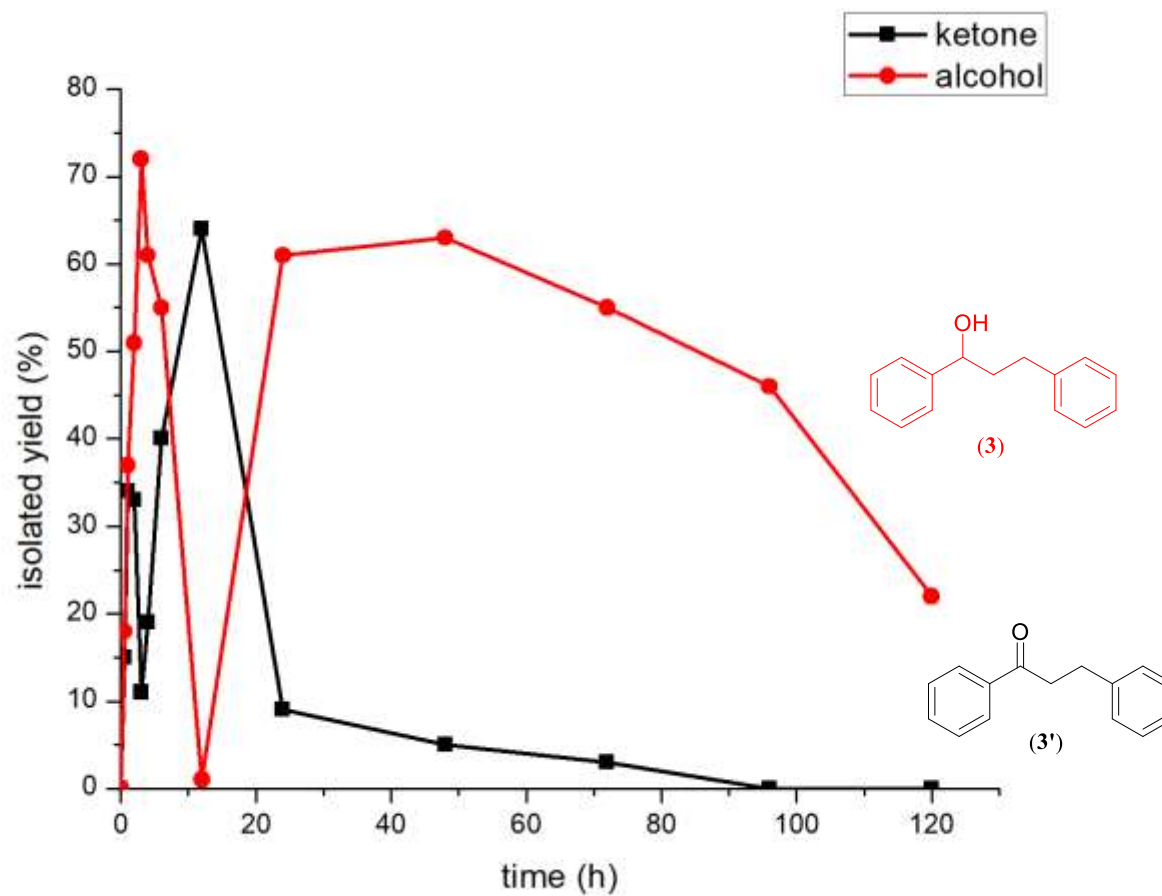
Company Name    : C.E. Instruments
Printed        : 5/30/2019 23:00
Instrument N.   : Instrument #1
Sample weight   : 1.108
    
```

Calib. method : using 'K Factors'

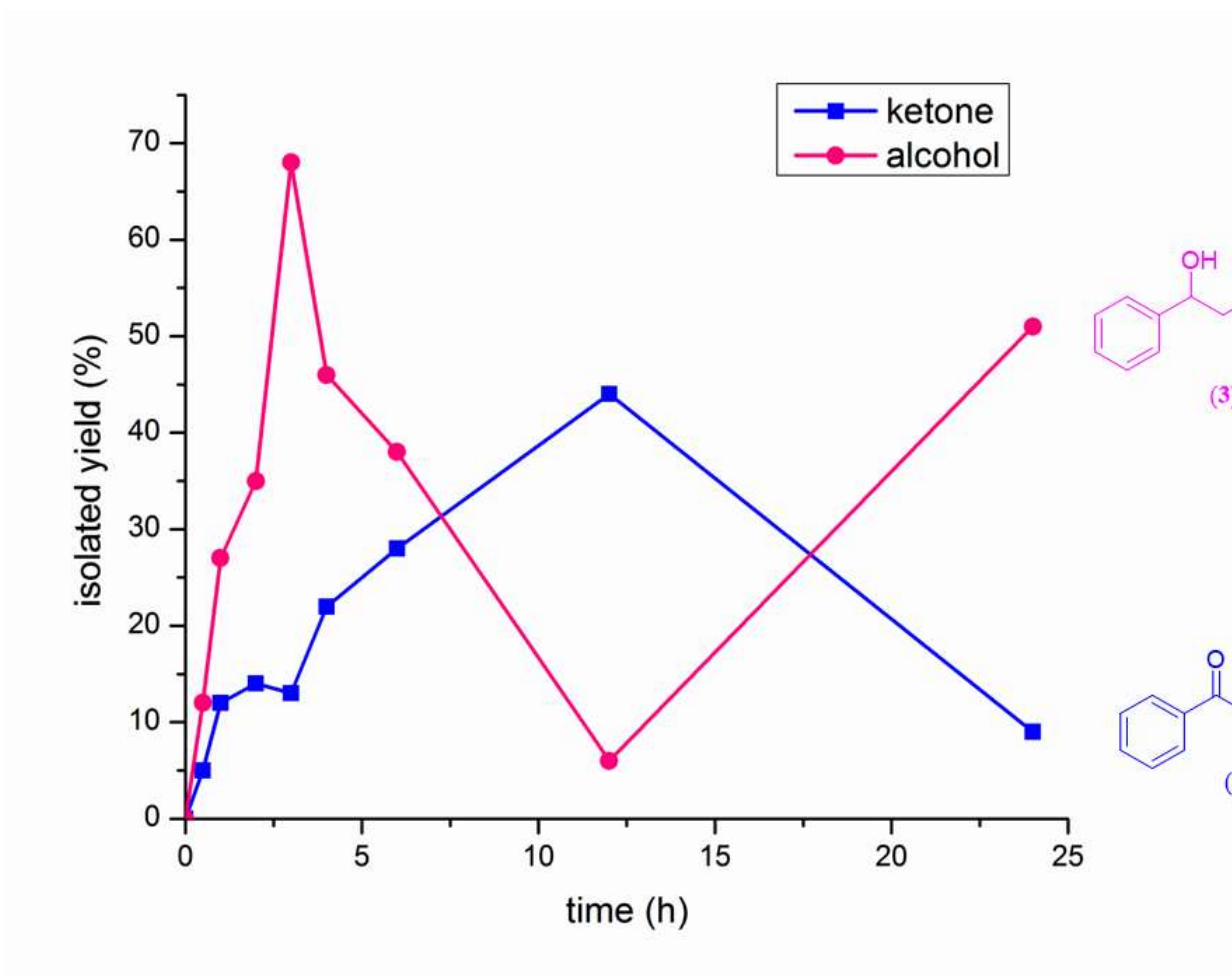
!!! Warning missing one or more peaks.

| Element Name | %       | Ret.Time | Area    | BC | Area ratio | K factor    |
|--------------|---------|----------|---------|----|------------|-------------|
| 1            | 0.0000  | 2        | 30781   | FU |            | 0.0000      |
| 2            | 0.0000  | 6        | 114524  | FU |            | 0.0000      |
| Carbon       | 81.1336 | 63       | 2401468 | RS | 1.000000   | .267139E+07 |
| Hydrogen     | 5.8671  | 184      | 444371  | RS | 5.404196   | .683575E+07 |
| Totals       | 87.0006 |          | 2991144 |    |            |             |

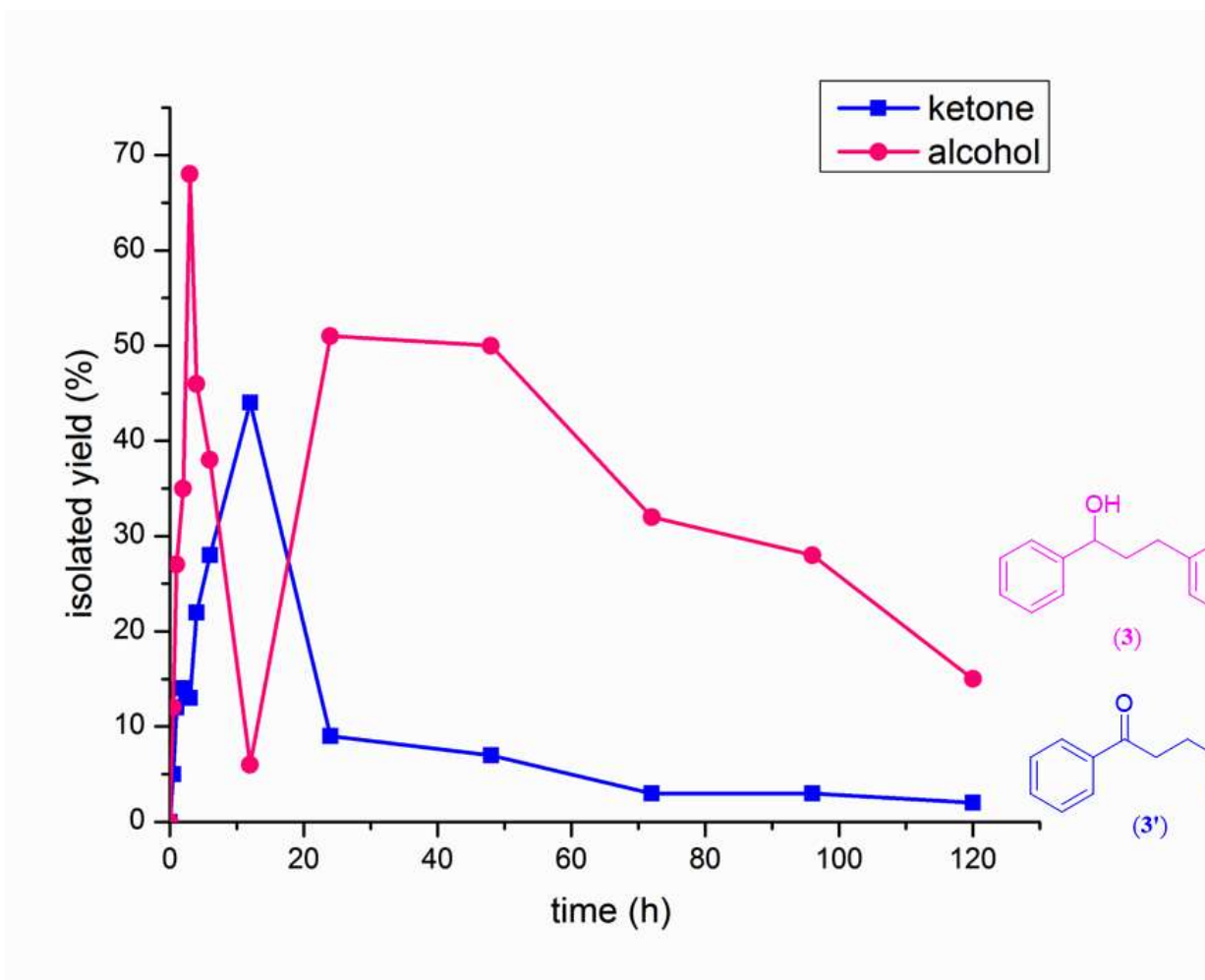
Figure S123. Elemental analysis data of (12).



**Figure S124.** An overlay of the formation of (3) and (3') as a function of time in the reaction of 1-phenylethanol and benzyl alcohol as catalyzed by the Ru–NHC complex (1c).



**Figure S125.** An overlay of the formation of (3) and (3') as a function of time in the reaction of 1-phenylethanol and benzyl alcohol as catalyzed by the Ru–NHC complex (2c).



**Figure S126.** An overlay of the formation of (3) and (3') as a function of time in the reaction of 1-phenylethanol and benzyl alcohol as catalyzed by the Ru-NHC complex (2c).

PG-ST-01-200-01

Current Data Parameters  
NAME PG-ST-01-200-01  
EXPNO 3  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20210126  
Time\_ 22.56  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDC13  
NS 16  
DS 0  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 30.72  
DW 50.000 usec  
DE 6.50 usec  
TE 292.7 K  
D1 1.0000000 sec  
TDO 1

==== CHANNEL f1 =====  
SFO1 500.1330885 MHz  
NUC1 1H  
P1 13.35 usec  
PLW1 16.00000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1300000 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

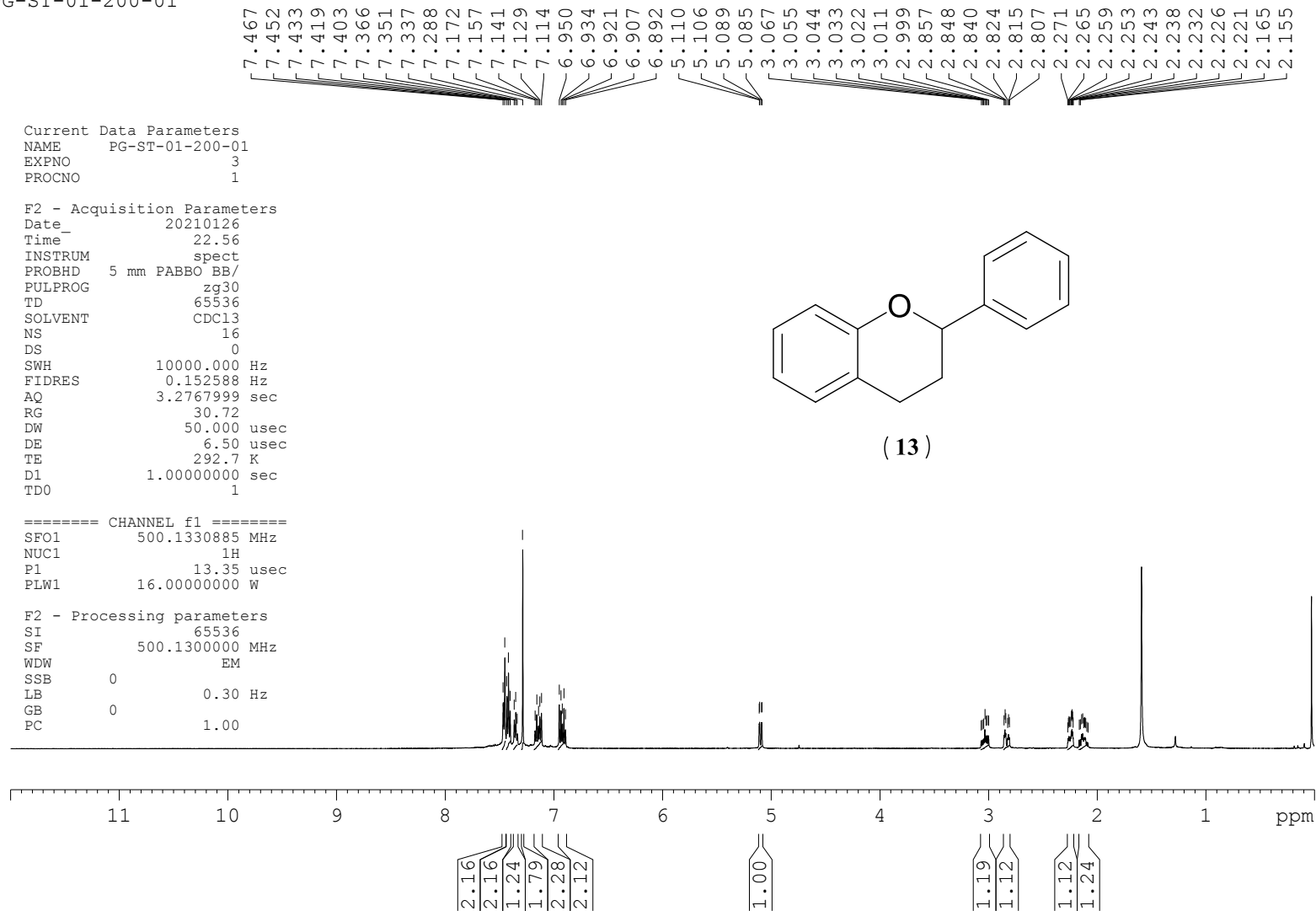


Figure S127. <sup>1</sup>H NMR spectrum of (13) in CDCl<sub>3</sub>.



PG-ST-01-200-01

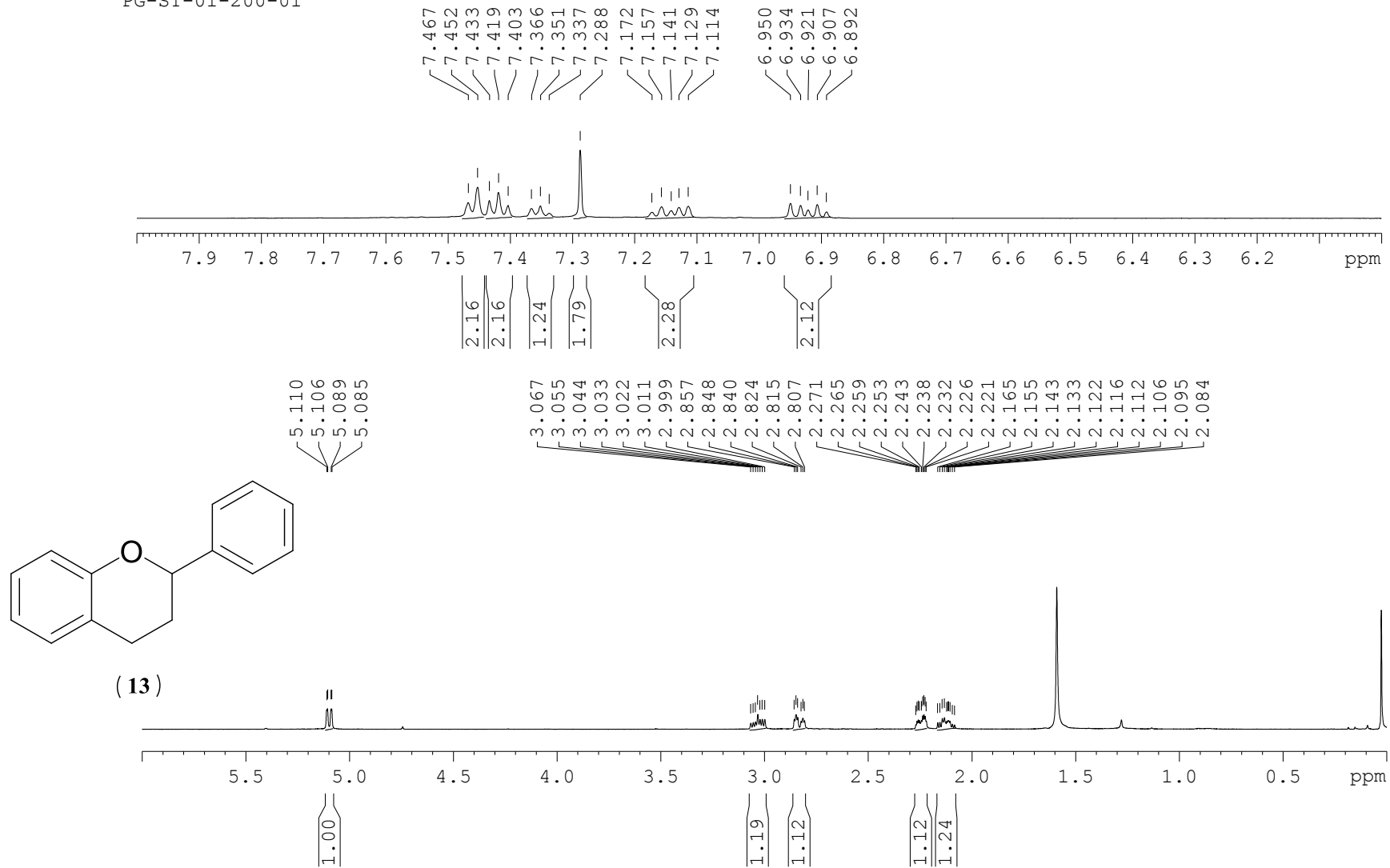


Figure S128. Expanded  $^1\text{H}$  NMR spectrum of (13) in  $\text{CDCl}_3$ .

PG-ST-01-200-01-13C

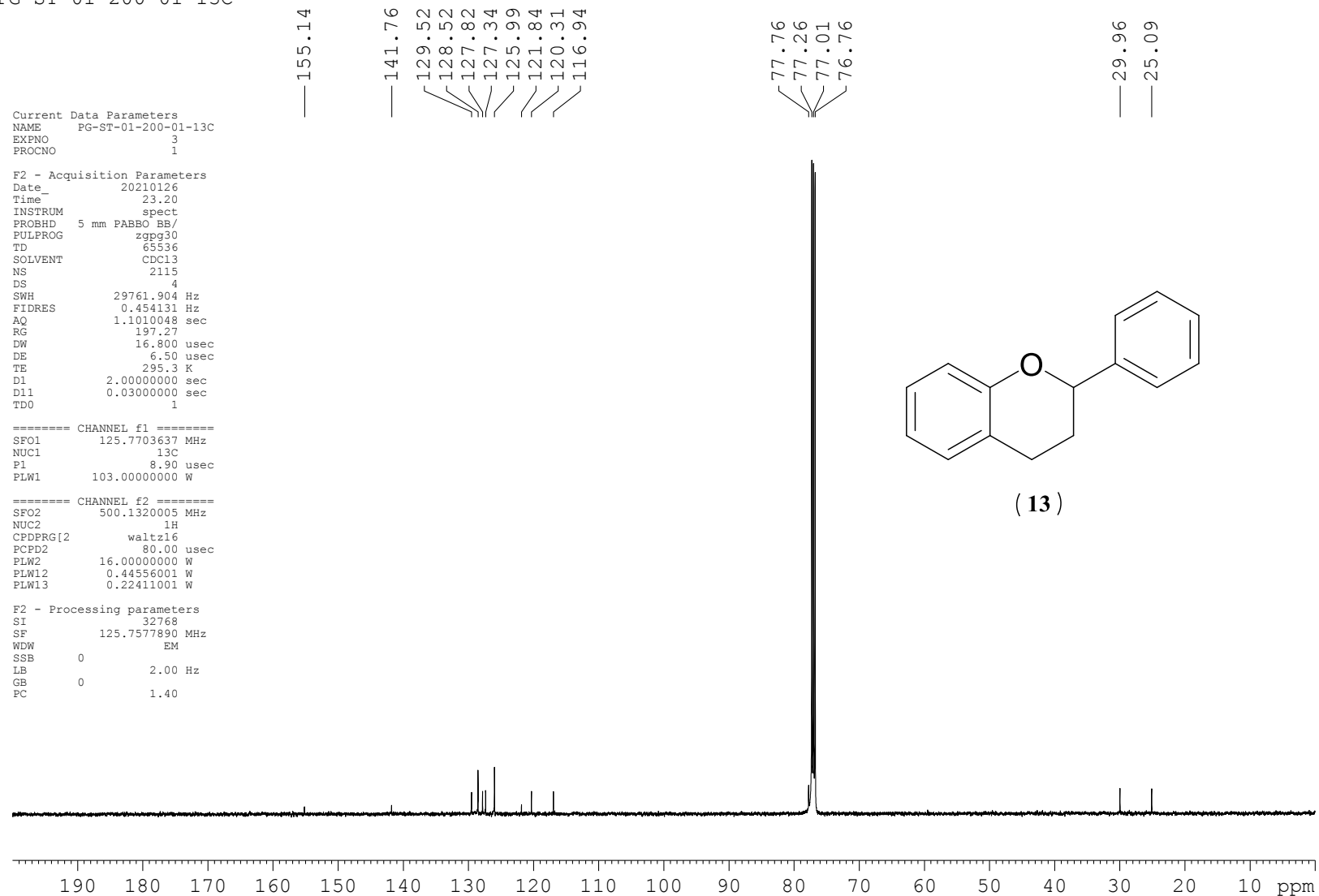


Figure S129.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (13) in  $\text{CDCl}_3$ .

PG-ST-01-200-01-13C

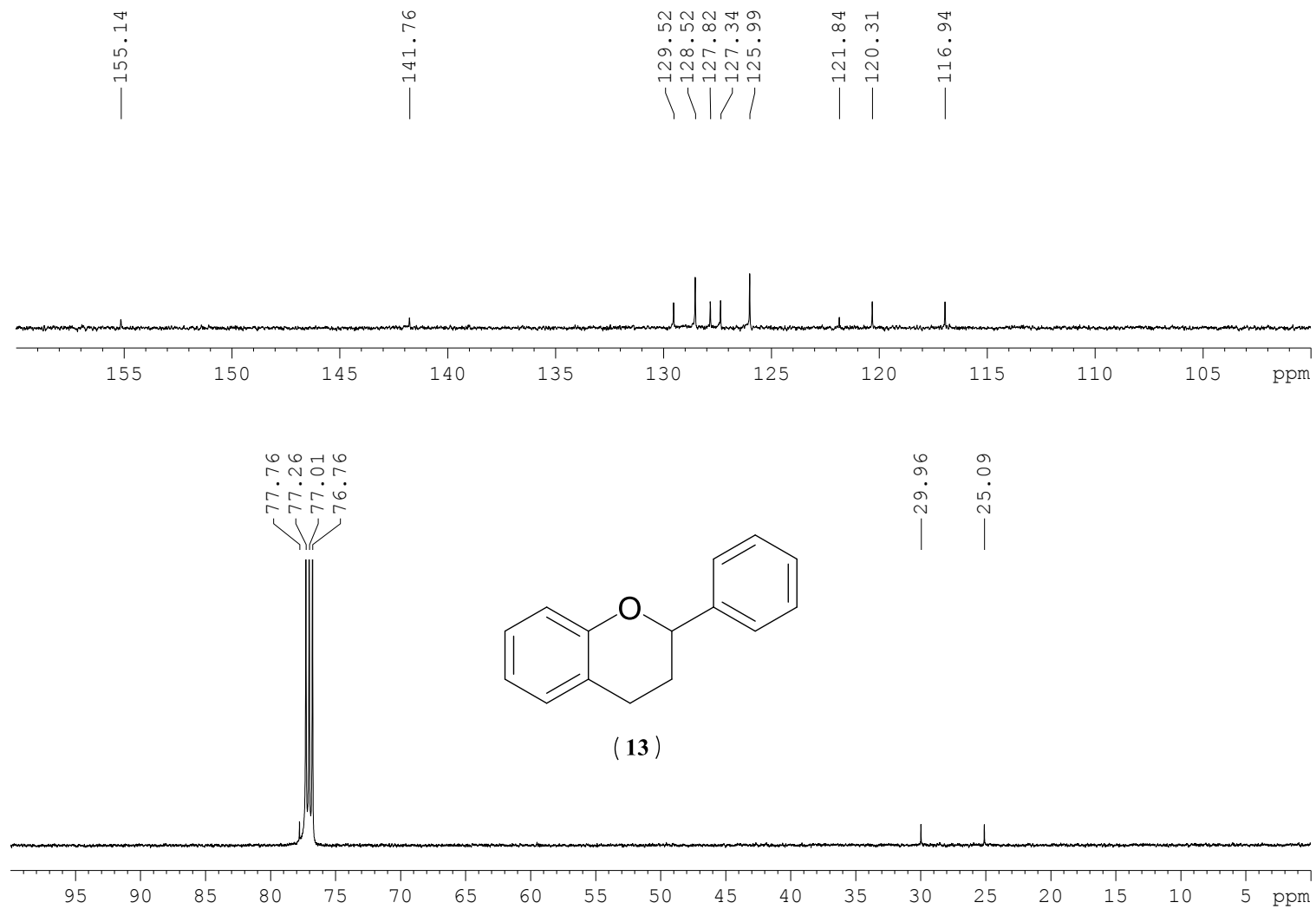


Figure S130. Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (13) in  $\text{CDCl}_3$ .

File: I:\GCMS-GATA-2020\JAN-2021\PG-ST-01-204.D  
Operator: J.ZH  
Acquired: 10 Feb 2021 16:00 using AcqMethod: COMBIMETHOD-2020.M  
Instrument: GCMS  
Sample Name: PG-ST-01-204  
Misc Info:  
Vial Number: 4

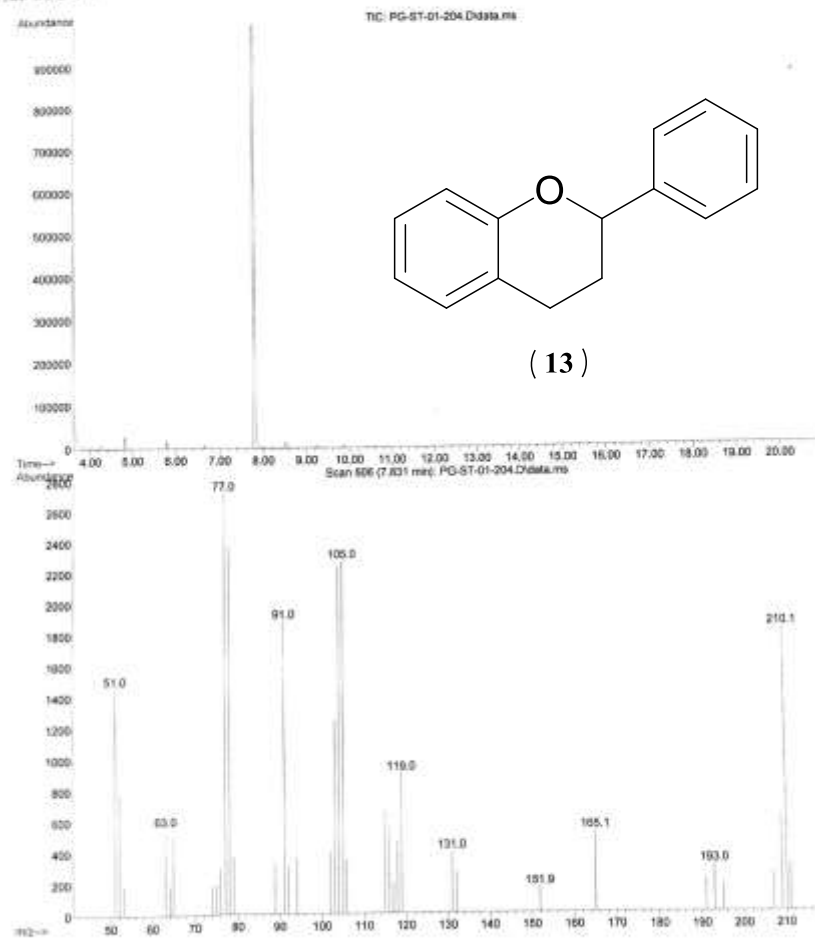
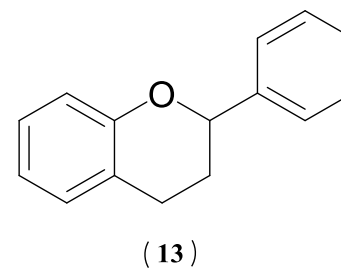
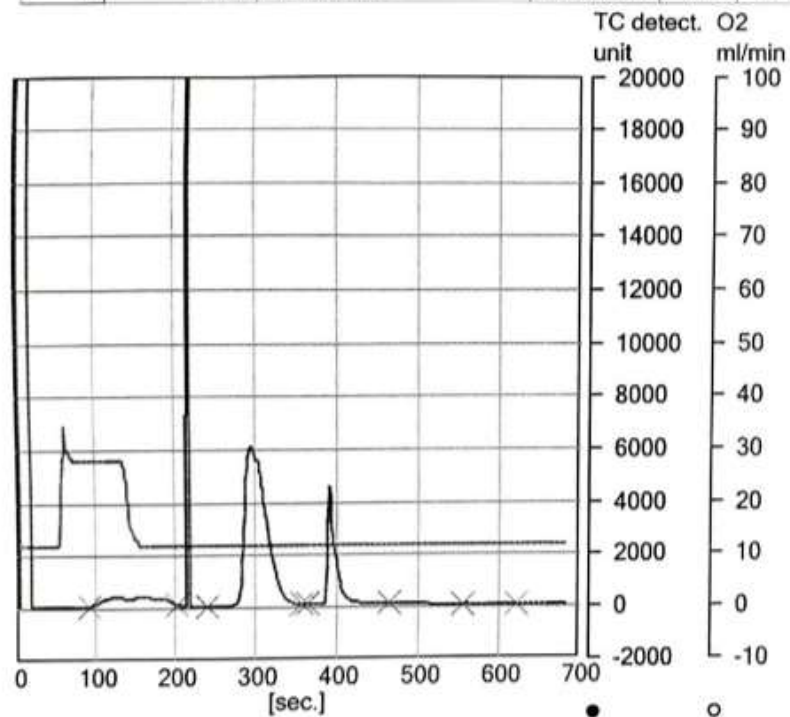


Figure S131. GCMS trace in EtOAc of (13) showing the  $M^+$  peak at  $m/z$  210.

| No. | Weight [mg] | Name           | Method     | N Area | C Area | H Area | N [%] | C [%] | H [%] | Date       | Time  |
|-----|-------------|----------------|------------|--------|--------|--------|-------|-------|-------|------------|-------|
| 45  | 0.8080      | PG-ST-01-200-1 | 2mgChem80s | 2 741  | 18 143 | 4 862  | 0.00  | 79.34 | 5.581 | 29-01-2021 | 21:54 |



Name: eassuperuser, Access: VarioMICRO administrator

30-01-2021 15:40:25

varioMICRO V4.0.1 (aeb1e0e)2015-10-12, CHNS Mode, Ser. No.: 15154051  
Elementar Analysensysteme GmbH

Page 1 (of 1)

Figure S132. Elemental analysis data of (13).

PG-ST-01-219-1-1H

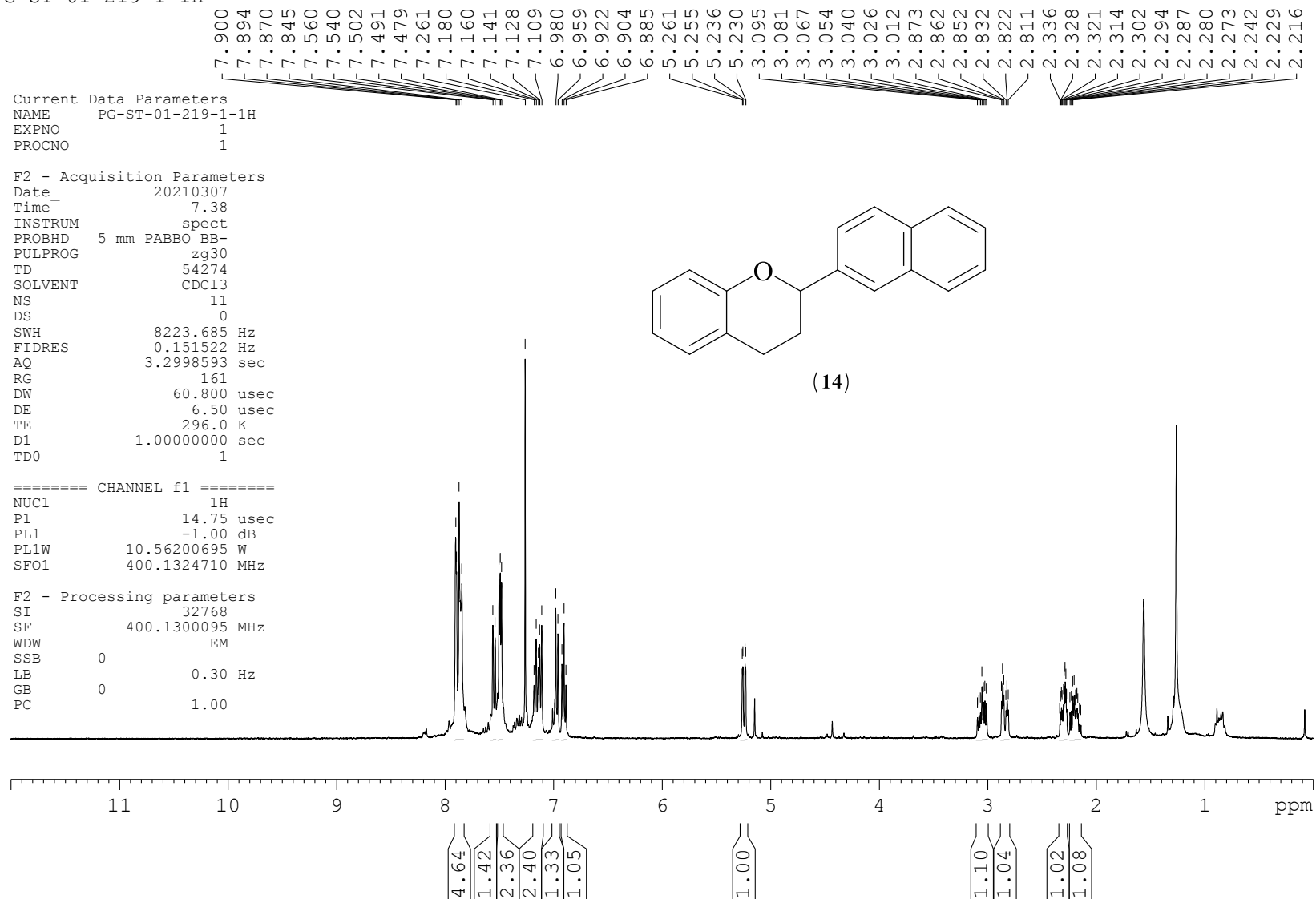
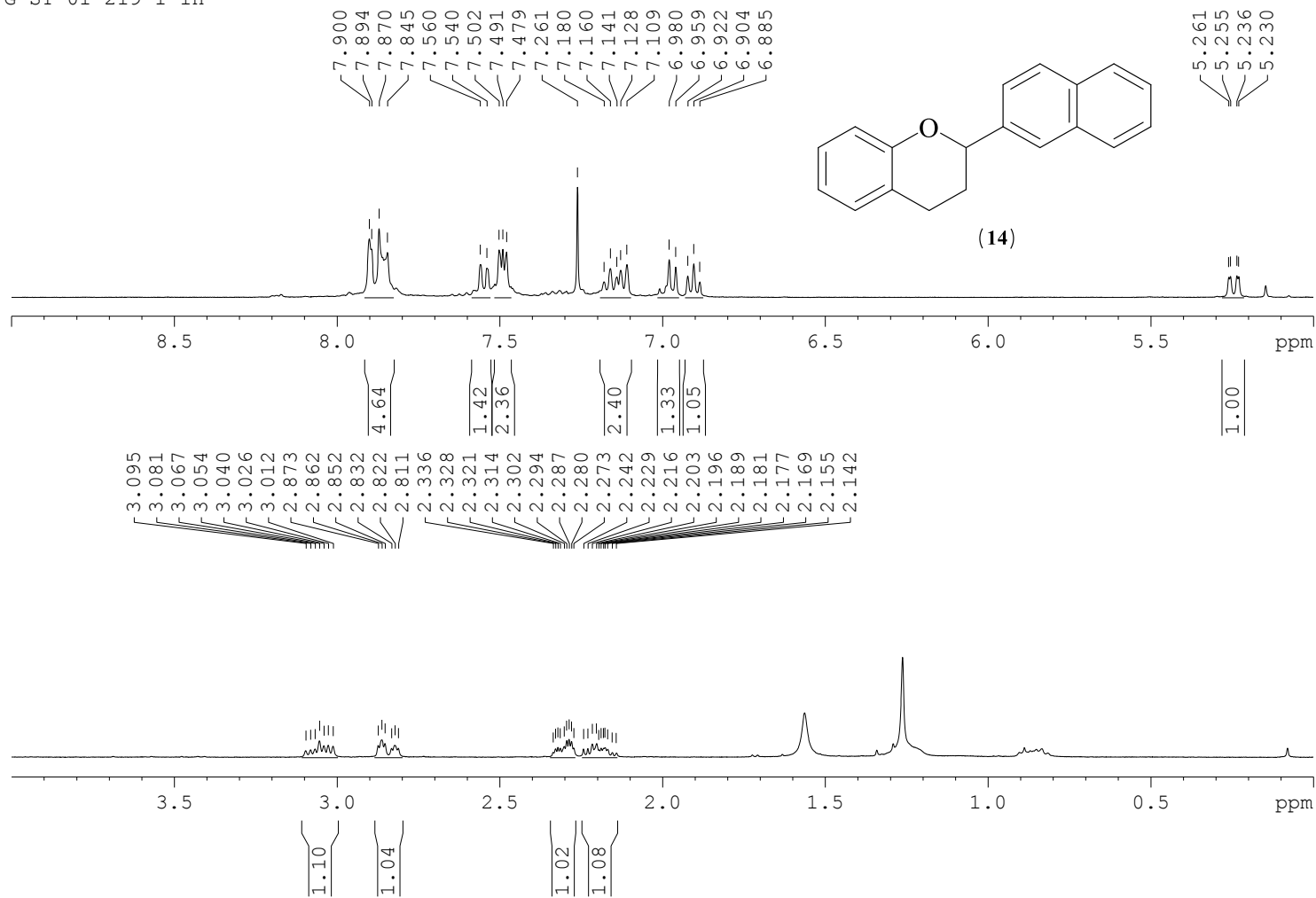


Figure S133. <sup>1</sup>H NMR spectrum of (14) in CDCl<sub>3</sub>.

PG-ST-01-219-1-1H



**Figure S134.** Expanded  $^1\text{H}$  NMR spectrum of (14) in  $\text{CDCl}_3$ .

PG-ST-01-219-01-13C

Current Data Parameters  
NAME PG-ST-01-219-01-13C  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20210310  
Time 2.52  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 253  
DS 0  
SWH 29761.904 Hz  
FIDRES 0.454131 Hz  
AQ 1.1010048 sec  
RG 197.27  
DW 16.800 usec  
DE 6.50 usec  
TE 295.6 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TD0 1

==== CHANNEL f1 =====  
SFO1 125.7703637 MHz  
NUC1 13C  
P1 8.90 usec  
PLW1 103.0000000 W

==== CHANNEL f2 =====  
SFO2 500.1320005 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 80.00 usec  
PLW2 16.00000000 W  
PLW12 0.44556001 W  
PLW13 0.22411001 W

F2 - Processing parameters  
SI 32768  
SF 125.7577890 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

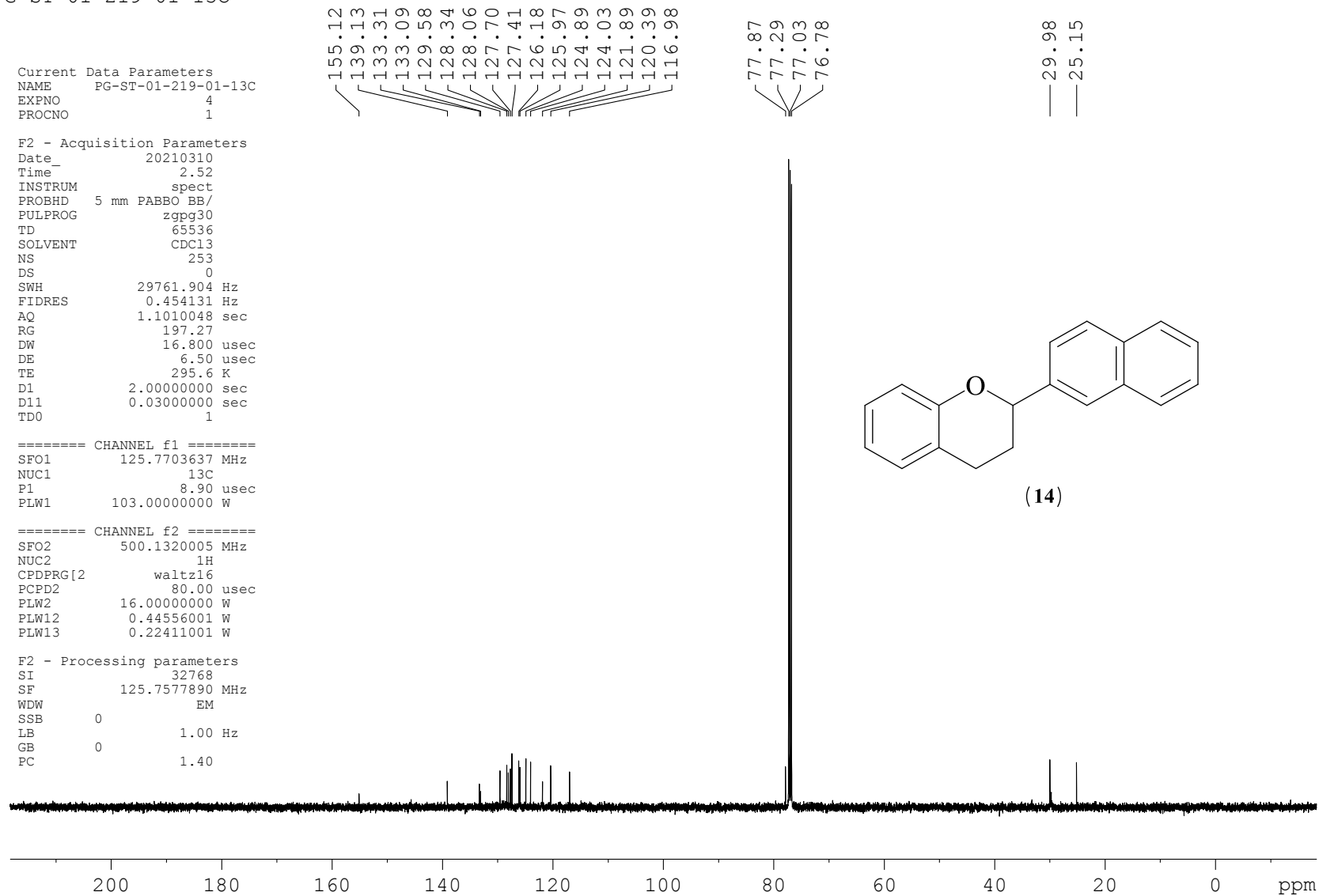


Figure S135.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (14) in  $\text{CDCl}_3$ .



PG-ST-01-219-01-13C

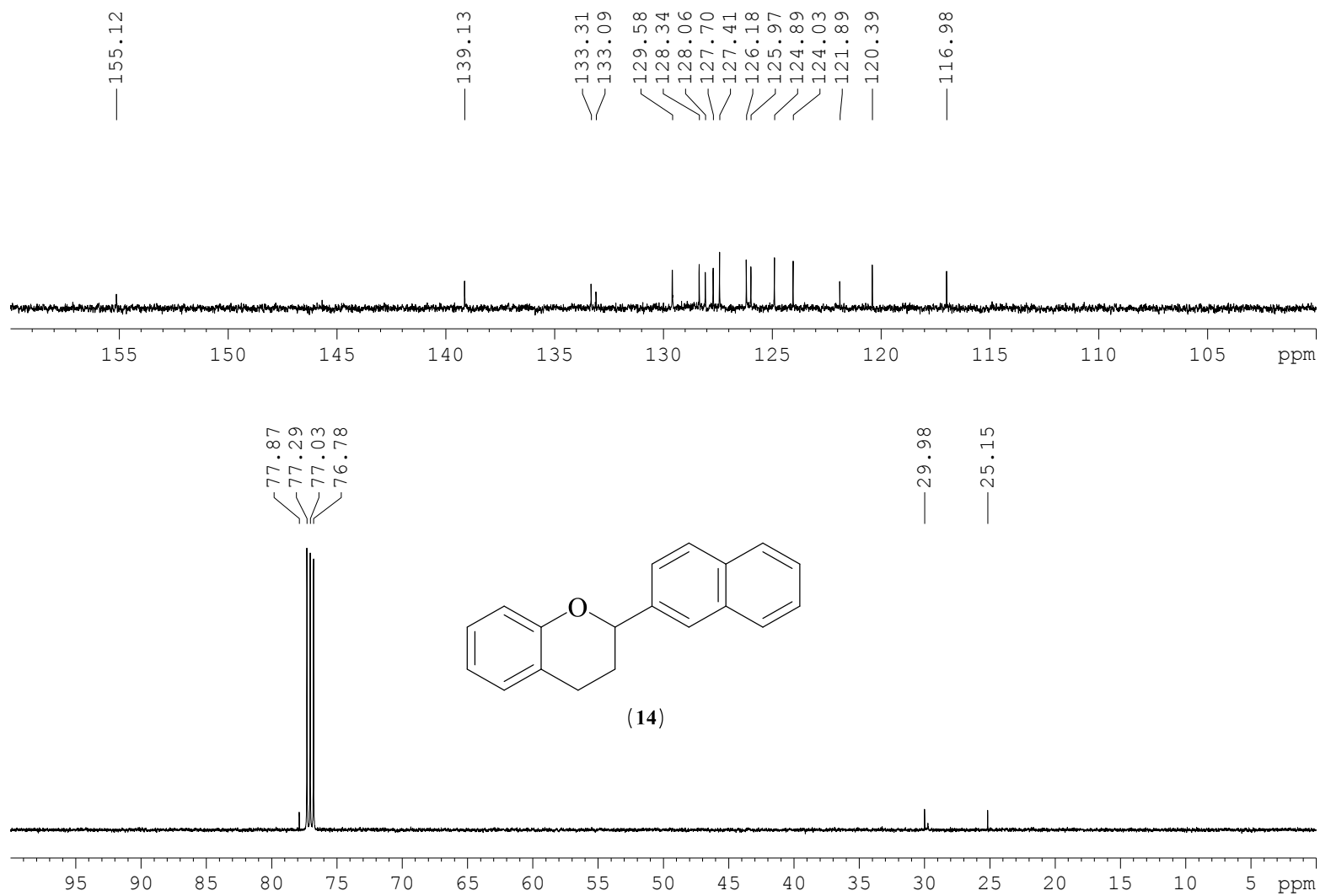


Figure S136. Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (14) in  $\text{CDCl}_3$ .

File :F:\GCMS-DATA-2021\MARCH 2021\PG-ST-01-219-1.D  
Operator : SACHIN  
Acquired : 9 Mar 2021 00:05 using AcqMethod COMMONMETHOD-2020.M  
Instrument : GCMS  
Sample Name: PG-ST-01-219-1  
Misc Info :  
Vial Number: 3

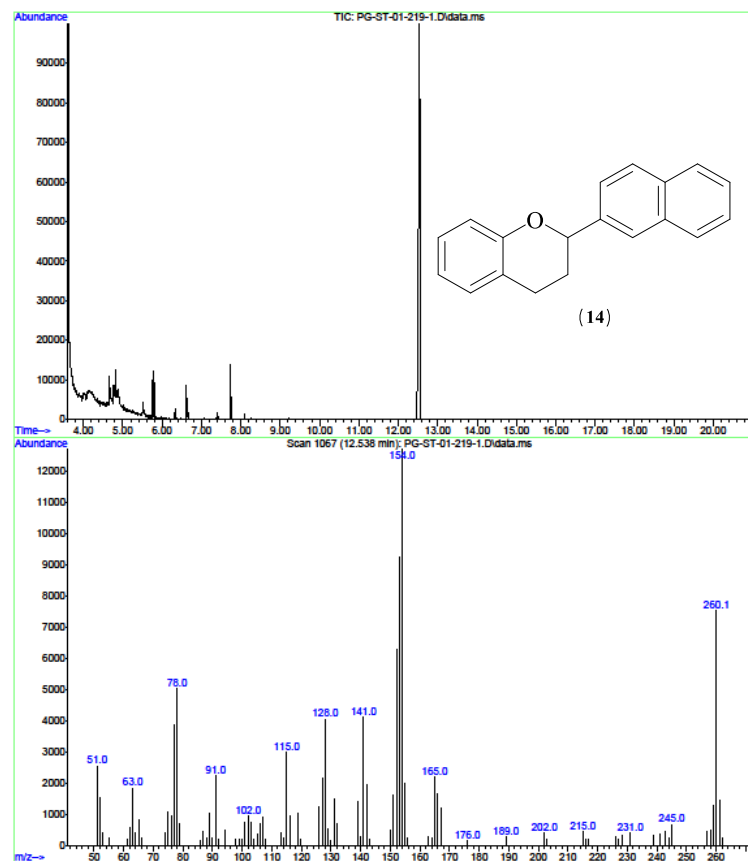


Figure S137. GCMS trace in EtOAc of (14) showing the M<sup>+</sup> peak at  $m/z$  260.

Document: SP-06-04-2021 (varioMICRO) from: ---,--- (modified)

SP18022016  
varioMICRO CHNS  
serial number: 15154051

Graphic report

| No. | Weight [mg] | Name              | Method     | N Area | C Area | H Area | N [%] | C [%] | H [%] | Date       | Time  |
|-----|-------------|-------------------|------------|--------|--------|--------|-------|-------|-------|------------|-------|
| 23  | 1.0800      | PG-ST-01-222-02-1 | 2mgChem80s | 1 971  | 25 238 | 6 949  | 0.00  | 81.68 | 5.662 | 06-04-2021 | 17:45 |

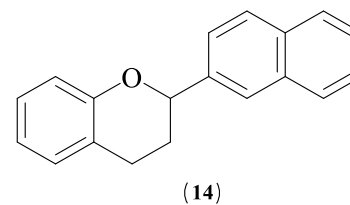
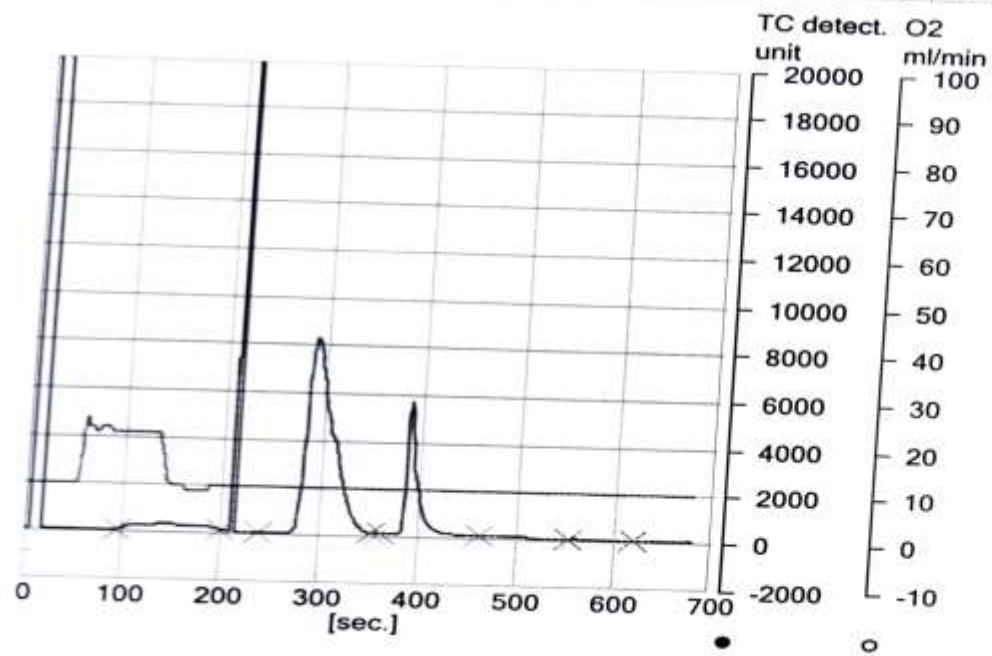


Figure S138. Elemental analysis data (14).

PG-ST-01-230-05

Current Data Parameters  
NAME PG-ST-01-230-05  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20210314  
Time\_ 22.26  
INSTRUM spect  
PROBHD 5 mm PABBO BB/  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 10000.000 Hz  
FIDRES 0.152588 Hz  
AQ 3.2767999 sec  
RG 157.24  
DW 50.000 usec  
DE 6.50 usec  
TE 295.8 K  
D1 1.00000000 sec  
TDO 1

==== CHANNEL f1 =====  
SFO1 500.1330885 MHz  
NUC1 1H  
P1 13.35 usec  
PLW1 16.00000000 W

F2 - Processing parameters  
SI 65536  
SF 500.1300000 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

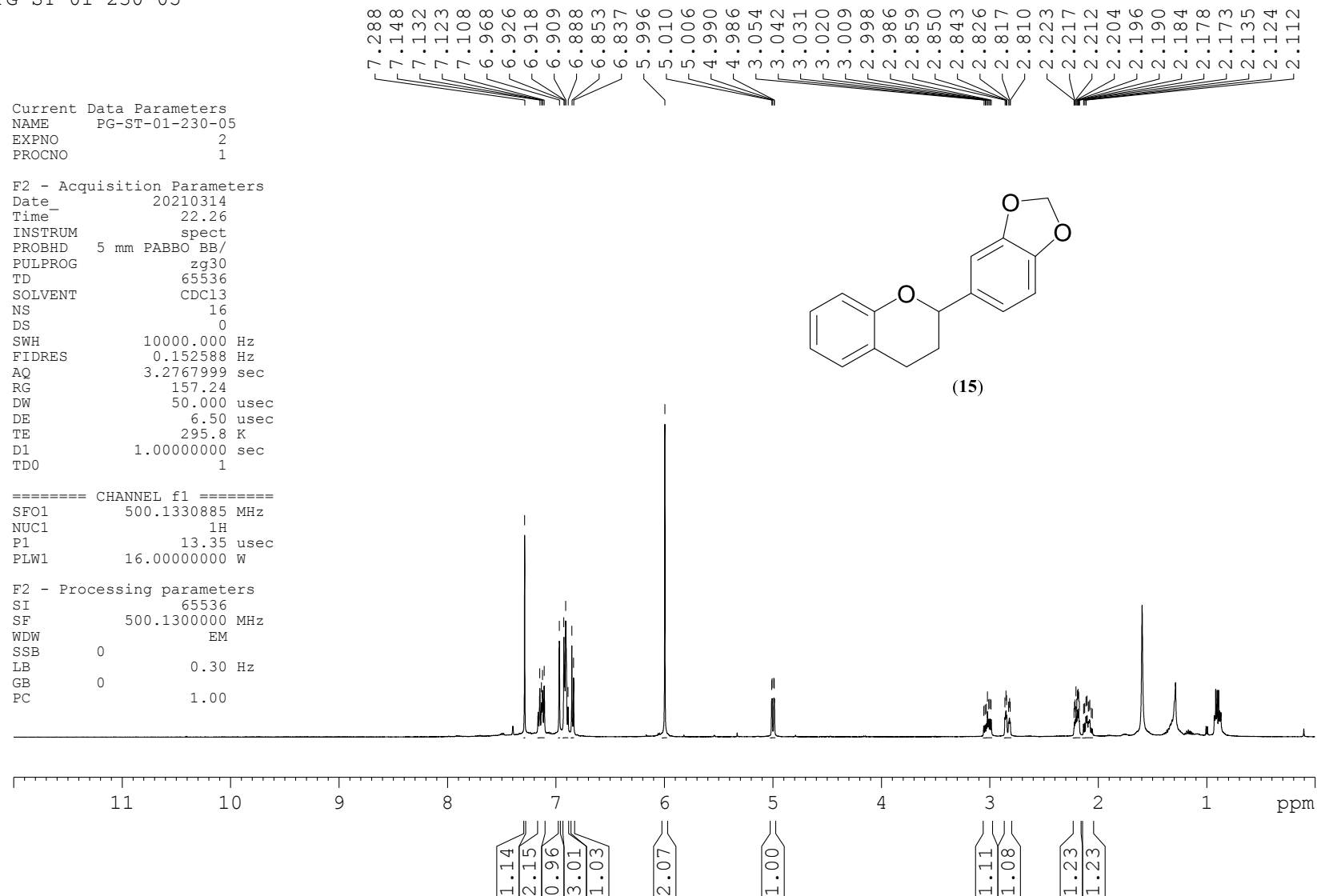
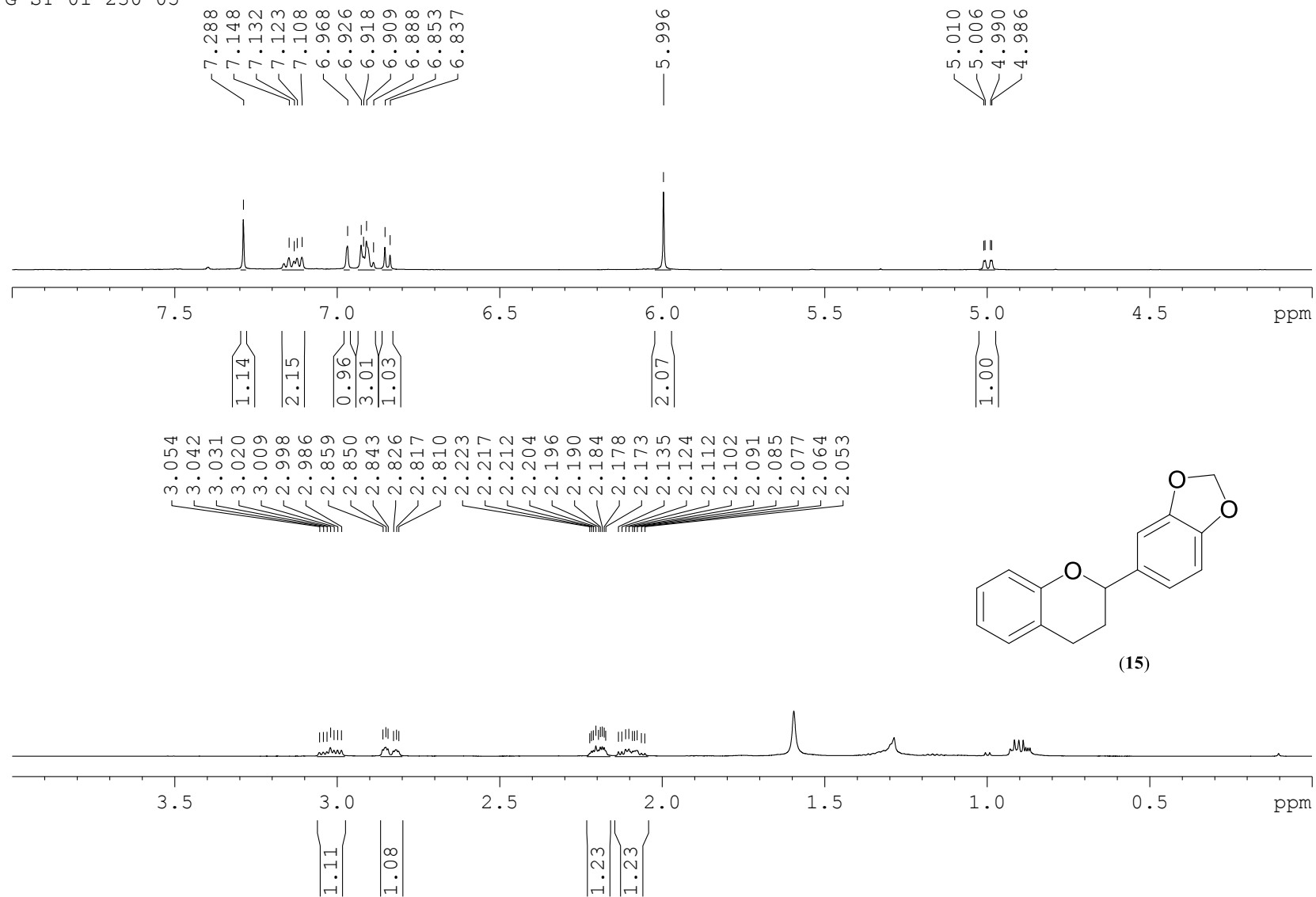


Figure S139. <sup>1</sup>H NMR spectrum of (15) in CDCl<sub>3</sub>.

PG-ST-01-230-05



**Figure S140.** Expanded  $^1\text{H}$  NMR spectrum of (15) in  $\text{CDCl}_3$ .

PG-ST-01-230-05-13C

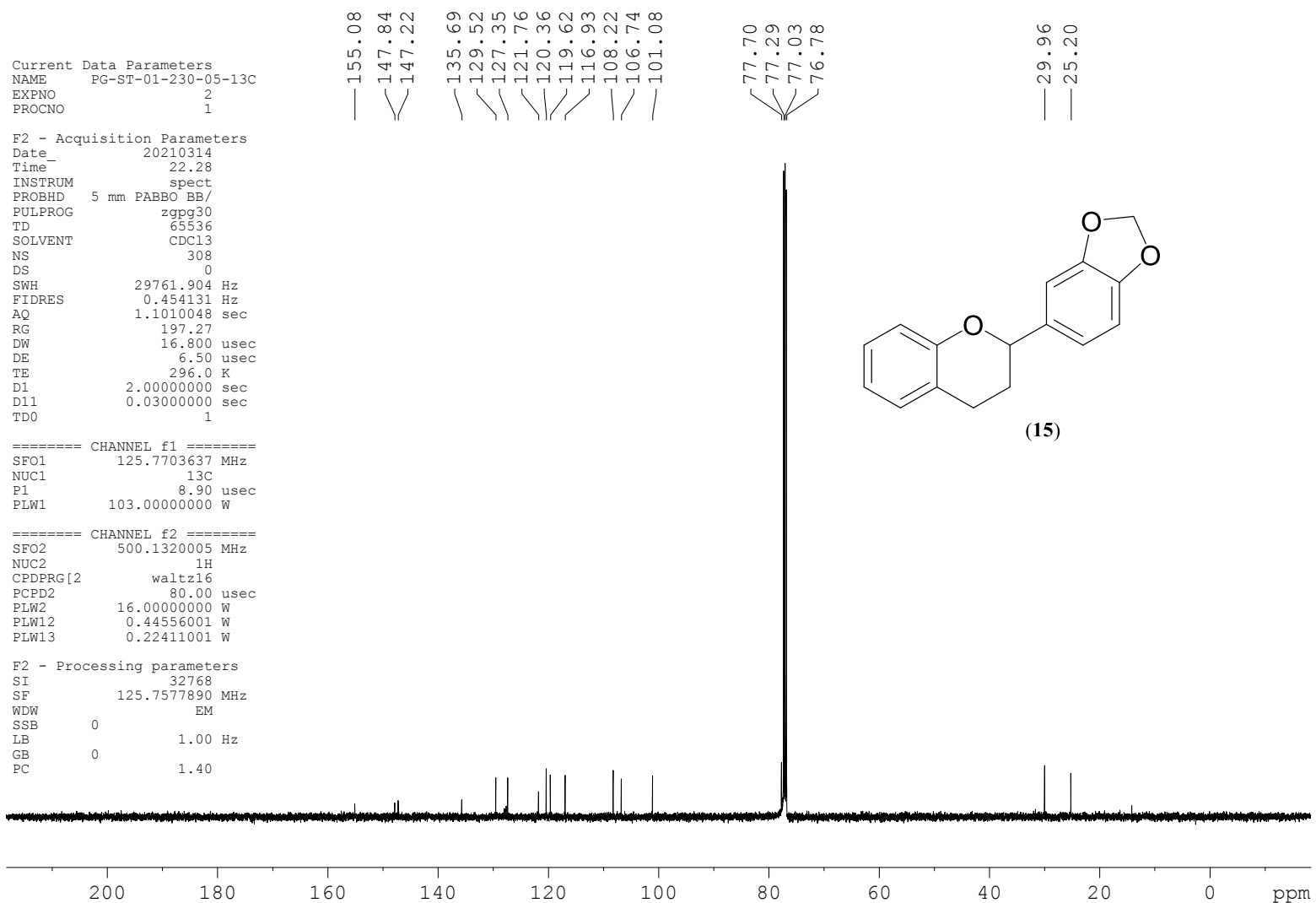


Figure S141.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (15) in  $\text{CDCl}_3$ .

PG-ST-01-230-05-13C

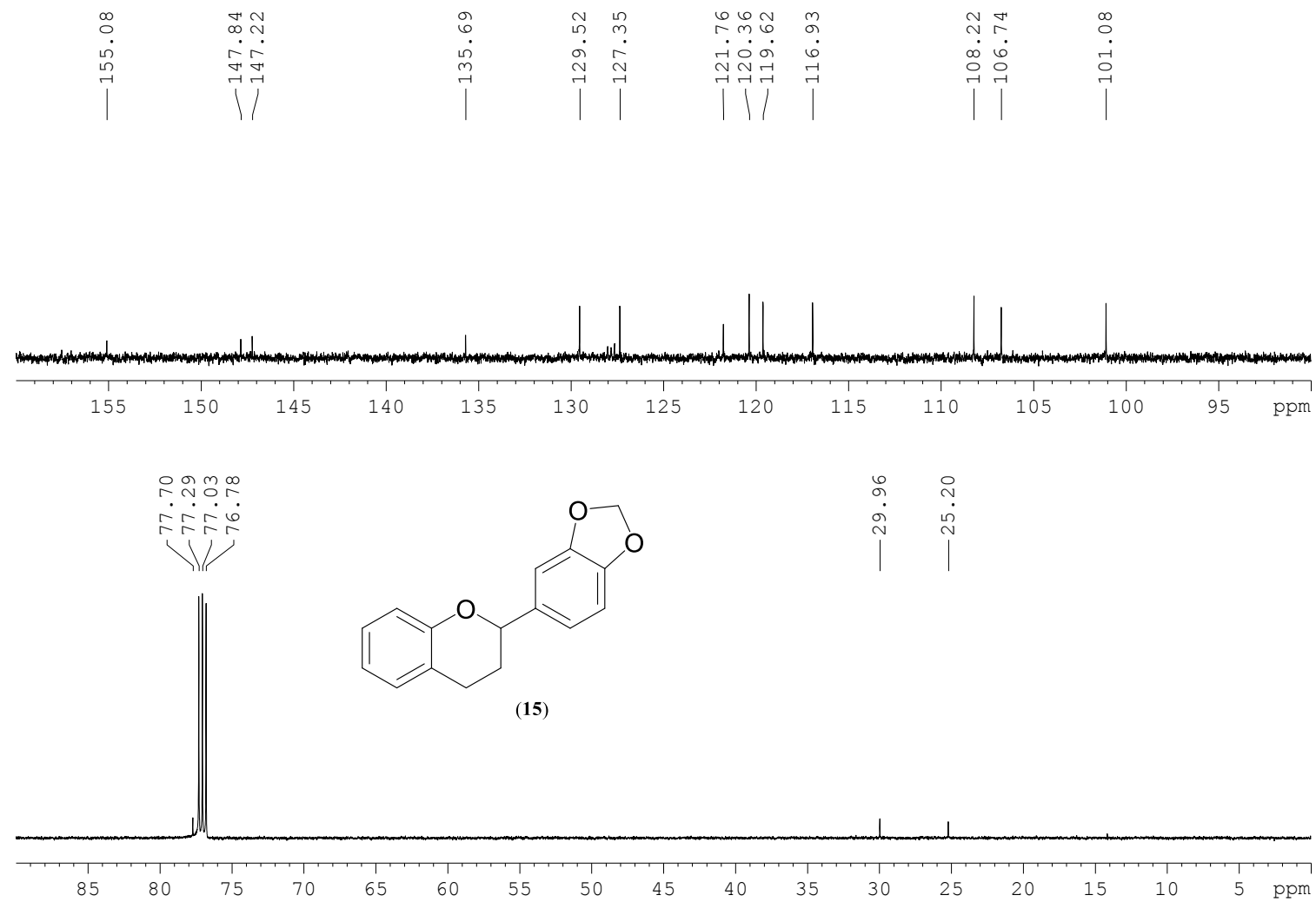
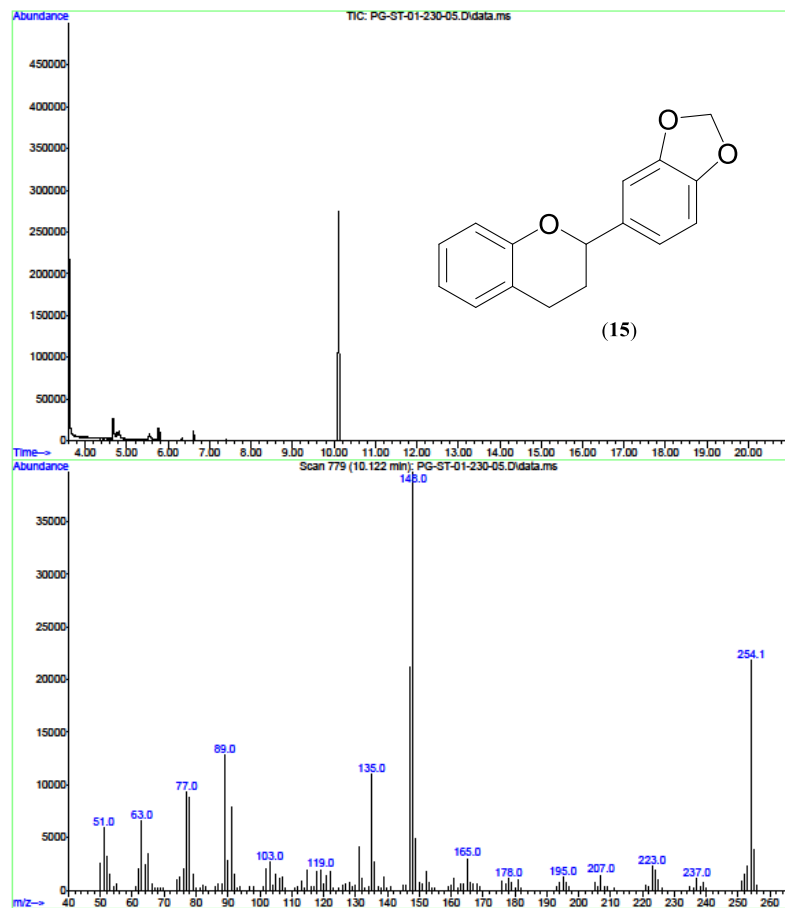


Figure S142. Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (15) in  $\text{CDCl}_3$ .

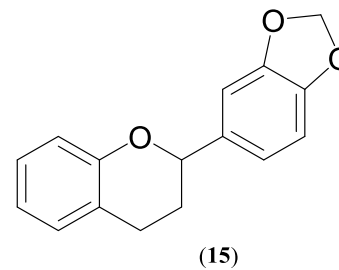
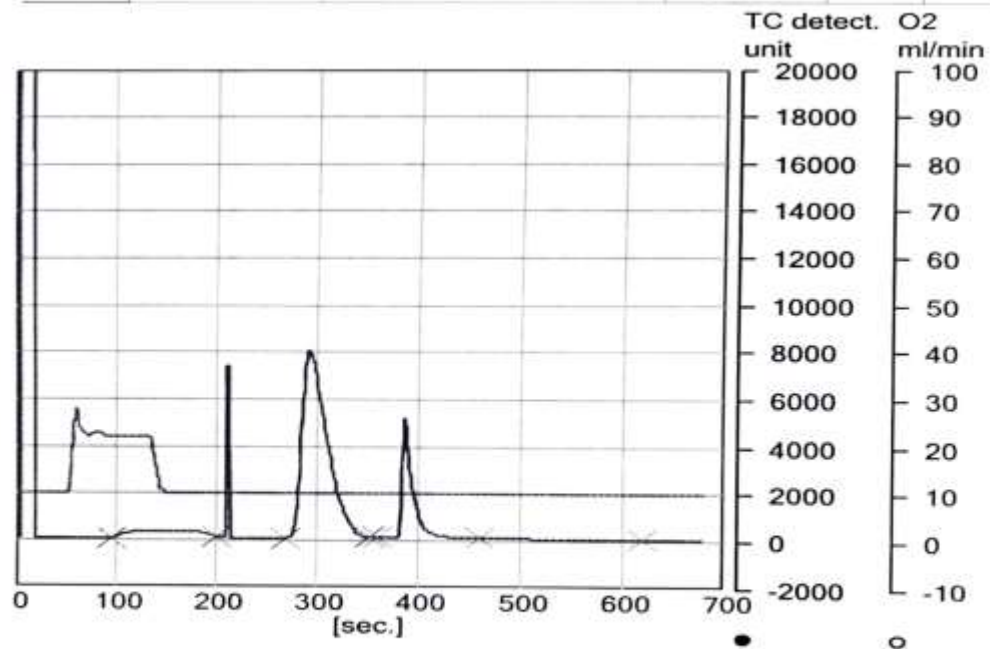
File :F:\GCMS-DATA-2021\MARCH 2021\PG-ST-01-230-05.D  
Operator : SACHIN  
Acquired : 15 Mar 2021 13:54 using AcqMethod COMMONMETHOD-2020.M  
Instrument : GCMS  
Sample Name : PG-ST-01-230-05  
Misc Info :  
Vial Number: 6



**Figure S143.** GCMS trace in EtOAc of (15) showing the M<sup>+</sup> peak at *m/z* 254.



| No. | Weight [mg] | Name              | Method     | N Area | C Area | H Area | N [%] | C [%] | H [%] | Date       | Time  |
|-----|-------------|-------------------|------------|--------|--------|--------|-------|-------|-------|------------|-------|
| 36  | 1.0540      | PG-ST-01-230-05-2 | 2mgChem80s | 2 174  | 22 272 | 6 036  | 0.00  | 74.42 | 4.292 | 01-04-2021 | 18:35 |



Name: eassuperuser, Access: VarioMICRO administrator

02-04-2021 09:55:13

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Figure S144. Elemental analysis data (15).

PG-ST-02-04-01-1H

Current Data Parameters  
NAME PG-ST-02-04-03-1H  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20210323  
Time 6.51  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 54274  
SOLVENT CDCl3  
NS 25  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2998593 sec  
RG 80.6  
DW 60.800 usec  
DE 6.50 usec  
TE 295.4 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.75 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300095 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
FC 1.00

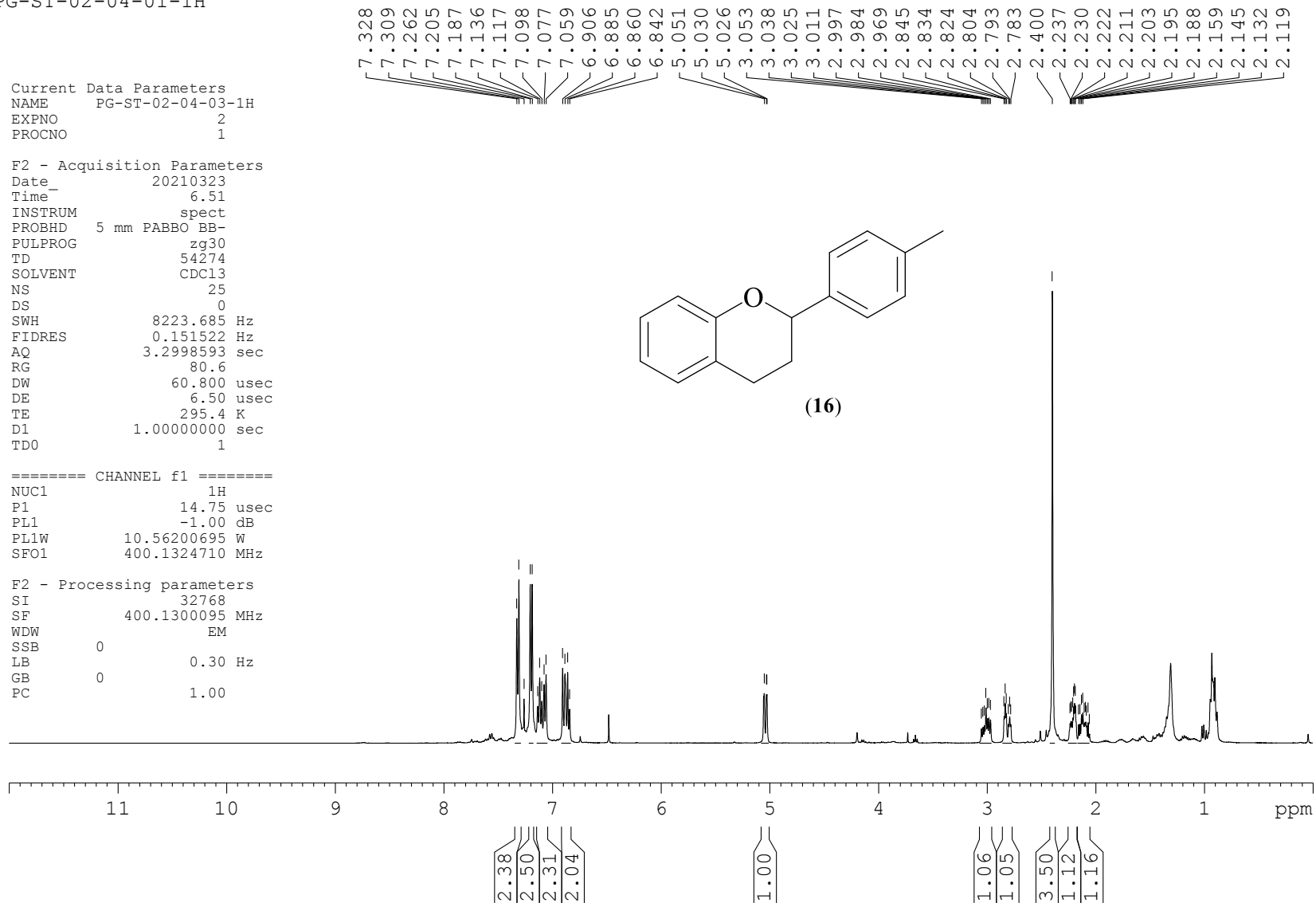


Figure S145. <sup>1</sup>H NMR spectrum of (16) in CDCl<sub>3</sub>.

PG-ST-02-04-01-1H

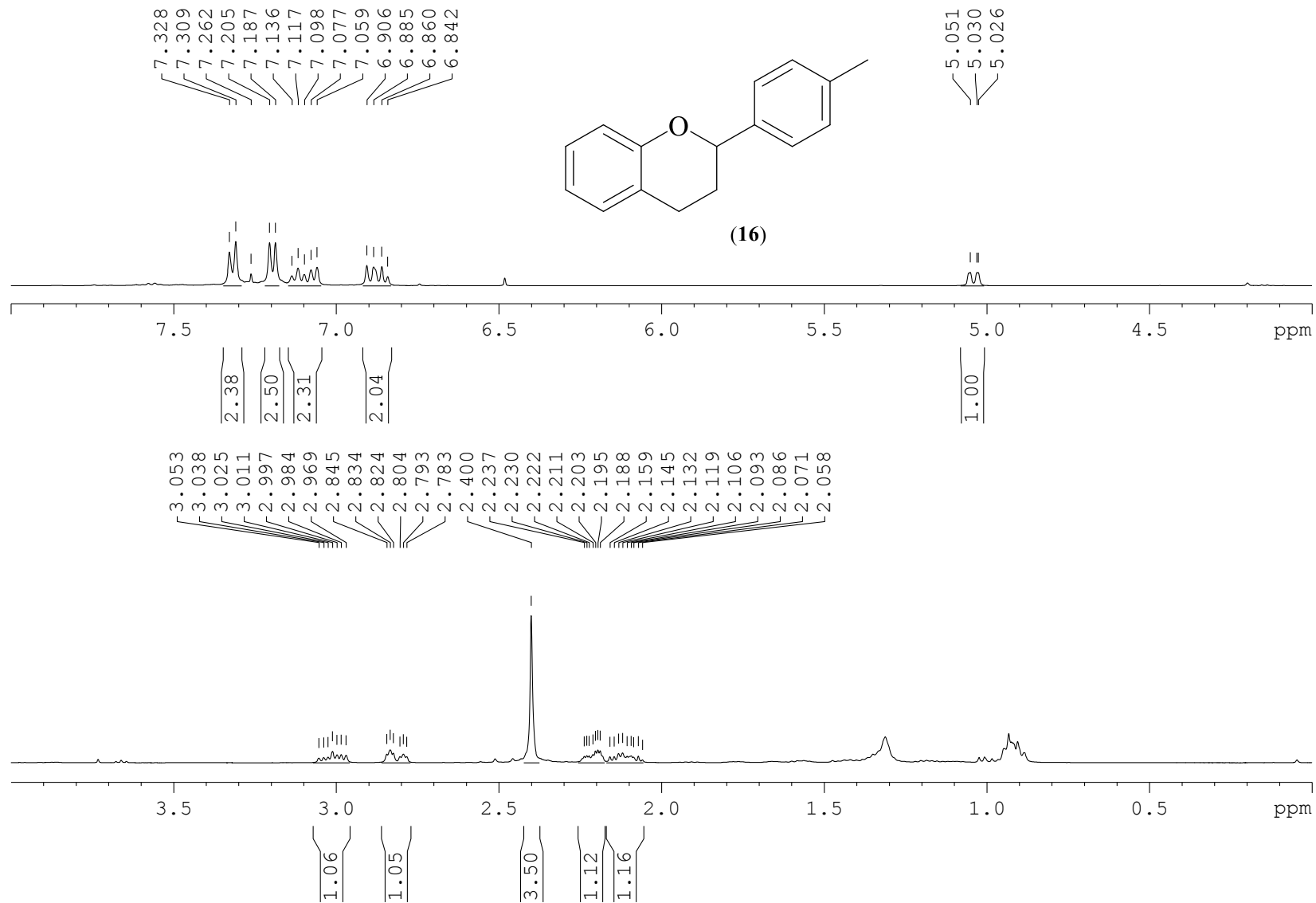


Figure S146. Expanded  $^1\text{H}$  NMR spectrum of (16) in  $\text{CDCl}_3$ .

PG-ST-02-04-01-13C

Current Data Parameters  
NAME PG-ST-02-04-03-13C  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20210323  
Time 7.07  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 250  
DS 0  
SWH 26041.666 Hz  
FIDRES 0.397364 Hz  
AQ 1.2582912 sec  
RG 1030  
DW 19.200 usec  
DE 6.50 usec  
TE 295.4 K  
D1 1.00000000 sec  
D11 0.03000000 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 8.50 usec  
PL1 -2.00 dB  
PL1W 56.53121948 W  
SFO1 100.6238364 MHz

===== CHANNEL f2 =====  
CPDPRG[2] waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -1.00 dB  
PL12 13.69 dB  
PL13 14.50 dB  
PL2W 10.56200695 W  
PL12W 0.35871249 W  
PL13W 0.29767781 W  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127690 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

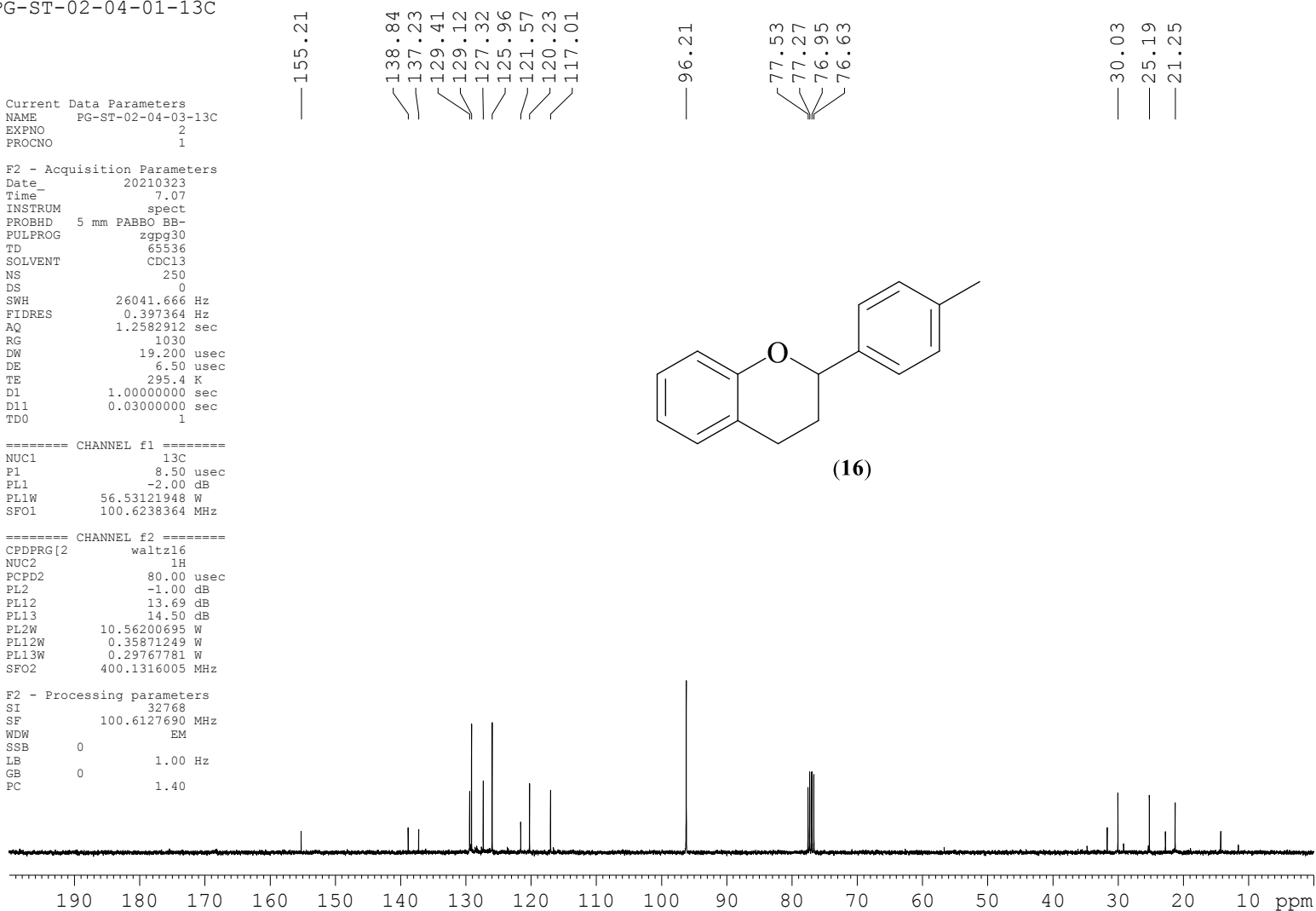
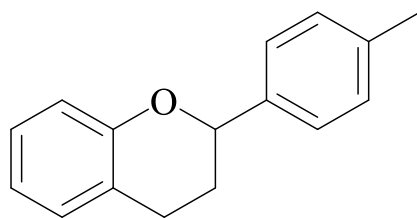
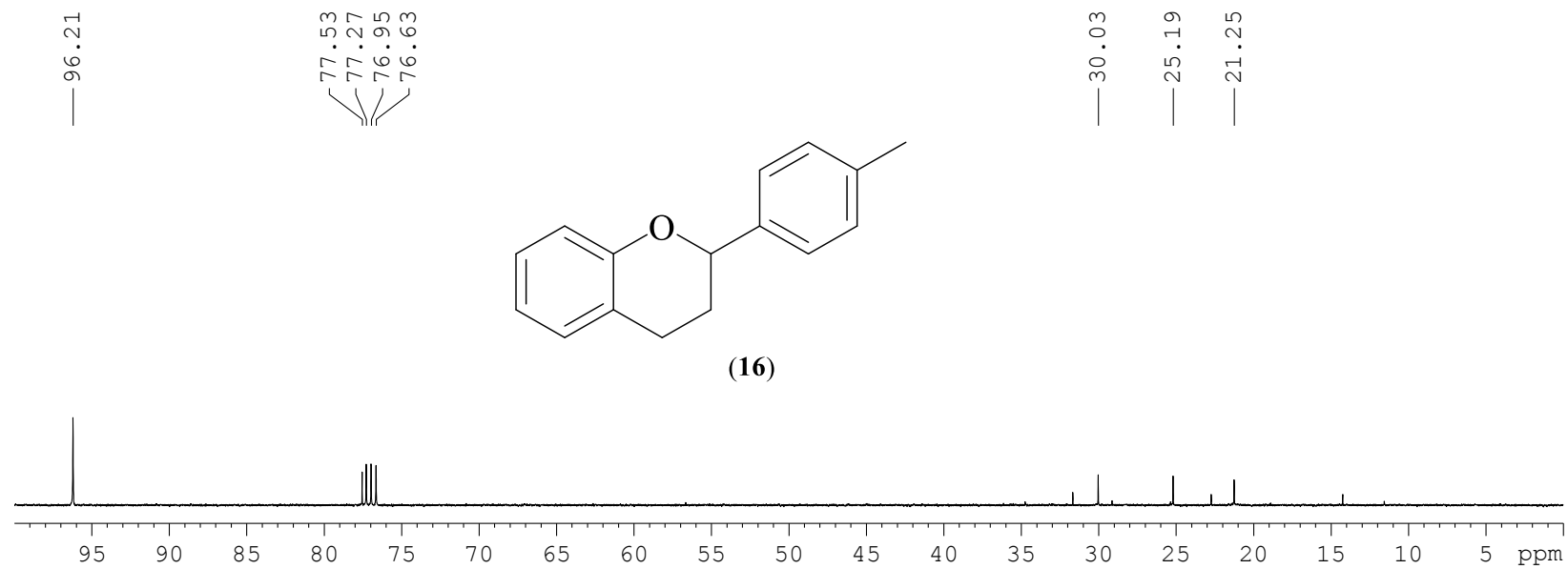
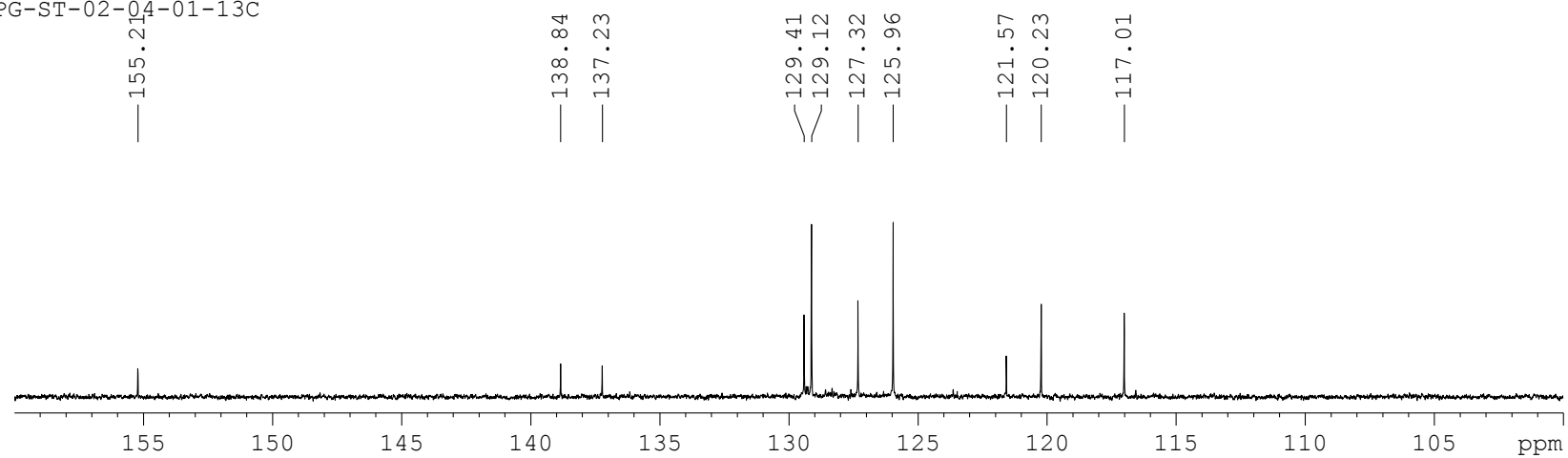


Figure S147.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (16) in  $\text{CDCl}_3$ .

PG-ST-02-04-01-13C



(16)

Figure S148. Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (16) in  $\text{CDCl}_3$ .

File :F:\GCMS-DATA-2020\JAN-2021\PG-ST-02-04-03.D  
Operator : AS  
Acquired : 23 Mar 2021 18:44 using AcqMethod COMMONMETHOD-2020.M  
Instrument : GCMS  
Sample Name: PG-ST-02-04-03  
Misc Info :  
Vial Number: 2

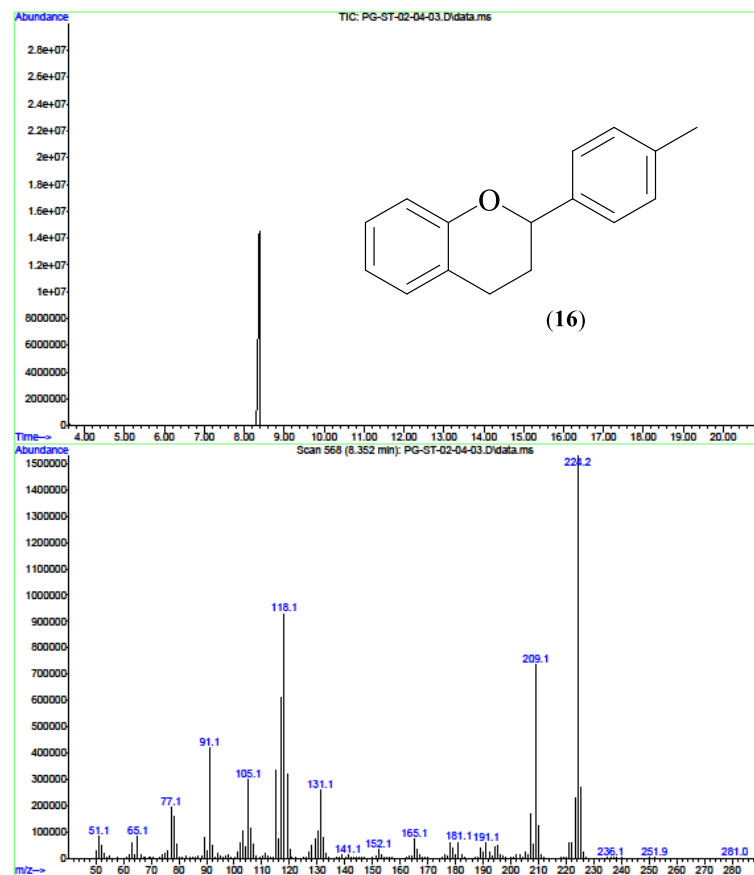
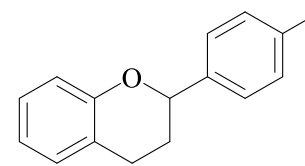
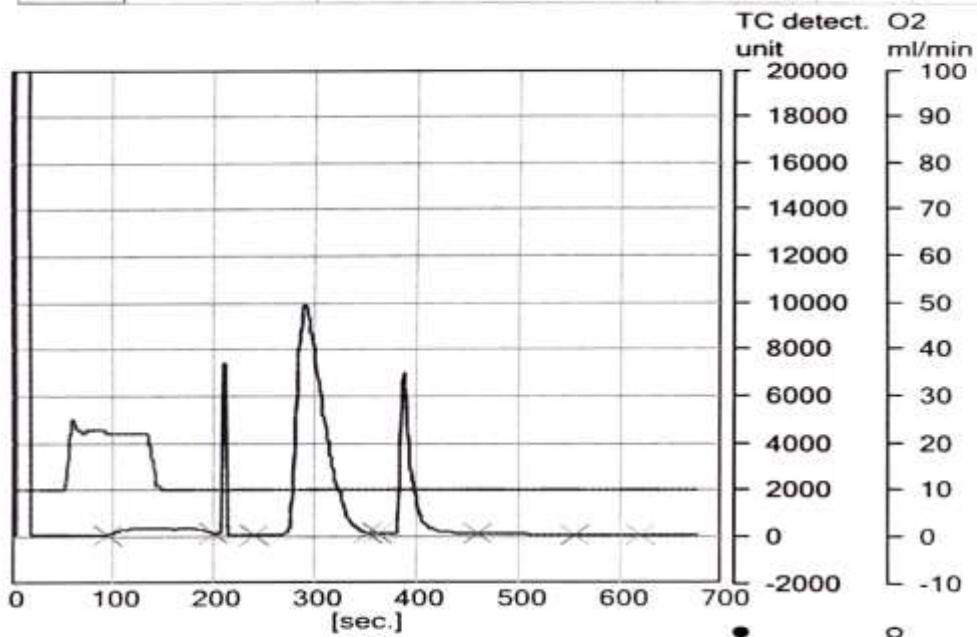


Figure S149. GCMS trace in EtOAc of (16) showing the  $M^+$  peak at  $m/z$  224.

| No. | Weight [mg] | Name             | Method     | N Area | C Area | H Area | N [%] | C [%] | H [%] | Date       | Time  |
|-----|-------------|------------------|------------|--------|--------|--------|-------|-------|-------|------------|-------|
| 38  | 1.2100      | PG-ST-02-04-01-2 | 2mgChem80s | 2 150  | 29 310 | 8 439  | 0.00  | 85.41 | 6.353 | 01-04-2021 | 18:58 |



(16)

Name: eassuperuser, Access: VarioMICRO administrator

02-04-2021 09:56:40

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Figure S150. Elemental analysis data (16).

PG-ST-02-05-06

Current Data Parameters  
NAME PG-ST-02-05-06  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20210324  
Time\_ 8.10  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 54274  
SOLVENT CDC13  
NS 25  
DS 0  
SWH 8223.685 Hz  
FIDRES 0.151522 Hz  
AQ 3.2998593 sec  
RG 80.6  
DW 60.800 usec  
DE 6.50 usec  
TE 295.8 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 14.75 usec  
PL1 -1.00 dB  
PL1W 10.56200695 W  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300095 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

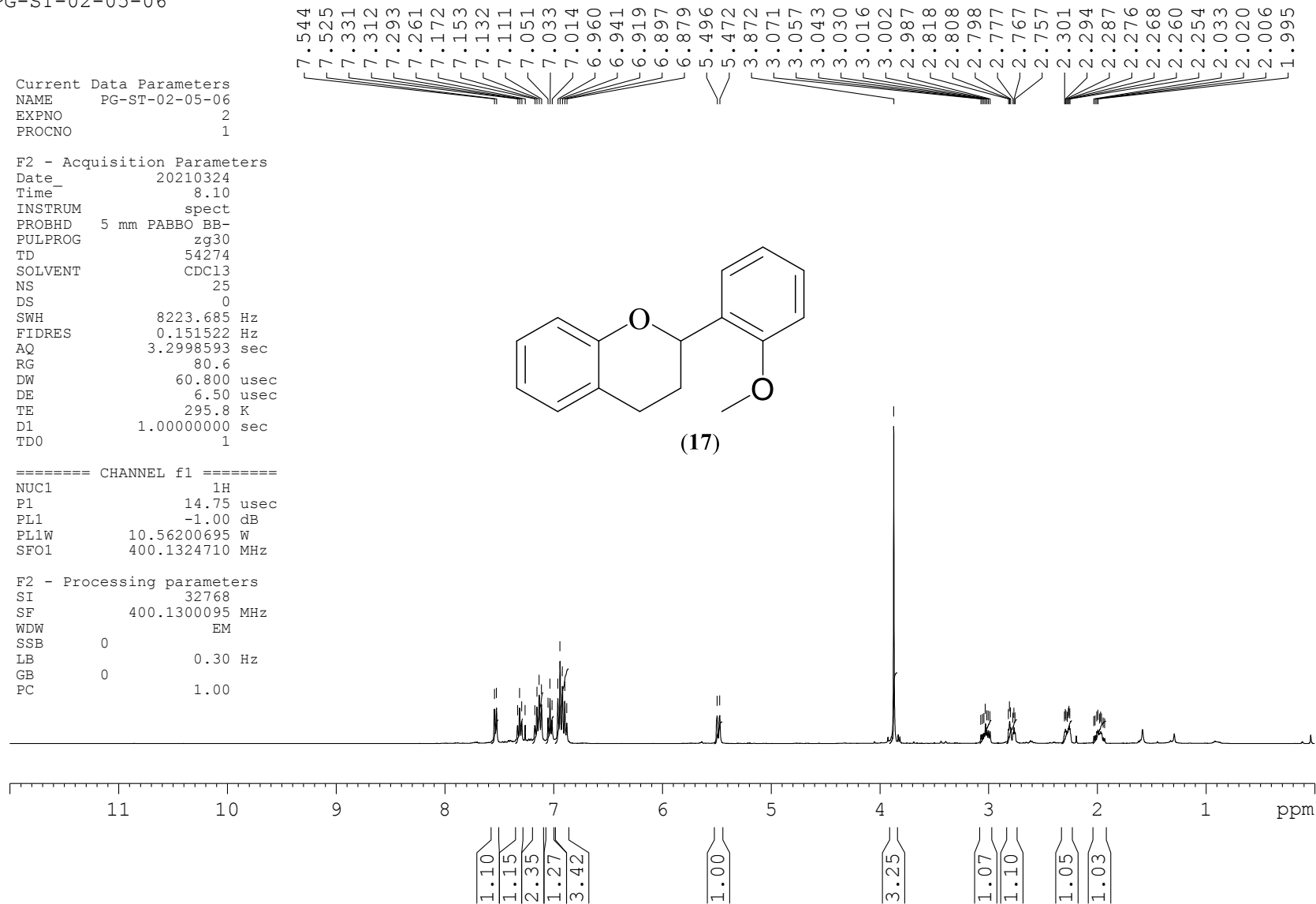


Figure S151. <sup>1</sup>H NMR spectrum of (17) in CDCl<sub>3</sub>.



PG-ST-02-05-06

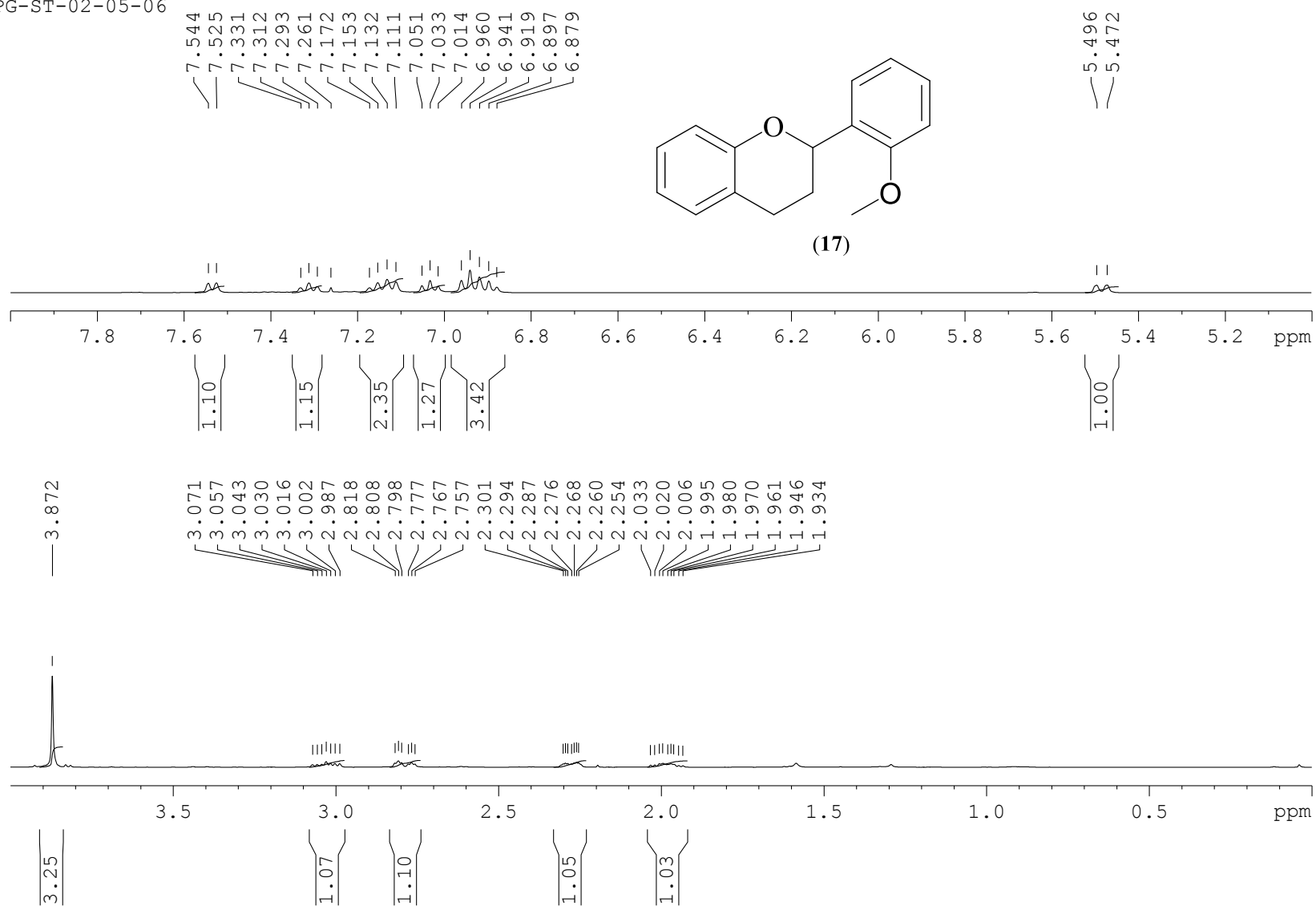


Figure S152. Expanded  $^1\text{H}$  NMR spectrum of (17) in  $\text{CDCl}_3$ .

PG-ST-02-05-06-13C

Current Data Parameters  
NAME PG-ST-02-05-06-13C  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20210324  
Time 8.20  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 354  
DS 0  
SWH 26041.666 Hz  
FIDRES 0.397364 Hz  
AQ 1.2582912 sec  
RG 1820  
DW 19.200 usec  
DE 6.50 usec  
TE 295.9 K  
D1 1.00000000 sec  
D11 0.03000000 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 8.50 usec  
PL1 -2.00 dB  
PL1W 56.53121948 W  
SFO1 100.6238364 MHz

===== CHANNEL f2 =====  
CPDPRG[2] waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 -1.00 dB  
PL12 13.69 dB  
PL13 14.50 dB  
PL2W 10.56200695 W  
PL12W 0.35871249 W  
PL13W 0.29767781 W  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127690 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

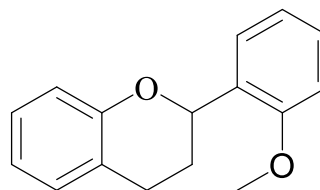
155.90  
155.53

130.28  
129.55  
128.53  
127.23  
126.47  
122.25  
120.80  
120.10  
116.90  
110.38

77.39  
77.07  
76.75  
72.40

55.42

28.58  
25.26



(17)

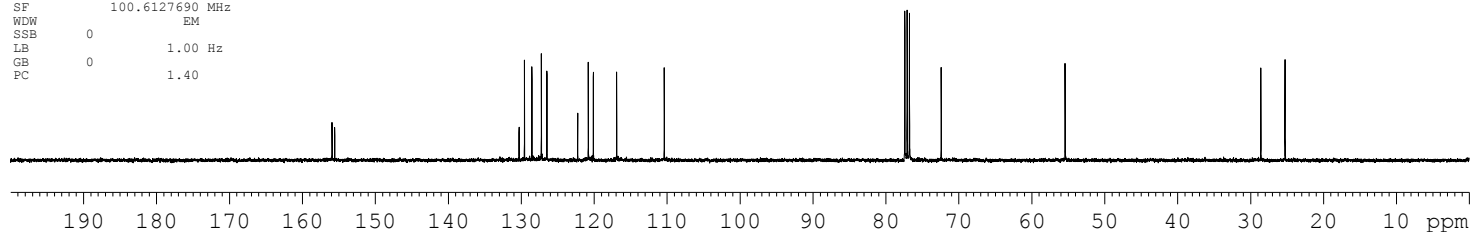
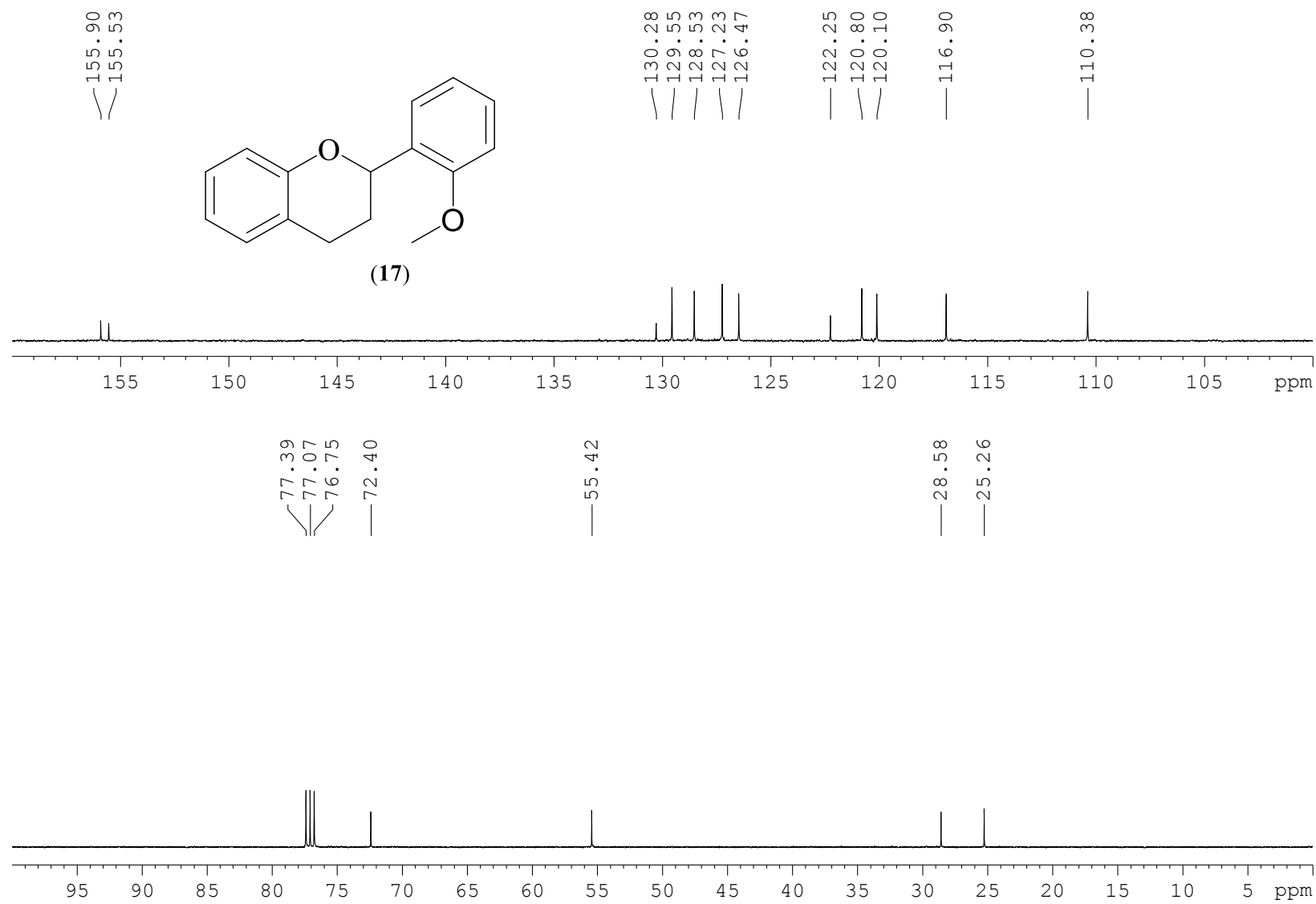


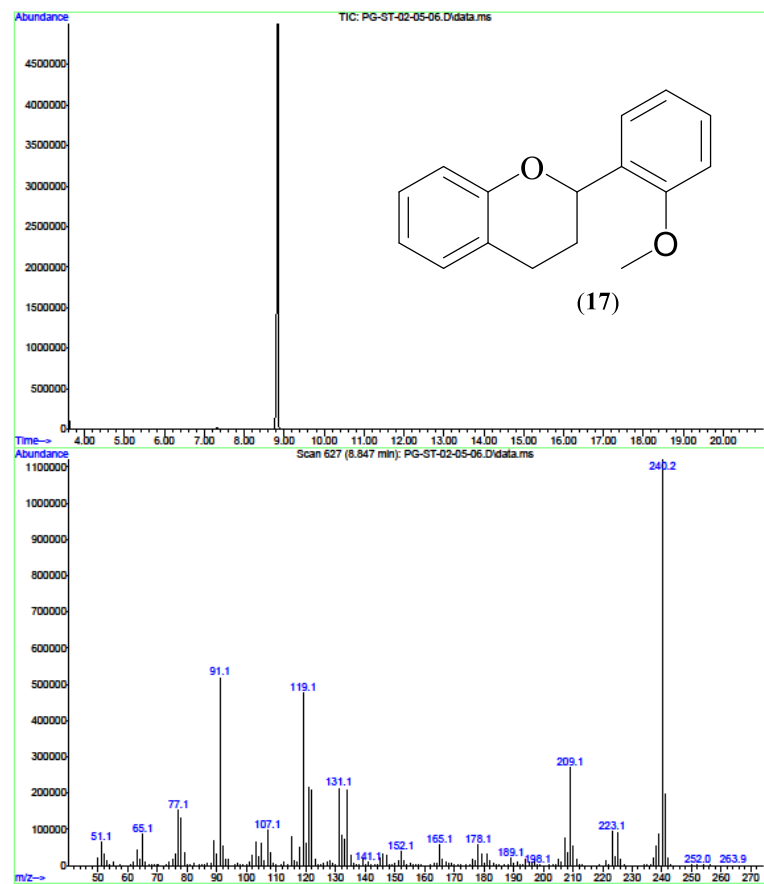
Figure S153.  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (17) in  $\text{CDCl}_3$ .

PG-ST-02-05-06-13C



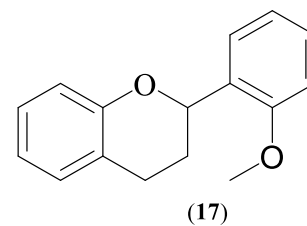
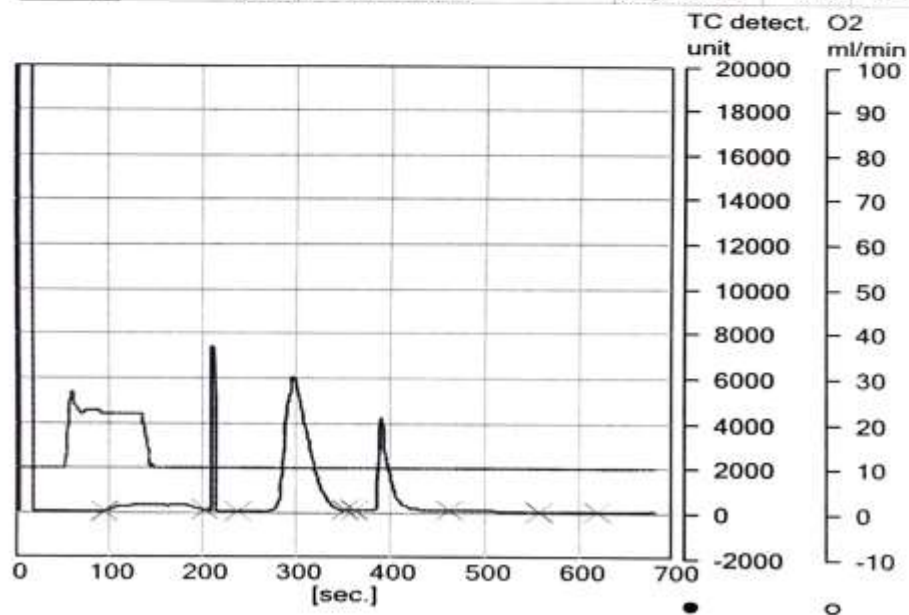
**Figure S154.** Expanded  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of (17) in  $\text{CDCl}_3$ .

File :F:\GCMS-DATA-2021\MARCH 2021\PG-ST-02-05-06.D  
Operator : RM  
Acquired : 24 Mar 2021 21:15 using AcqMethod COMMONMETHOD-2020.M  
Instrument : GCMS  
Sample Name : PG-ST-02-05-06  
Misc Info :  
Vial Number : 7



**Figure S155.** GCMS trace in EtOAc of (17) showing the  $M^+$  peak at  $m/z$  240.

| No. | Weight [mg] | Name             | Method     | N Area | C Area | H Area | N [%] | C [%] | H [%] | Date       | Time  |
|-----|-------------|------------------|------------|--------|--------|--------|-------|-------|-------|------------|-------|
| 40  | 0.7340      | PG-ST-02-05-06-2 | 2mgChem80s | 2 155  | 16 456 | 5 132  | 0.00  | 79.31 | 6.470 | 01-04-2021 | 19:21 |



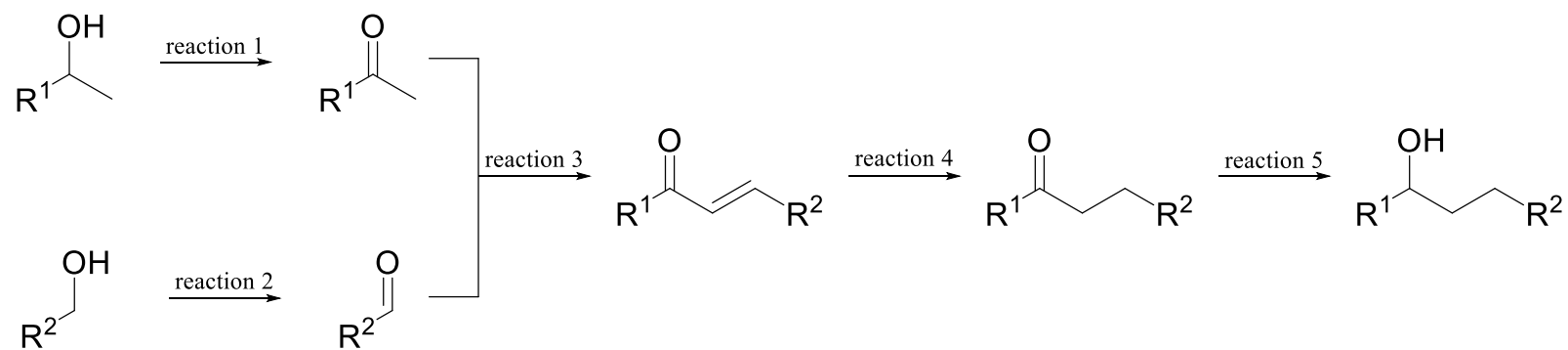
Name: eassuperuser, Access: VarioMICRO administrator

02-04-2021 09:57:19

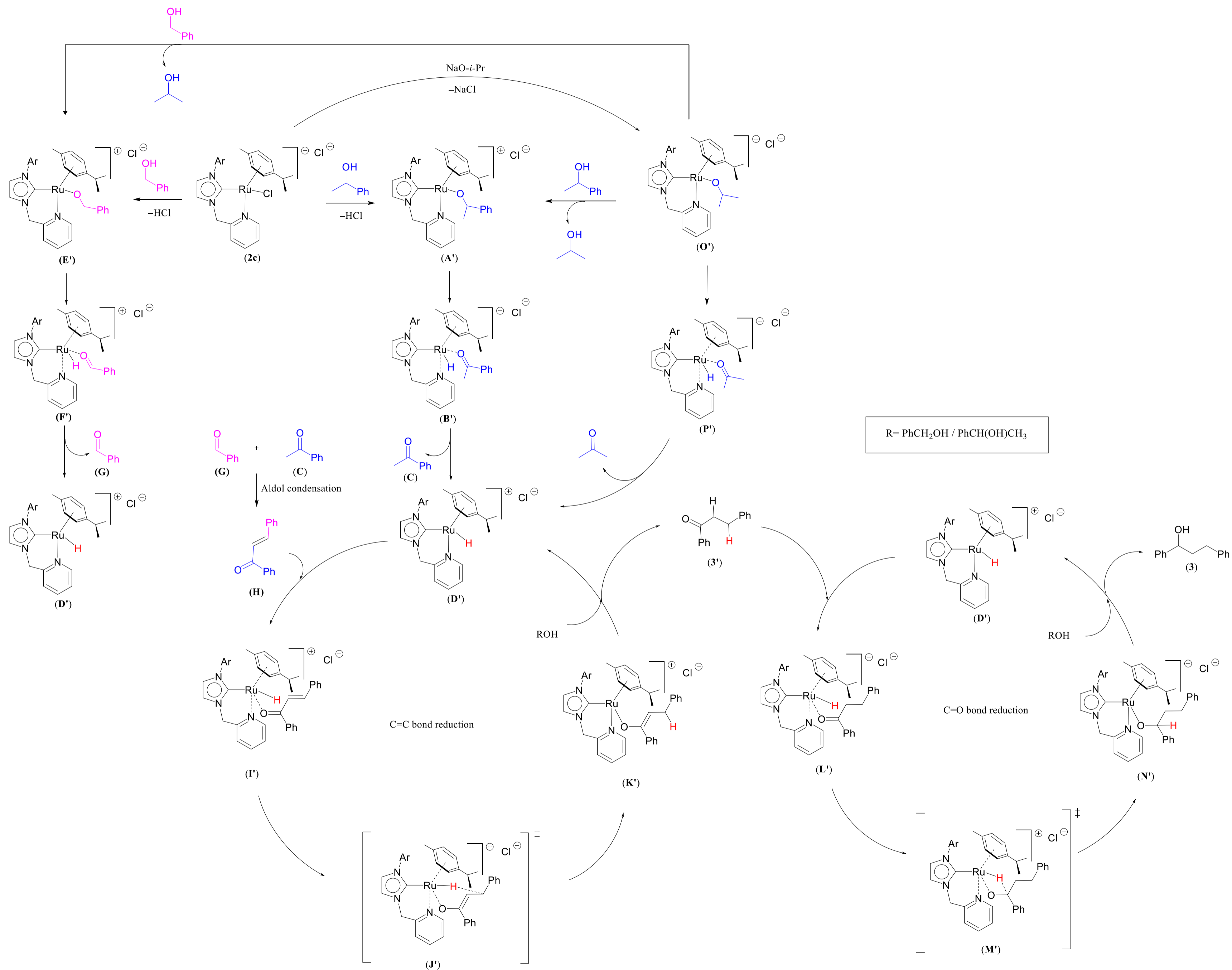
varioMICRO V4.0.1 (aeb1e0e)2015-10-12, CHNS Mode, Ser. No.: 15154051  
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Figure S156. Elemental analysis data (17).



**Scheme S1.** One pot tandem  $\beta$ -alkylation reaction of secondary alcohol involving five sequential reactions.



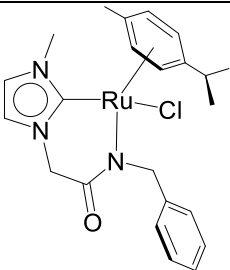
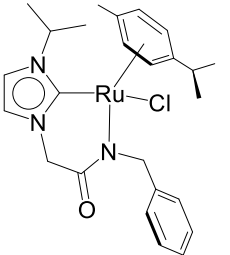
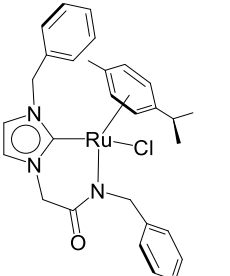
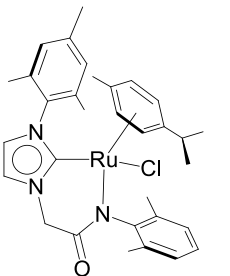
**Scheme S2.** Proposed mechanism for the Ru-NHC (**2c**) catalyzed one pot tandem  $\beta$ -alkylation reaction for representative substrates namely 1-phenylethanol and benzyl alcohol.

**Table S1.** X-ray crystallographic data for Ag–NHC complex (**2b**), Ru–NHC complexes (**1c**), (**1c'**) and **2c**.

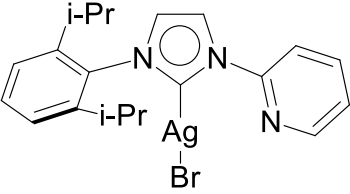
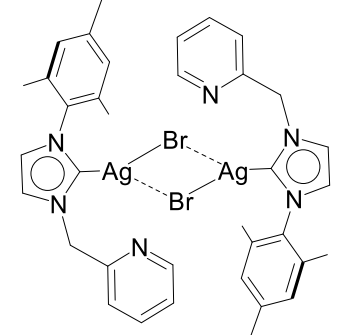
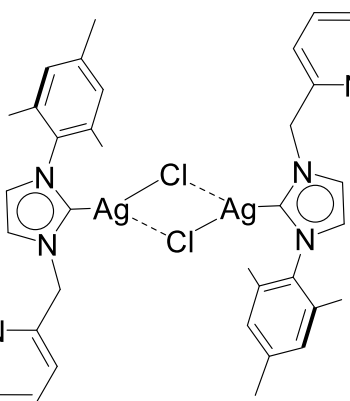
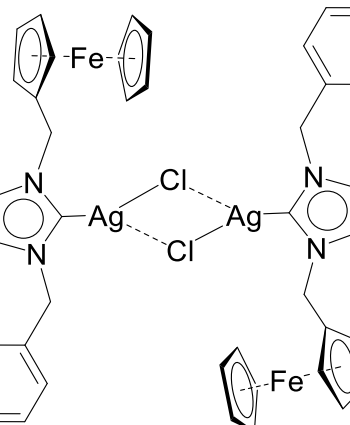
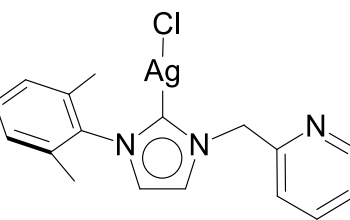
| compound                      | ( <b>1c</b> )  | ( <b>1c'</b> )                                       | ( <b>2b</b> )                                       | ( <b>2c</b> )   |
|-------------------------------|--|--|---|---|
| Lattice                       | Monoclinic   | Triclinic  | Monoclinic  | Triclinic   |
| Formula                       | C <sub>32</sub> H <sub>38</sub> N <sub>3</sub> ORuCl | C <sub>32</sub> H <sub>38</sub> N <sub>3</sub> ORuCl | C <sub>17</sub> H <sub>17</sub> N <sub>3</sub> AgCl | C <sub>27</sub> H <sub>31</sub> N <sub>3</sub> RuCl <sub>2</sub> •2H <sub>2</sub> O |
| Formula weight                | 617.17   | 617.17   | 406.65  | 605.55  |
| Space group                   | P121/n1  | P-1  | P121/n1   | P-1   |
| a/Å                           | 9.0361(6)  | 8.0275(3)  | 9.8162(2)   | 8.2367(3)   |
| b/Å                           | 30.505(2)  | 13.1953(5)   | 16.0104(3)  | 9.1208(3)   |
| c/Å                           | 10.5031(6)   | 15.2588(6)   | 10.8039(2)  | 17.8719(5)  |
| α/°                           | 90.000   | 103.407(3)   | 90.000  | 93.836(2)   |
| β/°                           | 93.623(5)  | 104.358(3)   | 90.098(2)   | 93.825(2)   |
| γ/°                           | 90.000   | 102.686(3)   | 90.000  | 97.623(3)   |
| V/Å <sup>3</sup>              | 2889.4(3)  | 1455.78(10)  | 1697.95(6)  | 1323.90(8)  |
| Z                             | 4  | 2  | 4   | 2   |
| Temperature (K)               | 150(2)   | 150(2)   | 150(2)  | 150(2)  |
| Radiation (λ, Å)              | 0.71073  | 0.71073  | 0.71073   | 0.71073   |
| ρ(calcd.), g cm <sup>-3</sup> | 1.419  | 1.408  | 1.591   | 1.519   |
| μ(Mo Kα), mm <sup>-1</sup>    | 0.665  | 0.660  | 1.343   | 0.824   |
| θ max, deg.                   | 25.00  | 25.00  | 31.101  | 24.999  |
| No. Of data                   | 3630   | 4556   | 4147  | 3922  |
| No. Of parameters             | 351  | 351  | 201   | 327   |
| R <sub>1</sub>                | 0.0617   | 0.0554   | 0.0268  | 0.0450  |
| wR <sub>2</sub>               | 0.1500   | 0.1422   | 0.0634  | 0.1094  |
| GOF                           | 1.061  | 1.068  | 1.054   | 1.054   |



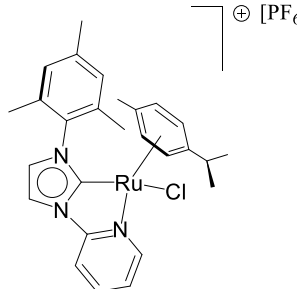
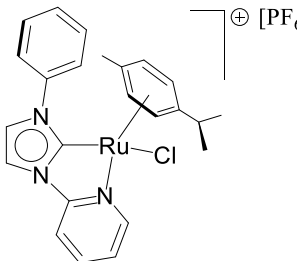
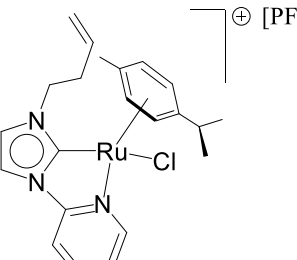
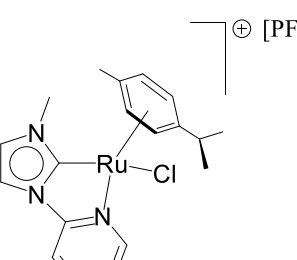
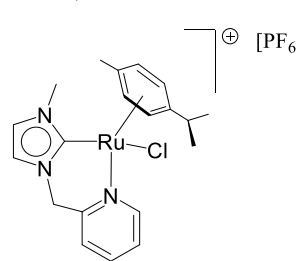
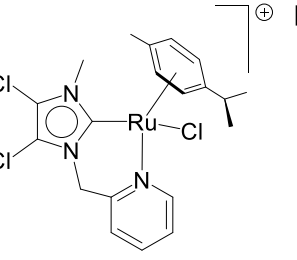
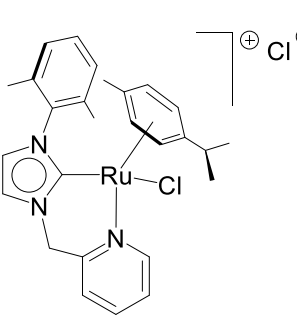
**Table S2.** A comparison of the metrical data of the neutral (amido-N functionalized NHC)Ru(*p*-cymene)Cl type complexes known in the literature is shown.

| S. No. | complex   | $d(\text{Ru}-\text{C}_{\text{carbene}})/(\text{\AA})$ | $d(\text{Ru}-\text{N})/(\text{\AA})$ | $d(\text{Ru}-\text{Cl})/(\text{\AA})$ | $d(\text{Ru}-\text{C}_{\text{centroid}})/(\text{\AA})$ | reference    |
|--------|---|---|--------------------------------------|---------------------------------------|--|--------------|
| 1.     |            | 2.0172(19)  | 2.1074(16)                           | 2.4404(7)                             | 1.706  | [6]          |
| 2.     |           | 2.033(5)  | 2.125(5)                             | 2.4256(14)                            | 1.712  | [6]          |
| 3.     |          | 2.019(3)  | 2.1074(16)                           | 2.4325(8)                             | 1.719  | [6]          |
| 4.     | <br>(1e) | 2.087(5)  | 2.153(4)                             | 2.4299(14)                            | 1.736  | present work |

**Table S3.** A comparison of the metrical data of the representative structurally characterized examples of neutral (unsubstituted picolyl functionalized NHC)AgCl and neutral (unsubstituted pyridyl functionalized NHC)AgCl type complexes known in the literature is shown.

| S. No. | complex   | $d(\text{Ag}-\text{C}_{\text{carbene}})$ / (Å) | $d(\text{Ag}-\text{X})$ (X=Br, Cl)/ (Å) | reference    |
|--------|---|--|---|--------------|
| 1.     |            | 2.075(7)                                       | 2.421(1)                                | [7]          |
| 2.     |           | 2.07(2)  | 2.373(4) and 2.952(4)                   | [7]          |
| 3.     |          | 2.0762(19)                                     | 2.0763(19) and 2.9916(6)                | [7]          |
| 4.     |          | 2.097(2)                                       | 2.4119(8) and 2.830(1)                  | [7]          |
| 5.     | <br>(2b) | 2.0813(17)                                     | 2.3684(5)                               | present work |

**Table S4.** A comparison of the metrical data of the representative structurally characterized examples of ionic (picolyl functionalized NHC)Ru(*p*-cymene)Cl type complexes known in the literature is shown.

| S. No. | complex  | $d(\text{Ru}-\text{C}_{\text{carbene}})/(\text{\AA})$ | $d(\text{Ru}-\text{N})/(\text{\AA})$ | $d(\text{Ru}-\text{Cl})/(\text{\AA})$ | $d(\text{Ru}-\text{C}_{\text{centroid}})/(\text{\AA})$ | reference    |
|--------|--|---|--------------------------------------|---------------------------------------|--|--------------|
| 1.     |  $\oplus [\text{PF}_6]$         | 2.029(3)  | 2.098(3)                             | 2.4615(7)                             | 2.7214(15)   | [8]          |
| 2.     |  $\oplus [\text{PF}_6]$        | 2.0145(19)  | 2.1055(16)                           | 2.4397(4)                             | 1.7155(8)  | [8]          |
| 3.     |  $\oplus [\text{PF}_6]$       | 2.033(2)  | 2.100(2)                             | 2.246(2)                              | 1.712  | [9]          |
| 4.     |  $\oplus [\text{PF}_6]$       | 2.009(2)  | 2.092(18)                            | 2.401(6)                              | 1.710(1)   | [10]         |
| 5.     |  $\oplus [\text{PF}_6]$       | 2.035(7)  | 2.095(6)                             | 2.389(2)                              | 1.710(3)   | [11]         |
| 6.     |  $\oplus [\text{PF}_6]$       | 2.023(5)  | 2.111(4)                             | 2.448(1)                              | 1.696(2)   | [11]         |
| 7.     |  $\oplus \text{Cl}^-$<br>(2c) | 2.052(4)  | 2.110(3)                             | 2.3936(9)                             | 1.704  | present work |

**Table S5.** Base variation study for the Ru–NHC (**1c**) catalyzed one pot tandem  $\beta$ -alkylation reaction for two representative substrates namely 1-phenylethanol and benzyl alcohol<sup>a</sup>.

CC(O)c1ccccc1 + OCC1=CC=CC=C1
 $\xrightarrow[\text{toluene, 110 }^\circ\text{C, base, time (h)}]{\text{1 mol \% (1c)}}$ 
CC(O)CC1=CC=CC=C1 + CC(=O)CC1=CC=CC=C1
  
(3) (3')

---

*yield<sup>b</sup>*

| S.No | base                           | time (h) | yield (%) |          |
|------|--------------------------------|----------|-----------|----------|
|      |                                |          | <br>(3)   | <br>(3') |
| 1    | NaOH                           | 6        | 37        | 52       |
| 2    | KO- <i>t</i> -Bu               | 6        | 63        | 17       |
| 3    | Et <sub>3</sub> N              | 6        | ND        | ND       |
| 4    | K <sub>2</sub> CO <sub>3</sub> | 6        | ND        | ND       |
| 5    | KOH                            | 6        | 69        | 19       |
| 6    | KOH                            | 3        | 55        | 18       |
| 7    | NaO <i>i</i> Pr                | 6        | 55        | 40       |
| 8    | NaO <i>i</i> Pr                | 3        | 72        | 11       |

(a). Reaction conditions: 1:1:1 ratio of 1°-alcohol:2°-alcohol:base 1.00 mmol, 1 mol % of (**1c**), 2.0 mL of toluene at 110 °C for time (h). (b). Isolated yields (%).

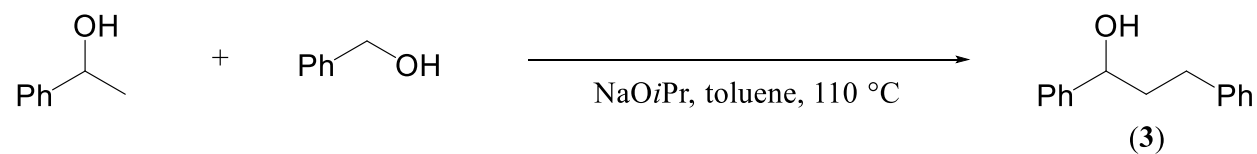
**Table S6.** Time variation study for the Ru–NHC (**1c/2c**) catalyzed one pot tandem  $\beta$ -alkylation reaction for two representative substrates namely 1-phenylethanol and benzyl alcohol<sup>a</sup>.

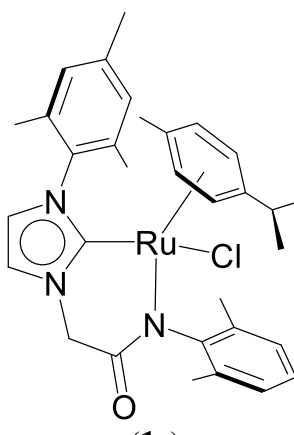
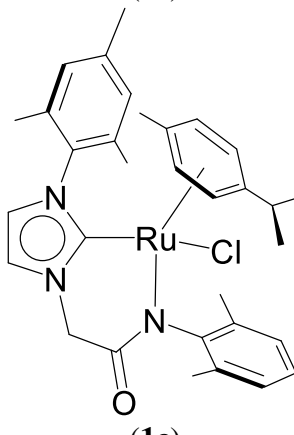
Reaction scheme: 1-phenylethanol + benzyl alcohol  $\xrightarrow[NaO-i-Pr, \text{ toluene, } 110^\circ\text{C, time (h)}]{1 \text{ mol } \% \text{ (1c/2c)}}$  1-phenyl-3-phenylpropan-1-ol (**3**) + 1-phenyl-3-phenylpropan-1-one (**3'**)

|      |             | yield <sup>b</sup> |             |             |             |
|------|-------------|--------------------|-------------|-------------|-------------|
| S.No | time<br>(h) | <b>(3)</b>         |             | <b>(3')</b> |             |
|      |             | <b>(1c)</b>        | <b>(2c)</b> | <b>(1c)</b> | <b>(2c)</b> |
| 1    | 0.5         | 18                 | 12          | 15          | 5           |
| 2    | 1           | 37                 | 27          | 34          | 12          |
| 3    | 2           | 51                 | 35          | 33          | 14          |
| 4    | 3           | 72                 | 68          | 11          | 13          |
| 5    | 4           | 61                 | 46          | 19          | 22          |
| 6    | 6           | 55                 | 38          | 40          | 28          |
| 7    | 12          | 1                  | 6           | 64          | 32          |
| 8    | 24          | 61                 | 51          | 9           | 9           |
| 9    | 48          | 63                 | 50          | 5           | 7           |
| 10   | 72          | 55                 | 32          | 3           | 3           |
| 11   | 96          | 46                 | 28          | > 5         | 3           |
| 12   | 120         | 22                 | 15          | > 5         | 2           |

(a). Reaction conditions: 1:1:1 ratio of 1°-alcohol:2°-alcohol:NaOiPr 1.00 mmol, 1 mol % of (**1c/2c**), 2.0 mL of toluene at 110 °C for T hour. (b) Isolated yields (%).

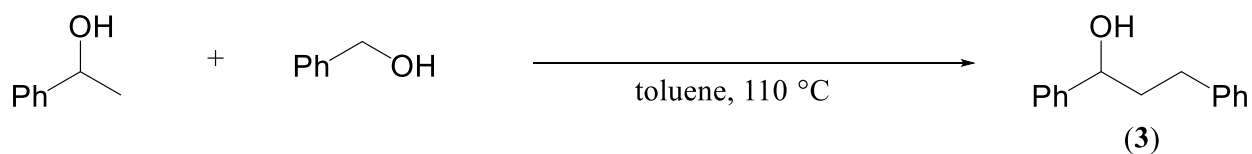
**Table S7.** Selected results of blank, control and Hg drop experiments for the one pot tandem  $\beta$ -alkylation reaction for two representative substrates namely 1-phenylethanol and benzyl alcohol<sup>a</sup>.



| S. No | metal complex  | Hg | yield <sup>b</sup> |
|-------|--|----|--------------------|
| 1     | -  |    | ND                 |
| 2     | [( <i>p</i> -cymene)RuCl <sub>2</sub> ] <sub>2</sub>   |    | 19                 |
| 3     | <br>( <b>1c</b> )  |    | 72                 |
| 4     | <br>( <b>1c</b> ) | Hg | 68                 |

(a). Reaction conditions: 1:1:1 ratio of 1°-alcohol:2°-alcohol:NaOiPr 1.00 mmol, 1 mol % of (**1c**), 2.0 mL of toluene at 110 °C for 3 hours. (b). Isolated yields (%).

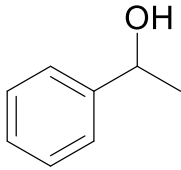
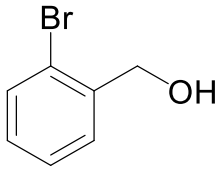
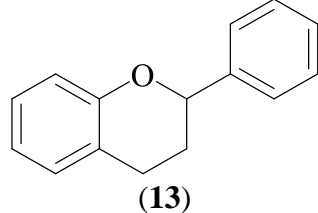
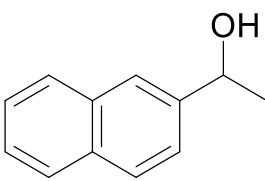
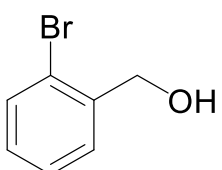
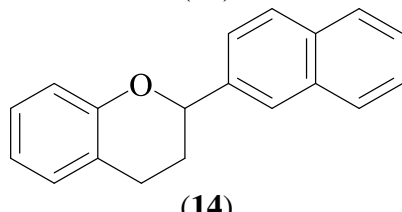
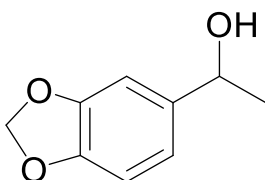
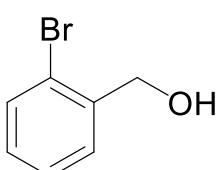
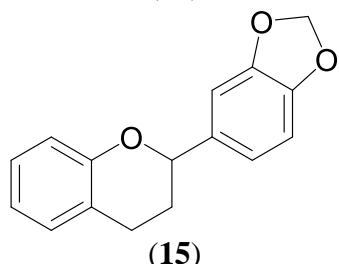
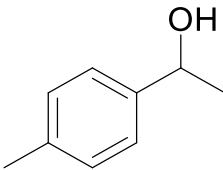
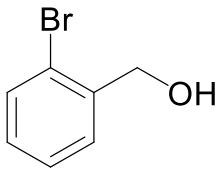
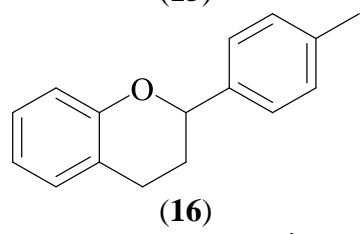
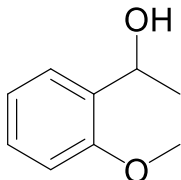
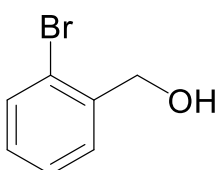
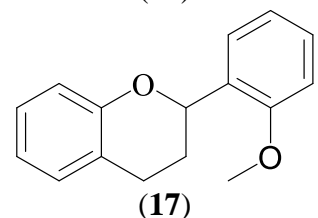
**Table S8.** Selected results of blank and control experiments for the one pot tandem  $\beta$ -alkylation reaction for two representative substrates namely 1-phenylethanol and benzyl alcohol<sup>a</sup>.



| S. No | metal complex | base   | yield <sup>b</sup> |
|-------|---------------|--------|--------------------|
| 1     | <p>(1c)</p>   | –      | ND                 |
| 2     | –             | NaOiPr | ND                 |

(a). Reaction conditions: 1:1:1 ratio of 1°-alcohol:2°-alcohol:NaOiPr 1.00 mmol, 1 mol % of (1c), 2.0 mL of toluene at 110 °C for 3 hours. (b). Isolated yields (%).

**Table S9.** Selected results for the Ru–NHC (**1–2**)c catalyzed one pot synthesis of flavan derivatives (**13–17**).

| S. No. | 2° alcohol  | 1° alcohol  | product  | Ru–NHC ( <b>1c</b> ) | Ru–NHC ( <b>2c</b> ) |
|--------|---|---|--|----------------------|----------------------|
|        |   |   |  | yield <sup>b</sup>   | yield <sup>b</sup>   |
| 13     |    |    | <br><b>(13)</b>   | 17                   | 10                   |
| 14     |    |    | <br><b>(14)</b>   | 36                   | 28                   |
| 15     |   |   | <br><b>(15)</b>  | 18                   | 15                   |
| 16     |  |  | <br><b>(16)</b> | 12                   | 11                   |
| 17     |  |  | <br><b>(17)</b> | 14                   | 13                   |

Reaction conditions: (a). Reaction conditions: 1:1:1 ratio of 1°-alcohol:2°-alcohol:base 1.00 mmol, 1 mol % of (**1c/2c**), 2.0 mL of toluene at 110 °C for 3 hours. (a). Reaction conditions: 20 mol % CuI, 20 mol % 2,2'-bipyridine, base 1.00 mmol, 1.0 mL of toluene at 110 °C for 24 hours. (c). Isolated yields (%).



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