

## Electronic Supplementary Information

# Solvent and Catalyst-Free Bromofunctionalization of Olefins Using a Mechanochemical Approach

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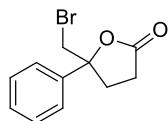
## (A) General Information

Unless otherwise specified, commercially available reagents were used directly without further purification. All reactions requiring anhydrous conditions were conducted by standard procedures under nitrogen atmosphere. The solvents were dried over a solvent purification system from Innovative Technology. Melting points were determined on a Buchi B-540b melting point apparatus.  $^1\text{H}$  NMR and  $^{13}\text{C}\{^1\text{H}\}$  NMR spectra were recorded on a Bruker AMX500 (500 MHz) spectrometer or a Bruker AMX400 (400 MHz) spectrometer. Proton and carbon chemical shifts are reported in parts per million (ppm) values downfield from TMS ( $\delta$  0.00) and referenced to residual protons in NMR solvents ( $\text{CDCl}_3$  at  $\delta$  7.26,  $\text{CD}_2\text{Cl}_2$  at  $\delta$  5.36) or carbon signals in NMR solvent ( $\text{CDCl}_3$  at  $\delta$  77.16,  $\text{CD}_2\text{Cl}_2$  at  $\delta$  55.42).  $^1\text{H}$  NMR data were reported as follows: chemical shift, multiplicity, coupling constants (Hz), and integration. High resolution mass spectra were obtained on a Thermo Finnigan MAT95XL Magnetic Sector mass spectrometer (ionization mode: EI) or a Thermo Q Exactive Hybrid Quadrupole-Orbitrap mass spectrometer (ESI). Analytical thin layer chromatography (TLC) was performed with Merck precoated TLC plates, silica gel 60F-254, layer thickness 0.25 mm. Flash chromatography separations were performed on Merck 60 (0.040–0.063 mm) mesh silica gel.

## (B) Mechanochemical Bromolactonization Reactions

**General Procedure.** Alkenoic acid **1** (0.2 mmol) and NBS (39.2 mg, 0.22 mmol) were added into a 10 mL zirconium oxide chamber. The milling chamber was then securely fastened into a Retsch mixer mill (MM 400) and set to oscillate at 30 Hz for 1 hour. On completion of reaction, the mill chamber was extracted with hexane or cyclohexane and the solution was filtered through a thin plug of celite. The filtrate was concentrated under reduced pressure to give the desired product **2** with high purity. No column chromatography was necessary.

### 5-(bromomethyl)-5-phenyldihydrofuran-2(3H)-one (**2a**)



Yield: 97% (49.5 mg, colorless oil)

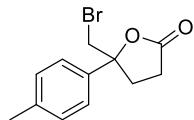
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.41 (d,  $J = 4.3$  Hz, 4H), 7.33-7.39 (m, 1H), 3.74 (d,  $J = 11.4$  Hz, 1H), 3.69 (d,  $J = 11.4$ , 1H), 2.77-2.86 (m, 2H), 2.49-2.61 (m, 2H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  175.7, 140.8, 129.0, 128.8, 125.0, 86.6, 41.2, 32.5, 29.2.

HRMS (ESI-Q-orbitrap) m/z:  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{11}\text{H}_{11}\text{BrO}_2$  276.98346, found 276.98365.

The data are in full accordance with the literature (Chen, T.; Foo, T. J. Y.; Yeung, Y.-Y. *ACS Catal.* **2015**, 5 (8), 4751–4755).

### 5-(bromomethyl)-5-(p-tolyl)dihydrofuran-2(3H)-one (**2b**)



Yield: 98% (52.7 mg, colorless oil)

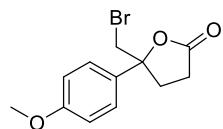
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.29 (d,  $J = 7.5$  Hz, 2H), 7.21 (d,  $J = 7.5$  Hz, 2H), 3.73 (d,  $J = 11.1$  Hz, 1H), 3.67 (d,  $J = 11.1$ , 1H), 2.74-2.84 (m, 2H), 2.48-2.58 (m, 2H), 2.36 (s, 3H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  175.8, 138.7, 137.8, 129.6, 125.0, 86.6, 41.2, 32.4, 29.2, 21.2.

HRMS (ESI-Q-orbitrap) m/z:  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{12}\text{H}_{13}\text{BrO}_2$  290.99911, found 290.99917.

The data are in full accordance with the literature (Chen, T.; Foo, T. J. Y.; Yeung, Y.-Y. *ACS Catal.* **2015**, 5 (8), 4751–4755).

**5-(bromomethyl)-5-(4-methoxyphenyl)dihydrofuran-2(3H)-one (**2c**)**



Yield: 87% (49.6 mg, colorless oil)

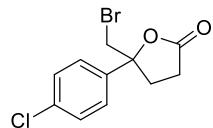
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.33 (d,  $J = 8.9$  Hz, 2H), 6.91 (d,  $J = 8.9$  Hz, 2H), 3.81 (s, 3H), 3.71 (d,  $J = 11.3$  Hz, 1H), 3.65 (d,  $J = 11.3$  Hz, 1H), 2.75–2.83 (m, 2H), 2.50–2.58 (m, 2H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  175.8, 159.8, 132.6, 126.4, 114.3, 86.5, 55.5, 41.3, 32.3, 29.3.

HRMS (ESI-Q-orbitrap) m/z:  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{12}\text{H}_{13}\text{BrO}_3$  306.99403, found 306.99378.

The data are in full accordance with the literature (Zhou, L.; Tan, C. K.; Jiang, X.; Chen, F.; Yeung, Y.-Y. *J. Am. Chem. Soc.* **2010**, 132 (44), 15474–15476.).

**5-(bromomethyl)-5-(4-chlorophenyl)dihydrofuran-2(3H)-one (**2d**)**



Yield: 98% (56.7 mg, colorless oil)

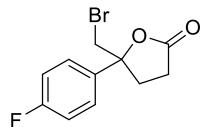
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.39 (d,  $J = 8.6$  Hz, 2H), 7.35 (d,  $J = 8.6$  Hz, 2H), 3.70 (d,  $J = 11.3$  Hz, 1H), 3.65 (d,  $J = 11.3$  Hz, 1H), 2.75–2.86 (m, 2H), 2.48–2.59 (m, 2H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  175.3, 139.4, 134.9, 129.2, 126.6, 86.1, 40.7, 32.5, 29.1.

HRMS (ESI-Q-orbitrap) m/z:  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{11}\text{H}_{10}\text{BrClO}_2$  312.94236, found 312.94231.

The data are in full accordance with the literature (Chen, T.; Foo, T. J. Y.; Yeung, Y.-Y. *ACS Catal.* **2015**, 5 (8), 4751–4755).

**5-(bromomethyl)-5-(4-fluorophenyl)dihydrofuran-2(3H)-one (**2e**)**



Yield: 90% (49.2 mg, colorless oil)

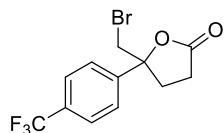
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.40 (d,  $J = 8.7$  Hz, 1H), 7.39 (d,  $J = 8.7$  Hz, 1H), 7.10 (t,  $J = 8.7$  Hz, 2H), 3.70 (d,  $J = 11.3$  Hz, 1H), 3.65 (d,  $J = 11.3$  Hz, 1H), 2.76–2.87 (m, 2H), 2.49–2.60 (m, 2H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  175.4, 163.8, 161.8, 136.7, 136.6, 127.1, 127.0, 116.0, 115.9, 86.2, 41.0, 32.5, 29.2.

HRMS (ESI-Q-orbitrap) m/z:  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{11}\text{H}_{10}\text{BrFO}_2$  294.97404, found 294.97435.

The data are in full accordance with the literature (Zhou, L.; Tan, C. K.; Jiang, X.; Chen, F.; Yeung, Y.-Y. *J. Am. Chem. Soc.* **2010**, 132 (44), 15474–15476.).

**5-(bromomethyl)-5-(4-(trifluoromethyl)phenyl)dihydrofuran-2(3H)-one (**2f**)**



Yield: 98% (63.3 mg, colorless oil)

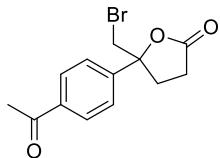
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.67 (d,  $J = 8.2$  Hz, 2H), 7.55 (d,  $J = 8.2$  Hz, 2H), 3.72 (d,  $J = 11.5$  Hz, 1H), 3.70 (d,  $J = 11.5$  Hz, 1H), 2.78–2.88 (m, 2H), 2.50–2.60 (m, 2H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  175.1, 144.8, 131.0 (q,  $J = 33$  Hz), 126.0 (q,  $J = 4$  Hz), 125.6, 123.9 (q,  $J = 272$  Hz), 86.0, 40.4, 32.6, 29.0.

HRMS (ESI-Q-orbitrap) m/z:  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{12}\text{H}_{10}\text{BrF}_3\text{O}_2$  344.97085, found 344.97095.

The data are in full accordance with the literature (Zhou, L.; Tan, C. K.; Jiang, X.; Chen, F.; Yeung, Y.-Y. *J. Am. Chem. Soc.* **2010**, 132 (44), 15474–15476.).

**5-(4-acetylphenyl)-5-(bromomethyl)dihydrofuran-2(3H)-one (**2g**)**



Yield: 81% (48.1 mg, white solid).

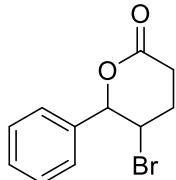
IR (KBr): 3020, 1785, 1737, 1685, 1270, 1216, 1162, 760, 668 cm<sup>-1</sup>.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 8.00 (d, *J* = 8.6 Hz, 2H), 7.52 (d, *J* = 8.6 Hz, 2H), 3.73 (d, *J* = 11.4 Hz, 1H), 3.70 (d, *J* = 11.4 Hz, 1H), 2.76-2.89 (m, 2H), 2.62 (s, 3H), 2.50-2.60 (m, 2H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 197.4, 175.2, 145.8, 137.4, 129.0, 125.4, 86.3, 40.5, 32.6, 29.1, 26.8.

HRMS (ESI-Q-orbitrap) m/z: [M + Na]<sup>+</sup> calcd for C<sub>13</sub>H<sub>13</sub>BrO<sub>3</sub> 318.99403, found 318.99413.

**5-bromo-6-phenyltetrahydro-2*H*-pyran-2-one **2h****



Yield: 78%, d.r. > 99:1 (39.7 mg, white solid)

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.36-7.43 (m, 3H), 7.32 (d, *J* = 6.9 Hz, 2H), 5.56 (d, *J* = 6.4 Hz, 1H), 4.39 (dd, *J* = 11.0, 6.4 Hz, 1H), 2.95 (dt, *J* = 18.2, 8.2 Hz, 1H), 2.72 (dt, *J* = 18.2, 6.2 Hz, 1H), 2.38-2.46 (m, 1H), 2.23-2.31 (m, 1H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 169.2, 137.4, 129.2, 128.9, 126.5, 85.7, 47.3, 28.5, 27.7.

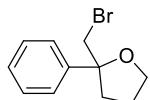
HRMS (ESI-Q-orbitrap) m/z: [M + Na]<sup>+</sup> calcd for C<sub>11</sub>H<sub>11</sub>BrO<sub>2</sub> 276.98346, found 276.98327.

The data are in full accordance with the literature (Zhou, L.; Tan, C. K.; Jiang, X.; Chen, F.; Yeung, Y.-Y. *J. Am. Chem. Soc.* **2010**, *132* (44), 15474–15476.).

### (C) Mechanochemical Bromocycloetherification Reactions

**General Procedure.** Olefinic mono-alcohol **5** (0.2 mmol) and NBS (39.2 mg, 0.22 mmol) were added into a 10 mL zirconium oxide chamber. The milling chamber was then securely fastened into a Retsch mixer mill (MM 400) and set to oscillate at 30 Hz, for 1 hour. On completion of reaction, the ball mill chamber was extracted with hexane and the solution was filtered through a thin plug of celite. The filtrate was concentrated under reduced pressure to give the desired product **6** in high purity. No column chromatography was necessary.

#### 2-(bromomethyl)-2-phenyltetrahydrofuran (**6a**)



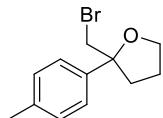
Yield: 98% (47.2 mg, colorless oil).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.40-7.44 (m, 2H), 7.38-7.38 (m, 2H), 7.26-7.30 (m, 1H), 4.06-4.12 (m, 1H), 3.92-3.97 (m, 1H), 3.66 (s, 2H), 2.40-2.46 (m, 1H), 2.23-2.30 (m, 1H), 2.02-2.10 (m, 1H), 1.80-1.89 (m, 1H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  144.1, 128.4, 127.5, 125.7, 85.4, 68.8, 42.3, 36.6, 26.3.

The data are in full accordance with the literature (Greb, M.; Hartung, J.; Köhler, F.; Špehar, K.; Kluge, R.; Csuk, R. *European J. Org. Chem.* **2004**, 2004 (18), 3799–3812.).

#### 2-(bromomethyl)-2-(p-tolyl)tetrahydrofuran (**6b**)



Yield: 99% (50.5 mg, colorless oil).

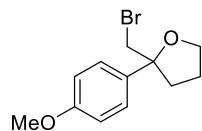
IR (KBr): 3017, 1216, 750  $\text{cm}^{-1}$ .

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.31 (d, *J* = 7.5 Hz, 2H), 7.17 (d, *J* = 7.5 Hz, 2H), 4.08 (dd, *J* = 14.5, 7.3 Hz, 1H), 3.93 (dd, *J* = 14.5, 7.3 Hz, 1H), 3.64 (s, 2H), 2.37-2.44 (m, 1H), 2.35 (s, 3H), 2.21-2.28 (m, 1H), 2.00-2.09 (m, 1H), 1.80-1.89 (m, 1H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 141.1, 137.2, 129.1, 125.6, 85.3, 68.7, 42.4, 36.5, 26.3, 21.2.

HRMS (ESI-Q-orbitrap) m/z: [M-H<sub>2</sub>O+H]<sup>+</sup> calcd for C<sub>12</sub>H<sub>15</sub>BrO 237.02734, found 237.02724.

### 2-(bromomethyl)-2-(4-methoxyphenyl)tetrahydrofuran (**6c**)



Yield: 76% (41.2 mg, colorless oil).

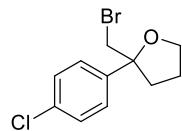
IR (KBr): 3019, 1510, 1249, 1216, 763, 669 cm<sup>-1</sup>.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.33 (d, *J* = 8.7 Hz, 2H), 6.88 (d, *J* = 8.7 Hz, 2H), 4.07 (dd, *J* = 14.7, 7.4 Hz, 1H), 3.92 (dd, *J* = 14.7, 7.4 Hz, 1H), 3.81 (s, 3H), 3.62 (s, 2H), 2.36-2.43 (m, 1H), 2.20-2.67 (m, 1H), 2.00-2.09 (m, 1H), 1.809-1.89 (m, 1H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 159.0, 136.0, 126.9, 113.7, 85.1, 68.7, 55.4, 42.5, 36.4, 26.3.

HRMS (ESI-Q-orbitrap) m/z: [M-H<sub>2</sub>O+H]<sup>+</sup> calcd for C<sub>12</sub>H<sub>15</sub>BrO<sub>2</sub> 253.02225, found 253.02216.

### 2-(bromomethyl)-2-(4-chlorophenyl)tetrahydrofuran (**6d**)



Yield: 90% (49.6 mg, colorless oil).

IR (KBr): 3018, 1490, 1216, 1052, 752 cm<sup>-1</sup>.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.35 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 4.08 (dd, *J* = 14.5, 7.2 Hz, 1H), 3.92 (dd, *J* = 14.5, 7.2 Hz, 1H), 3.60 (s, 2H), 2.36-2.43 (m, 1H), 2.16-2.25 (m, 1H), 2.01-2.10 (m, 1H), 1.79-1.89 (m, 1H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  142.6, 133.4, 128.5, 127.2, 85.1, 68.9, 41.9, 36.7, 26.2.

HRMS (ESI-Q-orbitrap) m/z:  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{11}\text{H}_{12}\text{BrClO}$  276.98107, found 276.98107.

2-(bromomethyl)-2-(4-fluorophenyl)tetrahydrofuran (**6e**)



Yield: 93% (48.2 mg, colorless oil).

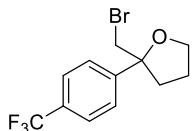
IR (KBr): 3017, 2981, 1604, 1508, 1210, 1052, 838, 756  $\text{cm}^{-1}$ .

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.38 (m, 2H), 7.03 (t,  $J = 8.3$  Hz, 2H), 4.08 (dd,  $J = 14.5, 7.4$  Hz, 1H), 3.92 (dd,  $J = 14.5, 7.4$  Hz, 1H), 3.61 (s, 2H), 2.37-2.44 (m, 1H), 2.19-2.26 (m, 1H), 2.01-2.10 (m, 1H), 1.80-1.89 (m, 1H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  163.2, 161.2, 139.8, 139.7, 127.5, 127.4, 115.3, 115.1, 85.1, 68.8, 42.1, 36.7, 26.2.

HRMS (ESI-Q-orbitrap) m/z:  $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{11}\text{H}_{12}\text{BrFO}$  259.01283, found 259.01287.

2-(bromomethyl)-2-(4-(trifluoromethyl)phenyl)tetrahydrofuran (**6f**)



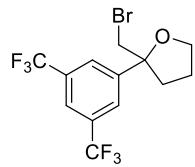
Yield: 98% (60.6 mg, colorless oil).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.61 (d,  $J = 8.3$  Hz, 2H), 7.54 (d,  $J = 8.3$  Hz, 2H), 4.08-4.13 (m, 1H), 3.92-3.97 (m, 1H), 3.63 (d,  $J = 0.9$  Hz, 2H), 2.41-2.47 (m, 1H), 2.21-2.28 (m, 1H), 2.04-2.12 (m, 1H), 1.80-1.90 (m, 1H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  148.2, 129.8 (q,  $J = 32$  Hz), 126.2, 125.4 (q,  $J = 4$  Hz), 124.4 (q,  $J = 271$  Hz), 85.3, 69.0, 41.6, 36.9, 26.2.

The data are in full accordance with the literature (Greb, M.; Hartung, J.; Köhler, F.; Špehar, K.; Kluge, R.; Csuk, R. *European J. Org. Chem.* **2004**, 2004 (18), 3799–3812.).

**2-(3,5-bis(trifluoromethyl)phenyl)-2-(bromomethyl)tetrahydrofuran (**6g**)**



Yield: 98% (73.9 mg, colorless oil).

IR (KBr): 3019, 1377, 1280, 1216, 1180, 1139, 1055, 900, 844, 757, 669 cm<sup>-1</sup>.

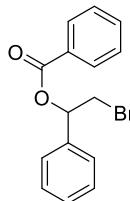
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.89 (s, 2H), 7.81 (s, 1H), 4.11-4.16 (m, 1H), 3.95-4.00 (m, 1H), 3.63 (d, *J* = 10.8 Hz, 1 H), 3.59 (d, *J* = 10.8, 1H), 2.47-2.54 (m, 1H), 2.24-2.30 (m, 1H), 2.08-2.17 (m, 1H), 1.84-1.93 (m, 1H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 147.0, 131.7 (q, *J* = 33 Hz), 126.2, 123.4 (q, *J* = 273 Hz), 121.6-121.8 (m), 85.0, 69.2, 41.0, 37.1, 26.3.

## (D) Mechanochemical Intermolecular Bromoesterification Reactions

**General Procedure.** Carboxylic acid **7** (0.2 mmol), alkene **8** (0.22 mmol) and DBDMH (62.9 mg, 0.22 mmol) were added into a 10 mL zirconium oxide chamber. The milling chamber was then securely fastened into a Retsch mixer mill (MM 400) and set to oscillate at 30 Hz, for 1 hour. On completion of reaction, the ball mill chamber was extracted with hexane and the solution was filtered through a thin plug of celite. The filtrate was concentrated under reduced pressure to give the desired product **9**. Purification using flash chromatography through silica gel (hexane/ethyl acetate 40:1) was used to obtain the final product.

### 2-bromo-1-phenylethyl benzoate (**9aa**)



Yield: 92% (56.2 mg, colorless oil,  $R_f = 0.47$ , hexane/ethyl acetate = 10:1).

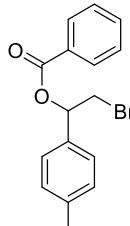
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.12-8.16 (m, 2H), 7.60 (tt,  $J = 7.4, 1.2$  Hz, 1H), 7.44-7.51 (m, 4H), 7.34-7.43 (m, 3H), 6.24 (dd,  $J = 8.0, 4.5$  Hz, 1H), 3.83 (dd,  $J = 10.9, 8.0$  Hz, 1H), 3.75 (dd,  $J = 10.9, 4.5$  Hz, 1H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  165.5, 137.9, 133.4, 129.9, 129.8, 128.6, 126.6, 75.4, 34.6.

HRMS (ESI-Q-orbitrap) m/z:  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{15}\text{H}_{13}\text{BrO}_2$  326.99911, found 326.99884.

The data are in full accordance with the literature (Ng, W.-H.; Hu, R.-B.; Lam, Y.-P.; Yeung, Y.-Y. *Org. Lett.* **2020**, 22 (14), 5572–5576.).

### 2-bromo-1-(p-tolyl)ethyl benzoate (**9ab**)



Yield: 93% (59.3 mg, colorless oil,  $R_f$  = 0.30, hexane/dichloromethane = 2:1).

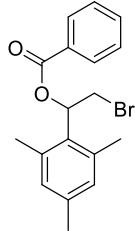
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.11 (d,  $J$  = 7.5 Hz, 2H), 7.58 (t,  $J$  = 7.6 Hz, 1H), 7.46 (t,  $J$  = 7.6 Hz, 2H), 7.34 (d,  $J$  = 7.8 Hz, 2H), 7.20 (d,  $J$  = 7.8 Hz, 2H), 6.18 (dd,  $J$  = 8.0, 4.5 Hz, 1H), 3.81 (dd,  $J$  = 10.8, 8.3 Hz, 1H), 3.71 (dd,  $J$  = 10.8, 4.5 Hz, 1H), 2.35 (s, 1H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  165.6, 138.9, 134.9, 133.4, 130.0, 129.6, 128.6, 126.6, 75.5, 34.6, 21.4.

HRMS (ESI-Q-orbitrap) m/z: [M + Na]<sup>+</sup> calcd for  $\text{C}_{16}\text{H}_{15}\text{BrO}_2$  341.01476, found 341.01511.

The data are in full accordance with the literature (Ng, W.-H.; Hu, R.-B.; Lam, Y.-P.; Yeung, Y.-Y. *Org. Lett.* **2020**, 22 (14), 5572–5576.).

### 2-bromo-1-mesitylethyl benzoate (**9ac**)



Yield: 99% (68.5 mg, colorless oil,  $R_f$  = 0.40, hexane/ethyl acetate = 20:1).

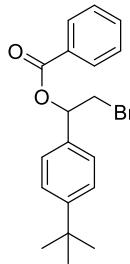
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.11–8.14 (m, 2H), 7.57–7.61 (m, 1H), 7.46–7.50 (m, 2H), 6.87 (s, 2H), 6.62 (dd,  $J$  = 10.1, 4.6 Hz, 1H), 4.07 (dd,  $J$  = 11.0, 10.1 Hz, 1H), 3.69 (dd,  $J$  = 11.0, 4.6 Hz, 1H), 2.55 (s, 6H), 2.27 (s, 3H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  165.6, 138.4, 136.8, 133.2, 130.6, 130.4, 129.9, 129.8, 128.6, 73.5, 32.0, 21.0, 20.8.

HRMS (ESI-Q-orbitrap) m/z: [M + Na]<sup>+</sup> calcd for  $\text{C}_{18}\text{H}_{19}\text{BrO}_2$  369.04606, found 369.04625.

The data are in full accordance with the literature (Ng, W.-H.; Hu, R.-B.; Lam, Y.-P.; Yeung, Y.-Y. *Org. Lett.* **2020**, 22 (14), 5572–5576.).

2-bromo-1-(4-(tert-butyl)phenyl)ethyl benzoate (**9ad**)



Yield: 90% (65.0 mg, colorless oil,  $R_f = 0.37$ , hexane/dichloromethane = 2:1).

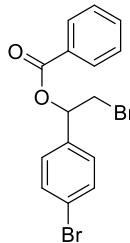
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.13 (d,  $J = 7.6$  Hz, 2H), 7.58 (t,  $J = 7.4$  Hz, 1H), 7.47 (t,  $J = 7.6$  Hz, 2H), 7.36-7.43 (m, 4H), 6.21 (dd,  $J = 8.3, 4.3$  Hz, 1H), 3.82 (dd,  $J = 10.9, 8.6$  Hz, 1H), 3.73 (dd,  $J = 10.9, 4.4$  Hz, 1H), 1.31 (s, 9H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  165.6, 152.0, 134.8, 133.4, 130.0, 128.6, 126.4, 125.8, 75.4, 34.8, 34.6, 31.4.

HRMS (ESI-Q-orbitrap) m/z:  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{19}\text{H}_{21}\text{BrO}_2$  383.06171, found 383.06263.

The data are in full accordance with the literature (Ng, W.-H.; Hu, R.-B.; Lam, Y.-P.; Yeung, Y.-Y. *Org. Lett.* **2020**, 22 (14), 5572–5576.).

2-bromo-1-(4-bromophenyl)ethyl benzoate (**9ae**)



Yield: 89% (68.3 mg, colorless oil,  $R_f = 0.30$ , hexane/dichloromethane = 2:1).

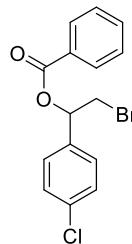
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.09-8.13 (m, 2H), 7.58-7.62 (m, 1H), 7.51-7.54 (m, 2H), 7.46-7.50 (m, 2H), 7.31-7.35 (m, 2H), 6.16 (dd,  $J = 7.5, 4.9$  Hz, 1H), 3.78 (dd,  $J = 10.9, 7.5$  Hz, 1H), 3.71 (dd,  $J = 10.9, 4.9$  Hz, 1H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  165.4, 136.9, 133.6, 132.1, 129.9, 129.6, 128.7, 128.4, 123.0, 74.7, 34.1.

HRMS (ESI-Q-orbitrap) m/z: [M + Na]<sup>+</sup> calcd for C<sub>15</sub>H<sub>12</sub>Br<sub>2</sub>O<sub>2</sub> 406.90765, found 406.90751.

The data are in full accordance with the literature (Ng, W.-H.; Hu, R.-B.; Lam, Y.-P.; Yeung, Y.-Y. *Org. Lett.* **2020**, 22 (14), 5572–5576.).

2-bromo-1-(4-chlorophenyl)ethyl benzoate (**9af**)



Yield: 88% (59.8 mg, colorless oil,  $R_f$  = 0.27, hexane/dichloromethane = 2:1).

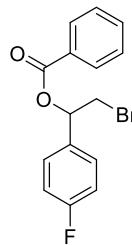
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  8.10–8.14 (m, 2H), 7.58–7.62 (m, 1H), 7.46–7.50 (m, 2H), 7.35–7.41 (m, 4H), 6.19 (dd,  $J$  = 7.5, 4.9 Hz, 1H), 3.79 (dd,  $J$  = 10.9, 7.5 Hz, 1H), 3.72 (dd,  $J$  = 10.9, 4.9 Hz, 1H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>):  $\delta$  165.4, 136.3, 134.8, 133.6, 129.9, 129.6, 129.1, 128.6, 128.1, 74.7, 34.2.

HRMS (ESI-Q-orbitrap) m/z: [M + Na]<sup>+</sup> calcd for C<sub>15</sub>H<sub>12</sub>BrClO<sub>2</sub> 362.95798, found 362.95726.

The data are in full accordance with the literature (Ng, W.-H.; Hu, R.-B.; Lam, Y.-P.; Yeung, Y.-Y. *Org. Lett.* **2020**, 22 (14), 5572–5576.).

2-bromo-1-(4-fluorophenyl)ethyl benzoate (**9ag**)



Yield: 93% (60.1 mg, colorless oil,  $R_f$  = 0.24, hexane/dichloromethane = 2:1).

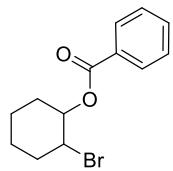
IR (KBr): 3020, 1722, 1511, 1268, 1216, 1109, 757, 669 cm<sup>-1</sup>.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 8.09-8.12 (m, 2H), 7.58-7.62 (m, 1H), 7.41-7.50 (m, 4H), 7.05-7.11 (m, 2H), 6.19 (dd, *J* = 7.6, 4.8 Hz, 1H), 3.79 (dd, *J* = 10.9, 7.6 Hz, 1H), 3.71 (dd, *J* = 10.9, 4.8 Hz, 1H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 165.5, 164.0, 162.0, 133.8, 133.7, 133.6, 130.0, 129.7, 128.7, 128.6, 128.5, 116.0, 115.8, 74.8, 34.4.

HRMS (ESI-Q-orbitrap) m/z: [M + Na]<sup>+</sup> calcd for C<sub>15</sub>H<sub>12</sub>BrFO<sub>2</sub> 344.98969, found 344.98986.

### 2-bromocyclohexyl benzoate (**9ah**)



Yield: 86% (48.6 mg, colorless oil, *R<sub>f</sub>* = 0.17, pure hexane).

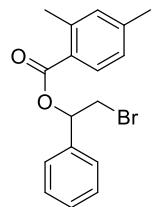
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 8.07 (d, *J* = 7.5 Hz, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 2H), 5.10-5.16 (m, 1H), 4.13-4.19 (m, 1H), 2.38-2.45 (m, 1H), 2.24-2.33 (m, 1H), 1.90-2.00 (m, 1H), 1.74-1.87 (m, 2H), 1.47-1.57 (m, 2H), 1.35-1.45 (m, 1H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 165.7, 133.2, 130.3, 129.9, 128.5, 76.5, 52.8, 35.7, 31.2, 25.6, 23.4.

HRMS (ESI-Q-orbitrap) m/z: [M + Na]<sup>+</sup> calcd for C<sub>13</sub>H<sub>15</sub>BrO<sub>2</sub> 305.01476, found 305.01457.

The data are in full accordance with the literature (Ng, W.-H.; Hu, R.-B.; Lam, Y.-P.; Yeung, Y.-Y. *Org. Lett.* **2020**, 22 (14), 5572–5576.).

### 2-bromo-1-phenylethyl 2,4-dimethylbenzoate (**9ba**)



Yield: 82% (54.6 mg, colorless oil, *R<sub>f</sub>* = 0.57, hexane/dichloromethane = 1:1).

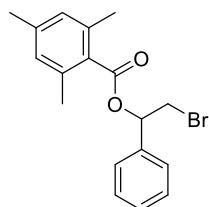
IR (KBr): 3019, 1717, 1615, 1256, 1216, 756 cm<sup>-1</sup>.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.99 (d, *J* = 7.8 Hz, 1H), 7.33-7.47 (m, 5H), 7.10 (d, *J* = 8.8 Hz, 2H), 6.20 (dd, *J* = 8.0, 4.6 Hz, 1H), 3.80 (dd, *J* = 10.9, 8.0 Hz, 1H), 3.73 (10.9, 4.6 Hz, 1H), 2.60 (s, 3H), 2.38 (s, 3H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 166.2, 143.1, 141.0, 138.1, 132.7, 131.2, 128.9, 126.7, 126.2, 75.2, 34.8, 22.1, 21.6.

HRMS (ESI-Q-orbitrap) m/z: [M + Na]<sup>+</sup> calcd for C<sub>17</sub>H<sub>17</sub>BrO<sub>2</sub> 355.03041, found 355.03042.

### 2-bromo-1-phenylethyl 2,4,6-trimethylbenzoate (**9ca**)



Yield: 90% (62.5 mg, colorless oil, *R<sub>f</sub>* = 0.43, hexane/ethyl acetate = 20:1).

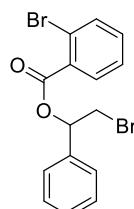
IR (KBr): 3020, 1726, 1612, 1262, 1216, 1167, 1079, 756, 669 cm<sup>-1</sup>.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.42-7.46 (m, 2H), 7.35-7.42 (m, 3H), 6.86 (s, 2H), 6.25 (dd, *J* = 8.7, 4.2 Hz, 1H), 3.78 (dd, *J* = 11.0, 8.7 Hz, 1H), 3.67 (dd, *J* = 11.0, 4.2 Hz, 1H), 2.29 (s, 3H), 2.24 (s, 6H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 169.2, 139.6, 137.7, 135.3, 130.6, 129.1, 128.9, 128.5, 127.1, 76.1, 34.0, 21.3, 19.9.

HRMS (ESI-Q-orbitrap) m/z: [M + Na]<sup>+</sup> calcd for C<sub>18</sub>H<sub>19</sub>BrO<sub>2</sub> 369.04606, found 369.04633.

### 2-bromo-1-phenylethyl 2-bromobenzoate (**9da**)



Yield: 90% (69.1 mg, colorless oil, *R<sub>f</sub>* = 0.32, hexane/dichloromethane = 2:1).

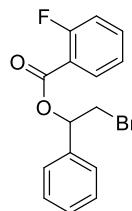
IR (KBr): 3019, 1735, 1249, 1215, 755, 669 cm<sup>-1</sup>.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.92 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.68 (d, *J* = 7.9 Hz, 1H), 7.46 (d, *J* = 7.6 Hz, 2H), 7.33-7.43 (m, 5H), 6.23 (dd, *J* = 8.0, 4.6 Hz, 1H), 3.82 (dd, *J* = 11.0, 8.0 Hz, 1H), 3.73 (dd, *J* = 11.0, 4.6 Hz, 1H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 164.9, 137.4, 134.7, 133.0, 131.8, 131.6, 129.1, 128.9, 127.4, 126.9, 122.1, 76.3, 34.3.

HRMS (ESI-Q-orbitrap) m/z: [M + Na]<sup>+</sup> calcd for C<sub>15</sub>H<sub>12</sub>Br<sub>2</sub>O<sub>2</sub> 406.90765, found 406.90749.

### 2-bromo-1-phenylethyl 2-fluorobenzoate (**9ea**)



Yield: 99% (63.9 mg, colorless oil, *R<sub>f</sub>* = 0.24, hexane/dichloromethane = 2:1).

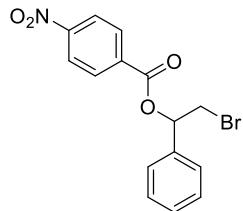
IR (KBr): 3020, 1718, 1615, 1216, 756 cm<sup>-1</sup>.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 8.02 (td, *J* = 7.6, 1.8 Hz, 1H), 7.52-7.57 (m, 1H), 7.45-7.49 (m, 2H), 7.34-7.42 (m, 3H), 7.23 (td, *J* = 7.8, 1.1 Hz, 1H), 7.16 (ddd, *J* = 10.8, 8.4, 0.9 Hz, 1H), 6.25 (dd, *J* = 8.1, 4.5 Hz, 1H), 3.81 (dd, *J* = 10.9, 8.1 Hz, 1H), 3.72 (dd, *J* = 10.9, 4.5 Hz, 1H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 163.3, 163.3, 163.2, 161.2, 137.6, 135.0, 134.9, 132.4, 129.0, 128.9, 128.9, 126.8, 124.2, 124.1, 118.4, 118.3, 117.3, 117.1, 75.9, 34.5.

HRMS (ESI-Q-orbitrap) m/z: [M + Na]<sup>+</sup> calcd for C<sub>15</sub>H<sub>12</sub>BrFO<sub>2</sub> 344.98969, found 344.98977.

### 2-bromo-1-phenylethyl 4-nitrobenzoate (**9fa**)



Yield: 69% (48.3 mg, colorless oil, *R<sub>f</sub>* = 0.20, hexane/dichloromethane = 2:1).

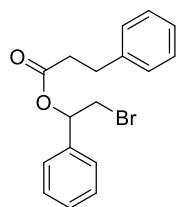
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 8.26-8.33 (m, 4H), 7.36-7.46 (m, 5H), 6.24 (dd, *J* = 8.4, 4.3 Hz, 1H), 3.84 (dd, *J* = 11.1, 8.4 Hz, 1H), 3.75 (dd, *J* = 11.1, 4.3 Hz, 1H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 163.7, 150.9, 137.2, 135.2, 131.1, 129.4, 129.1, 126.7, 123.8, 76.6, 34.1.

HRMS (ESI-Q-orbitrap) m/z: [M + Na]<sup>+</sup> calcd for C<sub>15</sub>H<sub>12</sub>BrNO<sub>4</sub> 371.98419, found 371.98425.

The data are in full accordance with the literature (Ng, W.-H.; Hu, R.-B.; Lam, Y.-P.; Yeung, Y.-Y. *Org. Lett.* **2020**, 22 (14), 5572–5576.).

### 2-bromo-1-phenylethyl 3-phenylpropanoate (**9ga**)



Yield: 96% (64.0 mg, yellow oil, *R<sub>f</sub>* = 0.33, hexane/ethyl acetate = 20:1).

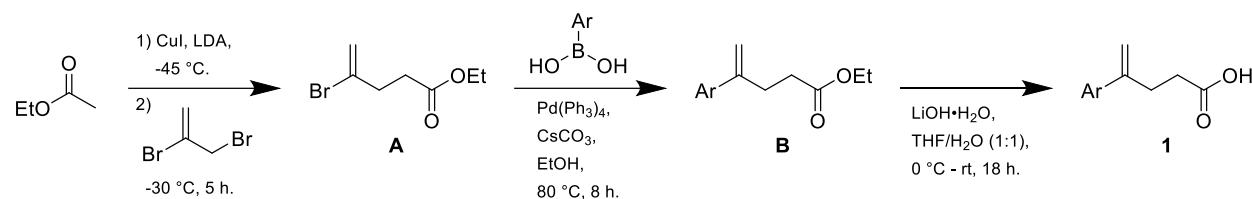
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.36-7.41 (m, 3H), 7.28-7.35 (m, 4H), 7.20-7.25 (m, 3H), 6.03 (dd, *J* = 8.2, 4.7 Hz, 1H), 3.65 (dd, *J* = 10.8, 8.2 Hz, 1H), 3.59 (dd, *J* = 10.8, 4.7 Hz, 1H), 2.97-3.07 (m, 2H), 2.70-2.83 (m, 2H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 171.8, 140.3, 137.7, 128.9, 128.8, 128.6, 128.4, 126.6, 126.4, 75.0, 35.9, 34.3, 30.9.

HRMS (ESI-Q-orbitrap) m/z: [M + Na]<sup>+</sup> calcd for C<sub>17</sub>H<sub>17</sub>BrO<sub>2</sub> 355.03041, found 355.03031.

The data are in full accordance with the literature (Shi, Y.; Wong, J.; Ke, Z.; Yeung, Y.-Y. *J. Org. Chem.* **2019**, 84 (7), 4017–4024.).

## (E) Synthesis of olefinic acid substrates **1**



### General procedure.

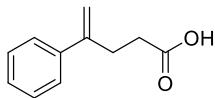
**1. Preparation of LDA solution:** n-Butyllithium solution 1.58 M in hexanes (47.5 mL, 75.0 mmol) was added to a solution of diisopropylamine (10.5 mL, 75.0 mmol) in THF (25.0 mL) at -78 °C. The resultant mixture was allowed to warm to room temperature over 2 hours.

**2. Enolate alkylation:** The LDA solution was added to a suspension of ethyl acetate (7.3 mL, 75.0 mmol) and copper (I) iodide (28.6 g, 150.0 mmol) in THF (75.0 mL) at -45 °C. The resultant solution was warmed to -30 °C. A solution of 2,3-dibromopropene (3.7 mL, 37.5 mmol) in THF (37.5 mL) was then added dropwise. The resultant mixture was stirred for an additional 5 hours at -30 °C. The reaction was then quenched using a saturated aqueous ammonium chloride solution (20.0 mL), acidified to pH 2 with 1 M aqueous hydrogen chloride solution, and extracted with diethyl ether (3 x 20.0 mL). The organic extracts were then washed with 28% aqueous ammonia solution (3 x 10.0 mL), dried over anhydrous sodium sulfate, filtereted, and concentrated *in vacuo* to give the intermediate **A**; which was used in the next step without further purification.

**3. Suzuki coupling:** A mixture of intermediate **A** (2.1 g, 10.0 mmol), aryl boronic acid (11.0 mmol), tetrakis(triphenylphosphine)-palladium(0) (0.6 g, 0.5 mmol), cesium carbonate (4.9 g, 15.0 mmol), in ethanol (50 mL) was degassed with nitrogen in a resealable tube. The sealed system was then heated to 80 °C for 8 hours. The resultant reaction mixture was filtered, concentrated *in vacuo* and purification by flash column chromatography in silica gel (hexane/ethyl acetate, 20:1) to give the intermediate **B**.

**4. Ester hydrolysis:** To a suspension of intermediate **B** (6.0 mmol), in a 1:1 mixture of THF and water (60.0 mL) at 0 °C, lithium hydroxide monohydrate (1.3 g, 30.0 mmol) was added in portions. The resultant mixture was warmed to room temperature and stirred for 18 hours. The product mixture was then acidified to pH < 4 with a 1 M hydrogen chloride aqueous solution, extracted with diethyl ether (3 x 10.0 mL) and washed with brine (3 x 5.0 mL). The organic fractions were concentrated *in vacuo* and recrystallized from a mixture of diethyl ether and hexane to give the alkenoic acid **1**.

4-phenylpent-4-enoic acid (**1a**)



White solid.

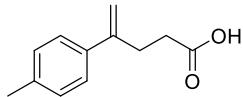
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  10.50 (br. s, 1H), 7.41 (d,  $J = 7.3$  Hz, 2H), 7.34 (t,  $J = 7.3$  Hz, 2H), 7.27-7.31 (m, 1H), 5.33 (s, 1H), 5.12 (s, 1H), 2.86 (t,  $J = 7.7$  Hz, 2H), 2.54 (t,  $J = 7.7$  Hz, 2H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  179.4, 146.6, 140.5, 128.6, 127.8, 126.2, 113.1, 33.1, 30.3.

HRMS (ESI-Q-orbitrap) m/z: [M - H]<sup>-</sup> calcd for  $\text{C}_{11}\text{H}_{12}\text{O}_2$  175.07645, found 175.07630.

The data are in full accordance with the literature (Zhou, L.; Tan, C. K.; Jiang, X.; Chen, F.; Yeung, Y.-Y. *J. Am. Chem. Soc.* **2010**, *132* (44), 15474–15476.).

4-(p-tolyl)pent-4-enoic acid (**1b**)



White solid.

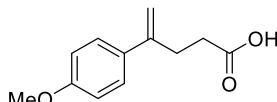
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  10.91 (br. s, 1H), 7.32 (d,  $J = 7.4$  Hz, 2H), 7.16 (d,  $J = 7.4$  Hz, 2H), 5.31 (s, 1H), 5.08 (s, 1H), 2.85 (t,  $J = 7.5$  Hz, 2H), 2.55 (t,  $J = 7.5$  Hz, 2H), 2.36 (s, 3H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  179.8, 146.4, 137.6, 137.5, 129.2, 126.1, 112.3, 33.2, 30.2, 21.2.

HRMS (ESI-Q-orbitrap) m/z: [M - H]<sup>-</sup> calcd for  $\text{C}_{12}\text{H}_{14}\text{O}_2$  189.09210, found 189.09202.

The data are in full accordance with the literature (Zhou, L.; Tan, C. K.; Jiang, X.; Chen, F.; Yeung, Y.-Y. *J. Am. Chem. Soc.* **2010**, *132* (44), 15474–15476.).

4-(4-methoxyphenyl)pent-4-enoic acid (**1c**)



White solid.

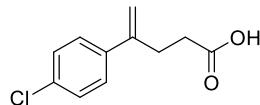
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.34 (d,  $J = 7.6$  Hz, 2H), 6.87 (d,  $J = 7.6$  Hz, 2H), 5.25 (s, 1H), 5.02 (s, 1H), 3.81 (s, 3H), 2.82 (t,  $J = 7.7$  Hz, 2H), 2.52 (t,  $J = 7.7$  Hz, 2H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $(\text{CD}_3)_2\text{SO}$ ):  $\delta$  174.1, 159.0, 145.8, 132.4, 127.1, 114.0, 110.8, 55.3, 32.9, 29.8.

HRMS (ESI-Q-orbitrap) m/z:  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{12}\text{H}_{14}\text{O}_3$  229.08352, found 229.08351.

The data are in full accordance with the literature (Zhou, L.; Tan, C. K.; Jiang, X.; Chen, F.; Yeung, Y.-Y. *J. Am. Chem. Soc.* **2010**, *132* (44), 15474–15476.).

#### 4-(4-chlorophenyl)pent-4-enoic acid (**1d**)



White solid.

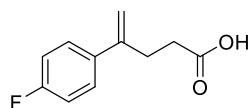
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  9.25 (br. s, 1H), 7.28–7.35 (m, 4H), 5.31 (s, 1H), 5.12 (m, 1H), 2.81 (t,  $J = 7.5$  Hz, 2H), 2.52 (t,  $J = 7.5$  Hz, 2H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  179.1, 145.5, 139.0, 133.6, 128.7, 127.5, 113.7, 32.9, 30.1.

HRMS (ESI-Q-orbitrap) m/z:  $[\text{M} - \text{H}]^-$  calcd for  $\text{C}_{11}\text{H}_{11}\text{ClO}_2$  209.03748, found 209.03744.

The data are in full accordance with the literature (Zhou, L.; Tan, C. K.; Jiang, X.; Chen, F.; Yeung, Y.-Y. *J. Am. Chem. Soc.* **2010**, *132* (44), 15474–15476.).

#### 4-(4-fluorophenyl)pent-4-enoic acid (**1e**)

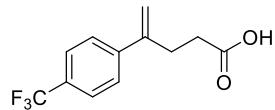


$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  11.02 (br. s, 1H), 7.37 (t,  $J = 6.3$  Hz, 2H), 7.03 (t,  $J = 7.9$  Hz, 2H), 5.28 (s, 1H), 5.10 (s, 1H), 2.82 (t,  $J = 7.5$  Hz, 2H), 2.53 (t,  $J = 7.5$  Hz, 2H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  179.8, 163.5, 161.6, 145.6, 136.6, 136.6, 127.9, 127.8, 115.5, 115.3, 113.1, 33.0, 30.3.

HRMS (ESI-Q-orbitrap) m/z: [M - H]<sup>-</sup> calcd for C<sub>11</sub>H<sub>11</sub>FO<sub>2</sub> 193.06703, found 193.06691.

4-(4-(trifluoromethyl)phenyl)pent-4-enoic acid (**1f**)



White solid.

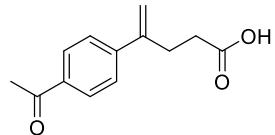
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.59 (d, *J* = 7.9 Hz, 2H), 7.50 (d, *J* = 7.9 Hz, 2H), 5.39 (s, 1H), 5.21 (s, 1H), 2.85 (t, *J* = 7.6 Hz, 2H), 2.53 (t, *J* = 7.6 Hz, 2H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 178.7, 145.6, 144.2, 129.8 (q, *J* = 32 Hz), 126.6, 125.6 (q, *J* = 3.6 Hz), 115.1, 32.8, 30.1.

HRMS (ESI-Q-orbitrap) m/z: [M - H]<sup>-</sup> calcd for C<sub>12</sub>H<sub>12</sub>F<sub>3</sub>O<sub>2</sub> 243.06384, found 243.06371.

The data are in full accordance with the literature (Zhou, L.; Tan, C. K.; Jiang, X.; Chen, F.; Yeung, Y.-Y. *J. Am. Chem. Soc.* **2010**, *132* (44), 15474–15476.).

4-(4-acetylphenyl)pent-4-enoic acid (**1g**)



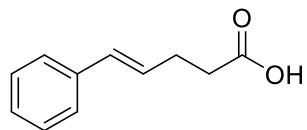
White solid.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.93 (d, *J* = 7.6 Hz, 2H), 7.49 (d, *J* = 7.4 Hz, 2H), 5.43 (s, 1H), 5.23 (s, 1H), 2.87 (t, *J* = 7.6 Hz, 2H), 2.61 (s, 3H), 2.54 (t, *J* = 7.6 Hz, 2H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 197.8, 177.6, 145.8, 145.3, 136.4, 128.7, 126.4, 115.2, 32.7, 30.0, 26.8.

HRMS (ESI-Q-orbitrap) m/z: [M+Na]<sup>+</sup> calcd for C<sub>13</sub>H<sub>14</sub>O<sub>3</sub> 241.08345, found 241.08342.

*(E)*-5-phenylpent-4-enoic acid **1h**



White solid.

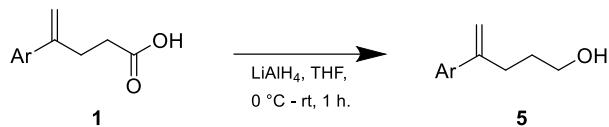
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.35 (d,  $J = 7.7$  Hz, 2H), 7.30 (t,  $J = 7.7$  Hz, 2H), 7.21 (t,  $J = 7.1$  Hz, 1H), 6.45 (d,  $J = 15.9$  Hz, 1H), 6.18-6.25 (m, 1H), 2.54-2.57 (m, 4H).

$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  179.1, 137.4, 131.3, 128.7, 128.1, 127.4, 126.2, 33.9, 28.0.

HRMS (ESI-Q-orbitrap) m/z:  $[\text{M}-\text{H}]^-$  calcd for  $\text{C}_{11}\text{H}_{12}\text{O}_2$  175.07645, found 175.07651.

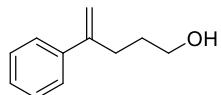
The data are in full accordance with the literature (Zhou, L.; Tan, C. K.; Jiang, X.; Chen, F.; Yeung, Y.-Y. *J. Am. Chem. Soc.* **2010**, *132* (44), 15474–15476.).

### Synthesis of olefinic mono-alcohol 5



**General procedure.** To a solution of **1** (3.0 mmol) in anhydrous THF (6.0 mL) at 0 °C lithium aluminum hydride (0.2 g, 6.0 mmol) was added in portions. The resultant suspension was warmed to room temperature and stirred for 1 hour. The reaction was then quenched with a 2 M aqueous solution of sodium hydroxide (5.0 mL), filtered through a thin pad of celite, and extracted with diethyl ether (3 x 5 ml). The organic fractions were dried with anhydrous sodium sulfate, filtered, and concentrated *in vacuo*. Purification by flash column chromatography through silica gel yielded the alkenoic alcohol **5**.

#### 4-phenylpent-4-en-1-ol (**5a**)



Colorless oil.

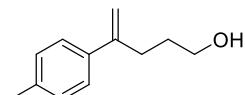
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.42 (d,  $J = 7.8$  Hz, 2H), 7.34 (t,  $J = 7.2$  Hz, 2H), 7.25-7.30 (m, 1H), 5.31 (s, 1H), 5.11 (s, 1H), 3.65 (t,  $J = 6.5$  Hz, 2H), 2.61 (t,  $J = 7.5$  Hz, 2H), 1.68-1.77 (m, 3H).

$^{13}\text{C}\{^1\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  148.02, 141.1, 128.4, 127.5, 126.2, 112.7, 62.4, 31.6, 31.2.

HRMS (ESI-Q-orbitrap) m/z:  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{11}\text{H}_{14}\text{O}$  185.09369, found 185.09375.

The data are in full accordance with the literature (Hornback, J. M.; Proehl, G. S. *J. Am. Chem. Soc.* **1979**, *101* (24), 7367–7373.)

#### 4-(p-tolyl)pent-4-en-1-ol (**5b**)



Yellow oil.

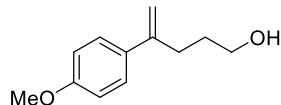
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.33 (d, *J* = 7.5 Hz, 2H), 7.15 (d, *J* = 7.5 Hz, 2H), 5.29 (s, 1H), 5.07 (s, 1H), 3.66 (t, *J* = 6.4 Hz, 2H), 2.60 (t, *J* = 7.5 Hz, 2H), 2.36 (s, 3H), 1.69–1.77 (m, 2H), 1.67 (br. s, 1H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 147.8, 138.1, 137.3, 129.1, 126.1, 111.9, 62.5, 31.6, 31.2, 21.2.

HRMS (ESI-Q-orbitrap) m/z: [M+Na]<sup>+</sup> calcd for C<sub>12</sub>H<sub>16</sub>O 199.10934, found 199.10952.

The data are in full accordance with the literature (Rösner, C.; Hennecke, U. *Org. Lett.* **2015**, *17* (13), 3226–3229.).

#### 4-(4-methoxyphenyl)pent-4-en-1-ol (**5c**)



Yellow oil.

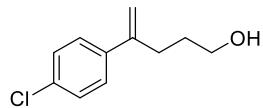
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.36 (d, *J* = 8.7 Hz, 2H), 6.86 (d, *J* = 8.7 Hz, 2H), 5.23 (s, 1H), 5.01 (s, 1H), 3.81 (s, 3H), 3.65 (t, *J* = 6.4 Hz, 2H), 2.57 (t, *J* = 7.5 Hz, 2H), 1.639–1.75 (m, 2H), 1.59 (br. s, 1H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 159.1, 147.29, 133.4, 127.3, 113.8, 111.1, 62.5, 55.4, 31.7, 31.3.

HRMS (ESI-Q-orbitrap) m/z: [M+H]<sup>+</sup> calcd for C<sub>12</sub>H<sub>16</sub>O<sub>2</sub> 193.12231, found 193.12252.

The data are in full accordance with the literature (Rösner, C.; Hennecke, U. *Org. Lett.* **2015**, *17* (13), 3226–3229.).

#### 4-(4-chlorophenyl)pent-4-en-1-ol (**5d**)



Colorless oil.

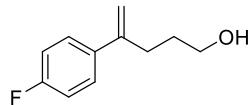
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.34 (d, *J* = 7.7 Hz, 2H), 7.29 (d, *J* = 7.7 Hz, 2H), 5.28 (s, 1H), 5.11 (s, 1H), 3.66 (t, *J* = 6.3 Hz, 2H), 2.58 (t, *J* = 7.5 Hz, 2H), 1.67–1.74 (m, 2H), 1.41 (br. s, 1H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 146.9, 139.5, 133.3, 128.6, 127.5, 113.2, 62.4, 31.6, 31.1.

HRMS (ESI-Q-orbitrap) m/z: [M+H]<sup>+</sup> calcd for C<sub>12</sub>H<sub>13</sub>ClO 197.07277, found 197.07285.

The data are in full accordance with the literature (Tsuji, N.; Kennemur, J. L.; Buyck, T.; Lee, S.; Prévost, S.; Kaib, P. S. J.; Bykov, D.; Farès, C.; List, B. *Science*. **2018**, *359* (6383), 1501–1505.).

**4-(4-fluorophenyl)pent-4-en-1-ol (**5e**)**



Yellow oil.

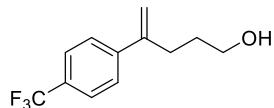
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.34–7.39 (m, 2H), 7.00 (t, *J* = 8.0 Hz, 2H), 5.24 (s, 1H), 5.07 (s, 1H), 3.64 (t, *J* = 6.3, 2H), 2.57 (t, *J* = 7.5 Hz, 2H), 1.80 (br. s, 1H), 1.70 (m, 2H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 163.3, 161.4, 137.1, 137.1, 127.8, 127.7, 115.3, 115.1, 112.6, 62.3, 31.7, 31.1.

HRMS (ESI-Q-orbitrap) m/z: [M+H]<sup>+</sup> calcd for C<sub>12</sub>H<sub>13</sub>FO 181.10232, found 181.10239.

The data are in full accordance with the literature (Rösner, C.; Hennecke, U. *Org. Lett.* **2015**, *17* (13), 3226–3229.).

**4-(4-(trifluoromethyl)phenyl)pent-4-en-1-ol (**5f**)**



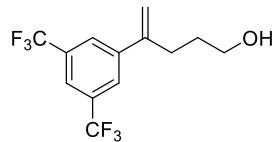
Colorless oil.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.58 (d, *J* = 8.4 Hz, 2H), 7.50 (d, *J* = 8.4 Hz, 2H), 5.36 (s, 1H), 5.20 (s, 1H), 3.67 (t, *J* = 6.4 Hz, 2H), 2.62 (t, *J* = 7.2 Hz, 2H), 1.68–1.75 (m, 2H), 1.50 (br. s, 1H).

<sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 147.0, 144.8, 129.5 (q, *J* = 32 Hz), 126.5, 125.4 (q, *J* = 4 Hz), 114.6, 62.3, 31.5, 31.1.

HRMS (ESI-Q-orbitrap) m/z: [M-H]<sup>-</sup> calcd for C<sub>12</sub>H<sub>13</sub>F<sub>3</sub>O 229.08457, found 229.08467.

**4-(3,5-bis(trifluoromethyl)phenyl)pent-4-en-1-ol (**5g**)**



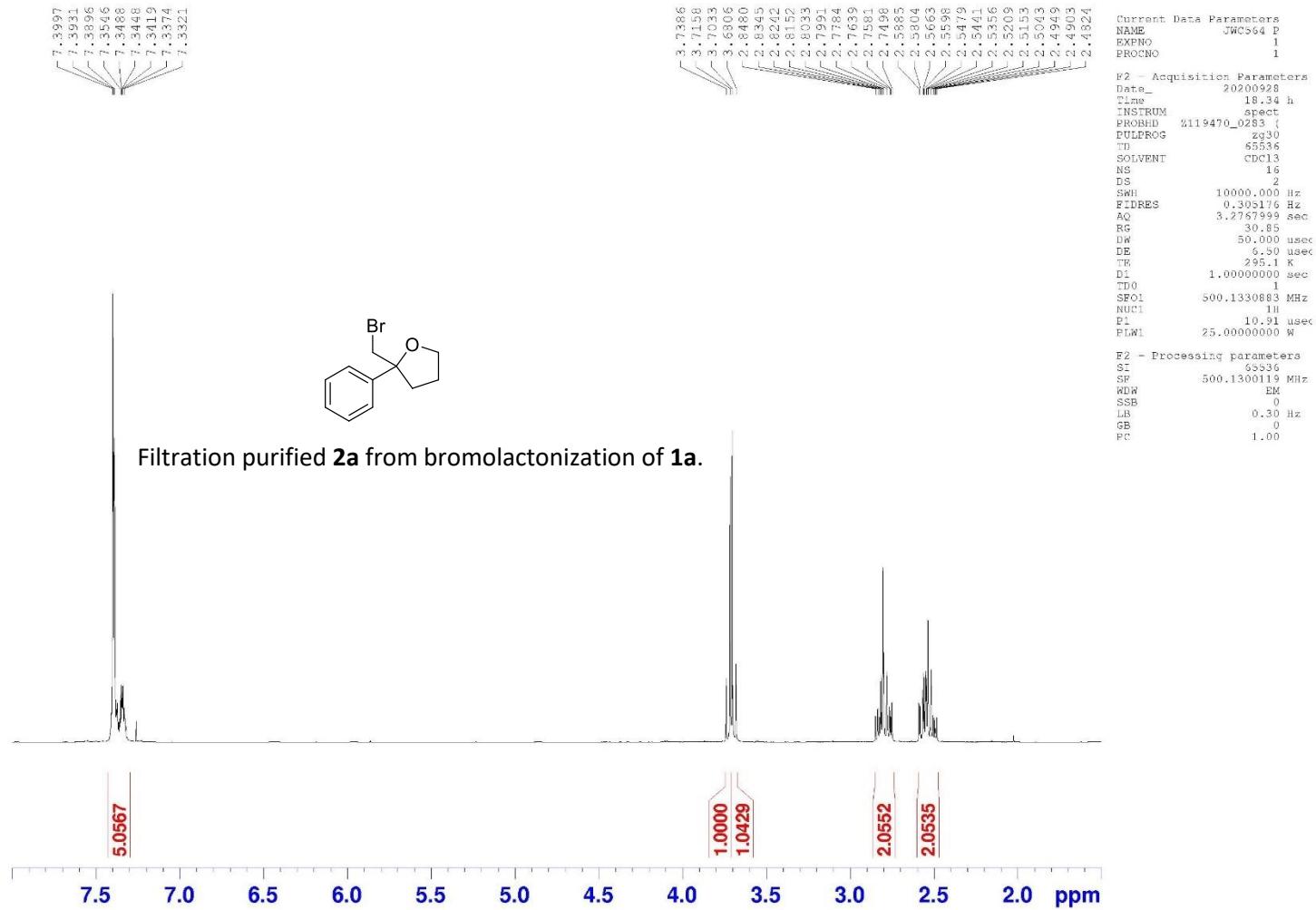
Colorless oil.

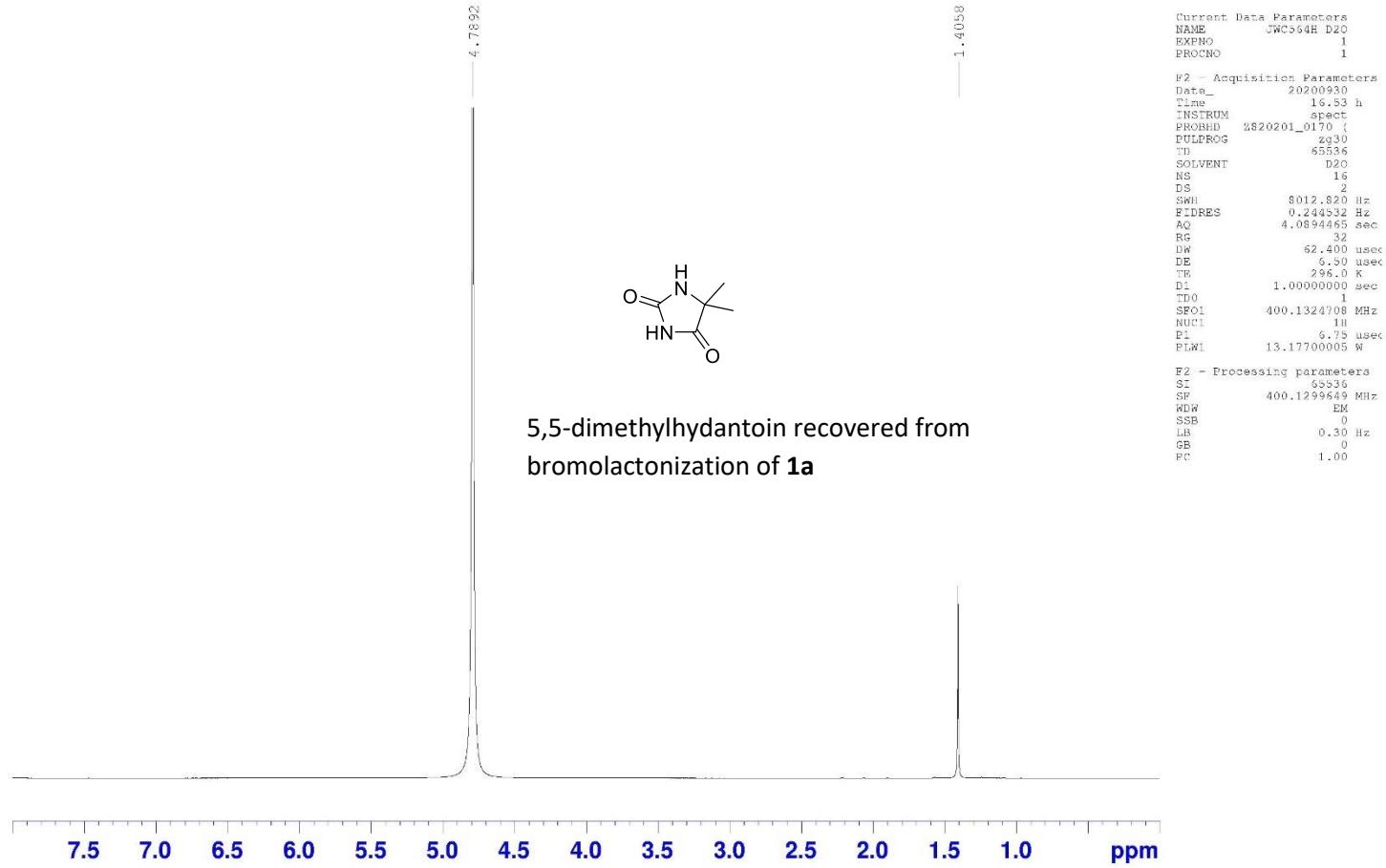
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.83 (s, 2H), 7.77 (s, 1H), 5.42 (s, 1H), 5.28 (s, 1H), 3.69 (t,  $J = 6.4$  Hz, 2H), 2.63 (t,  $J = 7.6$  Hz, 2H), 1.68-1.78 (m, 3H).

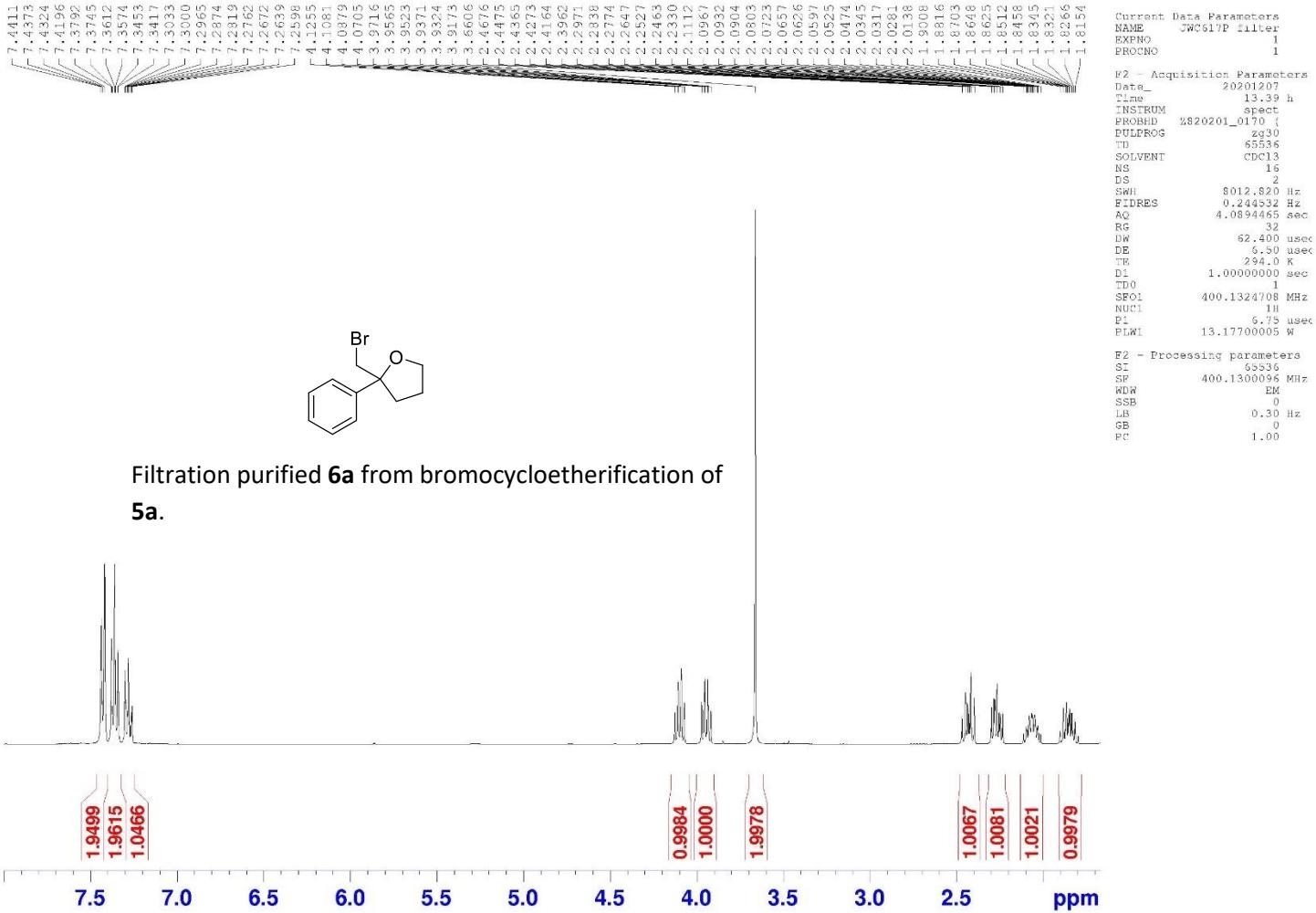
$^{13}\text{C}\{\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  145.7, 134.5, 131.8 (q,  $J = 33$  Hz), 126.3, 123.5 (q,  $J = 271$  Hz), 121.2 (m), 115.8, 62.1, 31.2, 30.9.

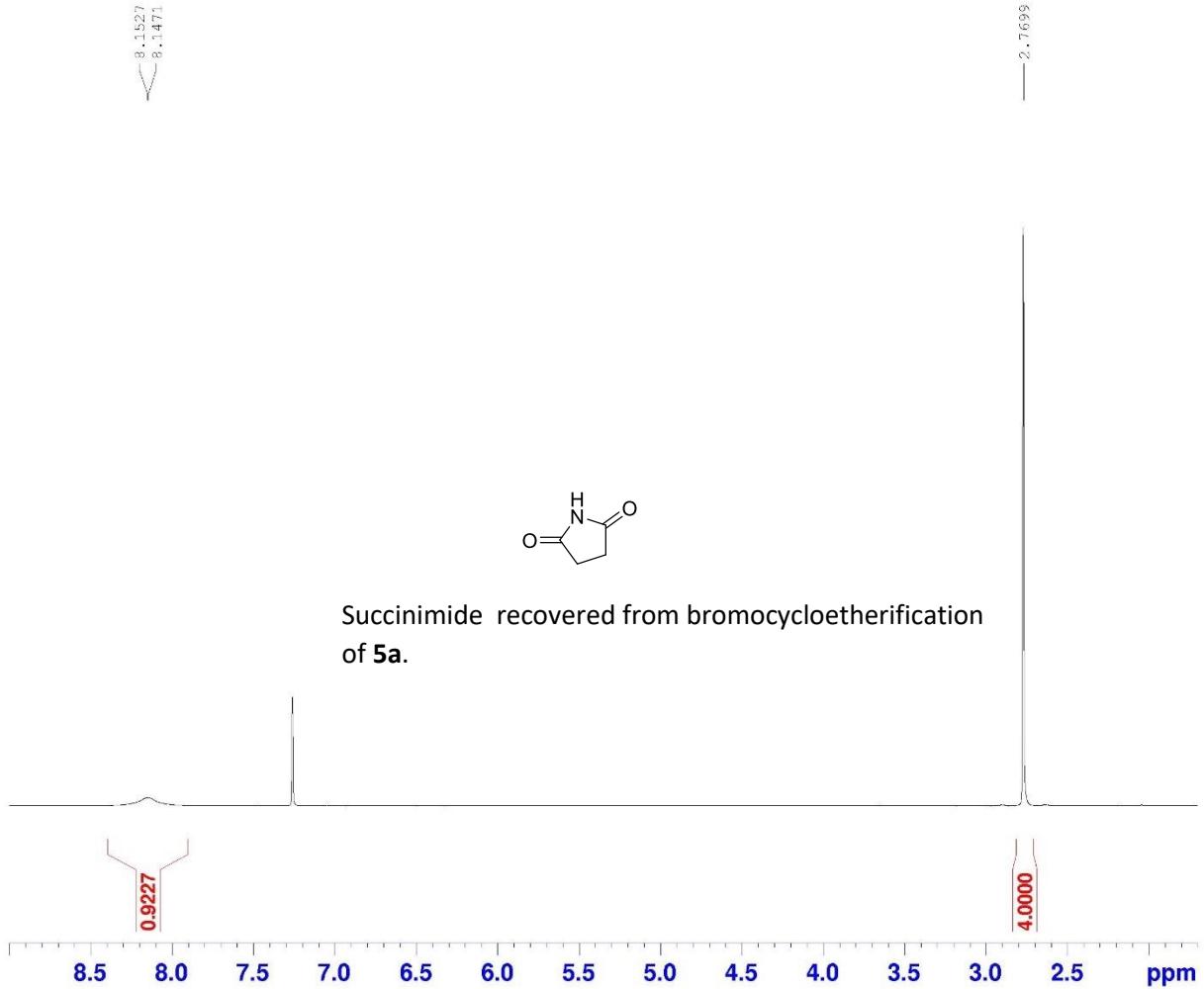
HRMS (ESI-Q-orbitrap) m/z: [M-H]<sup>-</sup> calcd for  $\text{C}_{13}\text{H}_{12}\text{F}_6\text{O}$  297.07196, found 297.07153.

**(F)  $^1\text{H}$  NMR of filter purified products and recovered imide by-products**









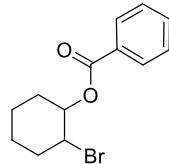
8.0782  
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 7.5506  
 7.4639  
 7.4193  
 7.4140

5.1495  
 5.1408  
 5.1316  
 5.1228  
 5.1135  
 5.1047  
 4.1195  
 4.1109  
 4.1098  
 4.1027  
 4.1007  
 4.1221  
 4.082  
 4.0199  
 4.0103  
 4.005  
 3.3025  
 3.3016  
 2.3097  
 2.2924  
 2.2933  
 2.2938  
 2.2810  
 2.2749  
 2.2713  
 2.2649  
 2.2616  
 1.9774  
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 1.9064  
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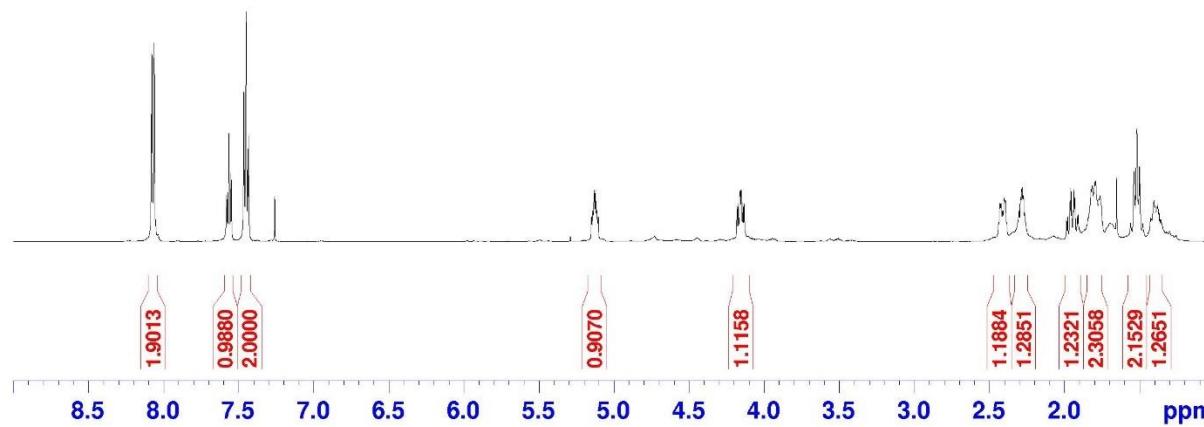
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 DATE: 10/16/2018  
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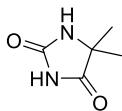
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 PROBPG: 00000000  
 TDRNG: 65536  
 SOLVENT: CDCl3  
 DR: 16  
 DS: 2  
 SW: 10000.00 Hz  
 FIDRES: 0.203174 Hz  
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 TS: 100000.00 sec  
 SWF: 50.000 usec  
 DRW: 6.50 usec  
 SWI: 1.00000000 sec  
 FID1: 500.1330833 MHz  
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 FID2: 25.000000000 sec

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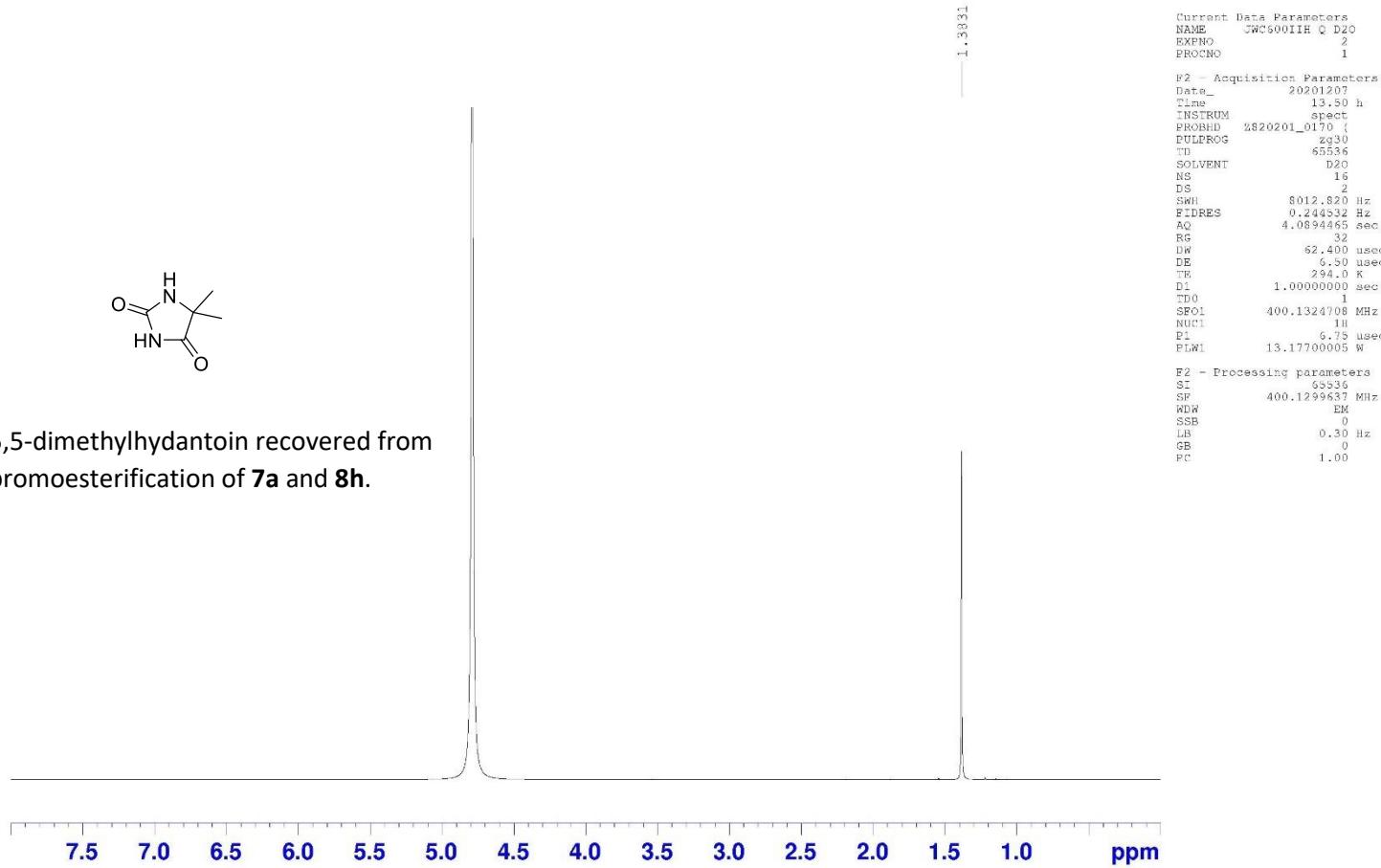


Filtration purified **9ah** from bromoesterification of **7a** and **8h**.

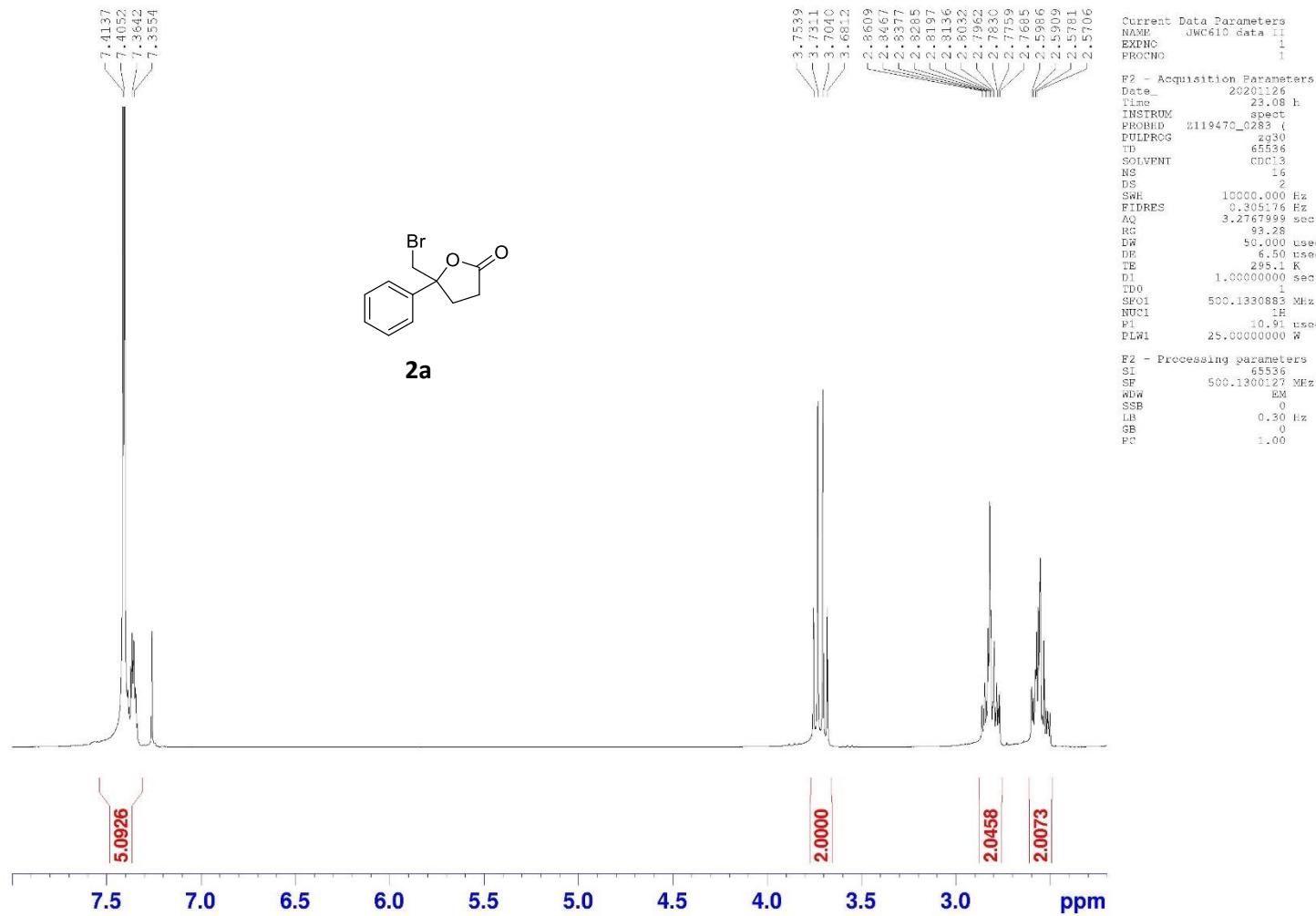


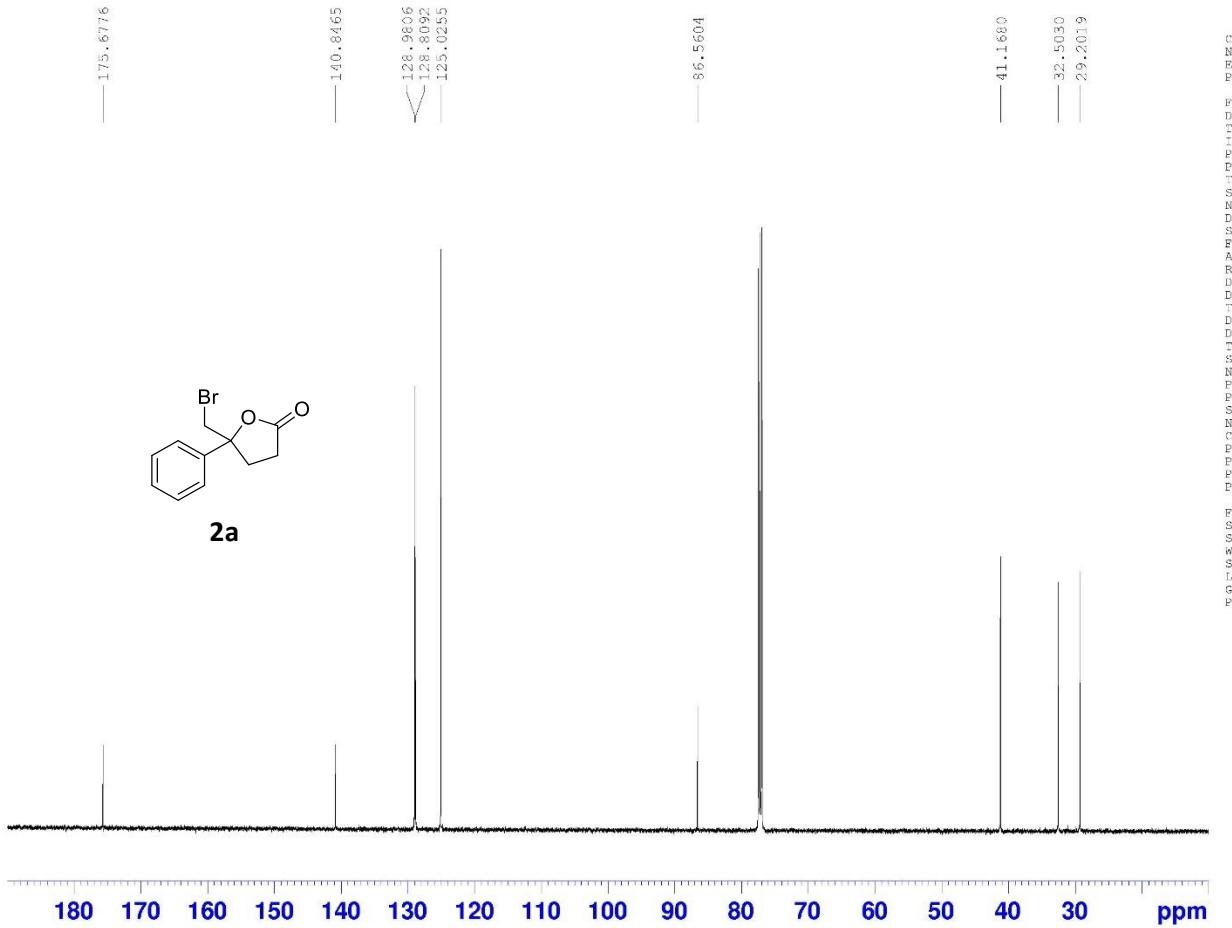


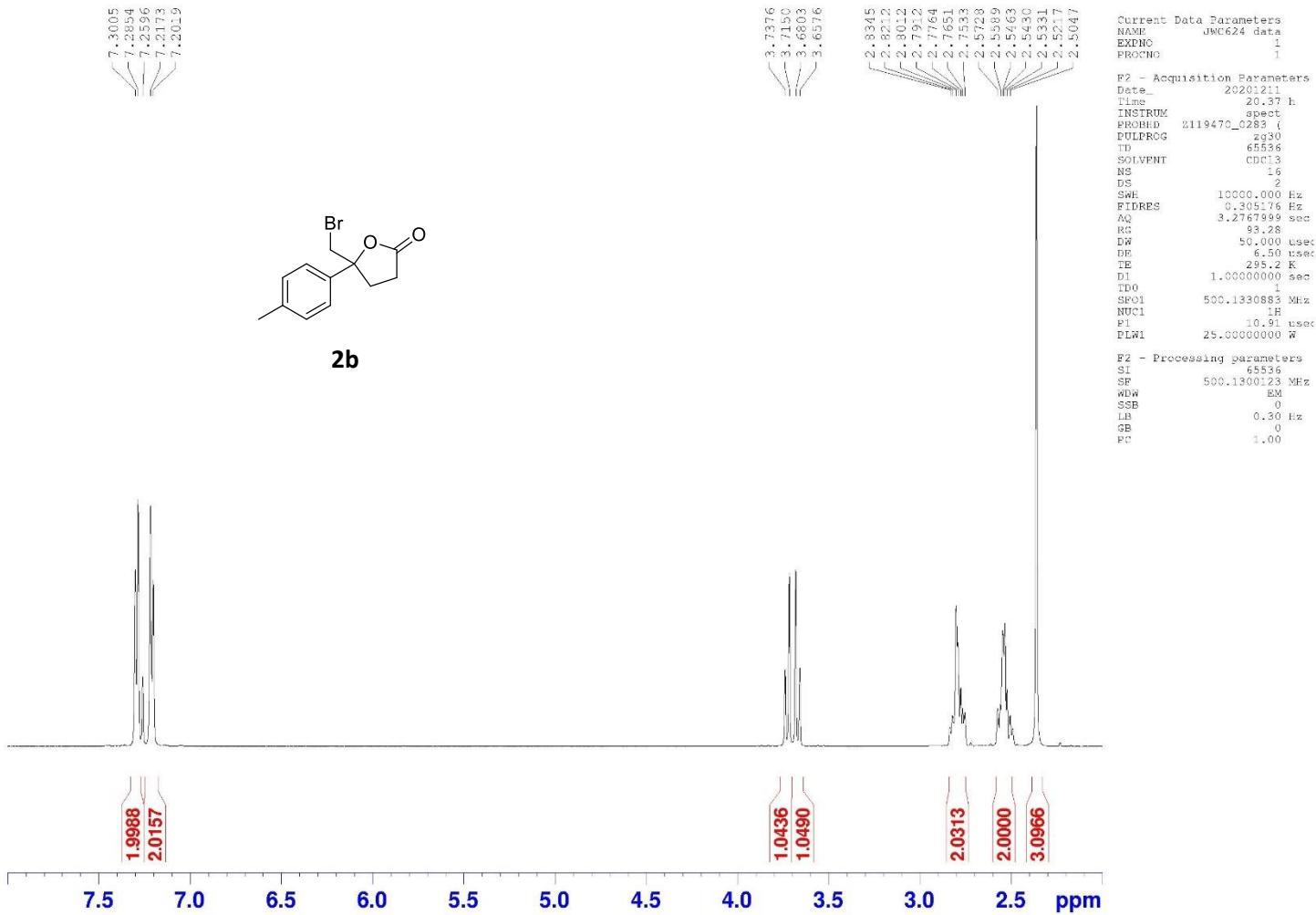
5,5-dimethylhydantoin recovered from bromoesterification of **7a** and **8h**.

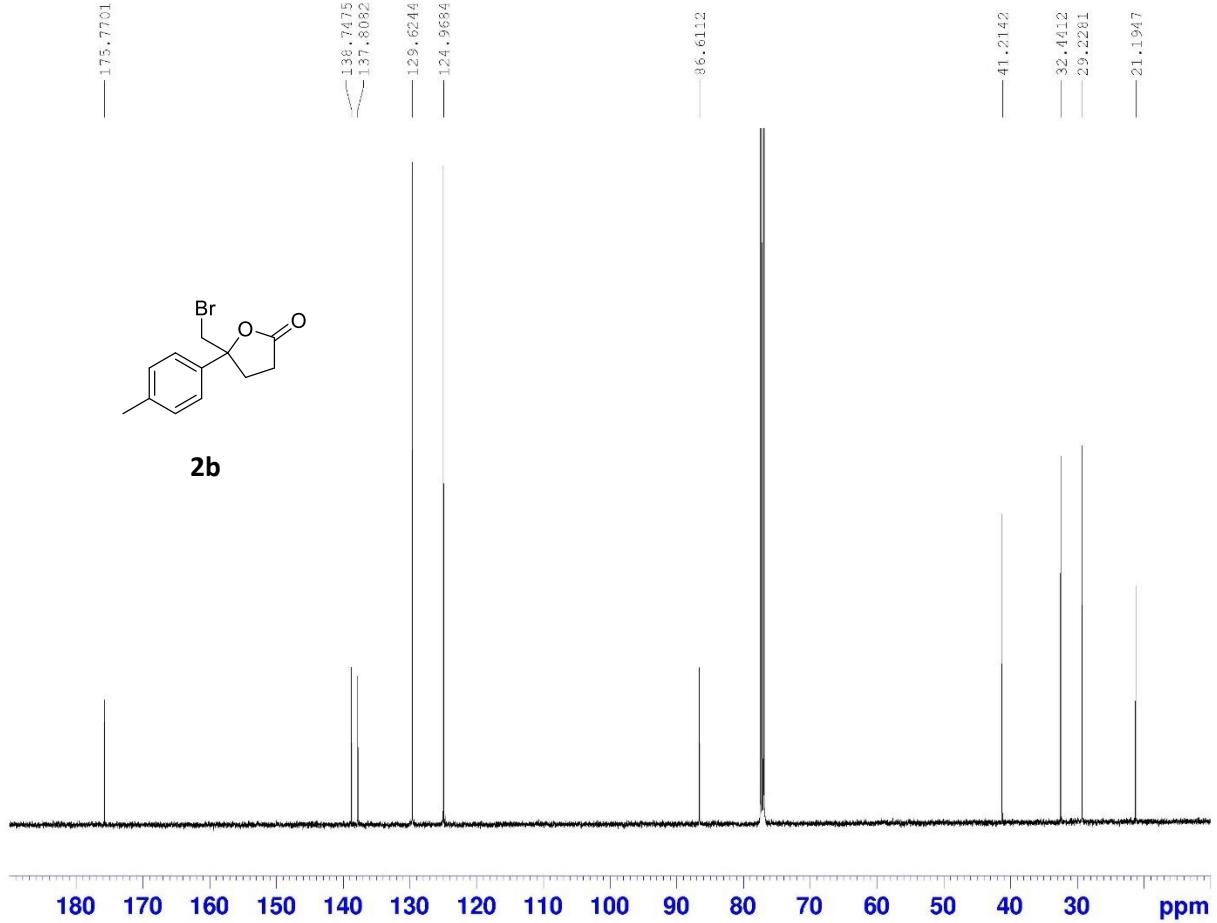


(G)  $^1\text{H}$  and  $^{13}\text{C}$  NMR Spectra



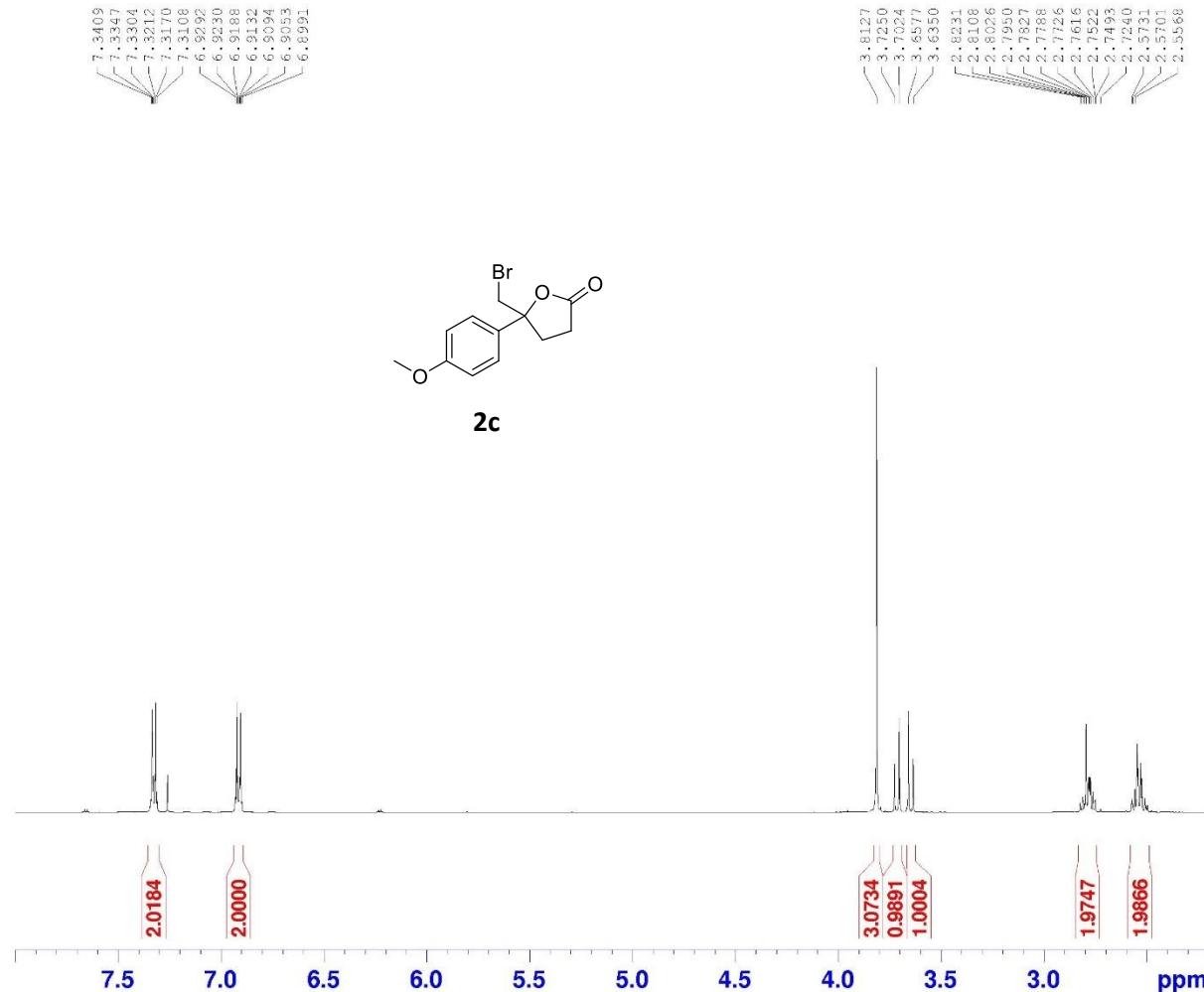






Current Data Parameters  
NAME JWC624\_data  
EXPNO 2  
PROCNO 1  
PULPROG zgpp30  
TD 65536  
T 1024  
SOLVENT CDCl3  
NS 1024  
DS 1  
SW0 29761.904 Hz  
FIDRES 0.908261 Hz  
AQ 1.1010048 sec  
RG 205.72  
DW 16.800 usec  
DE 6.50 usec  
TE 295.2 K  
D1 2.0000000 sec  
DL 0.03000000 sec  
DP0  
SW1 125.7703643 MHz  
NUC1 13C  
PL 9.75 usec  
PLW1 94.00000000 W  
SF02 500.1320005 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 80.00 usec  
PLW2 25.00000000 W  
PLW12 0.46495000 W  
PLW13 0.23387000 W

P2 - Processing parameters  
SI 32768  
SF 125.7665657 MHz  
WDW MM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



```

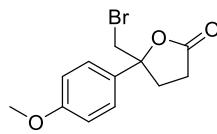
Current Data Parameters
NAME        JWCG468 data
EXPNO      1
PROCNO     1

F2 - Acquisition Parameters
Data_1      20201116
Time       18.22 h
INSTRUM   spect
PULPROG   z119470_02d1s
TD        65536
TDS       16
SWH      10000.000 Hz
ETDRES   0.305176 T
AQ        3.2787999 sec
RG        83.35
DW        50.0000 usec
DE        6.50
TE        295.2
TM        1.0000000 sec
D1        1.0000000 sec

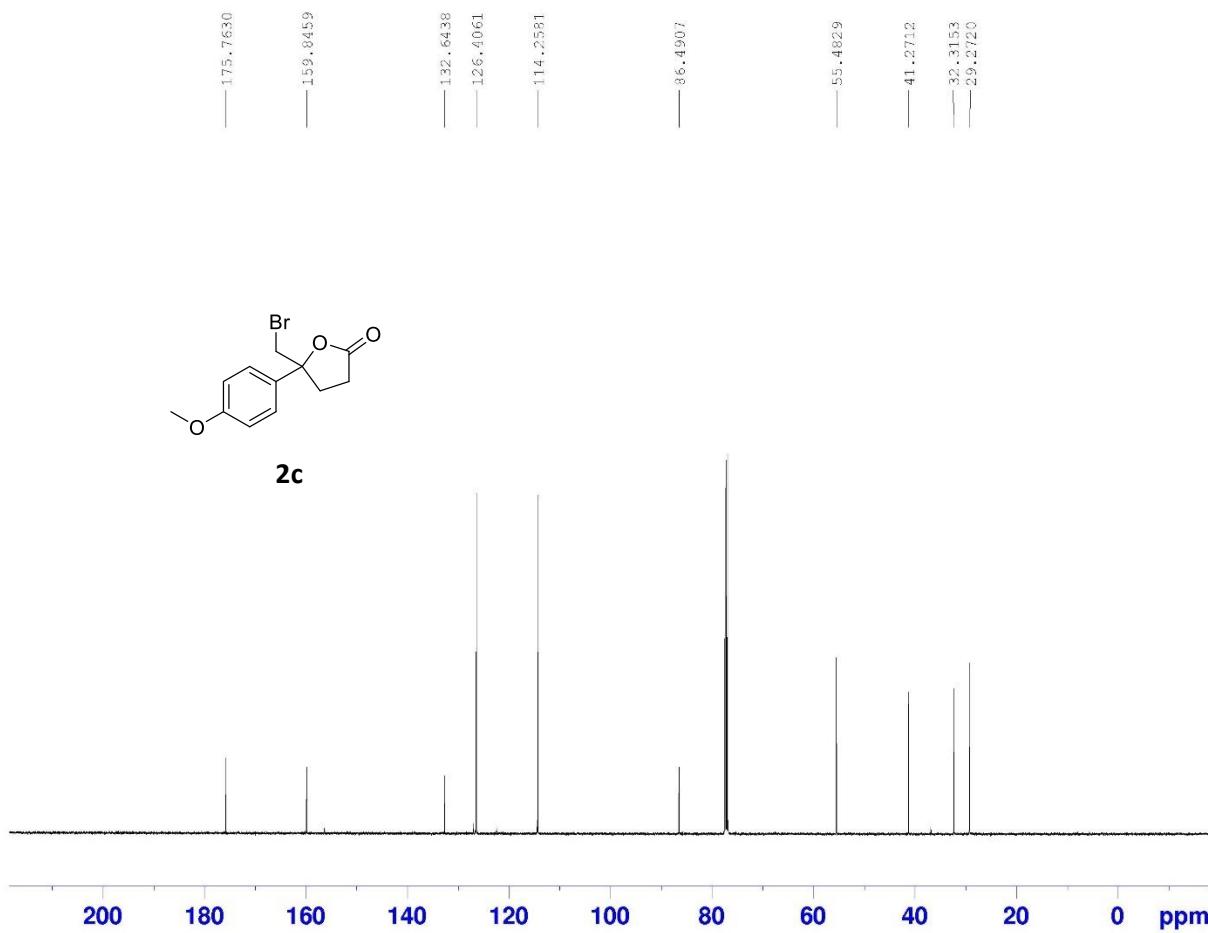
TDO      1
SFO1    500.1330883 MHz
NUC1      1H
P1        10.91 usec
PLW1    25.00000000 W

F2 - Processing parameters
SI        65536
SP      500.1300125 MHz
WDW      EM
SSSB      0
LB        0.30 Hz
GB        0
EC        1.00

```



**2c**

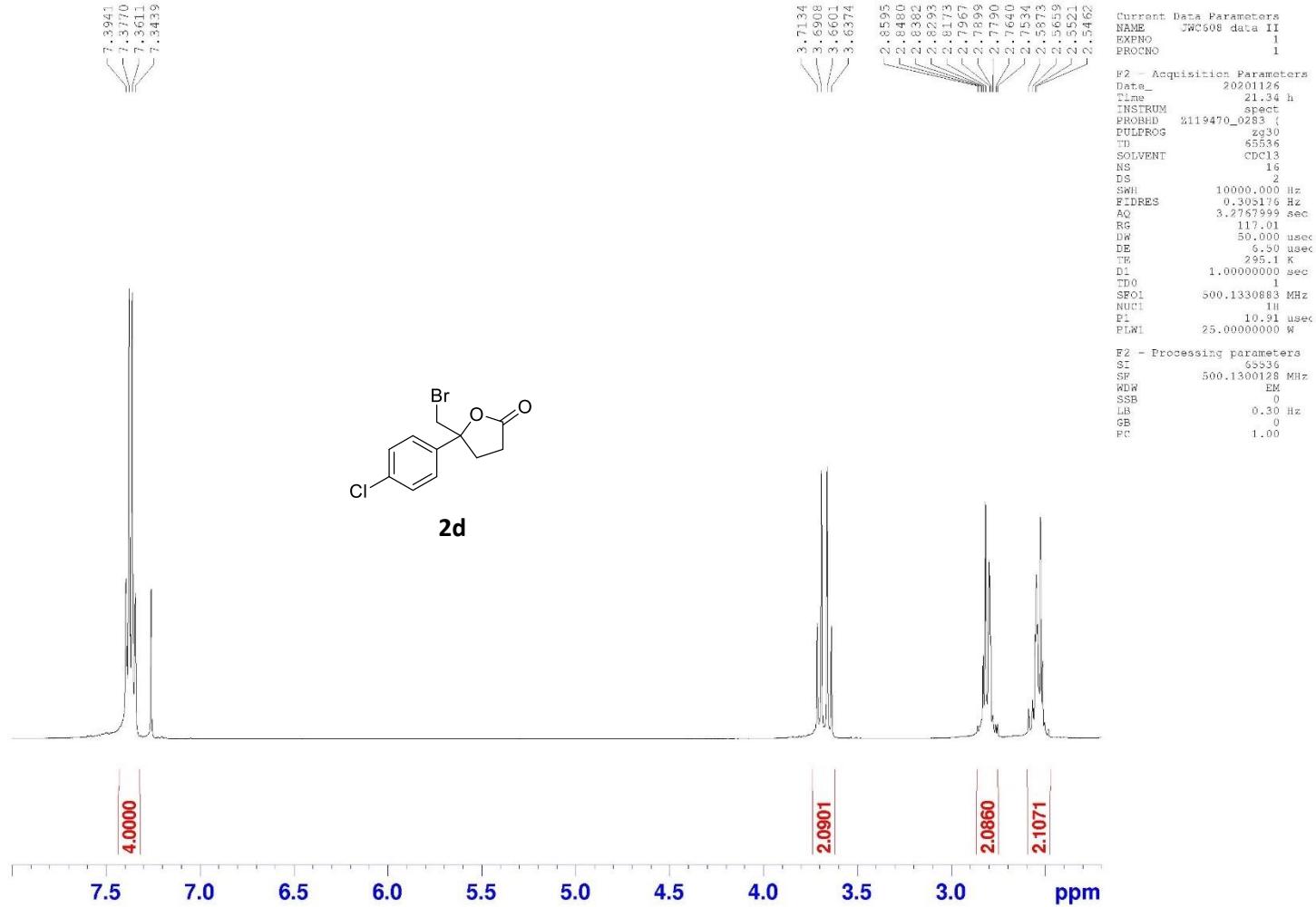


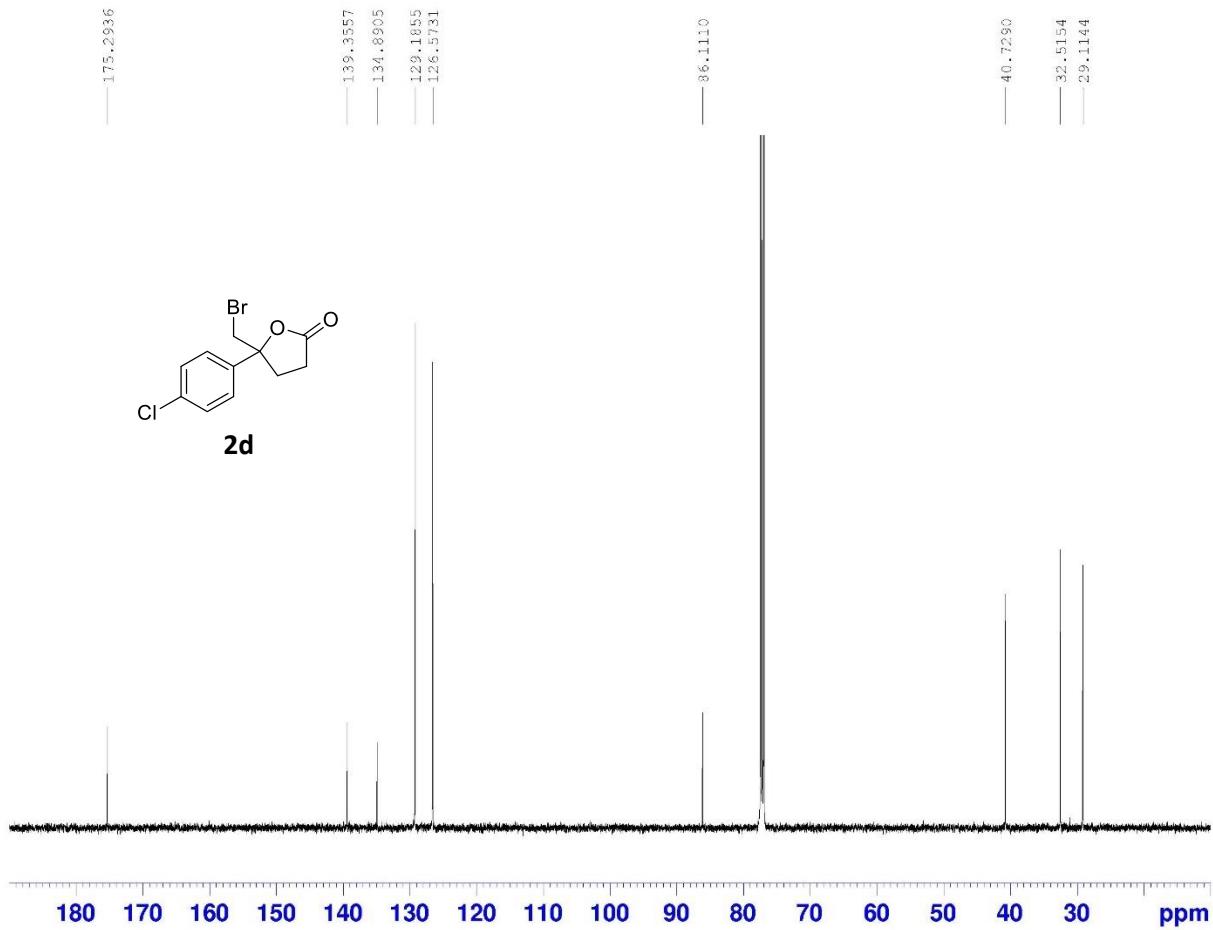
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Current Data Parameters
NAME      JWC468
EXPNO     2
PROCNO    1
Pulse 90°
TD        65536
SOLVENT   CDCl3
TMS
DS        700
TE        90.000000 sec
RG        205.72
DW        16.800 usec
DE        6.50 usec
TM        295.2 K
D1        2.0000000 sec
DL        0.0300000 sec
DPG      1
D1W      125.7703643 MHz
NUC1     13C
PL        9.75 usec
PLW1    94.00000000 w
SF02    500.1320005 MHz
NUC2     1H
CPDPRG[2] waltz16
PCPD2    80.00 usec
PLW2    25.00000000 w
PLW12   0.46495000 w
PLW13   0.23387000 w

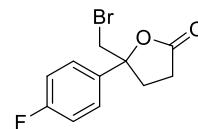
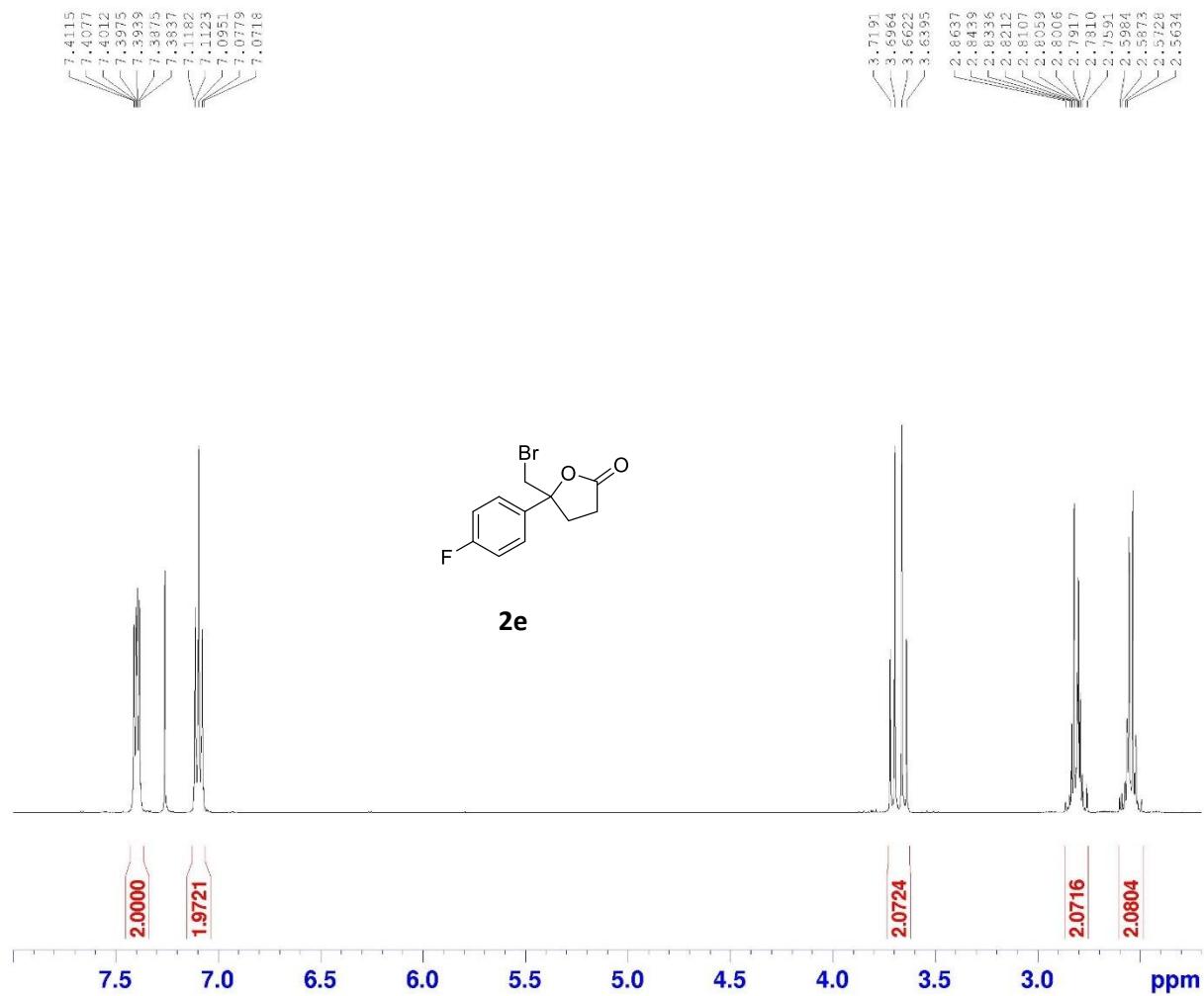
P2 - Processing parameters
SI        32768
SF        125.7577761 MHz
WDW
SSB
LB        1.00 Hz
GB
PC        1.40

```

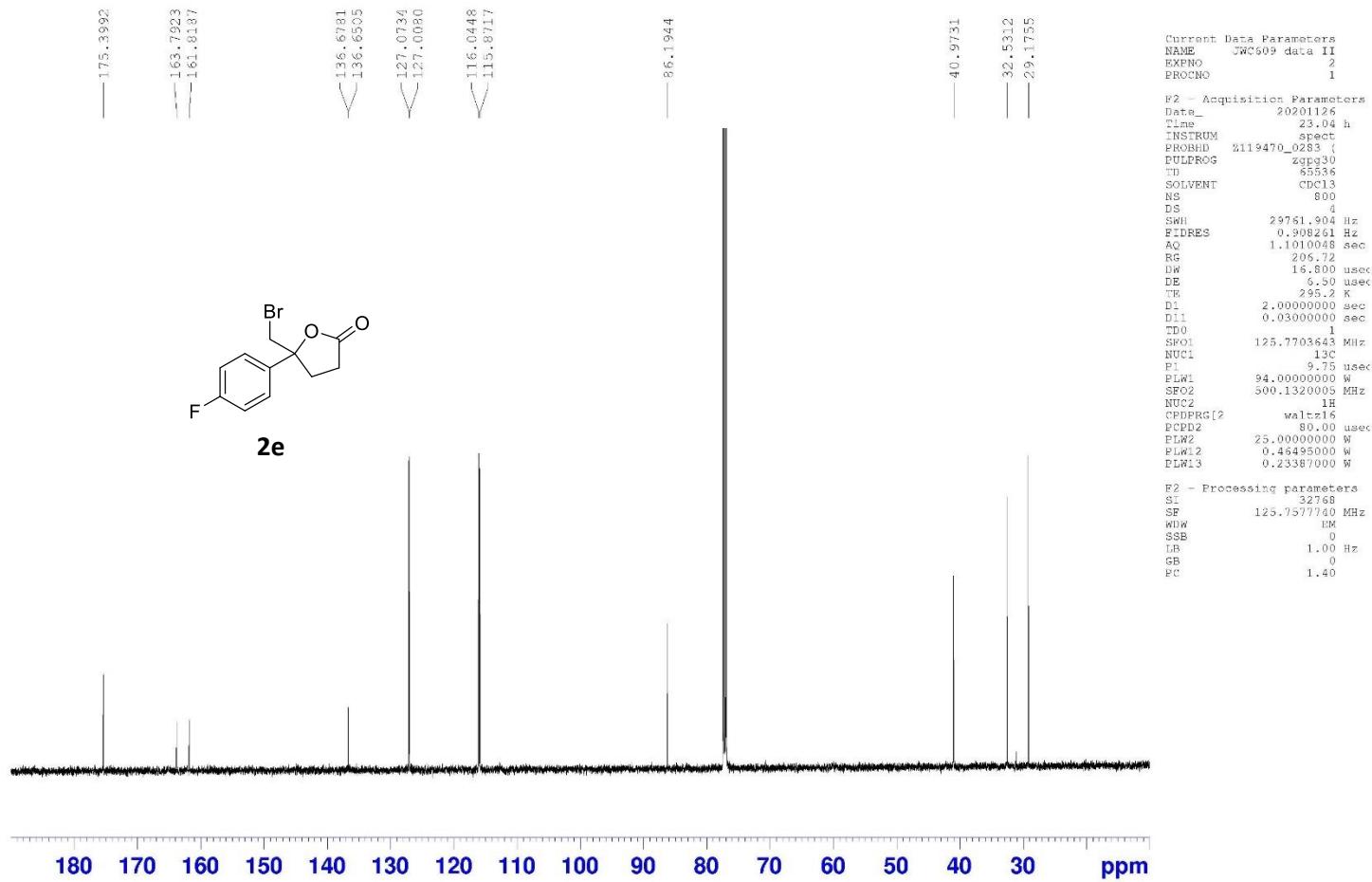


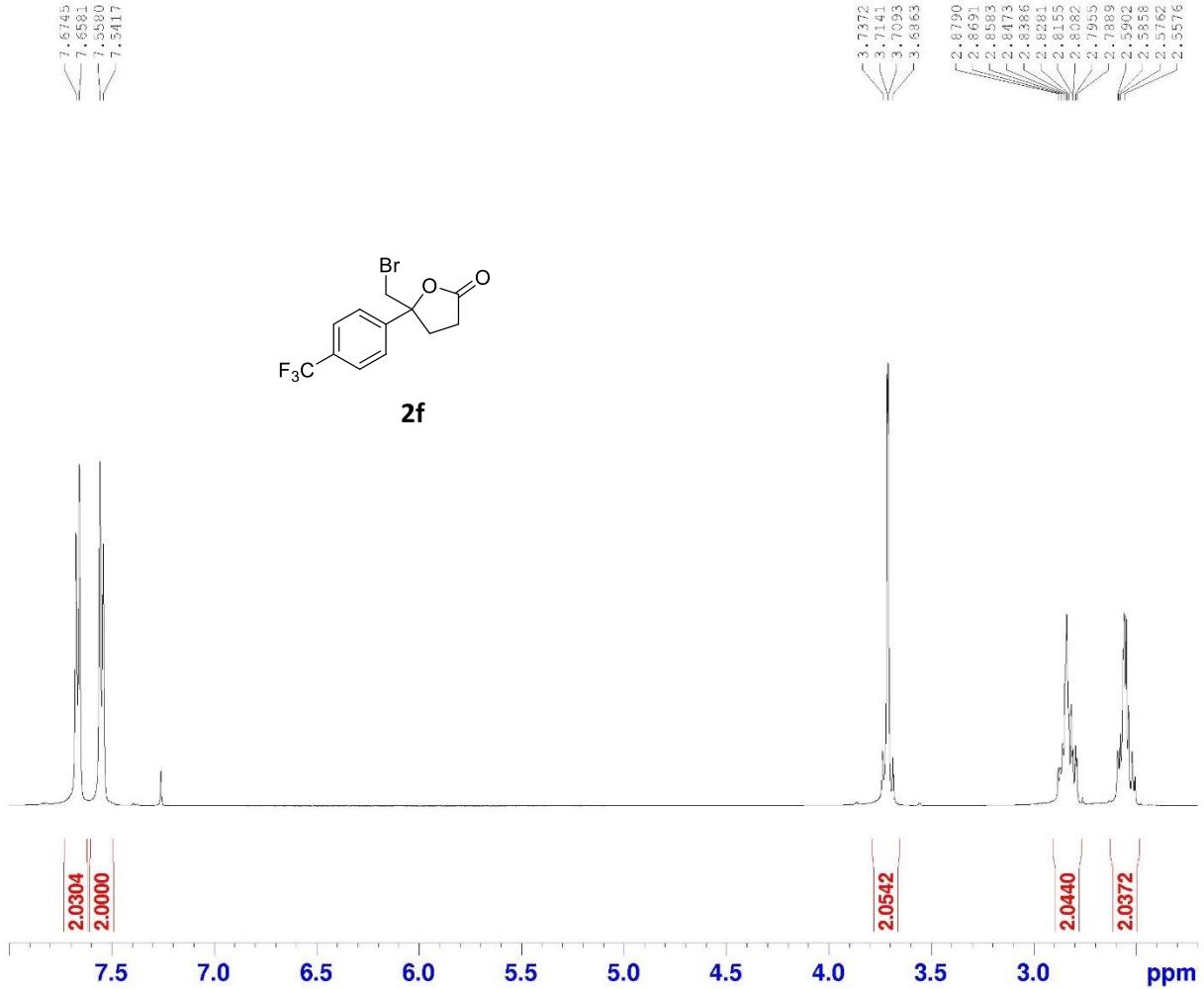


Current Data Parameters  
NAME JWC908 data II  
EXPNO 2  
PROCNO 1  
  
P2 - Acquisition Parameters  
Data\_20201126  
Time 22.17 h  
INSTRUM spect  
PROBHD Z119470\_0283  
PULPROG zgpp30  
TD 65536  
T1 65536  
SOLVENT CDCl3  
NS 600  
DS 8  
SW0 29761.904 Hz  
FIDRES 0.908261 Hz  
AQ 1.1010048 sec  
RG 205.72  
DW 16.800 usec  
DE 6.50 usec  
TE 295.2 K  
D1 2.0000000 sec  
DL 0.03000000 sec  
DP0  
SW1 125.7703643 MHz  
NUC1 13C  
P1 9.75 usec  
PLW1 94.00000000 w  
SF02 500.1320005 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 80.00 usec  
PLW2 25.00000000 w  
PLW12 0.46495000 w  
PLW13 0.23387000 w  
  
P2 - Processing parameters  
SI 32768  
SF 125.7577744 MHz  
WDW MM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



**2e**

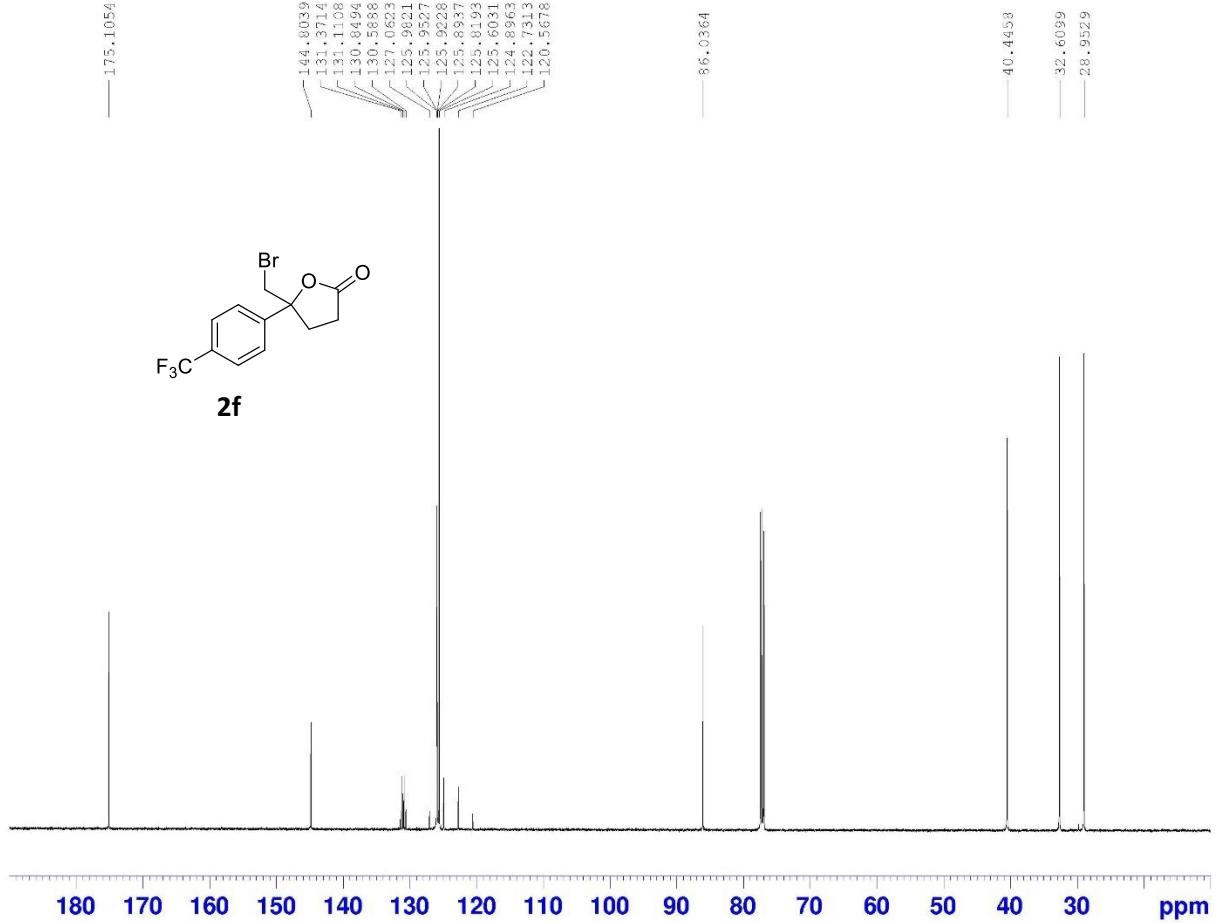


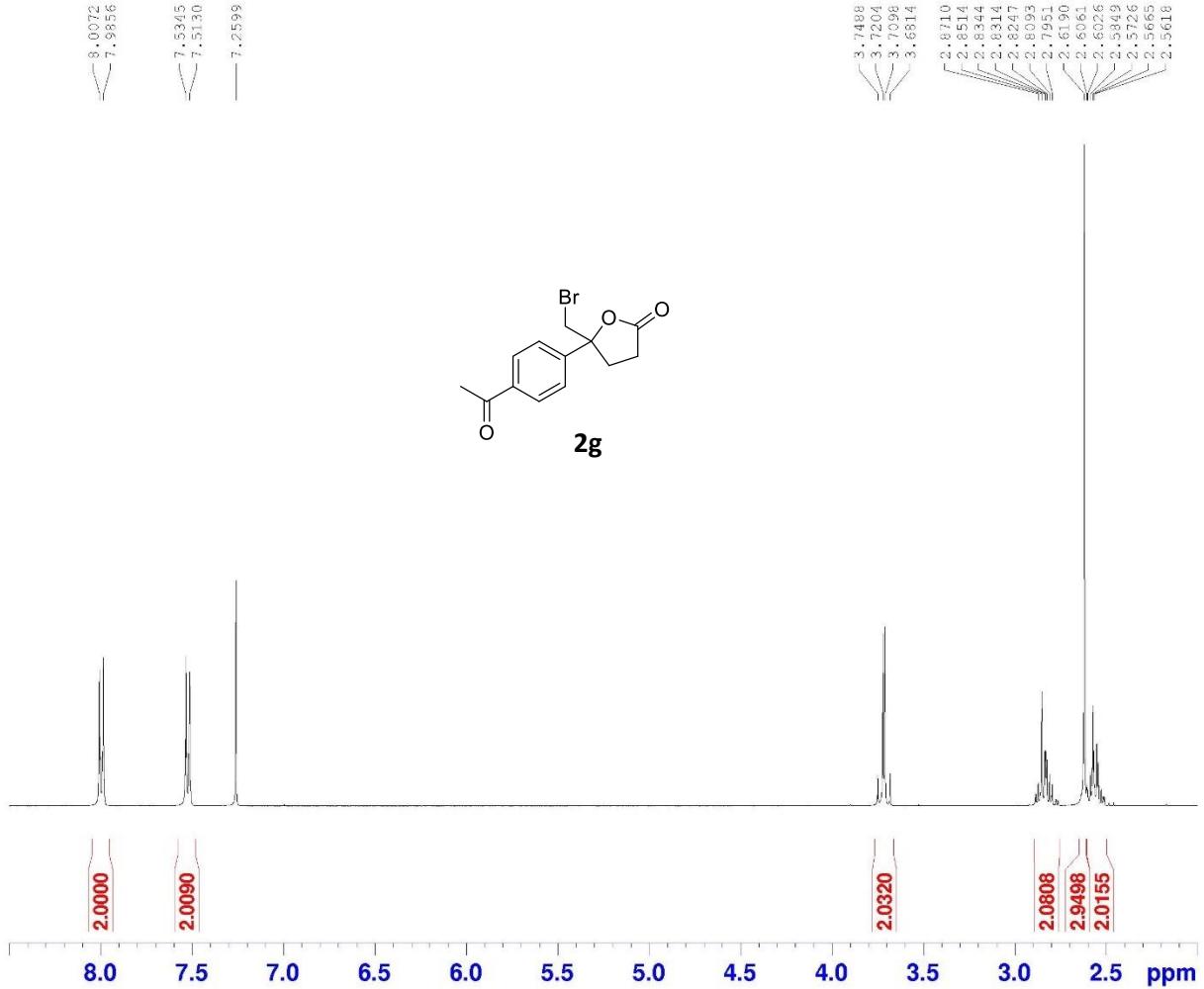


Current Data Parameters  
NAME JWC472 data  
EXPNO 2  
PROCNO 1

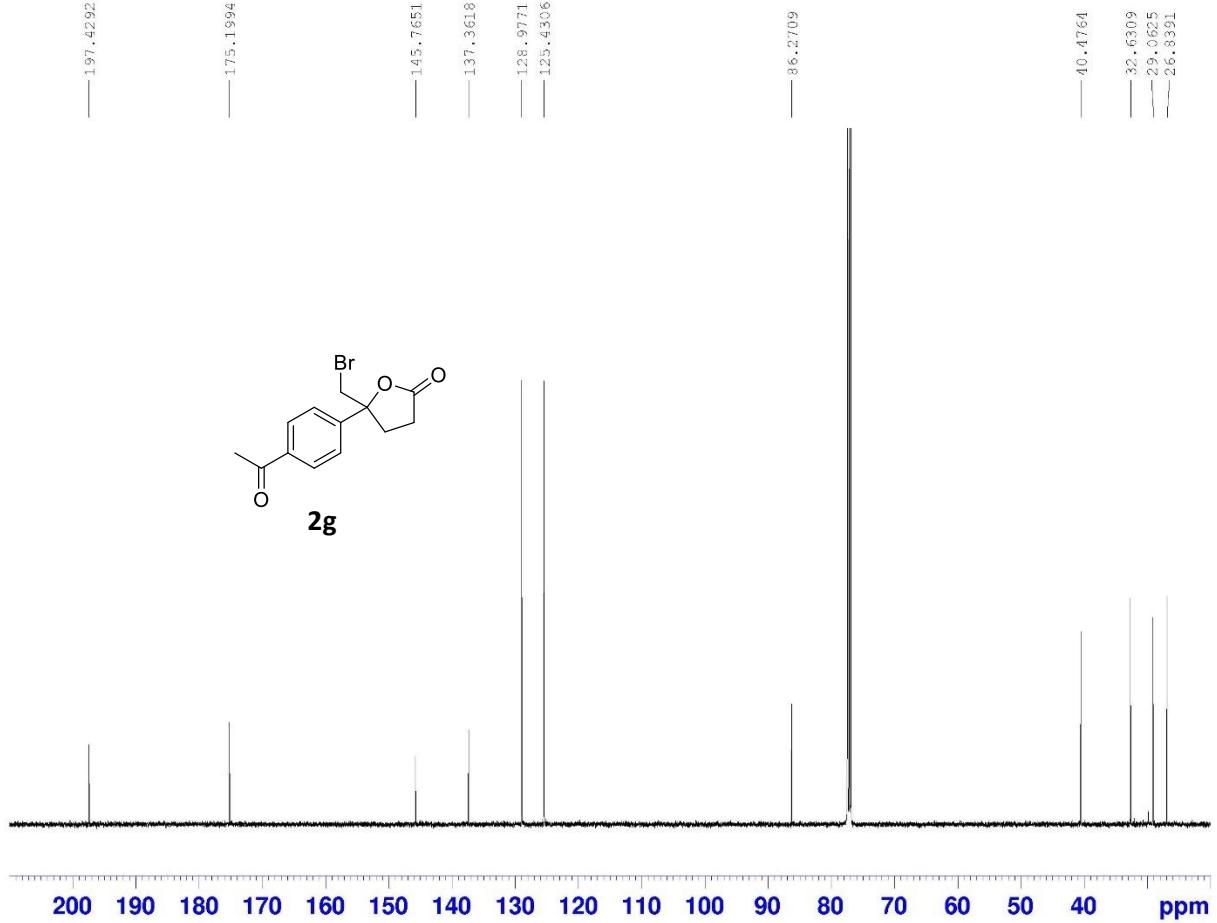
P2 - Acquisition Parameters  
Data- 20201116  
Time- 18.27 h  
INSTRUM spect  
PROBHD Z119470\_0283 {  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SW0 10000.000 Hz  
FIDRES 0.305176 Hz  
AQ 3.2757999 sec  
RG 30.85  
DW 50.000 usec  
DE 6.50 usec  
TE 295.2 K  
D1 1.0000000 sec  
TD0 500.1330683 MHz  
RNUC 1H  
P1 10.91 usec  
FWHM 25.0000000 W

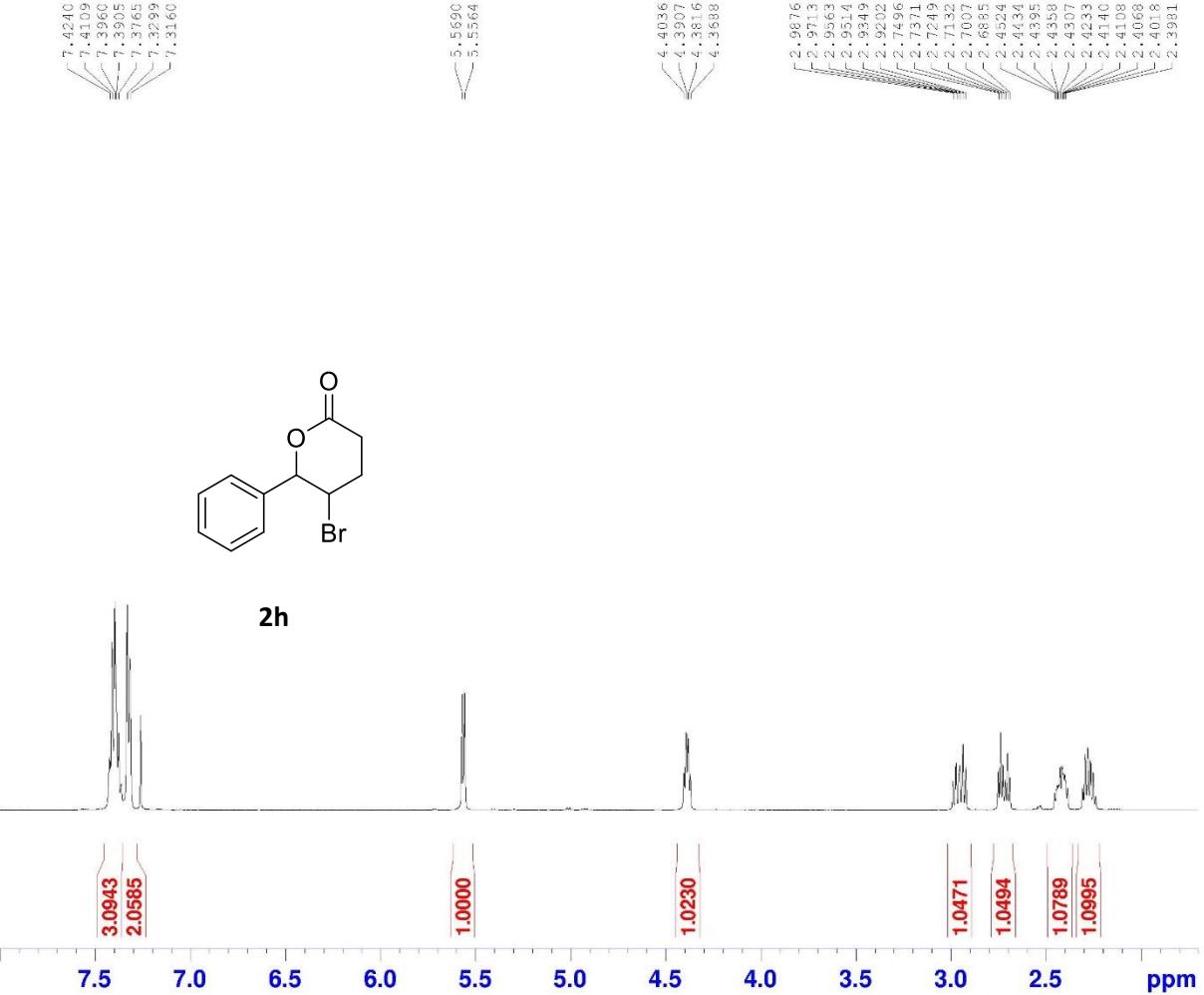
P2 - Processing parameters  
SI 65536  
SF 500.1300128 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

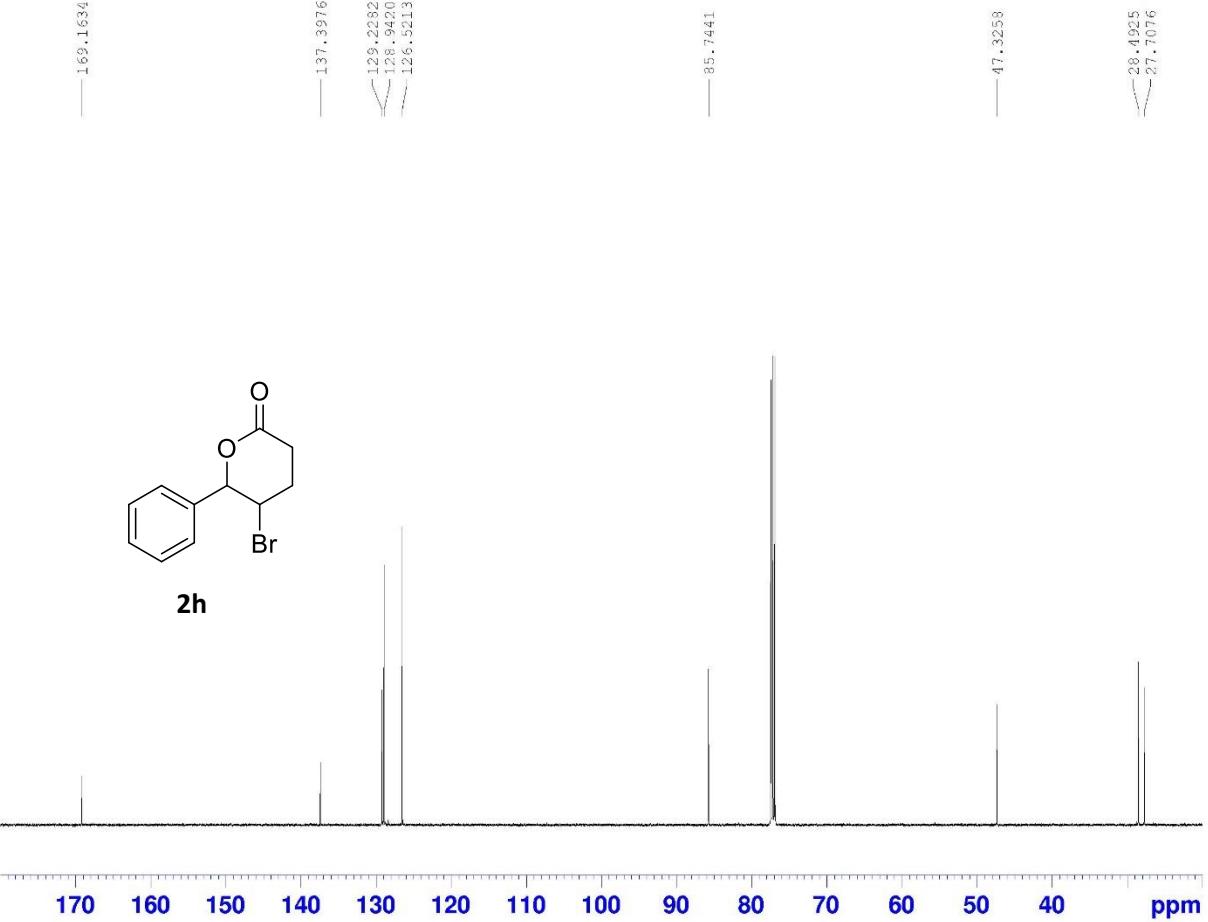




Current Data Parameters  
NAME JWC4731I.p  
EXPNO 1  
PROCNO 1  
P2 - Acquisition Parameters  
Data- 20200221  
Time 14.54 h  
INSTRUM spect  
PROBHD ZS20201\_0170 {  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SW0 8012.820 Hz  
FIDRES 0.244532 Hz  
AQ 4.0894465 sec  
RG 203  
DW 62.400 usec  
DE 6.50 usec  
TE 293.2 K  
D1 1.0000000 sec  
TD0 400.1324709 MHz  
RNUC 1H  
P1 6.75 usec  
FW1 13.17700005 W  
P2 - Processing parameters  
SI 65536  
SF 400.1300097 MHz  
RDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



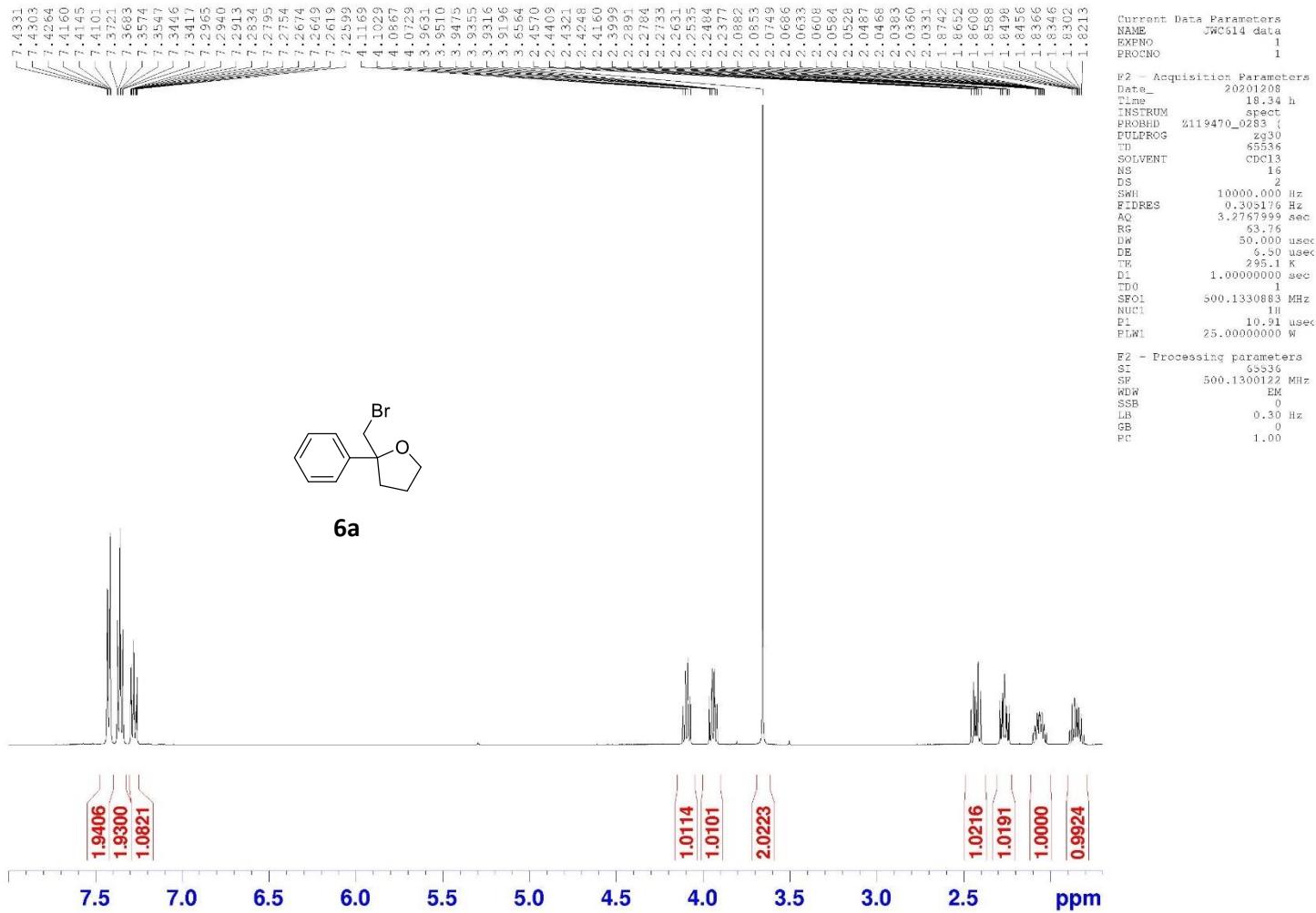


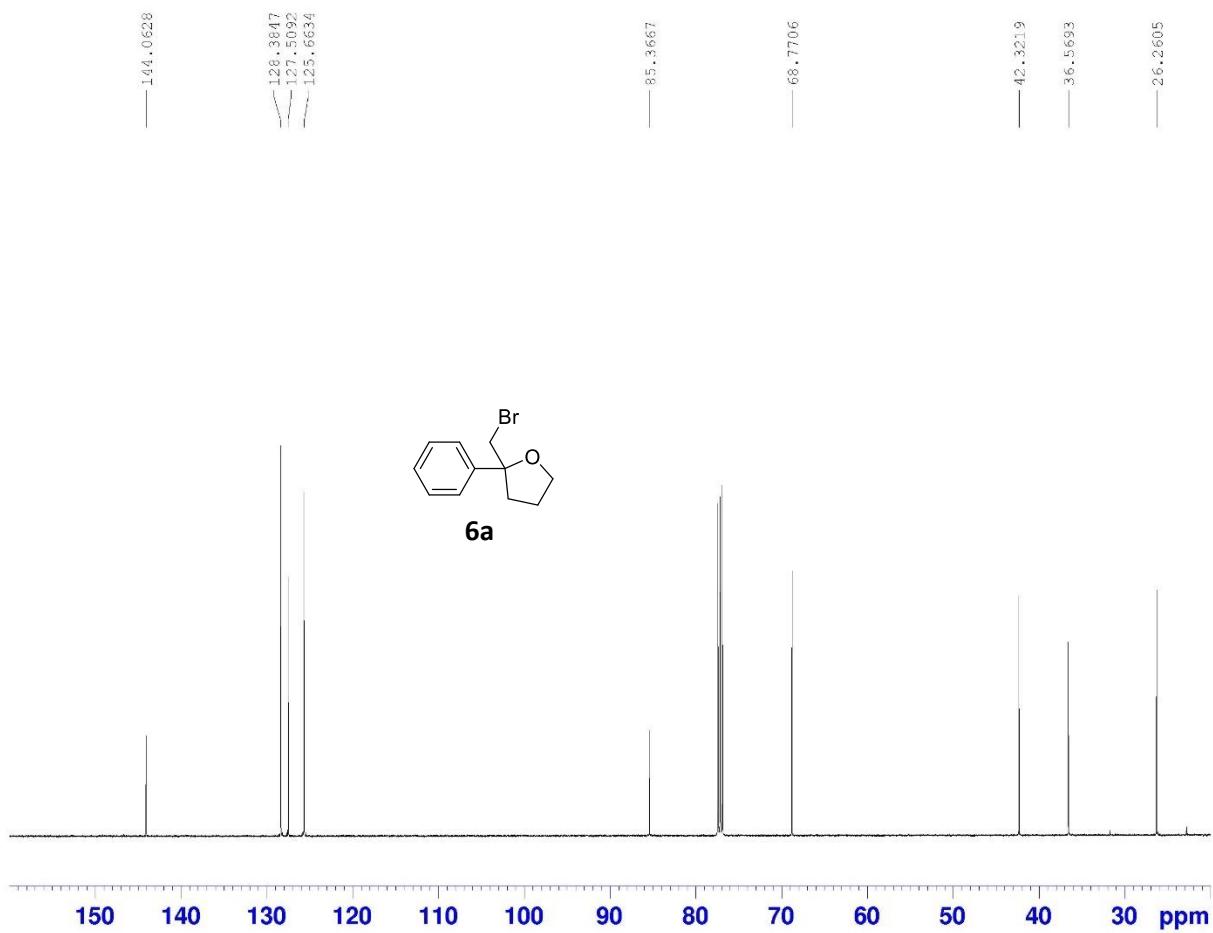


Current Data Parameters  
 NAME JWC634\_data  
 EXPNO 2  
 PROCNO 1

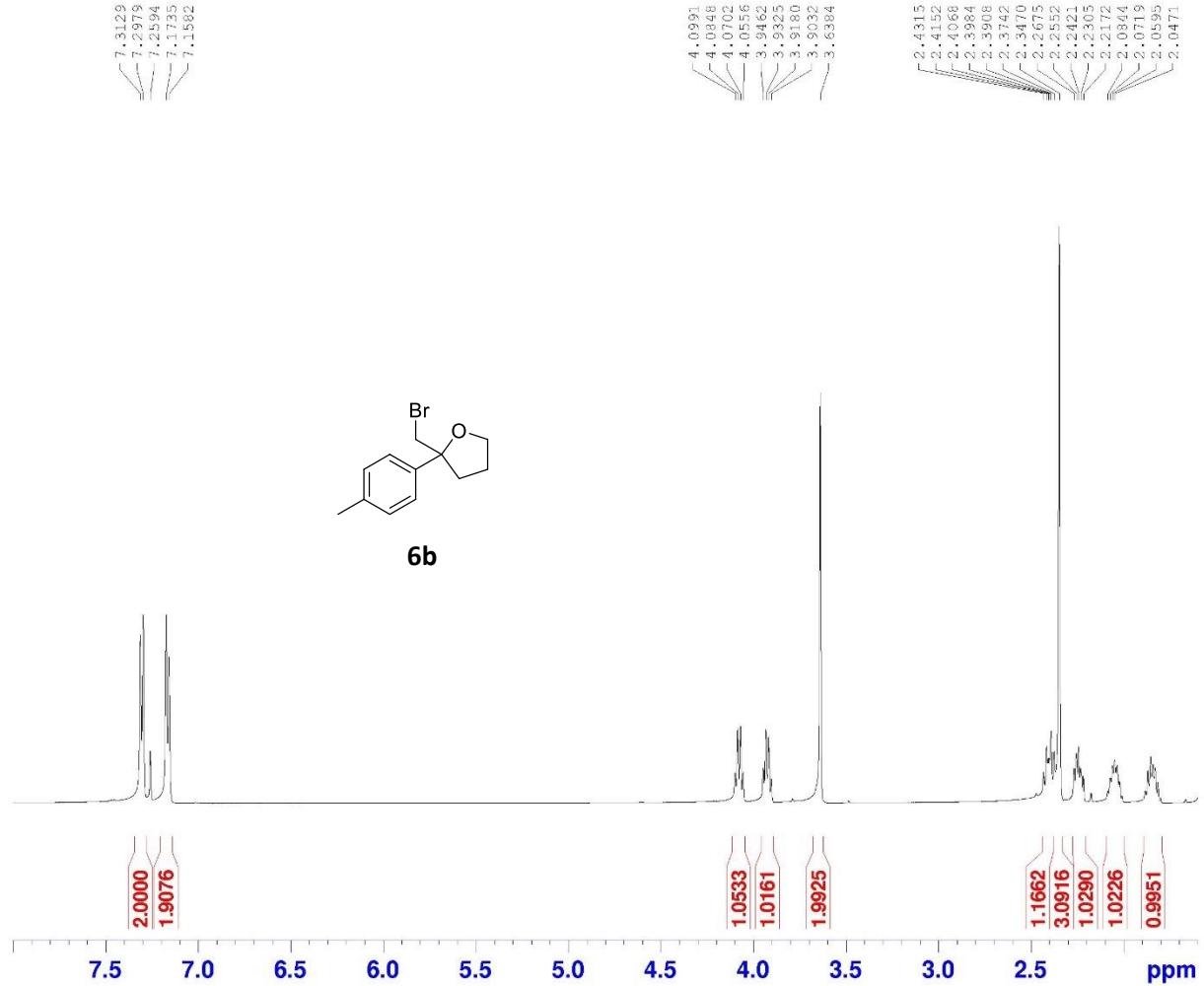
P2 - Acquisition Parameters  
 Date 20210113  
 Time 21.29 h  
 INSTRUM spect  
 PROBHD Z119470\_0283 {  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 RSL 1024  
 DS 1  
 SWH 29761.904 Hz  
 FIDRES 0.908261 Hz  
 AQ 1.1010048 sec  
 RG 142.5  
 DW 16.800 usec  
 DE 6.50 usec  
 TM 295.2 K  
 D1 2.00000000 sec  
 DL 0.03000000 sec  
 TDO 125.7703643 MHz  
 NUC1 13C  
 PL 9.75 usec  
 PLW1 94.00000000 W  
 SF02 500.1320005 MHz  
 NUC2 1H  
 CPDPFG[2] waltz16  
 PCPD2 80.00 usec  
 PLW2 25.00000000 W  
 PLW12 0.45495000 W  
 PLW13 0.23387000 W

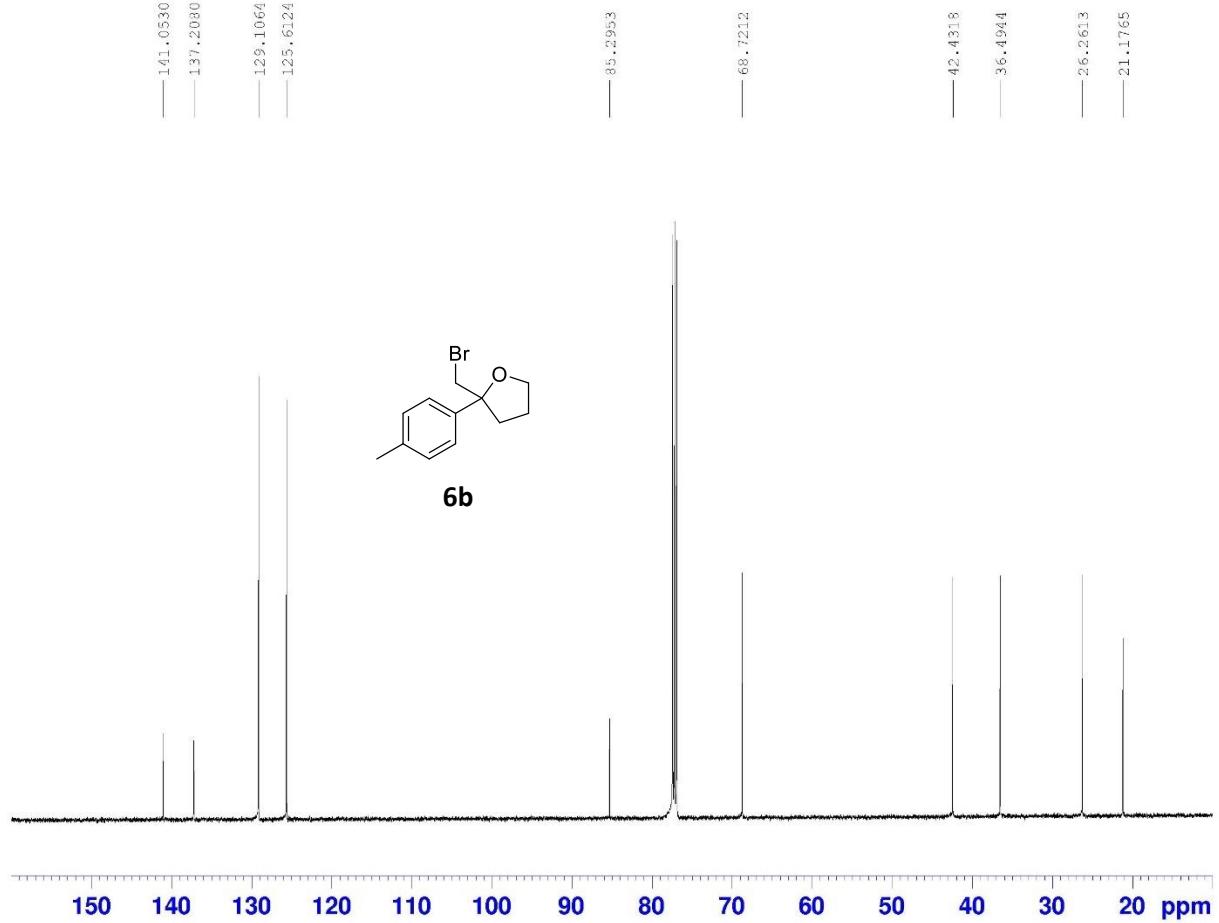
P2 - Processing parameters  
 SI 32768  
 SF 125.7577753 MHz  
 RDW 1MM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40



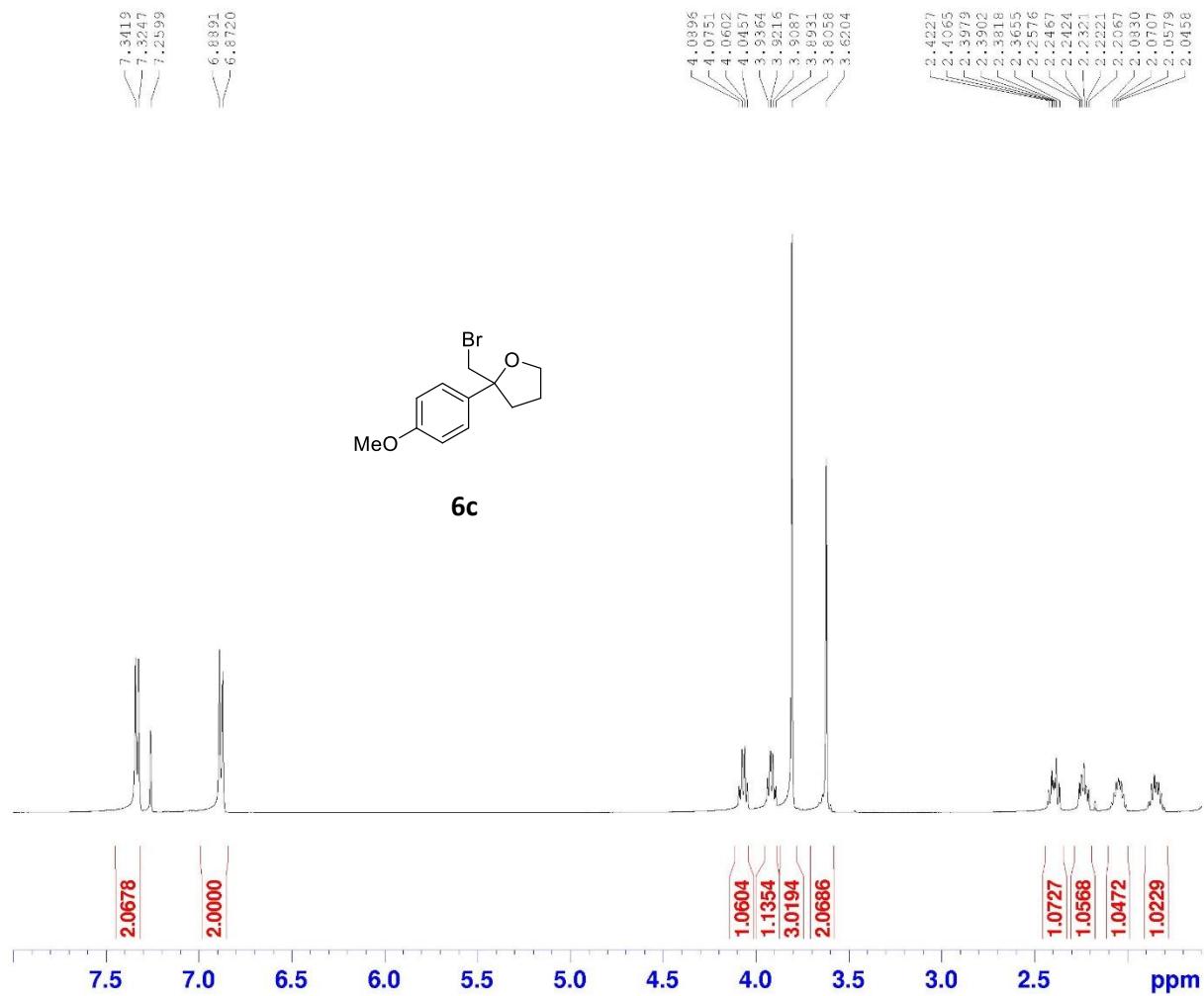


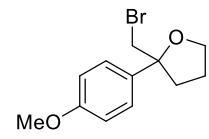
Current Data Parameters  
NAME JWC614 data  
EXPNO 2  
PROCNO 1  
  
P2 - Acquisition Parameters  
Data\_20201208  
Time 21.27 h  
INSTRUM spect  
PROBHD Z119470\_0283\_1  
PULPROG zgpp30  
TD 65536  
T1 1024  
SOLVENT CDCl3  
NS 1024  
DS 1  
SW0 29761.904 Hz  
FIDRES 0.908261 Hz  
AQ 1.1010048 sec  
RG 205.72  
DW 16.800 usec  
DE 6.50 usec  
TE 295.1 K  
D1 2.0000000 sec  
DL 0.0300000 sec  
DP0  
SW1 125.7703643 MHz  
NUC1 13C  
P1 9.75 usec  
PLW1 94.00000000 w  
SF02 500.1320005 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 80.00 usec  
PLW2 25.00000000 w  
PLW12 0.46495000 w  
PLW13 0.23387000 w  
  
P2 - Processing parameters  
SI 32768  
SF 125.7577772 MHz  
WDW MM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



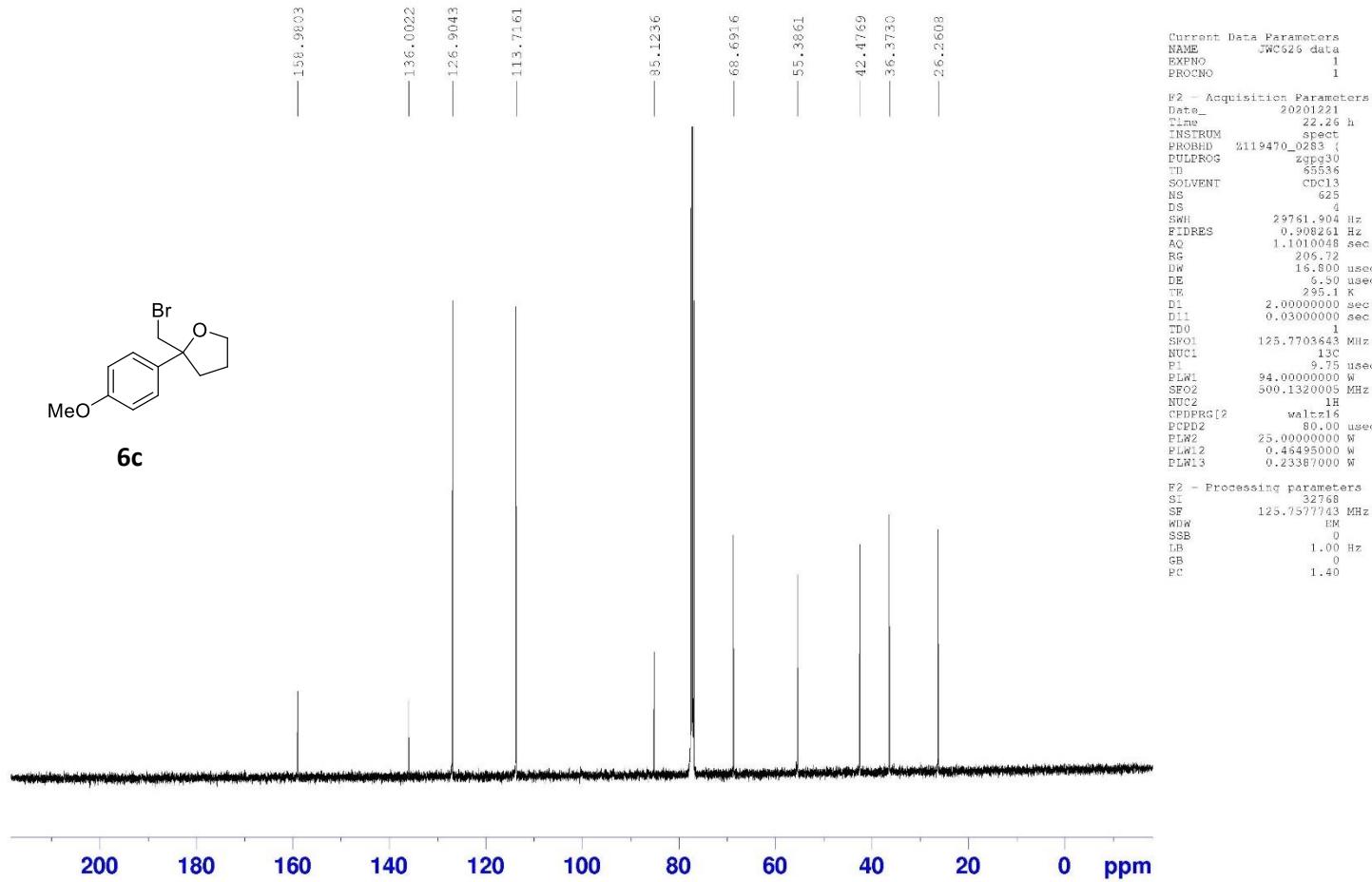


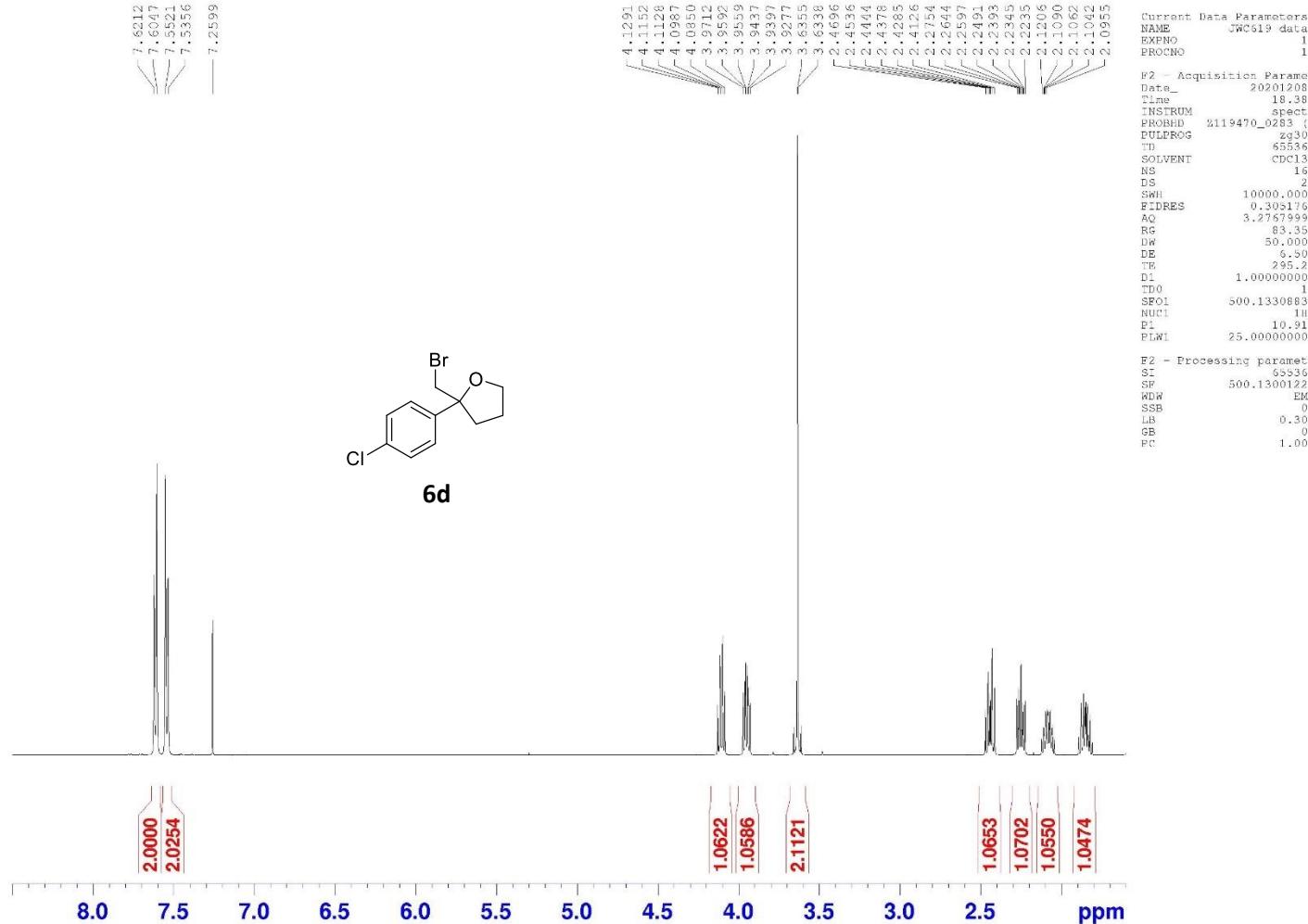
Current Data Parameters  
NAME JWC623 data  
EXPNO 1  
PROCNO 1  
  
P2 - Acquisition Parameters  
Data\_20201221  
Time 21.49 h  
INSTRUM spect  
PROBHD Z119470\_0283  
PULPROG zgpp30  
TD 65536  
SOLVENT CDCl3  
NS 1024  
DS 1  
SW0 29761.904 Hz  
FIDRES 0.908261 Hz  
AQ 1.1010048 sec  
RG 205.72  
DW 16.800 usec  
DE 6.50 usec  
TE 295.2 K  
D1 2.0000000 sec  
DL 0.0300000 sec  
DP0  
SW1 125.7703643 MHz  
NUC1 13C  
P1 9.75 usec  
PLW1 94.00000000 w  
SF02 500.1320005 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 80.00 usec  
PLW2 25.00000000 w  
PLW12 0.46495000 w  
PLW13 0.23387000 w  
  
P2 - Processing parameters  
SI 32768  
SF 125.7577752 MHz  
WDW MM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

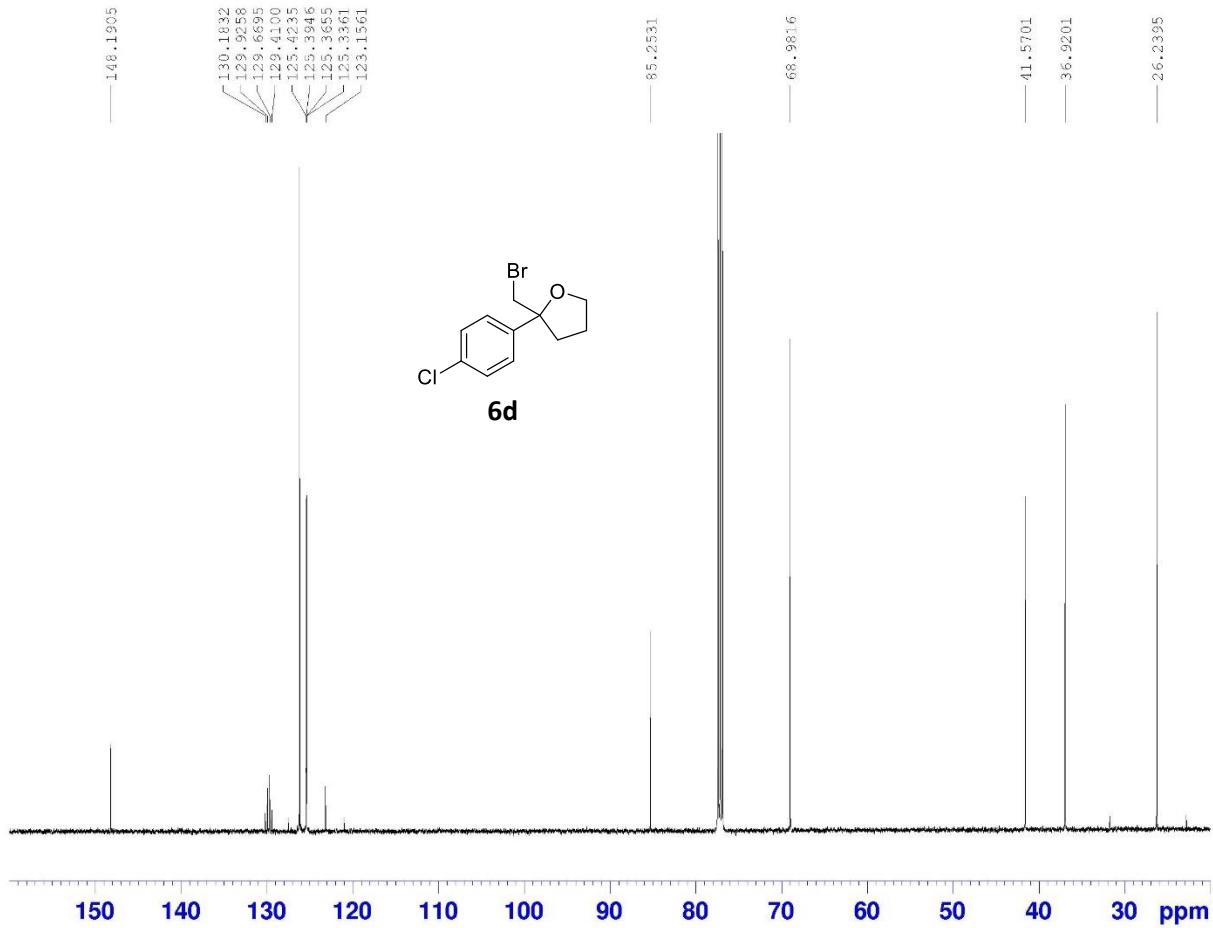


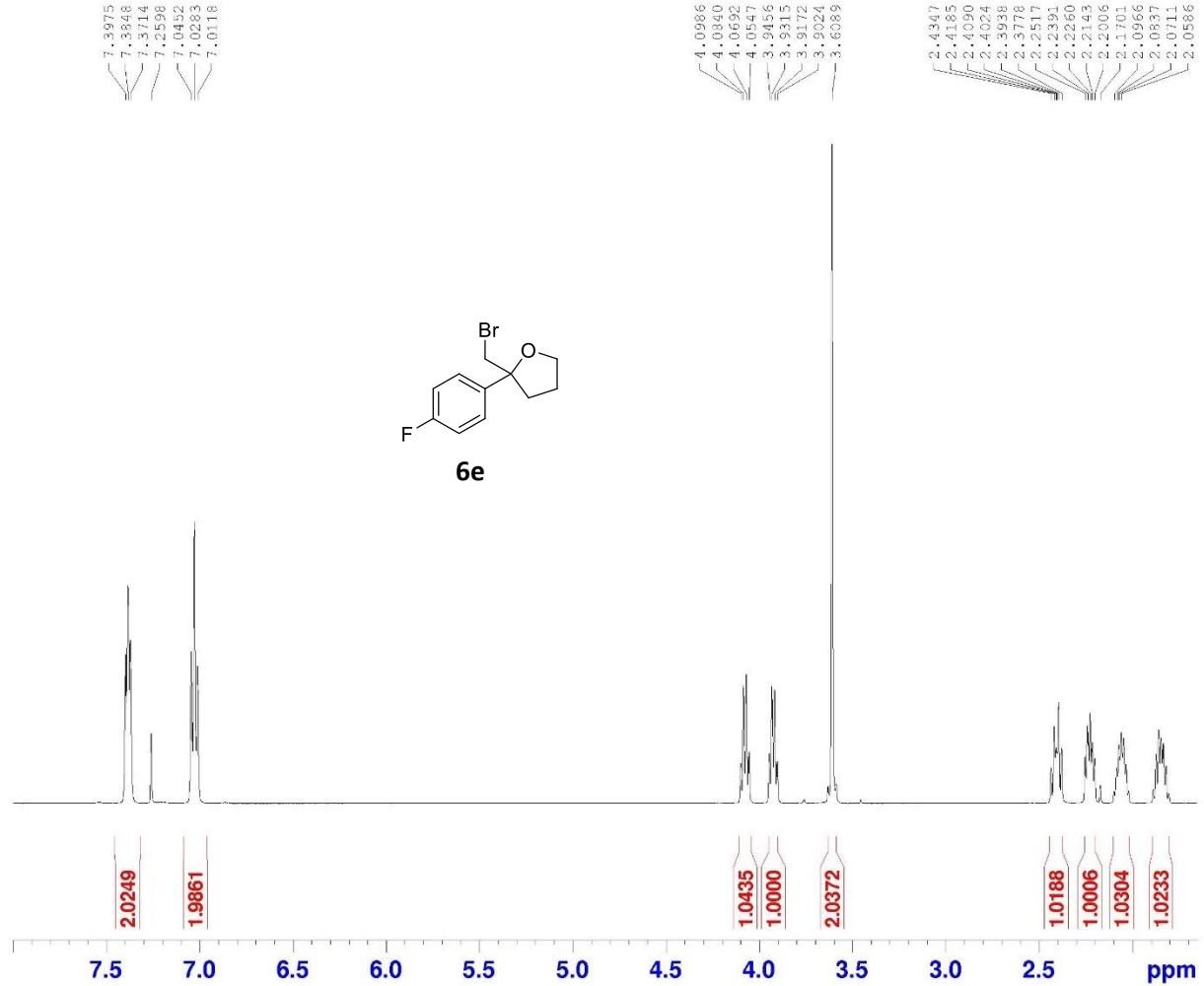


**6c**





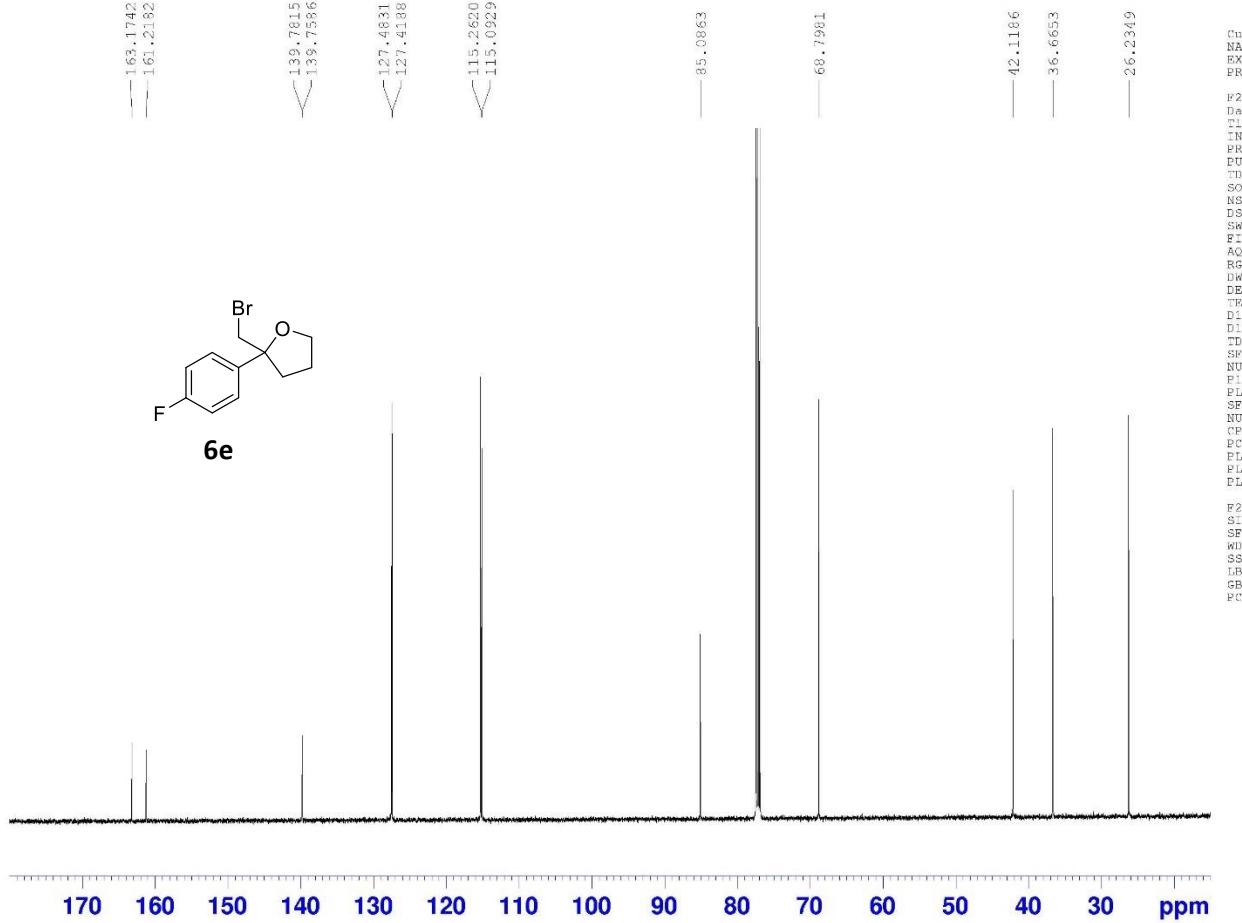


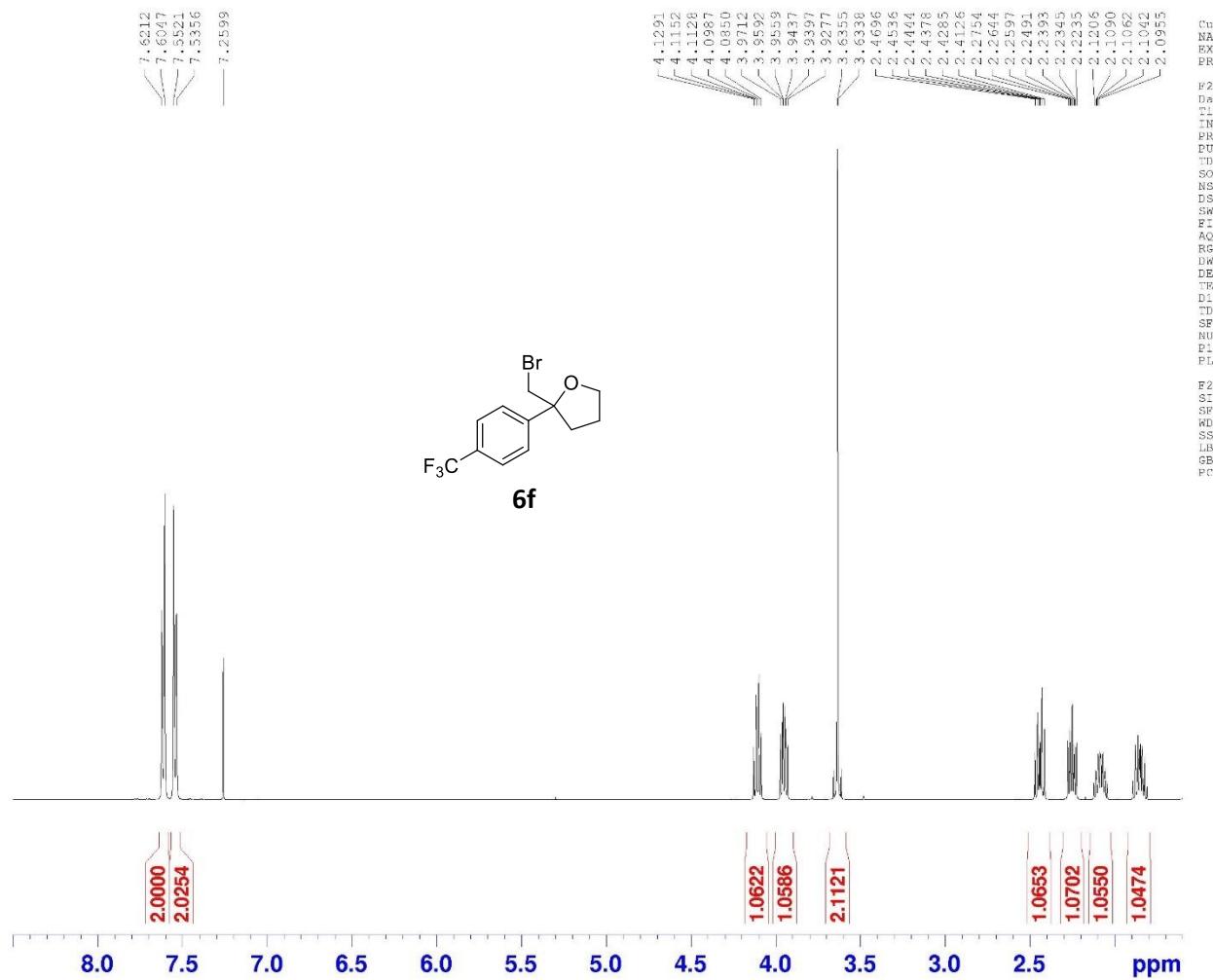


Current Data Parameters  
 NAME JWC620\_data  
 EXPNO 1  
 PROCNO 1

P2 - Acquisition Parameters  
 Data\_ 20201210  
 Time 22.38 h  
 INSTRUM spect  
 PROBHD Z119470\_0283 {  
 PULPROG zg30  
 TD 65536  
 T1 16  
 SOLVENT CDCl3  
 NS 16  
 DS 2  
 SWH 10000.000 Hz  
 FIDRES 0.305176 Hz  
 AQ 3.2757999 sec  
 RG 83.35  
 DW 50.000 usec  
 DE 6.50 usec  
 TE 295.1 K  
 D1 1.0000000 sec  
 ED0  
 SF01 500.1330883 MHz  
 RNUC 1H  
 F1 10.91 usec  
 FIDW 25.0000000 W

P2 - Processing parameters  
 SI 65536  
 SF 500.1300122 MHz  
 RDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 FC 1.00

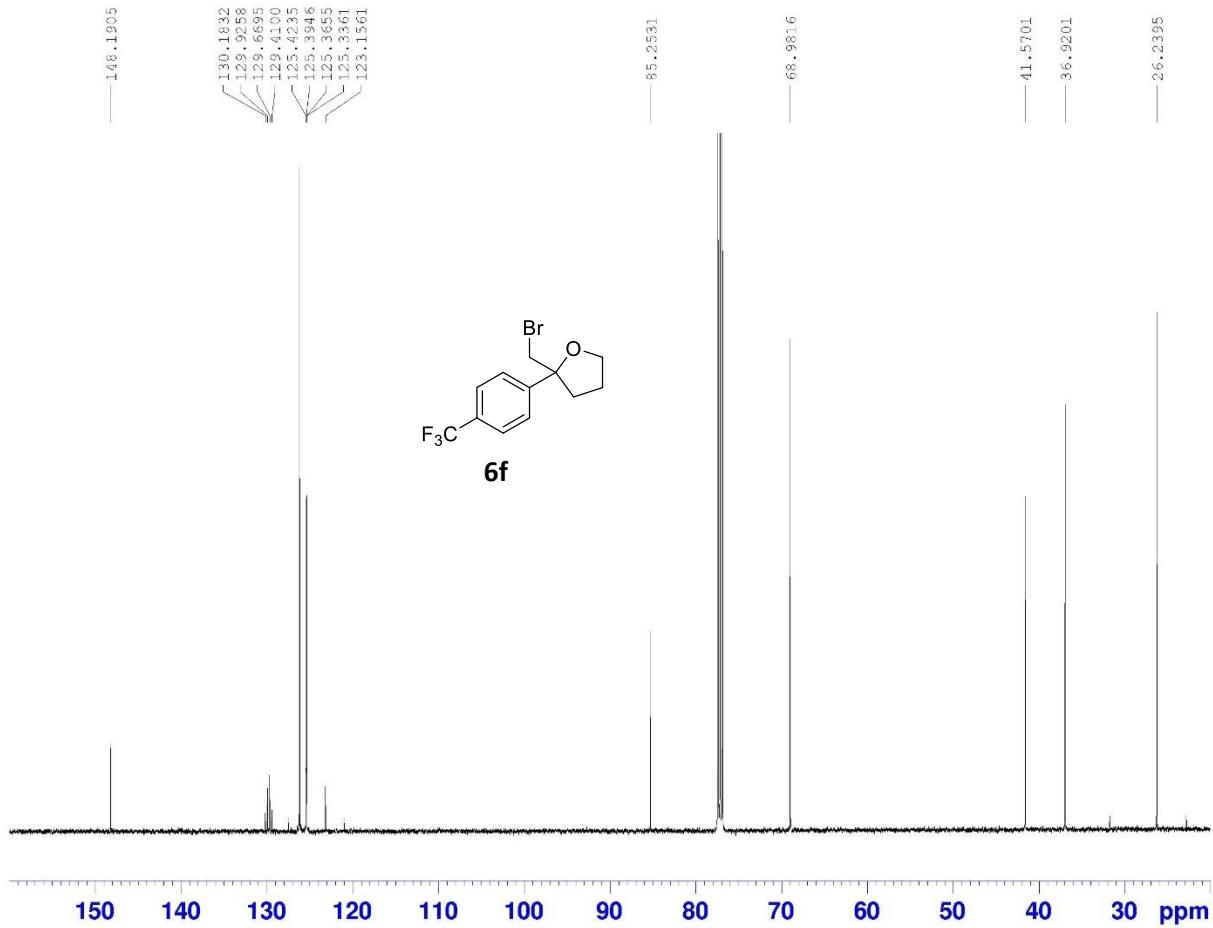


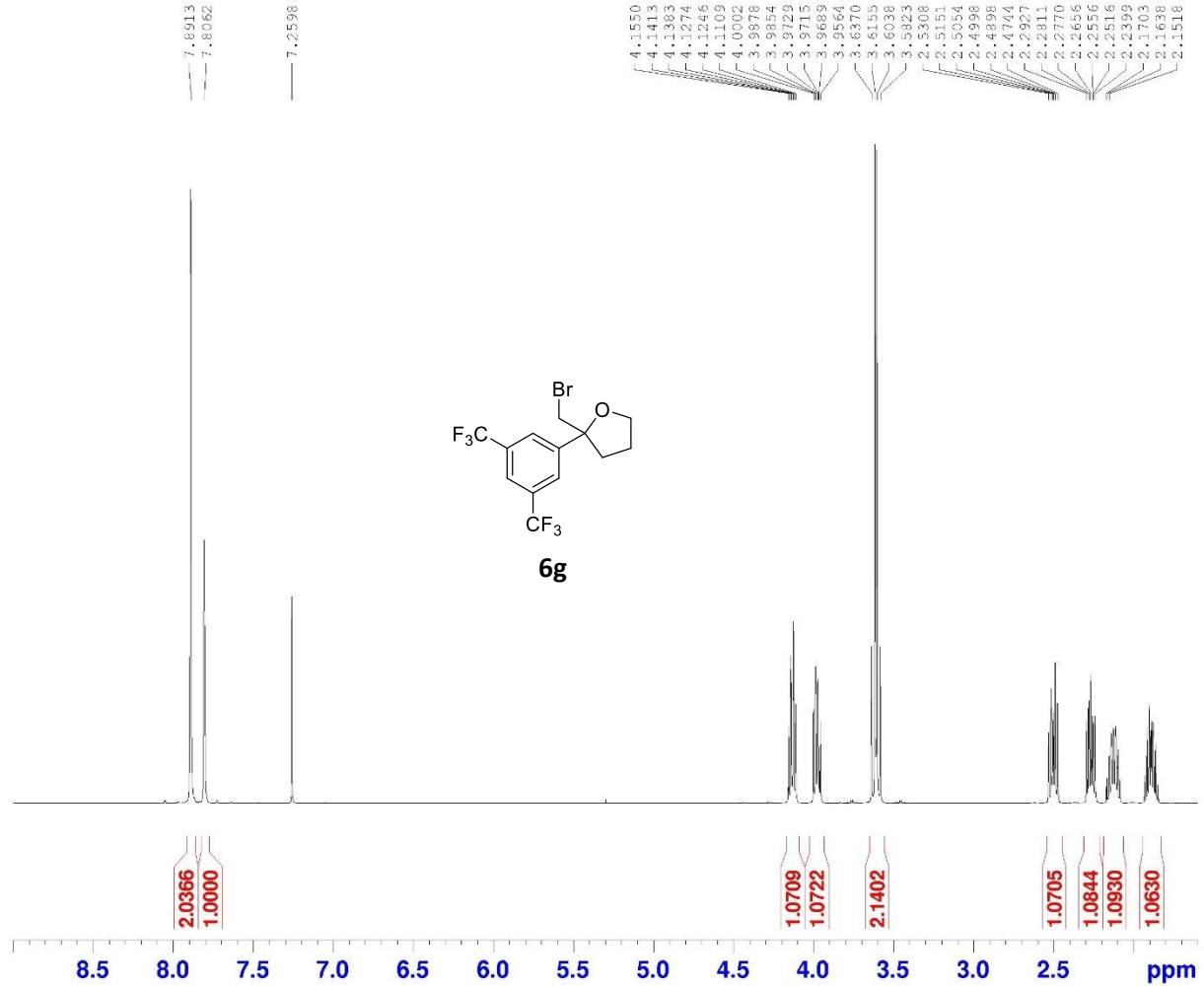


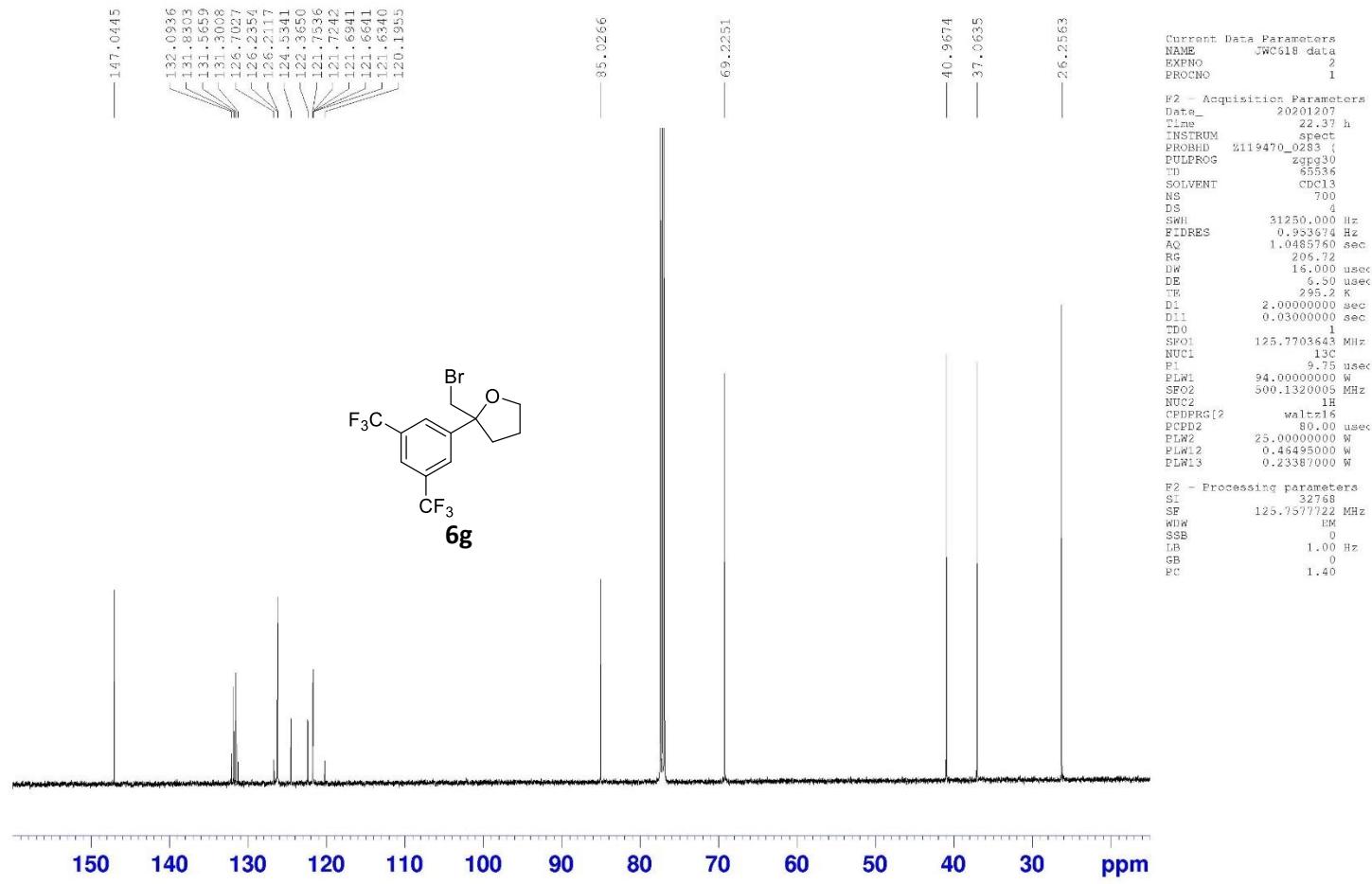
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NAME JWC619\_data  
EXPNO 1  
PROCNO 1

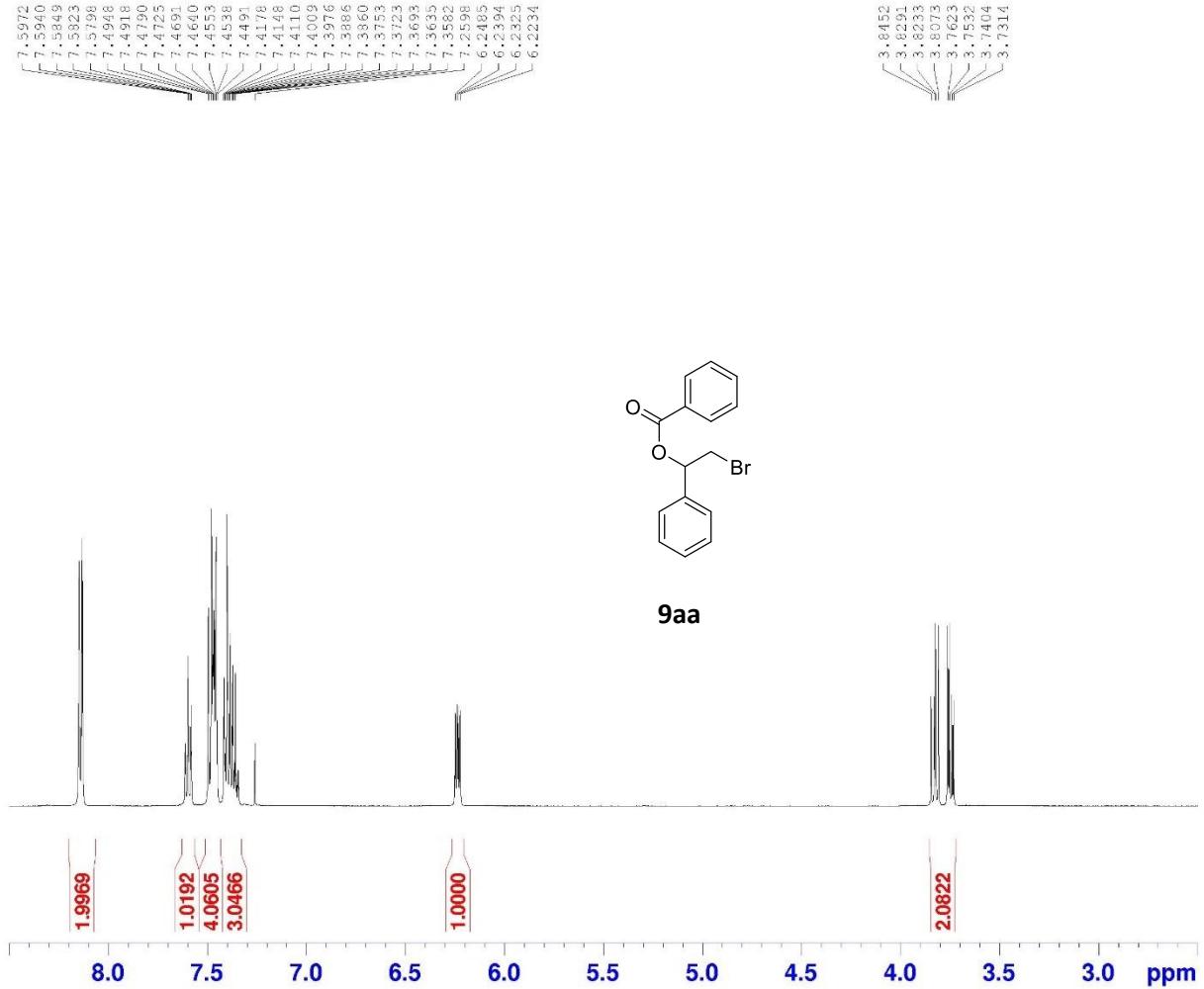
P2 - Acquisition Parameters  
Data\_ 20201208  
Time\_ 18.38 h  
INSTRUM spect  
PROBHD Z119470\_0283 {  
PULPROG zg30  
TD 65536  
T 10  
TE 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 10000.000 Hz  
FIDRES 0.305176 Hz  
AQ 3.2757999 sec  
RG 83.35  
DW 50.000 usec  
DE 6.50 usec  
TM 295.2 K  
D1 1.0000000 sec  
TD0 500.1330863 MHz  
RNUC 1H  
P1 10.91 usec  
FWHM 25.0000000 W

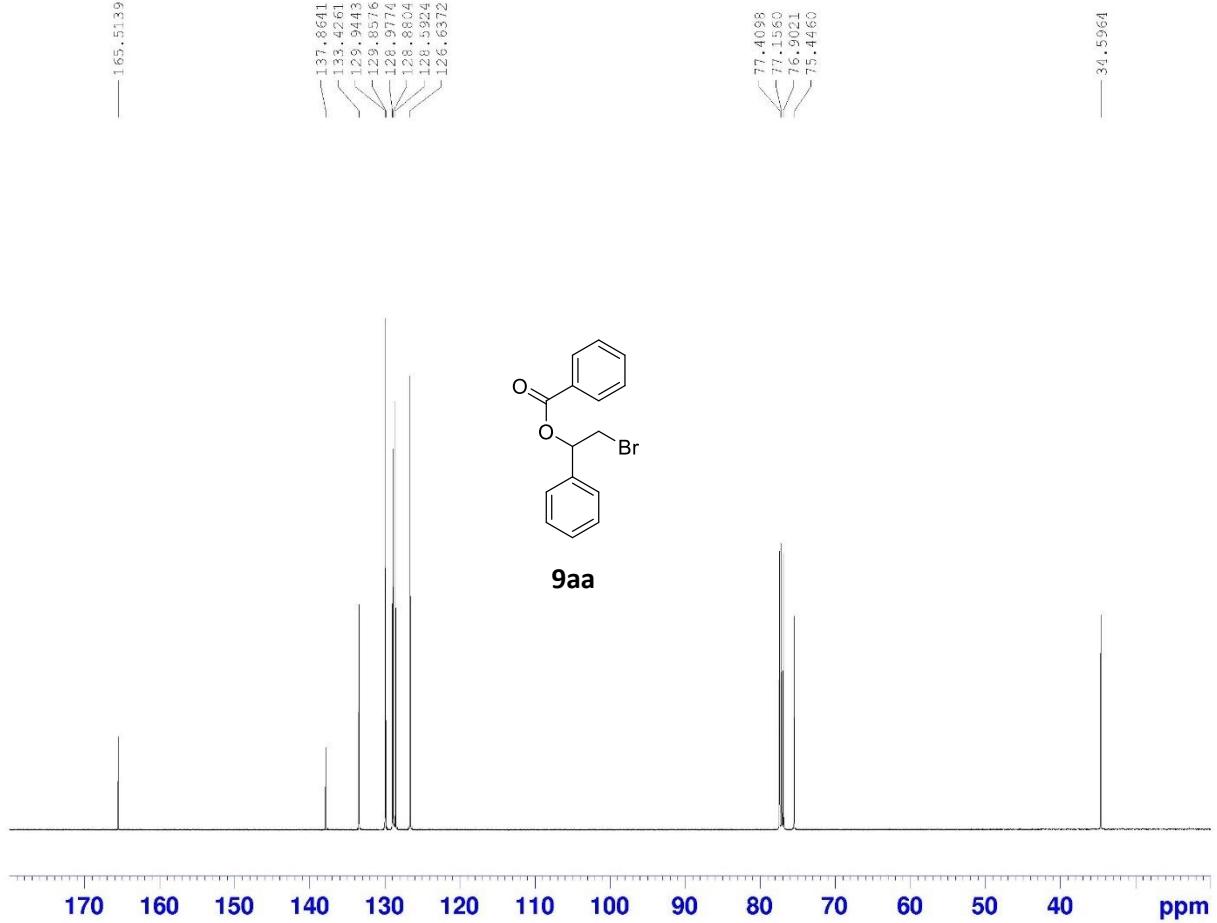
P2 - Processing parameters  
SI 65536  
SF 500.1300122 MHz  
RDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

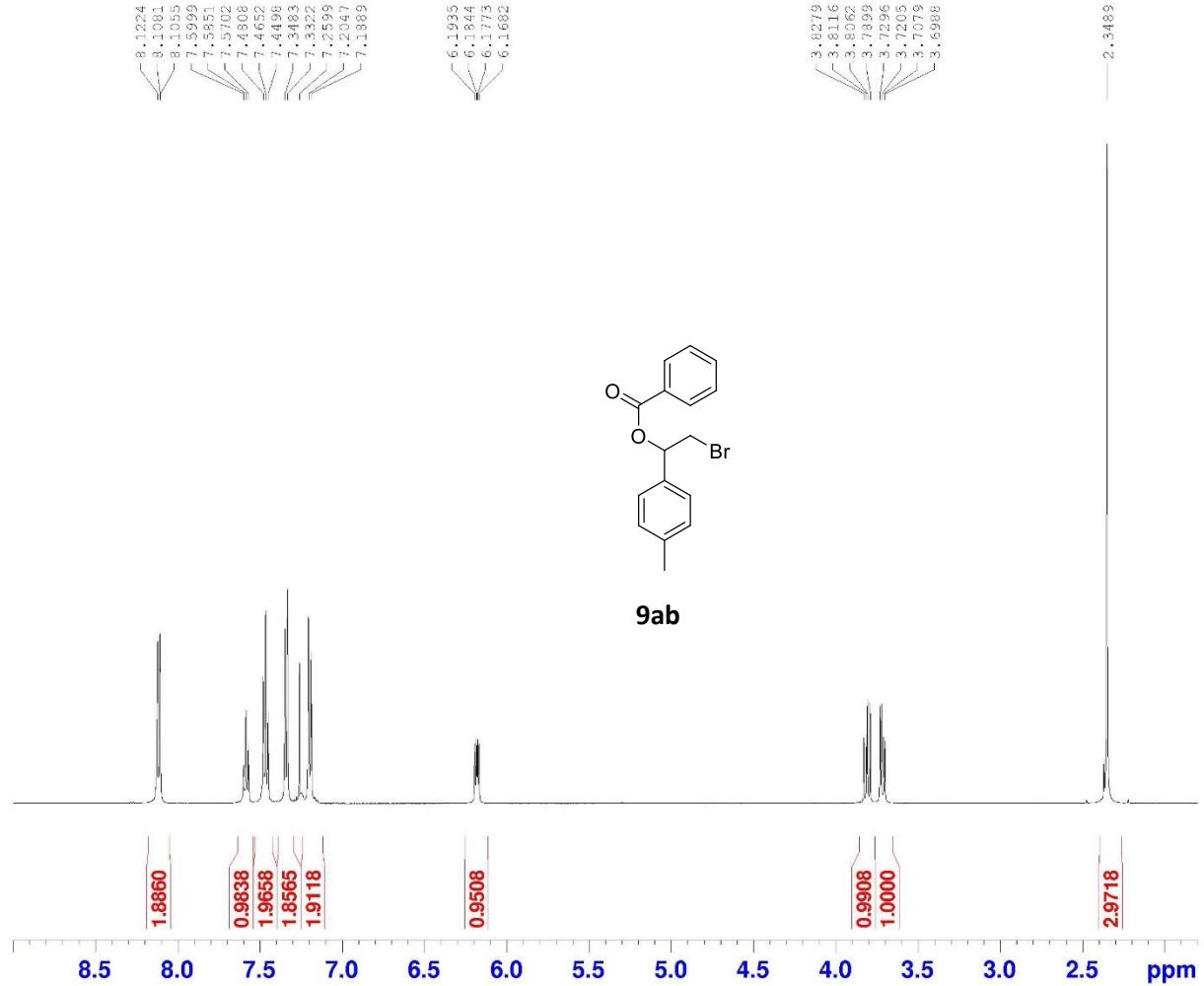


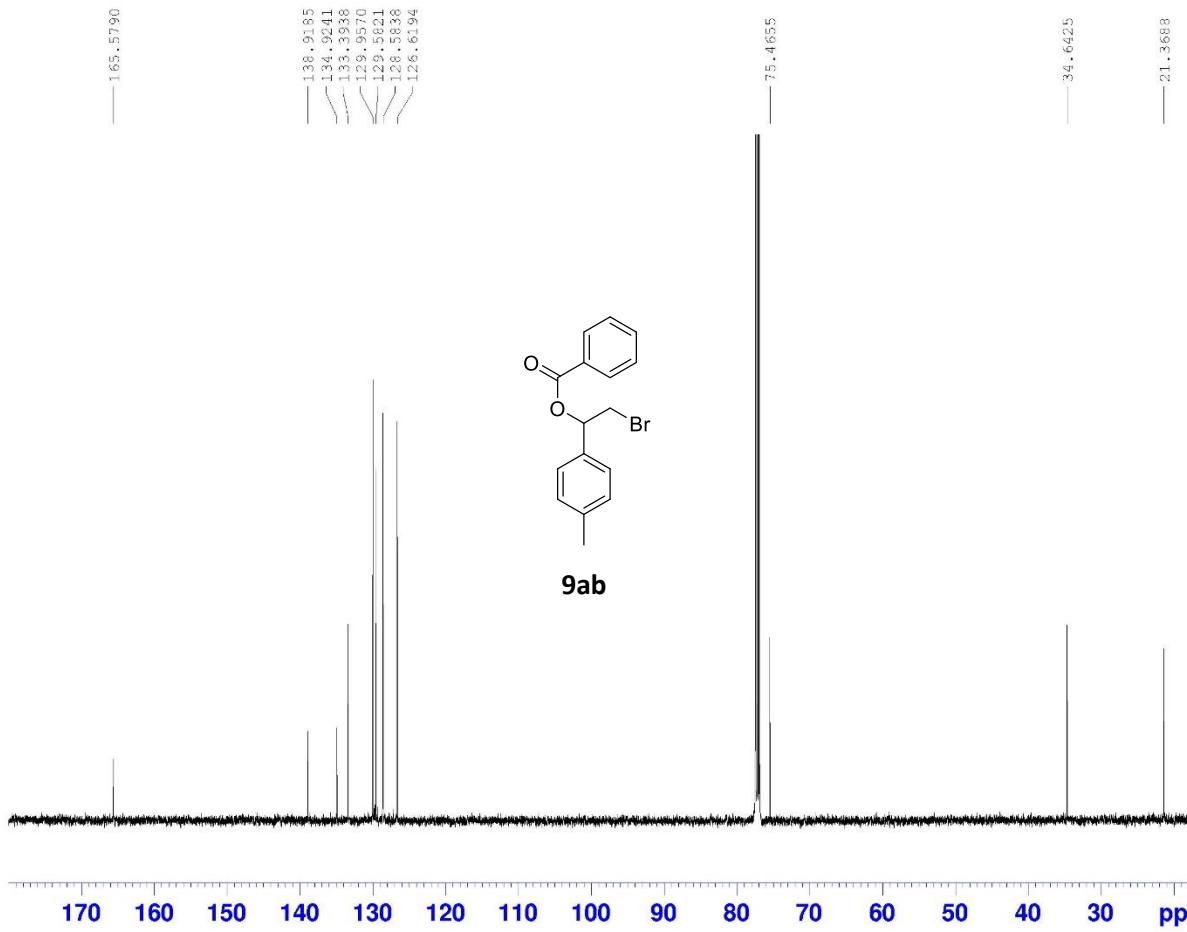








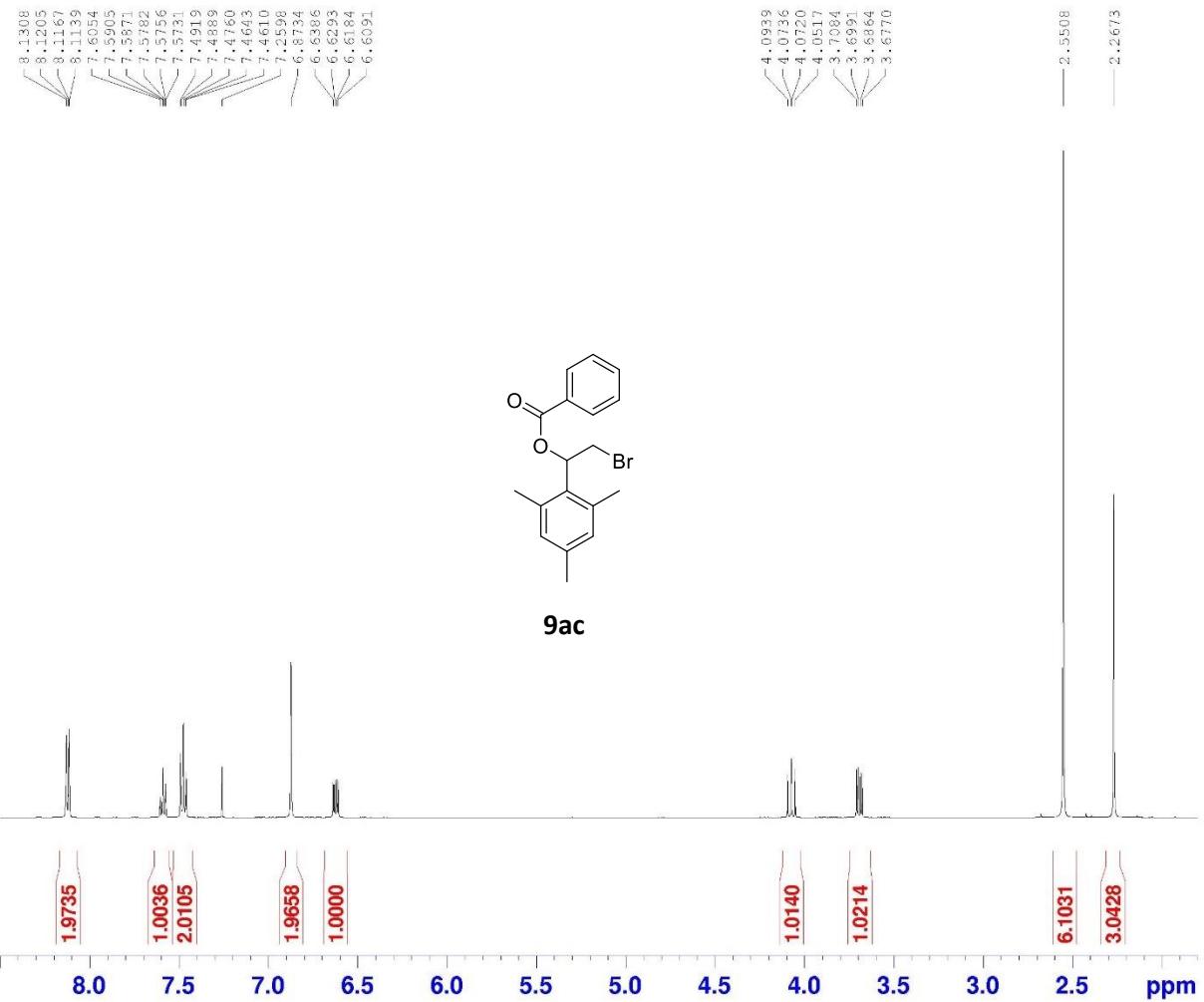


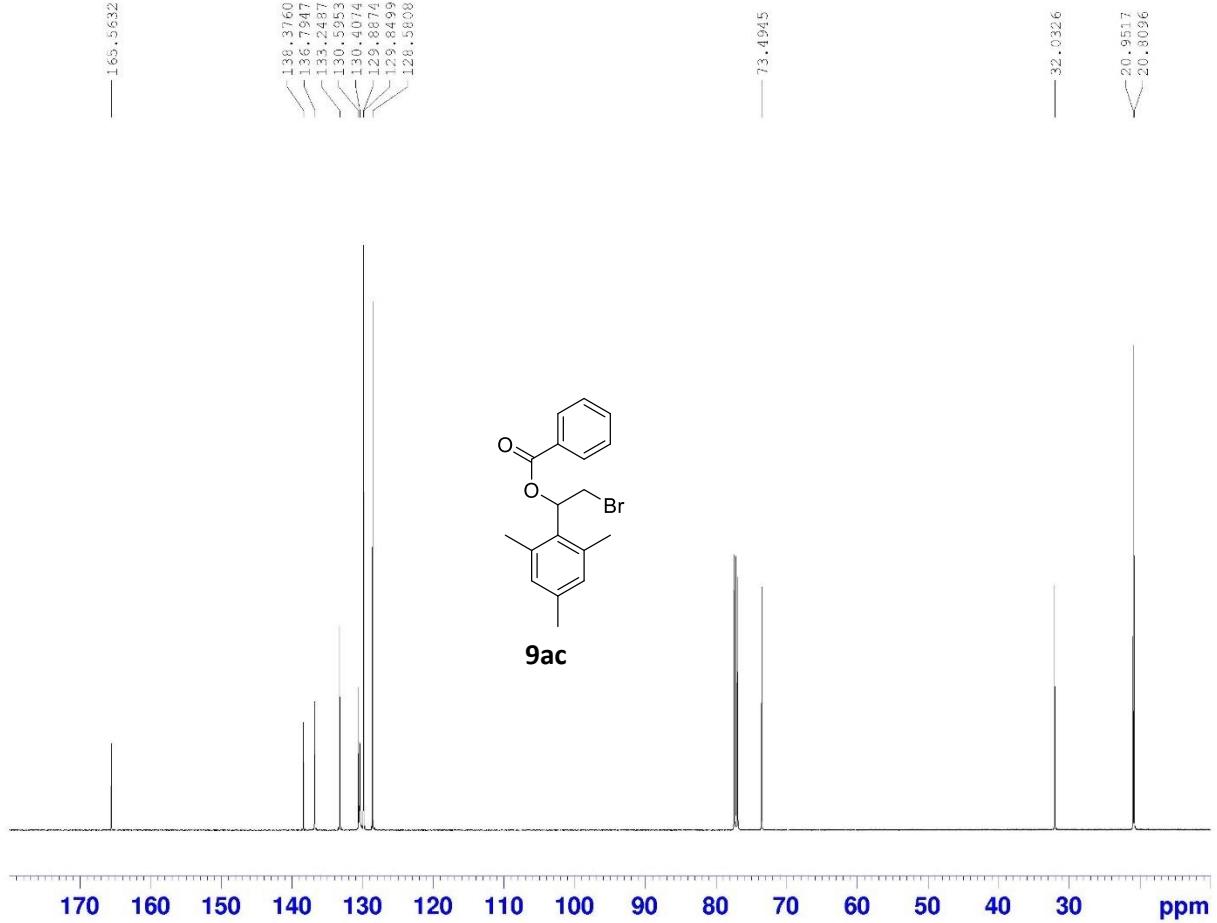


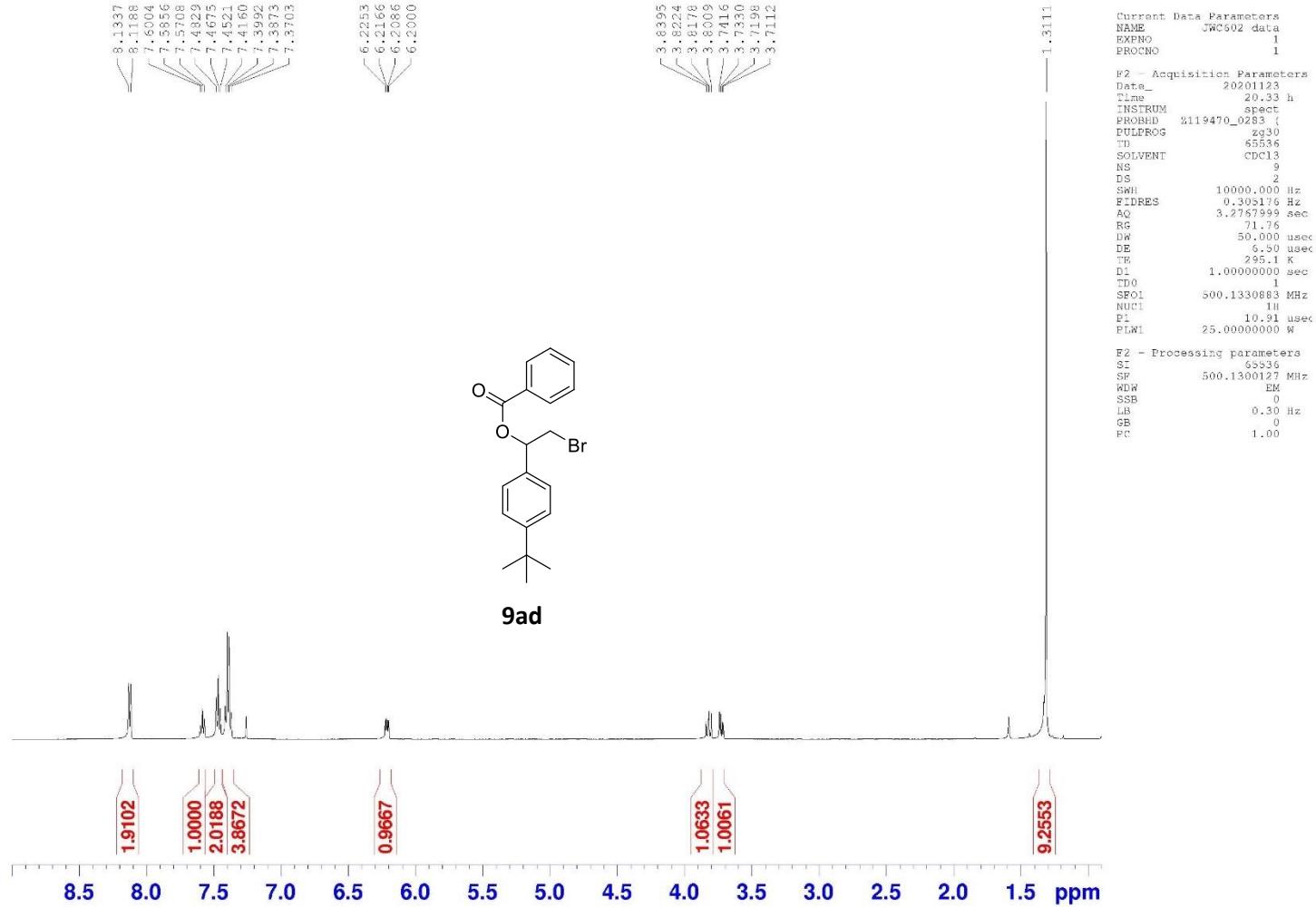
Current Data Parameters  
NAME JWC601 data  
EXPNO 2  
PROCNO 1

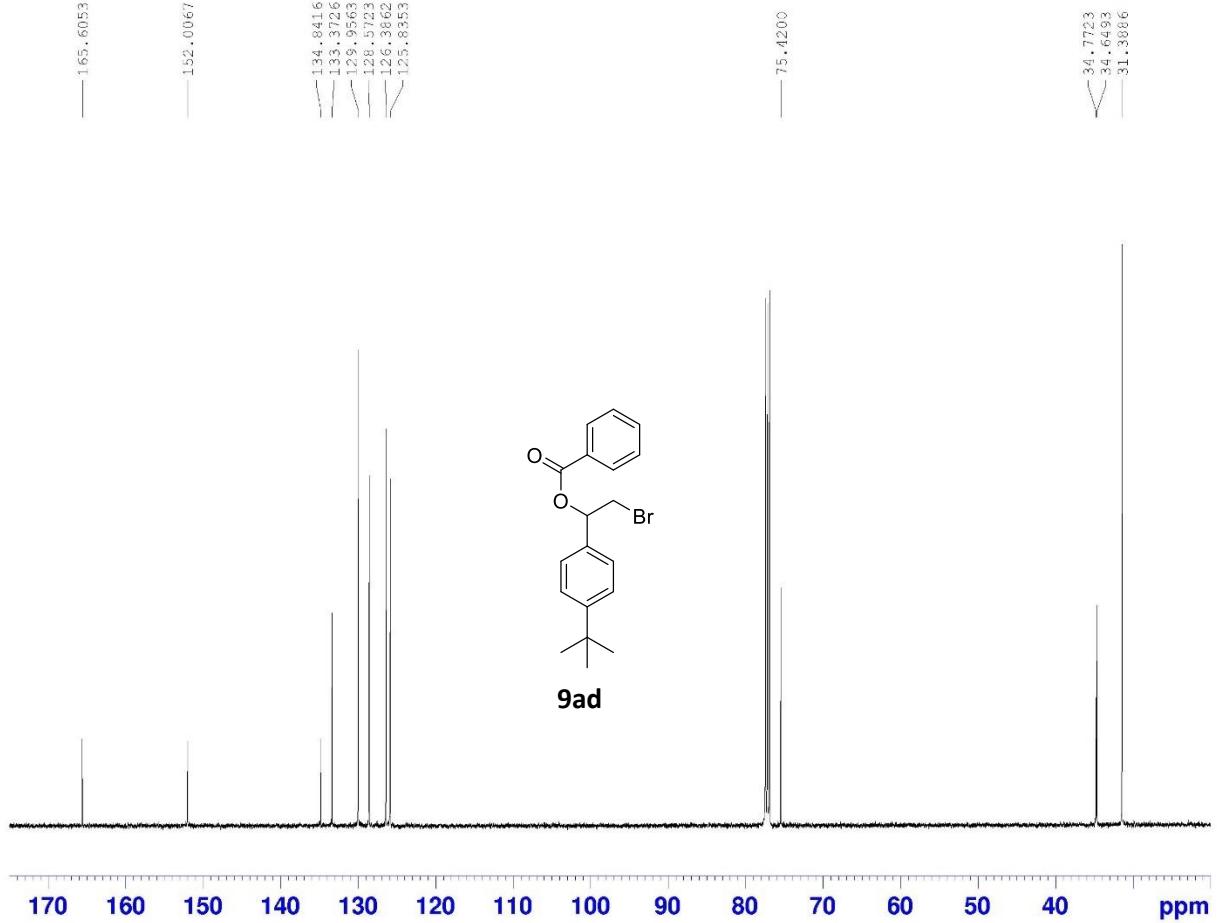
E2 - Acquisition Parameters  
Date\_ 20200123  
Time 21.47 h  
INSTRUM spect  
PROBHD Z119470\_0283 {  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 600  
DS 4  
SWH 29981.904 Hz  
ETDRES 0.308261 Hz  
AQ 1.1010048 sec  
RG 209.72  
DW 16.800 usec  
DE 6.50 usec  
TE 295.2 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TD0 125.7703643 MHz  
NUC1 13C  
PL 9.75 usec  
PLW1 94.00000000 W  
SFO2 500.1320005 MHz  
NUC2 1H  
CPDPFG[2] waltz16  
CPDP2 80.00 usec  
PLW2 25.00000000 W  
PLW12 0.45495000 W  
PLW13 0.23387000 W

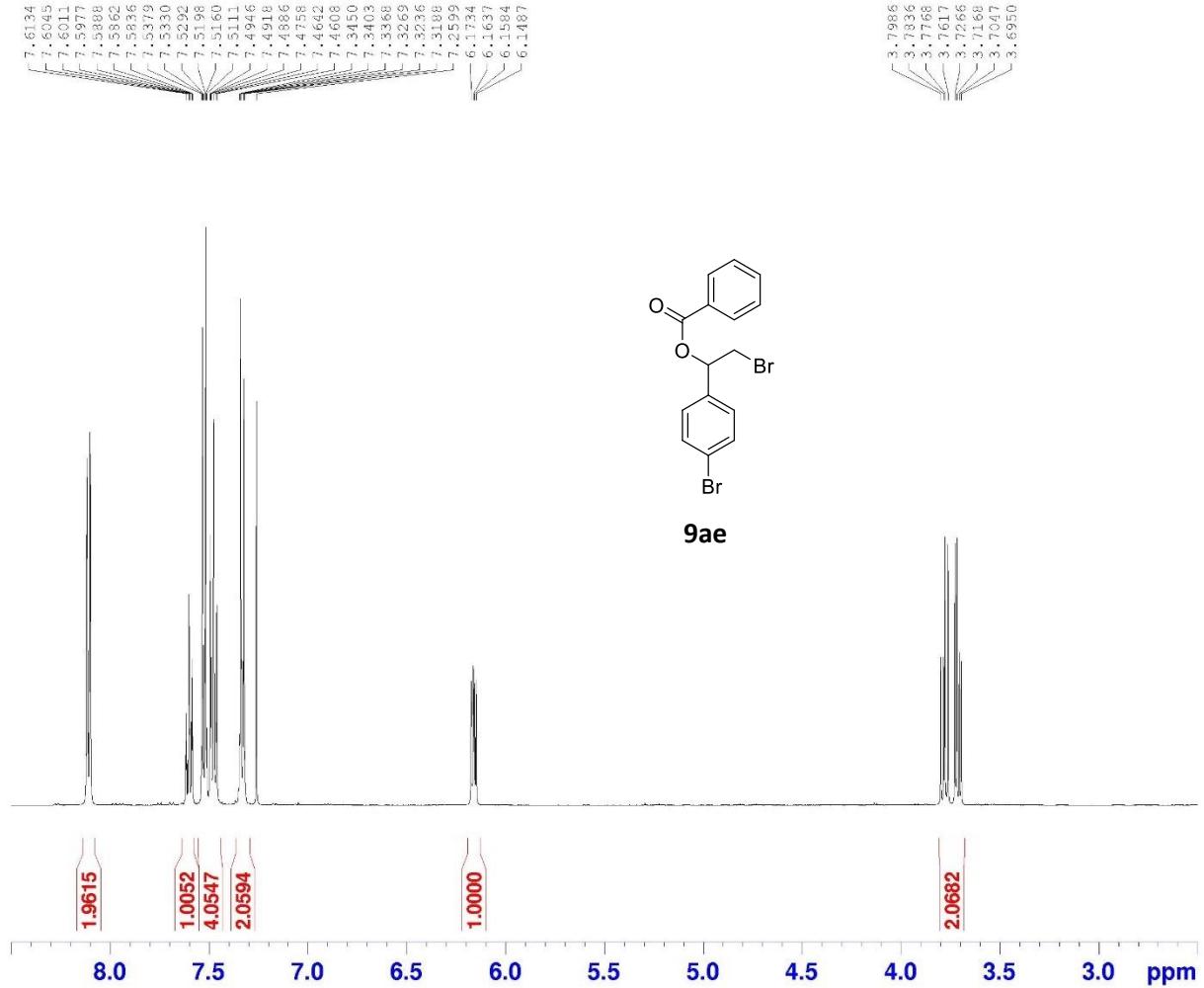
E2 - Processing parameters  
SI 32768  
SF 125.7577738 MHz  
WDW HM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

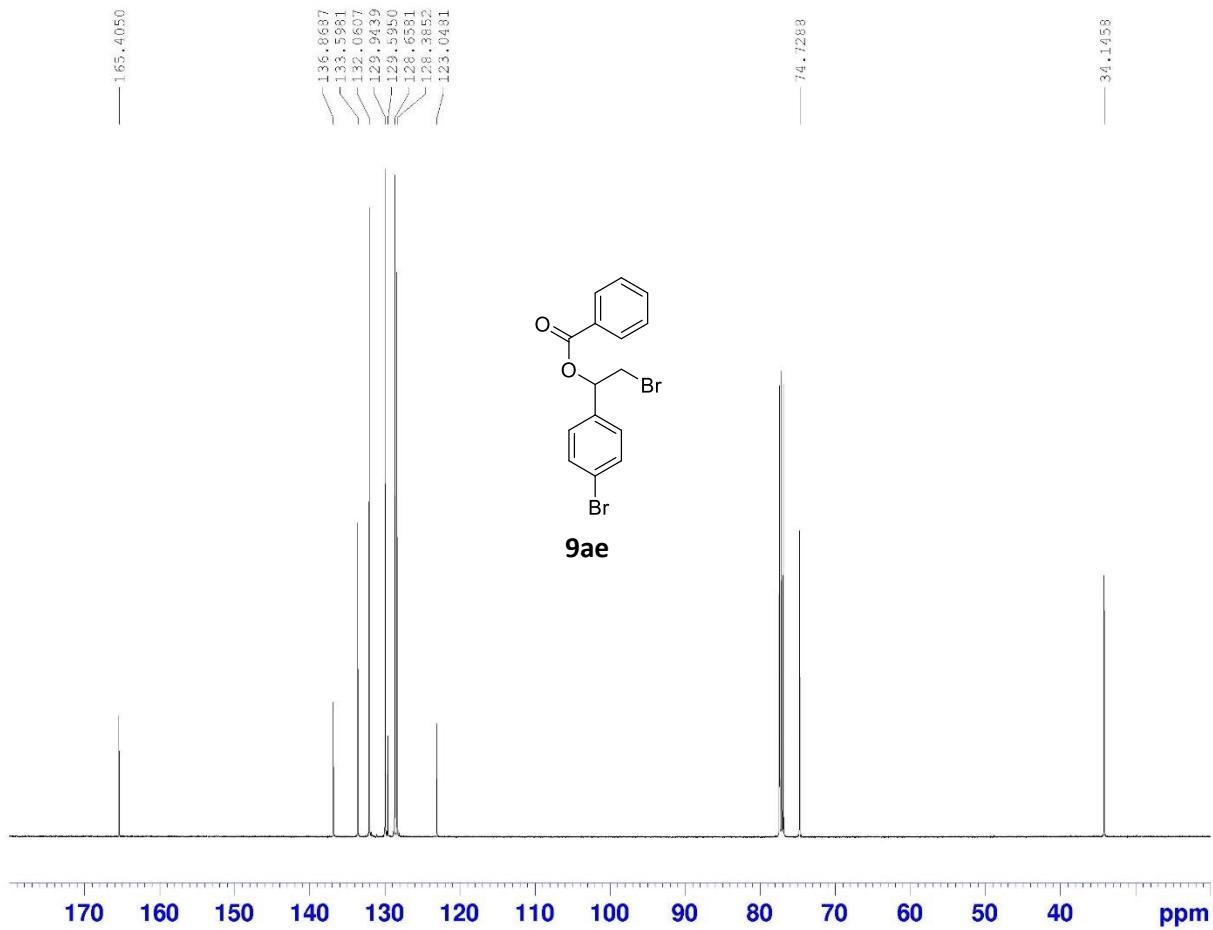


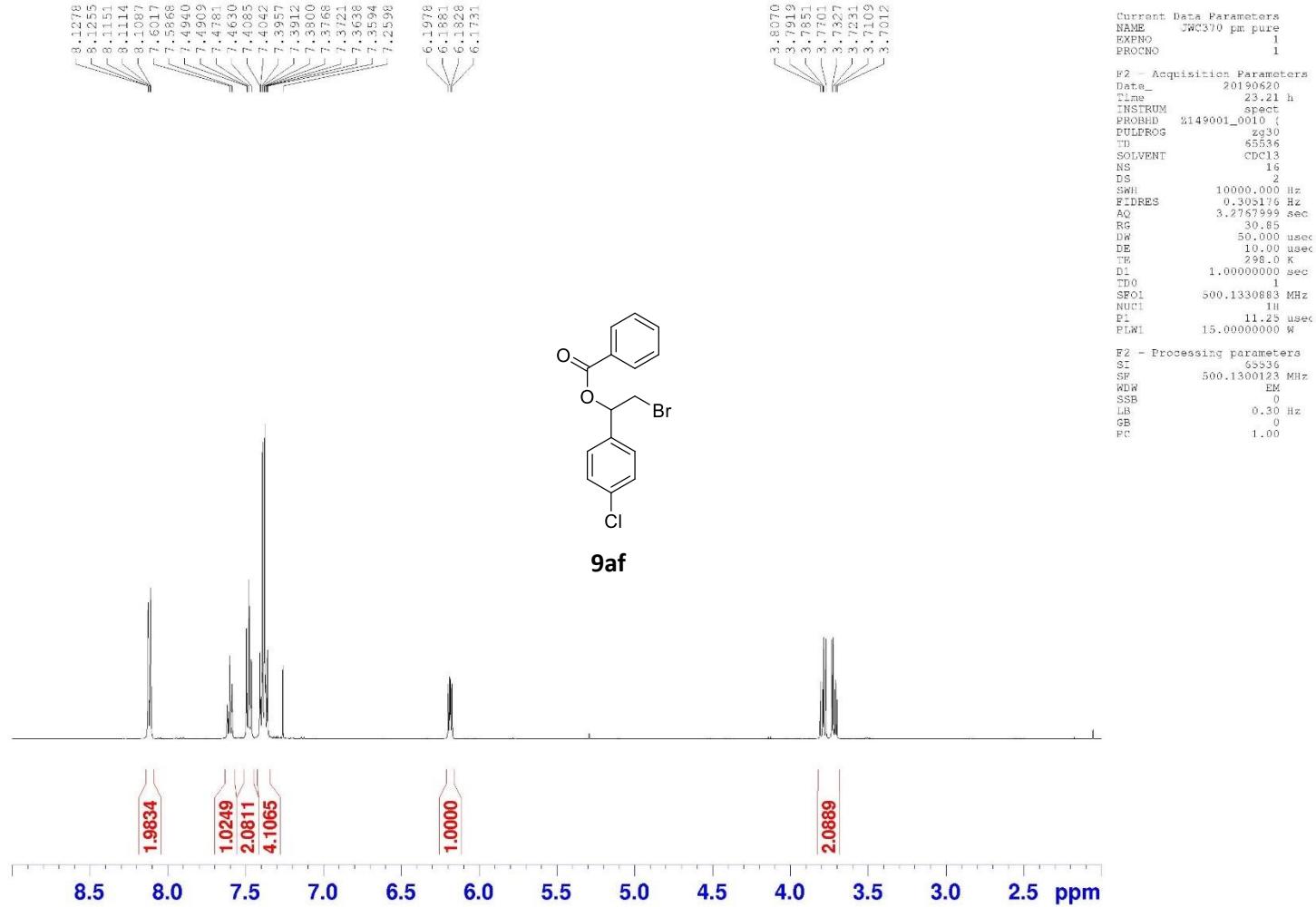


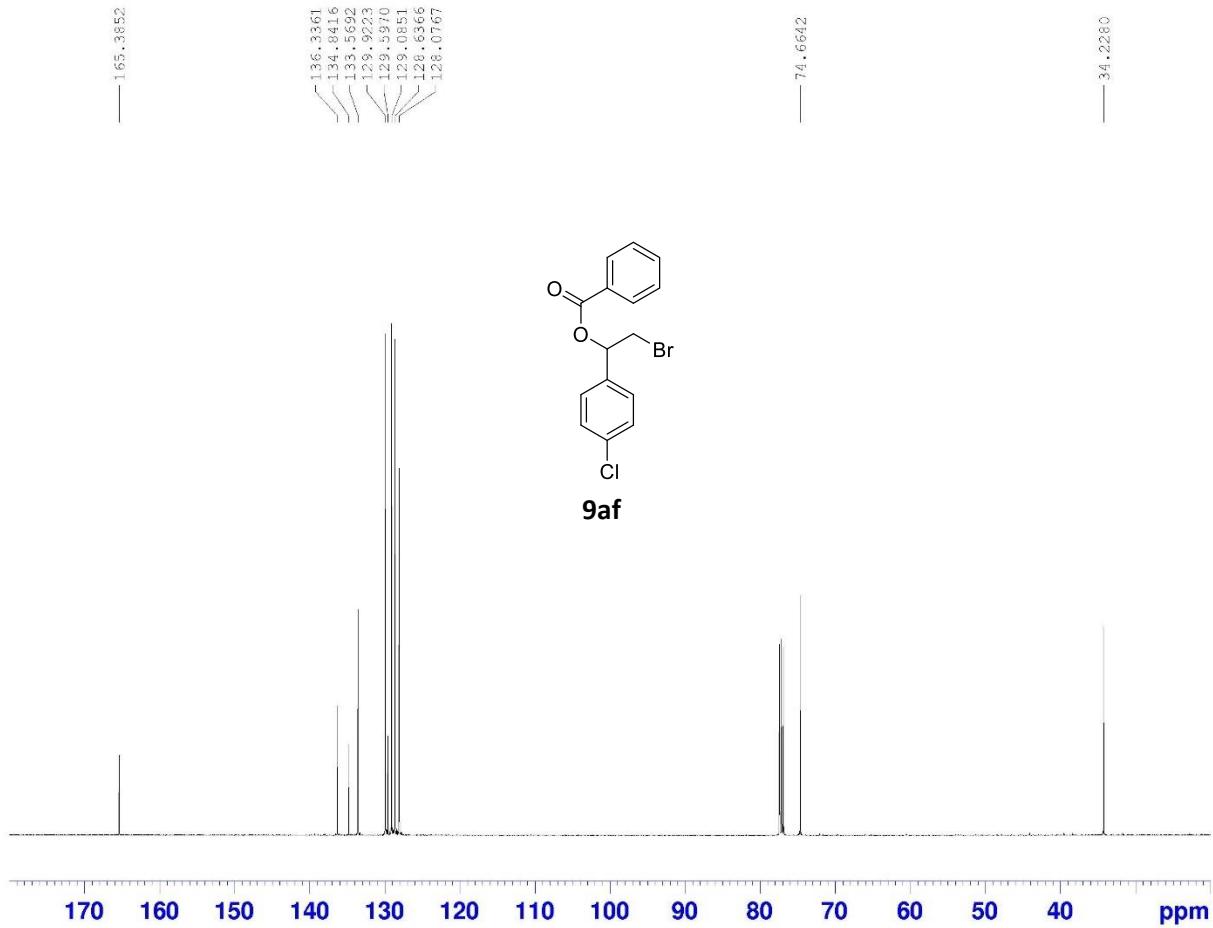




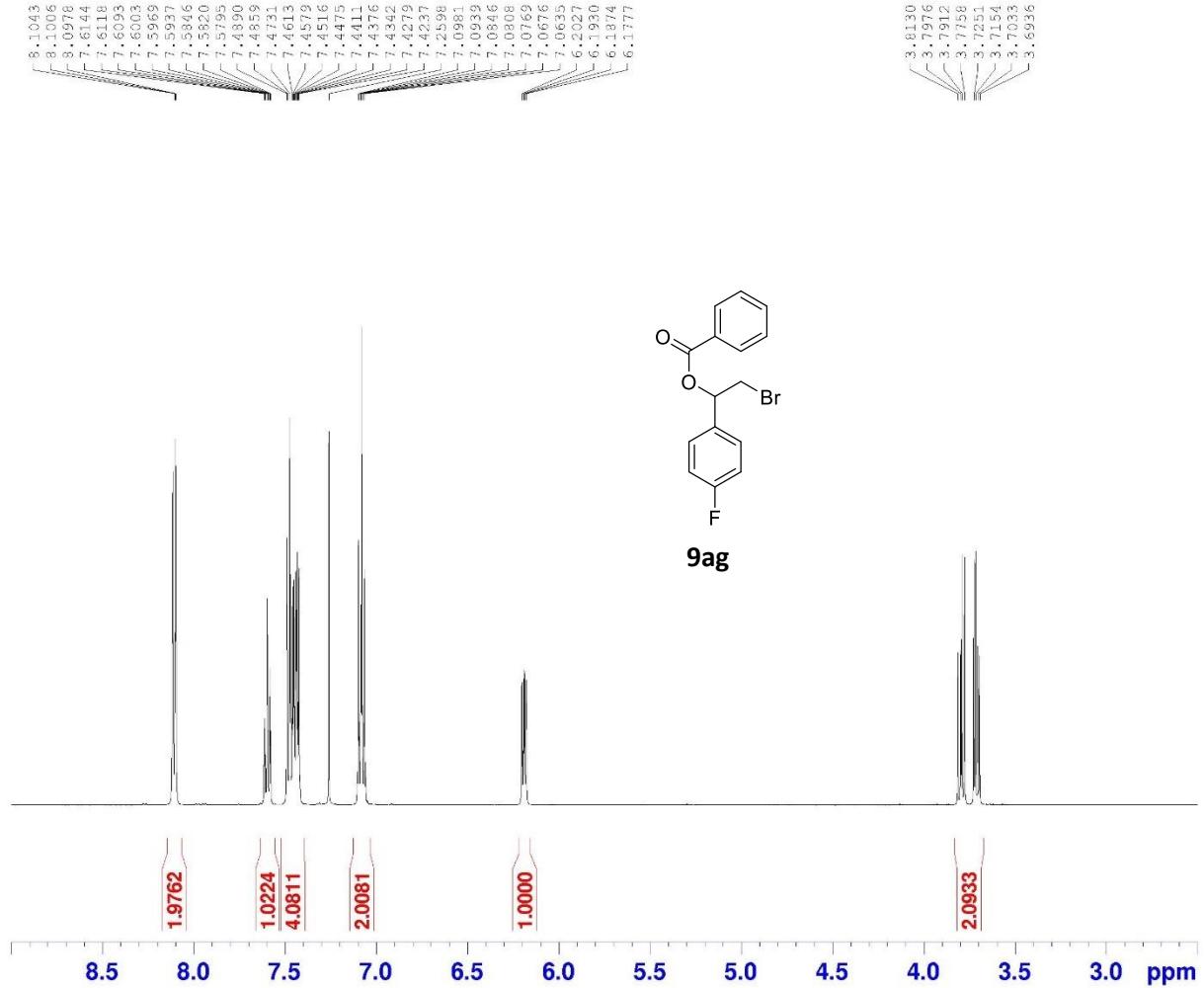


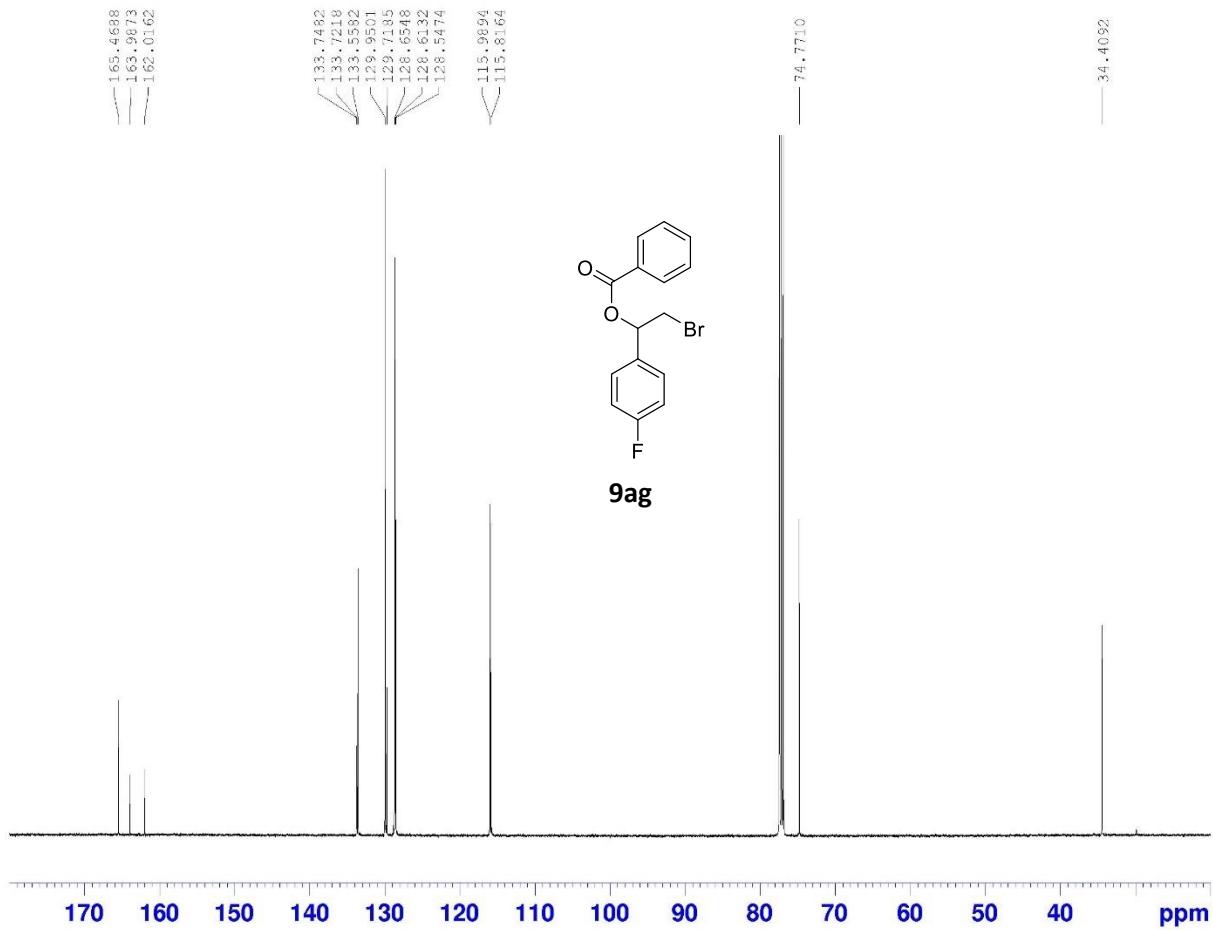




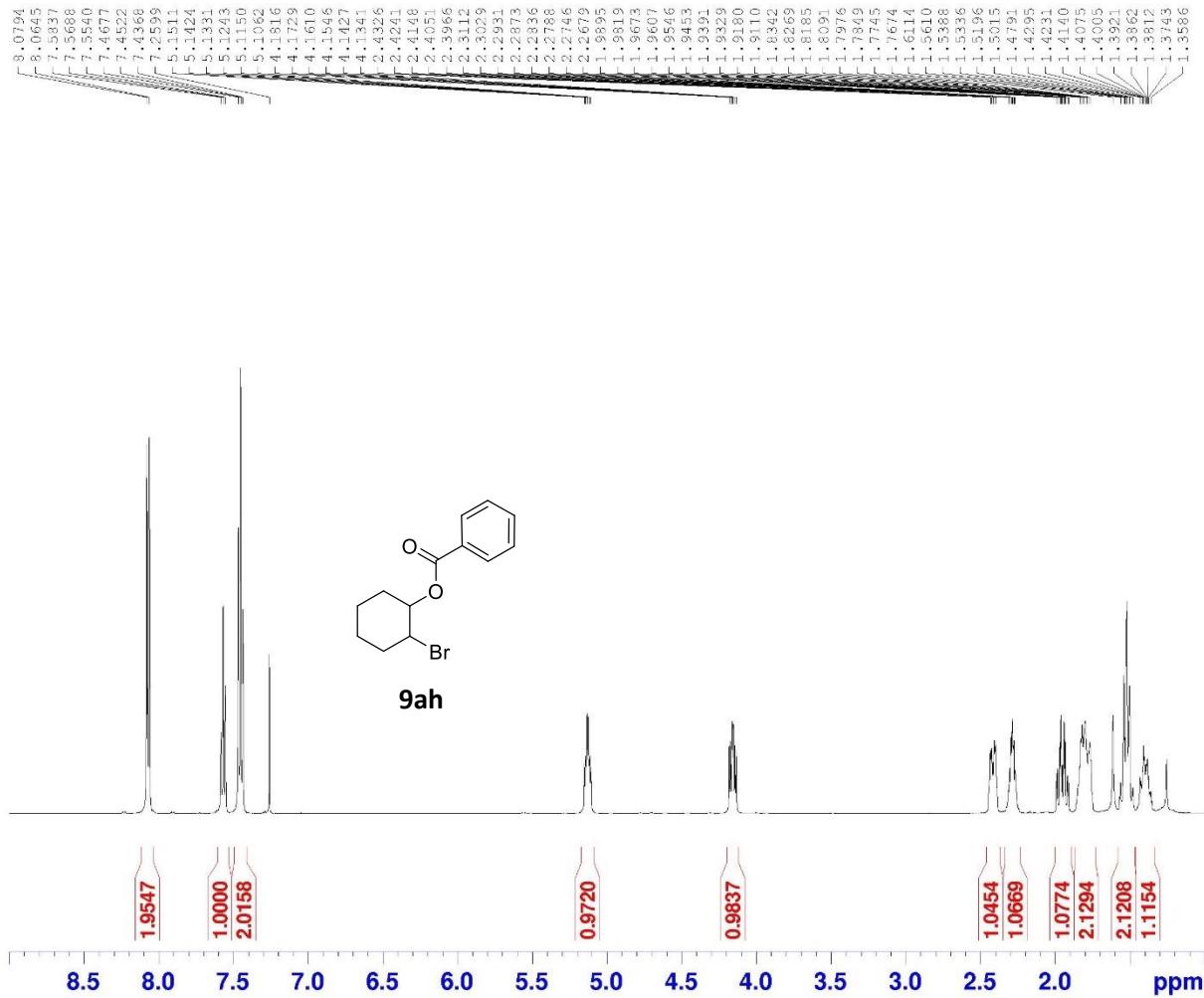


Current Data Parameters  
NAME JWC370 pm pure  
EXPNO 2  
PROCNO 1  
  
P2 - Acquisition Parameters  
Data\_ 20190621  
Time 0.03 h  
INSTRUM spect  
PROBHD Z149001\_0010 {  
PULPROG zgpp30  
TD 65536  
T1 65536  
SOLVENT CDCl3  
NS 600  
DS 4  
SW0 29761.904 Hz  
FIDRES 0.908261 Hz  
AQ 1.1010048 sec  
RG 205.72  
DW 16.800 usec  
DE 18.00 usec  
TE 298.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
DP0  
SW01 125.7703643 MHz  
NUC1 13C  
PL 10.00 usec  
PLW1 61.00000000 w  
SF02 500.1320005 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
CPDPD2 80.00 usec  
PLW2 15.00000000 w  
PLW12 0.29563000 w  
PLW13 0.14920001 w  
  
P2 - Processing parameters  
SI 32768  
SF 125.7577807 MHz  
WDW 0  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40





**9ag**



Current Data Parameters  
NAME JWC600 data  
EXPNO 1  
PROCNO 1

```

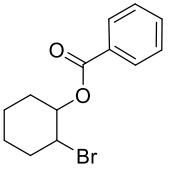
R2 - Acquisition Parameters
Date_   20201117
Time_   20.38 h
INSTRUM spect
PROBHD 2119470_0283 (I)
PULPROG zg30
TD      65536
SOLVENT CDC13
NS      16
DS      2
SWH    10000.000 Hz
FIDRES 0.305176 Hz
AQ     3.276599 sec
RG     83.35
DW     50.000 usec
DE     6.50 usec
TE     295.2 K
D1     1.0000000 sec
TDO     1
SF01 500.1330883 MHz
NUC1 1H
P1     10.91 usec
PLW1 25.0000000

```

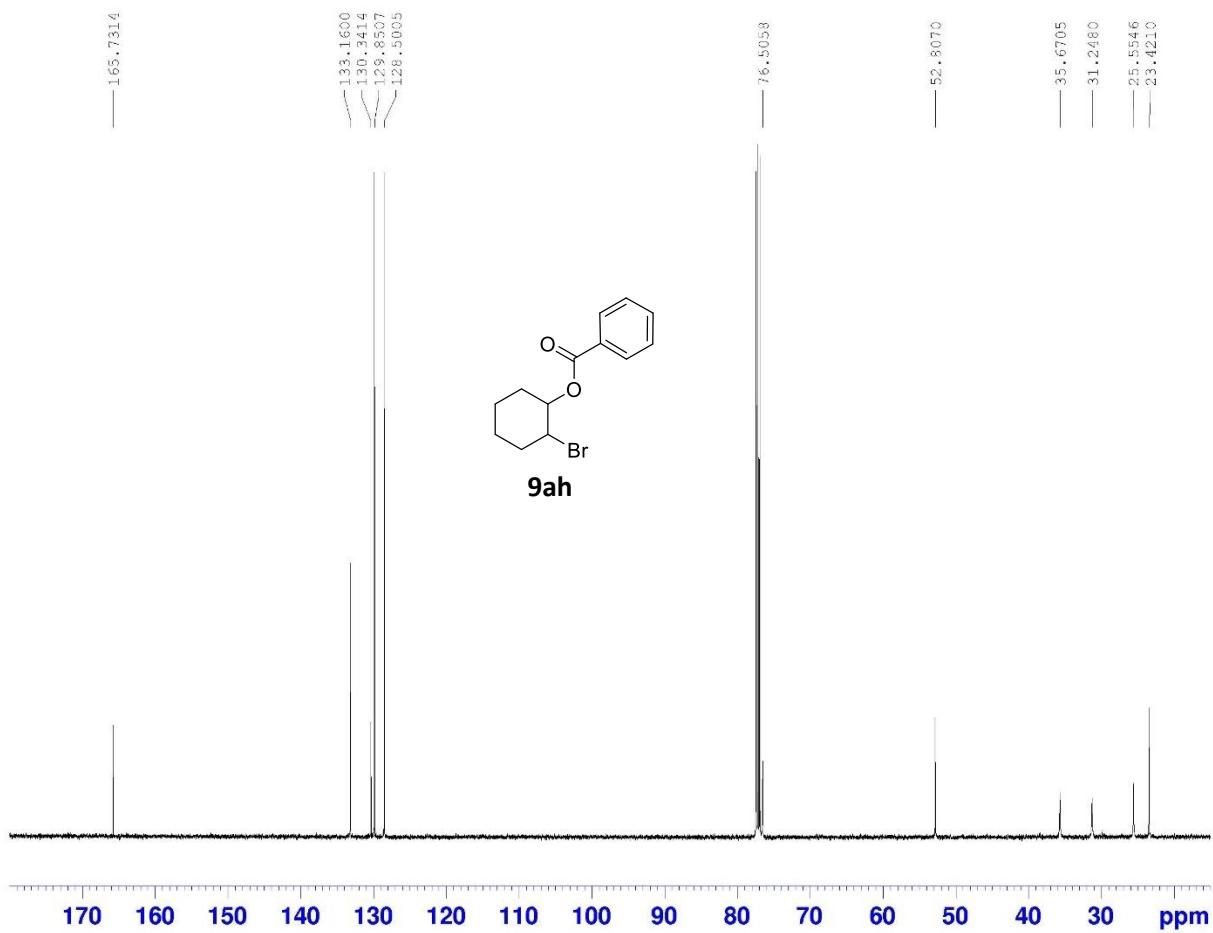
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F2 - Processing parameters
SI          65536
SF        500.1300125 MHz
WDW           EM
SSB            0
LB          0.30 Hz
GB            0
PC          1.00

```



9ah



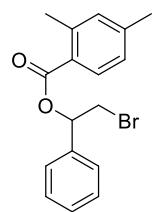
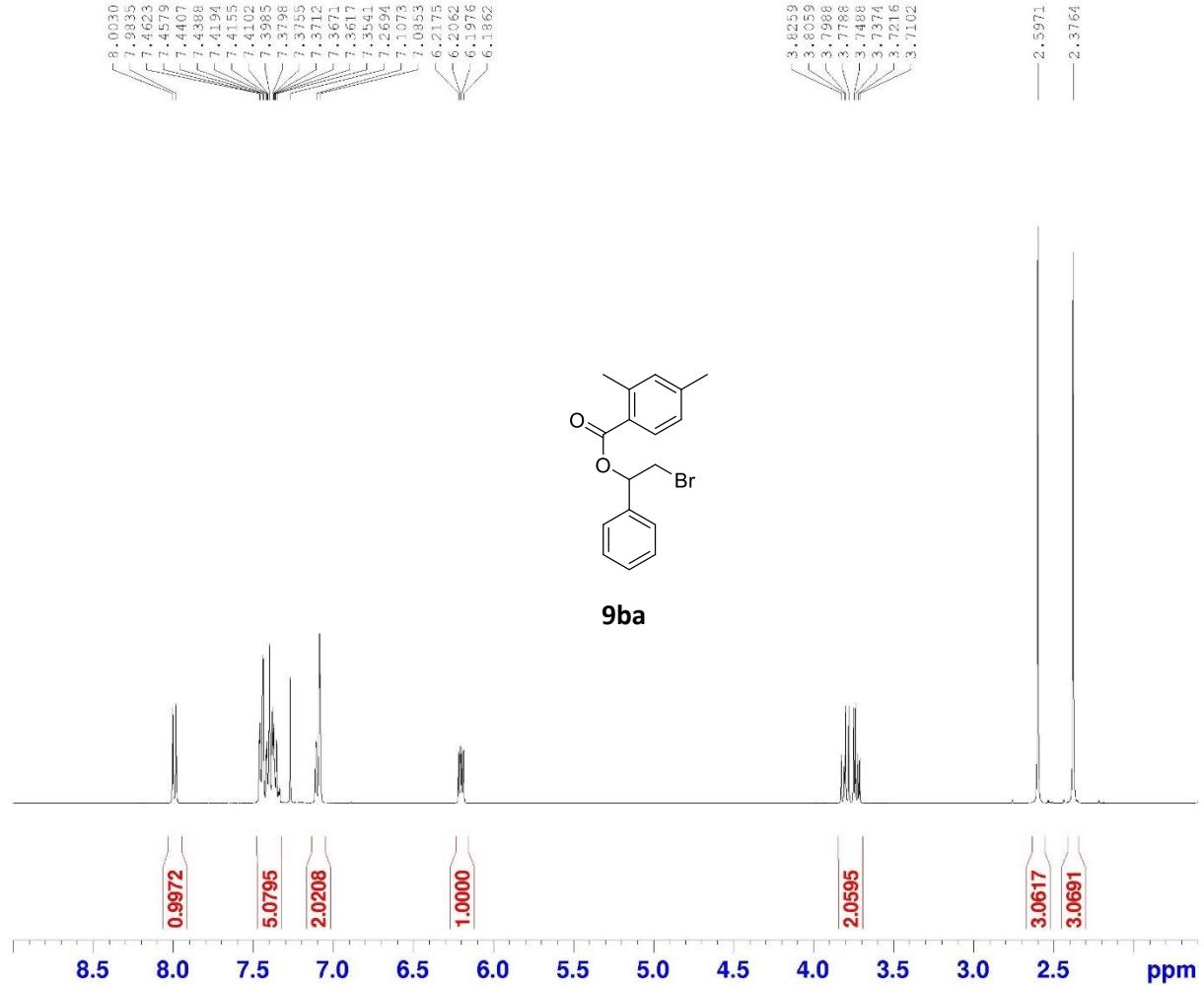
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Current Data Parameters
NAME      JWC500_data
EXPNO     2
PROCNO    1

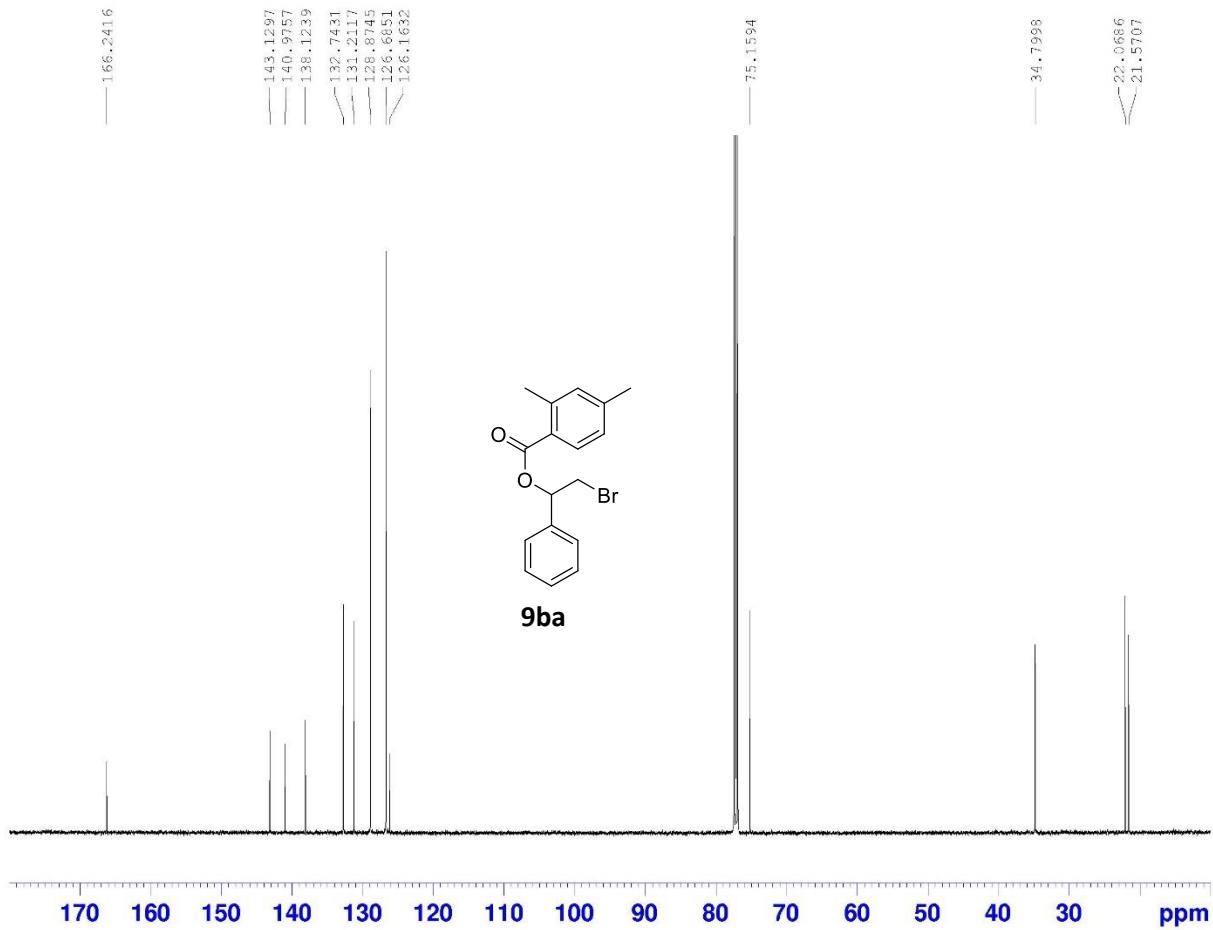
P2 - Acquisition Parameters
Date_   20201117
Time_   21.16 h
INSTRUM spect
PROBHD Z119470_0283 {
PULPROG zgpp30
TD      65536
SOLVENT CDCl3
NS      700
DS      4
SW0H   29751.904 Hz
FIDRES  0.908261 Hz
AQ      1.1010048 sec
RG      205.72
DW      16.800 usec
DE      6.50 usec
TE      295.1 K
D1      2.0000000 sec
T1L    0.0300000 sec
TD0
SW01   125.7703643 MHz
NUC1    13C
P1      9.75 usec
PLW1   94.00000000 w
SF02   500.1320005 MHz
NUC2    1H
CPDPRG[2] waltz16
PCPD2   80.00 usec
PLW2   25.00000000 w
PLW12  0.46495000 w
PLW13  0.23387000 w

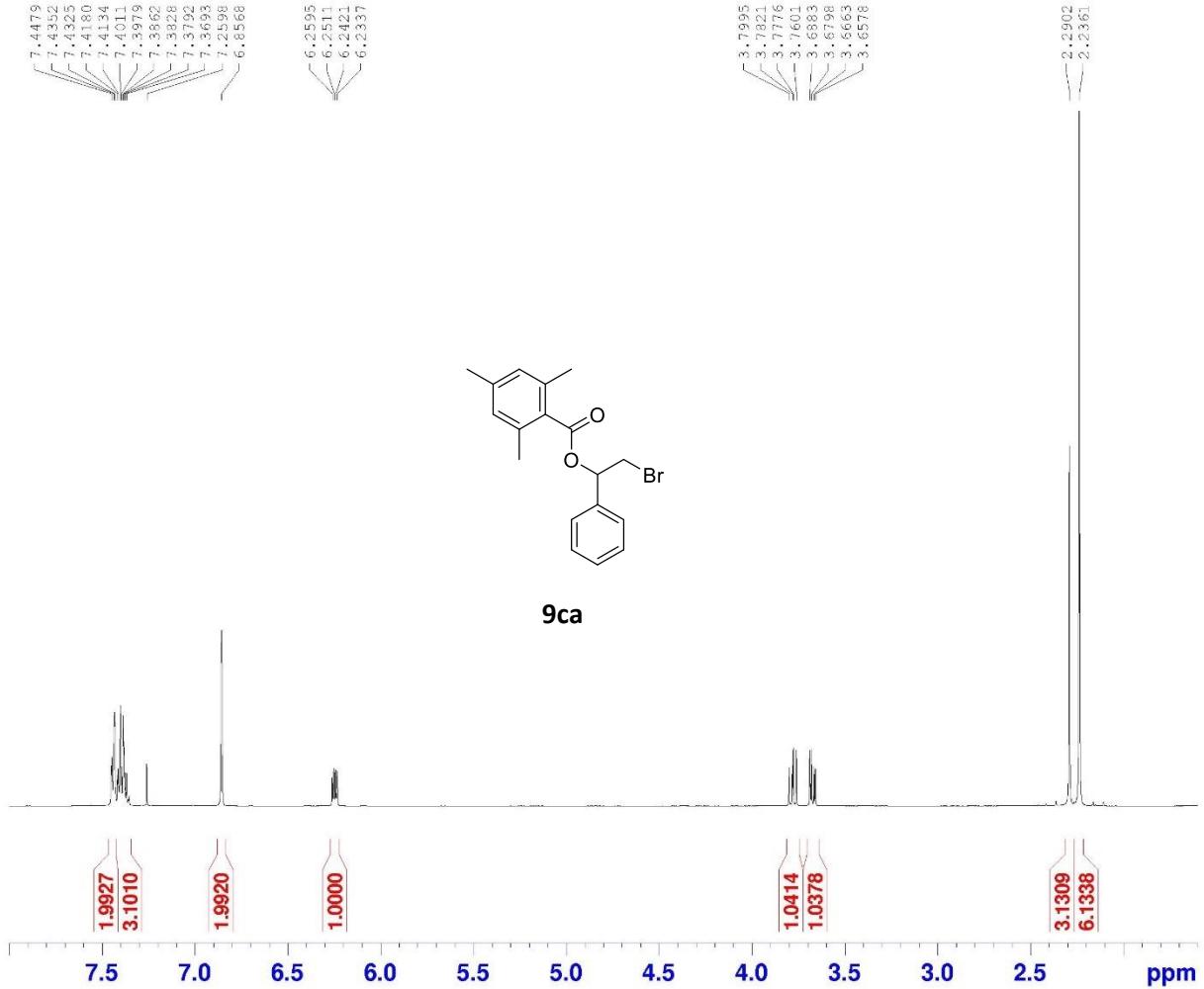
P2 - Processing parameters
SI      32768
SF      125.7577752 MHz
WDW
SSB
LB      1.00 Hz
GB      0
PC      1.40

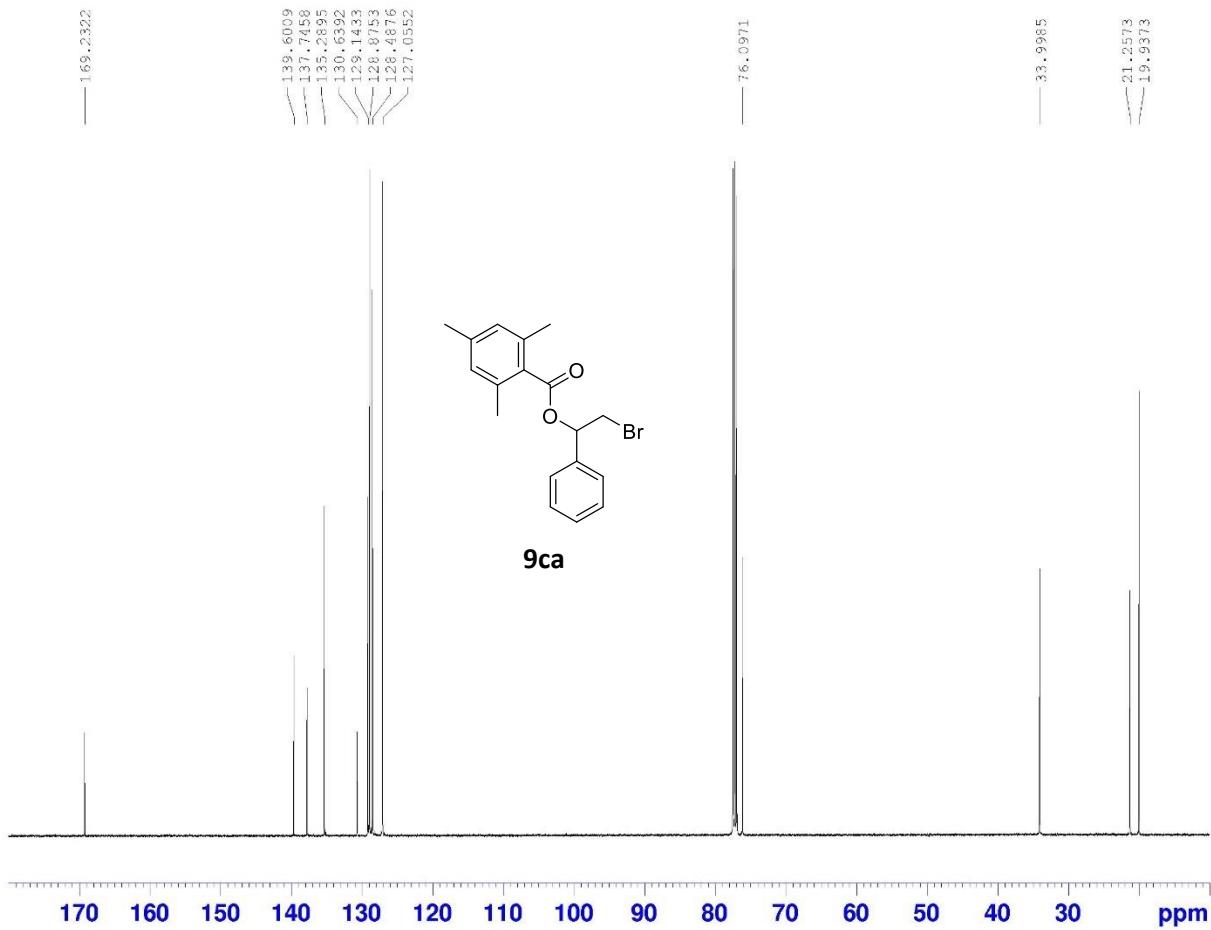
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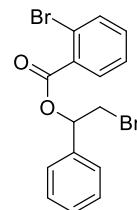
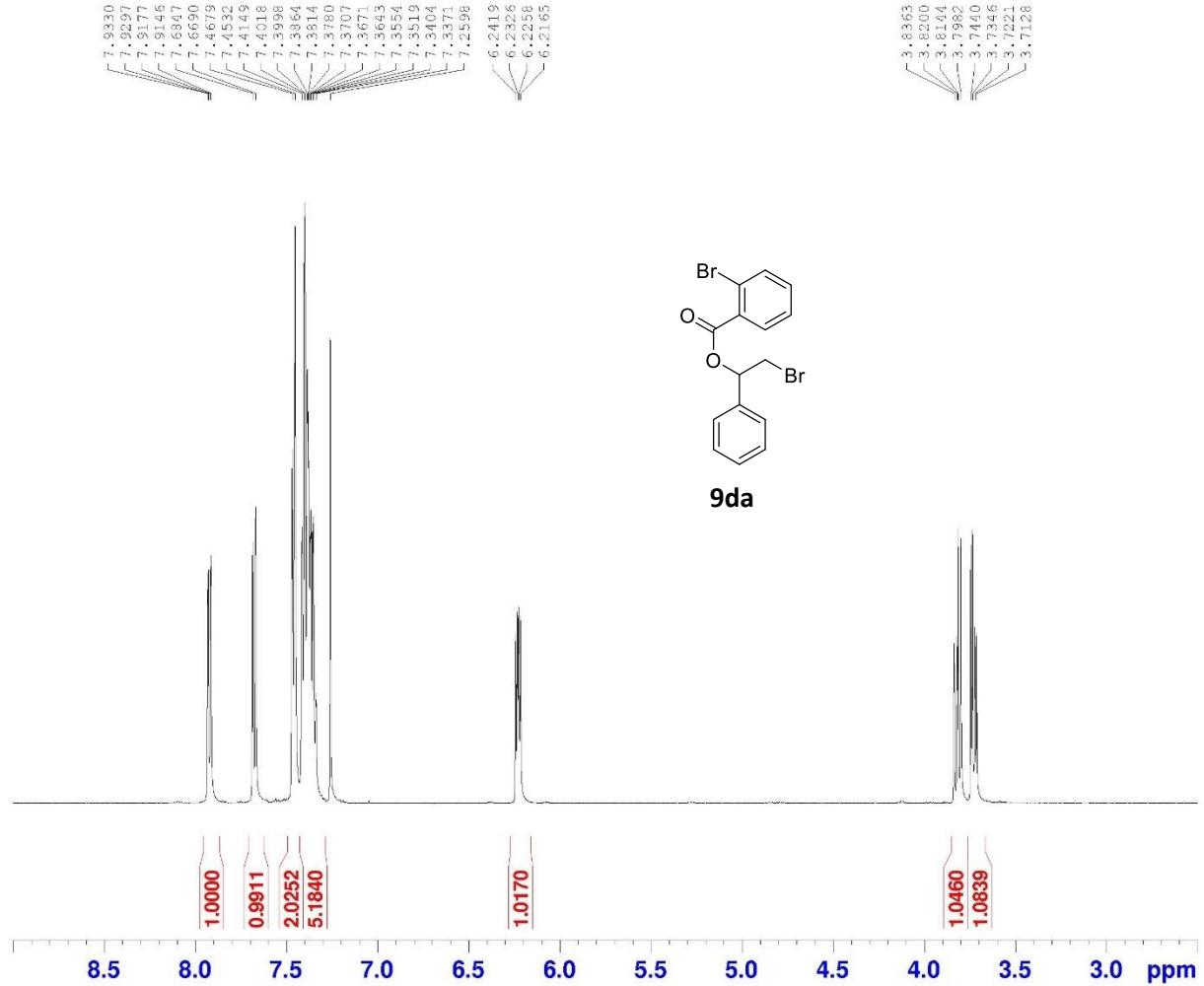


**9ba**

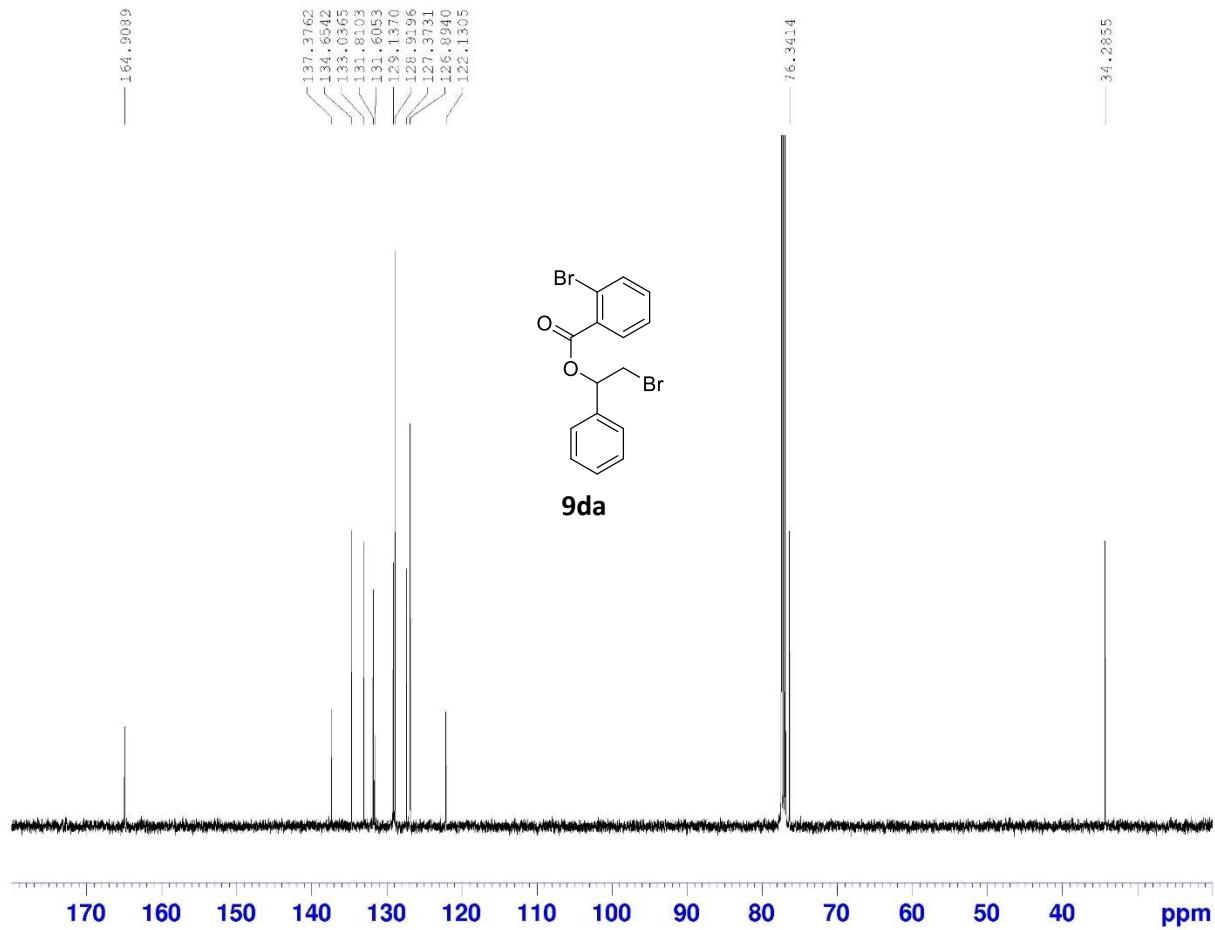


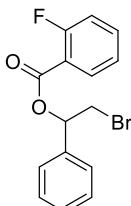
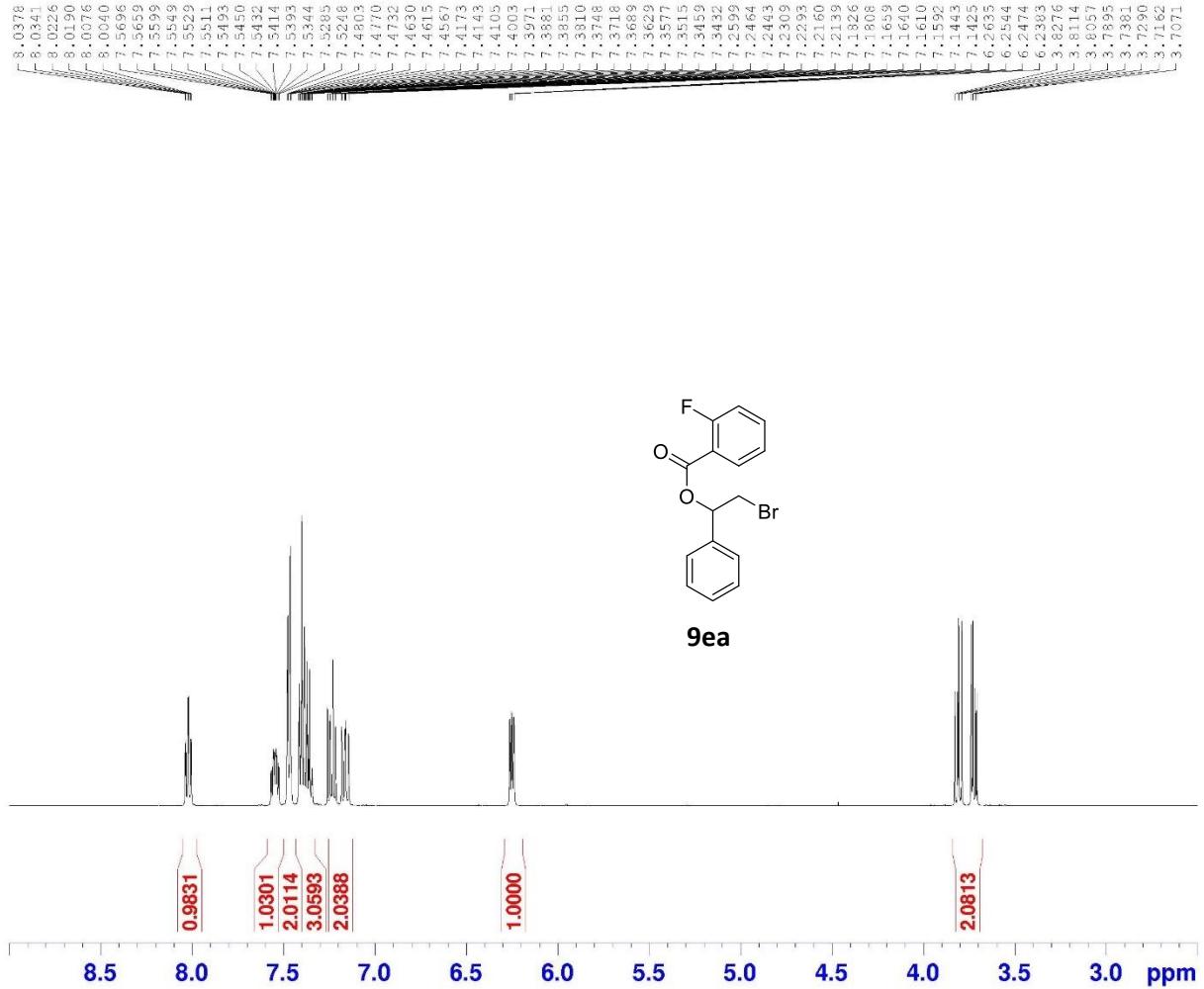




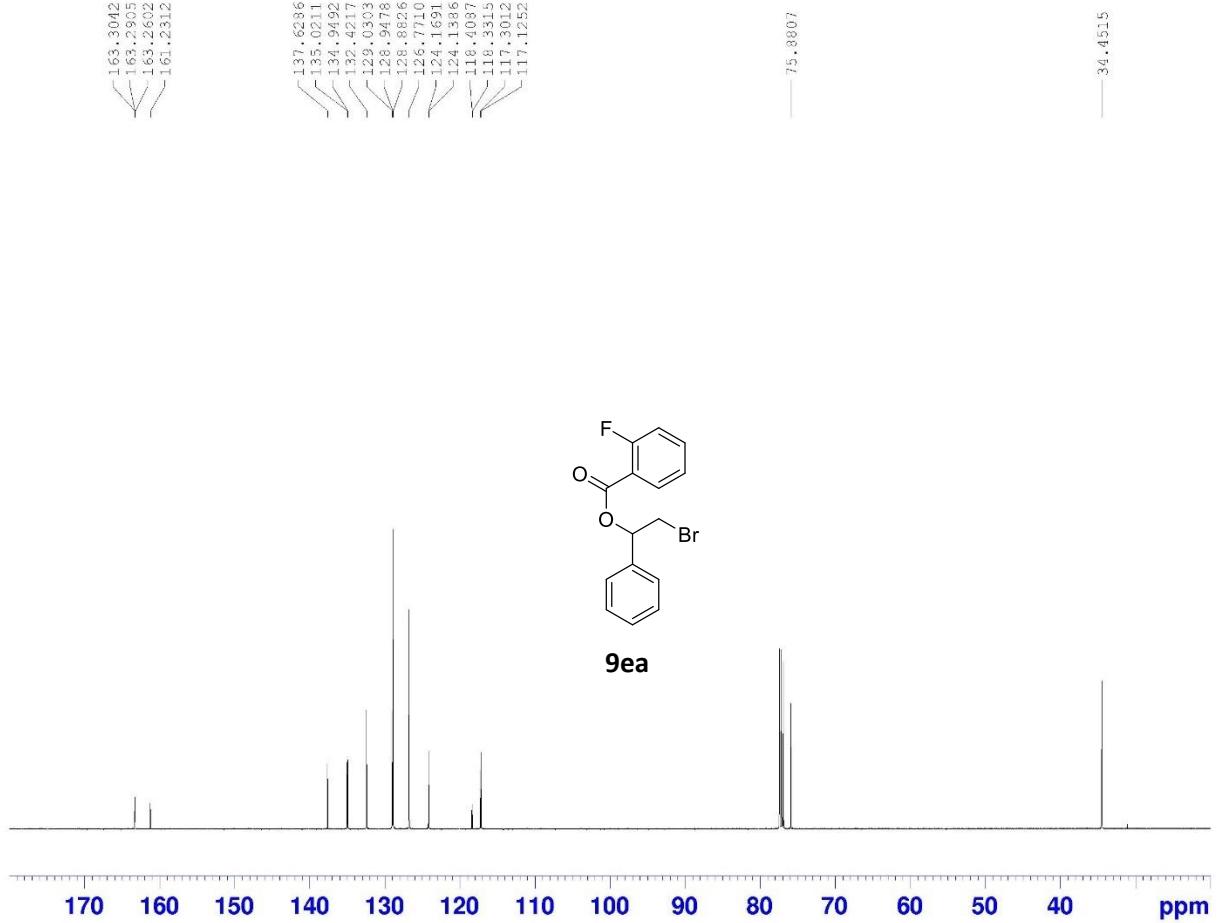


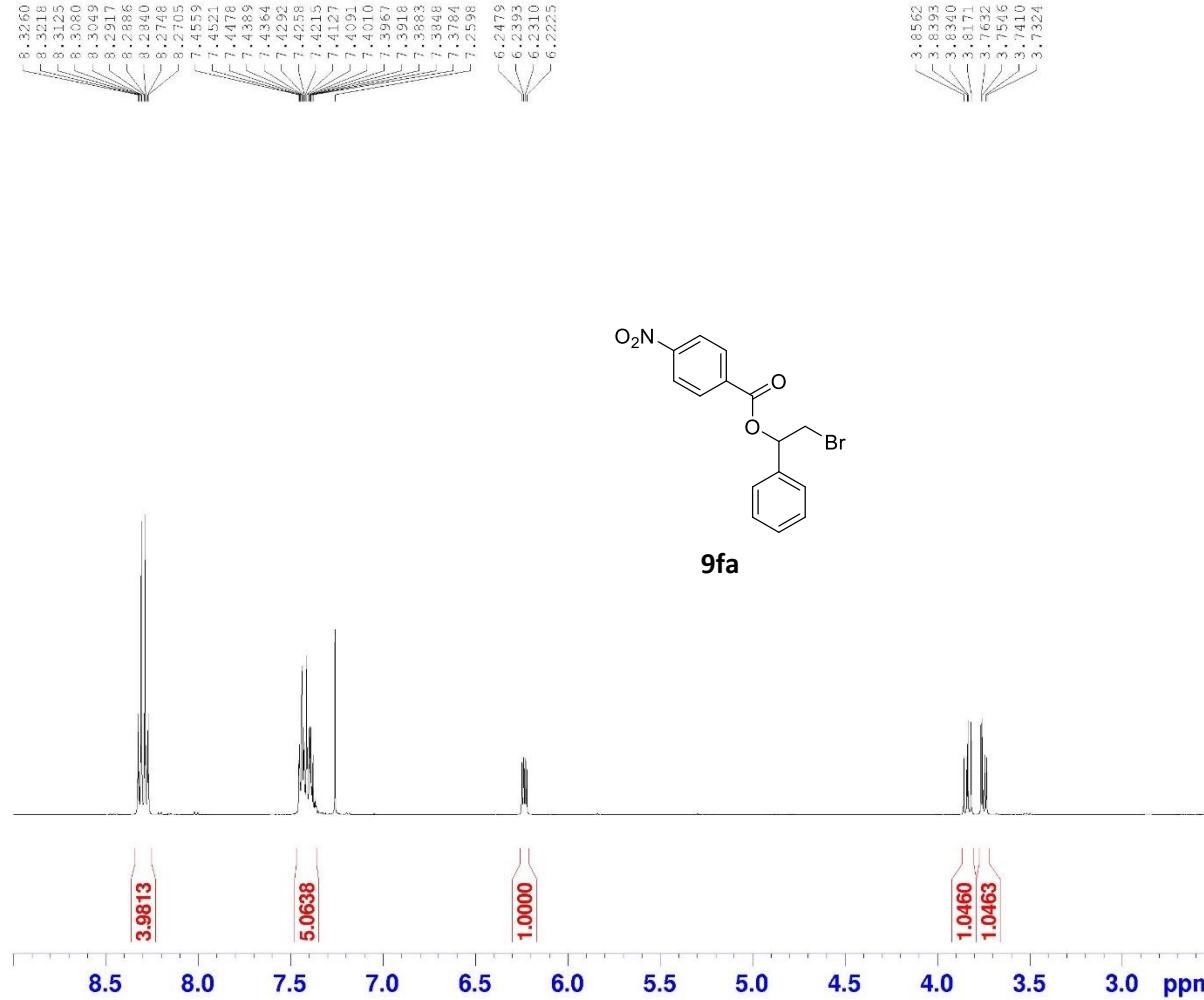
**9da**



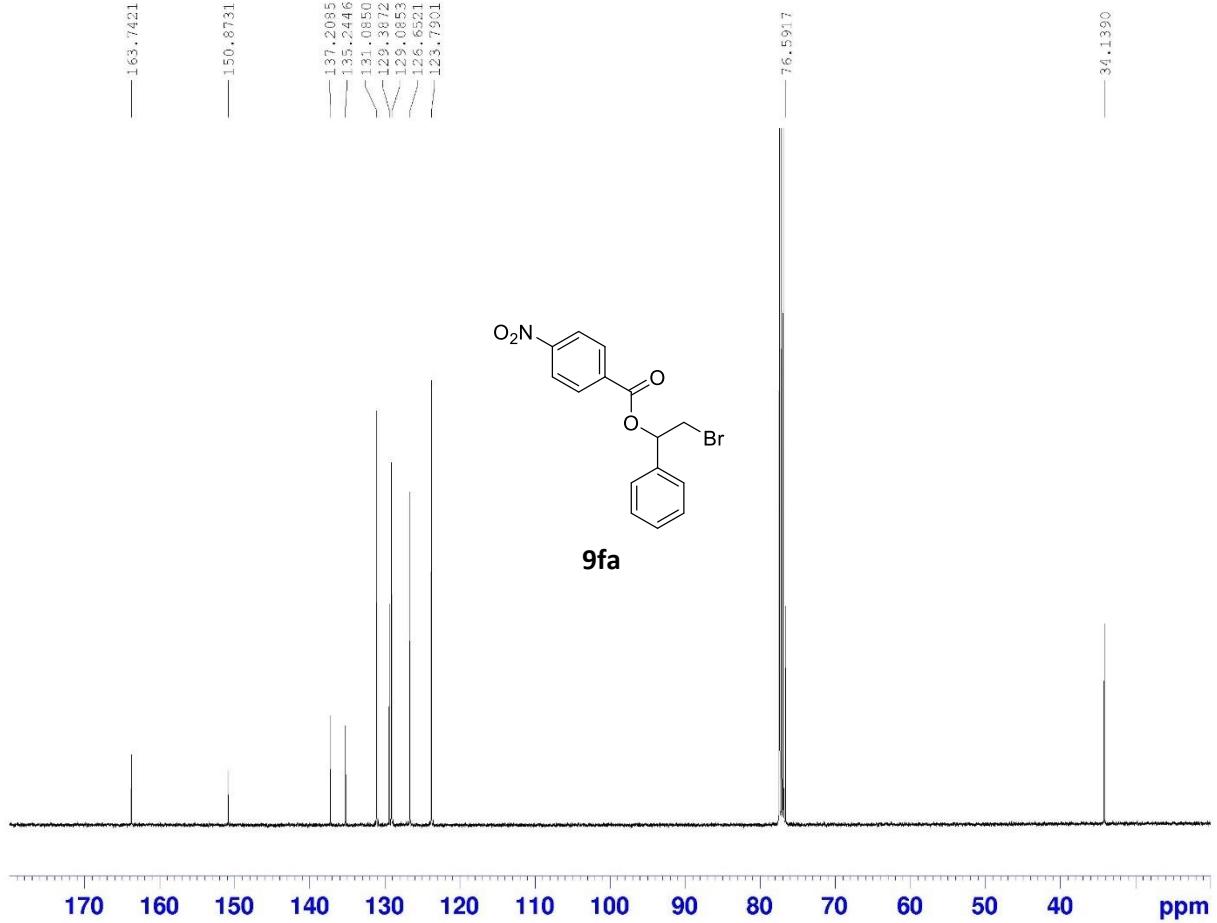


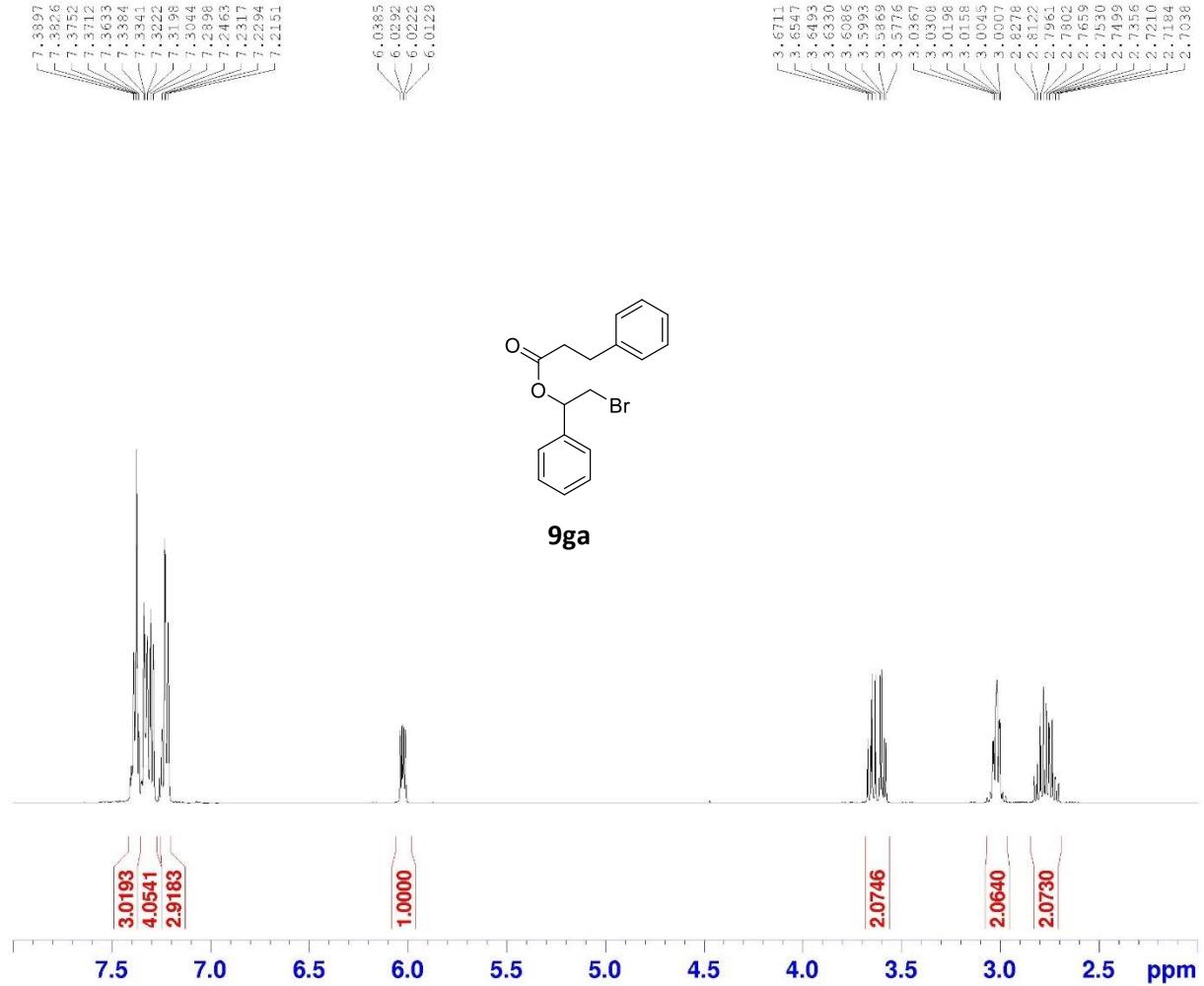
**9ea**



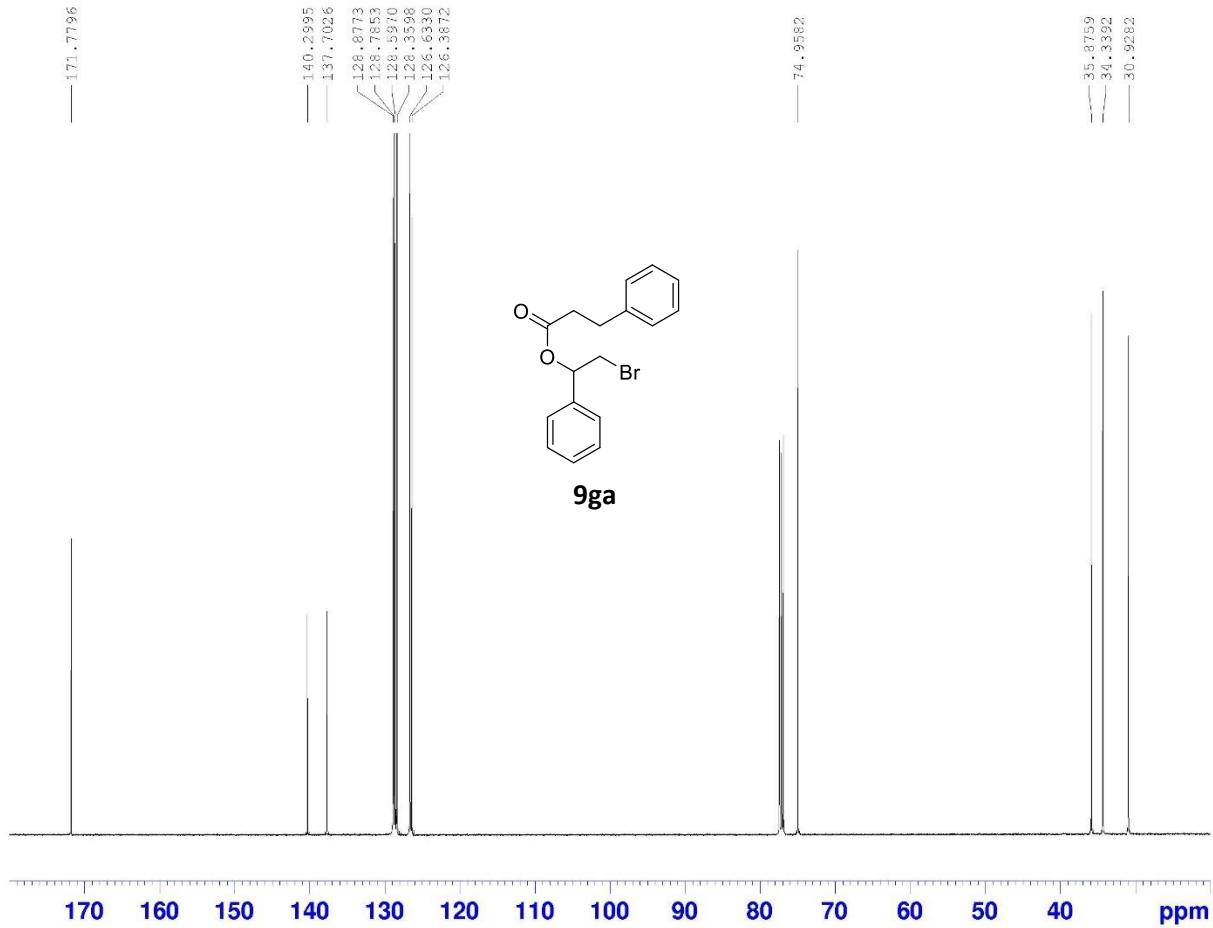


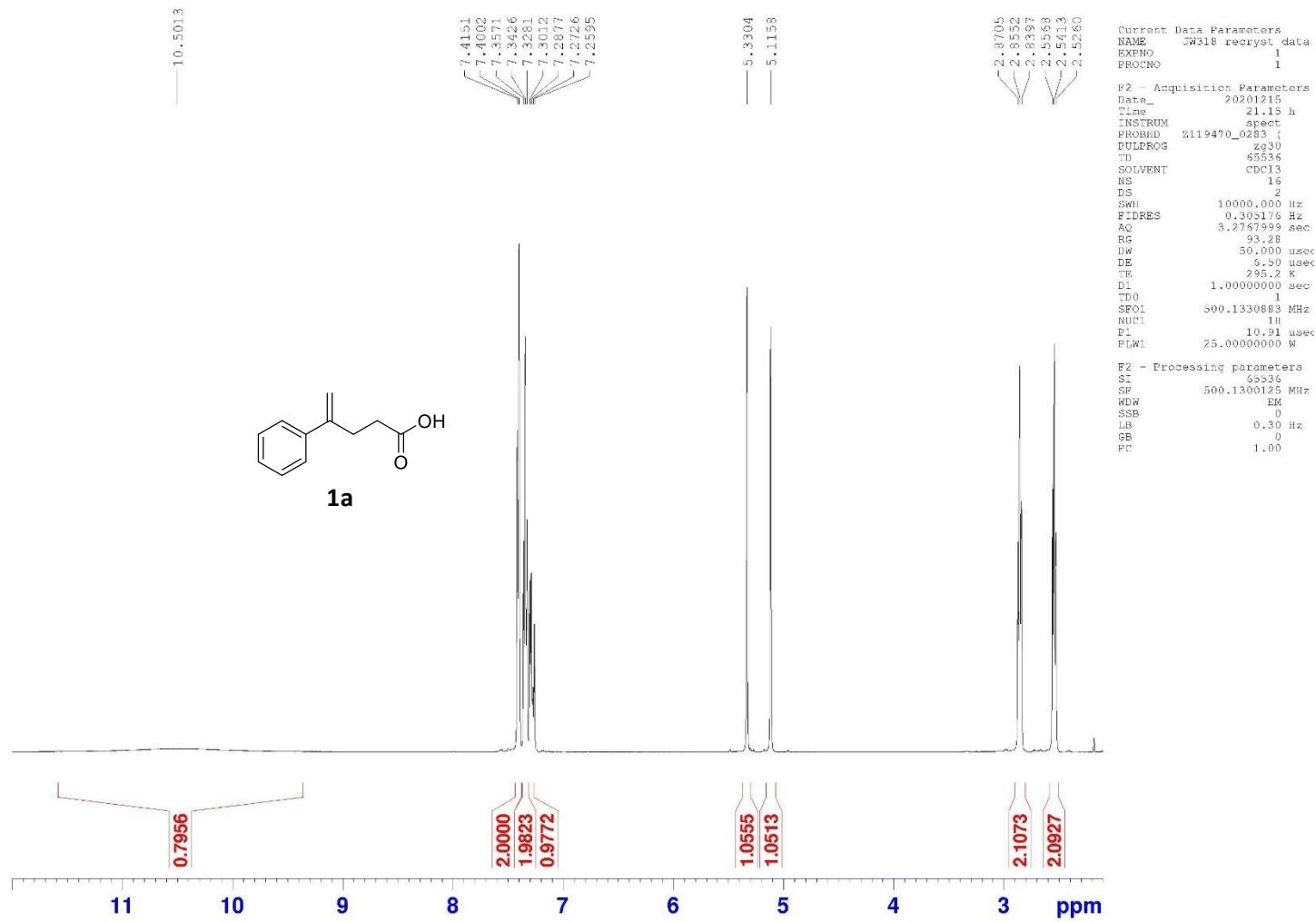
Current Data Parameters  
NAME :JWC379pm pure x  
EXPNO :1  
PROCNO :1  
  
P2 - Acquisition Parameters  
Data - 20190625  
Time - 17.33 h  
INSTRUM :spect  
PROBHD :Z149001\_0010 {  
PULPROG : zg30  
TD : 65536  
SOLVENT :CDC13  
NS : 16  
DS : 2  
SW0 : 10000.000 Hz  
FIDRES : 0.305176 Hz  
AQ : 3.2757999 sec  
RG : 30.85  
DW : 50.000 usec  
DE : 10.00 usec  
TE : 298.0 K  
D1 : 1.0000000 sec  
TD0 :  
SF01 : 500.1330683 MHz  
NUC1 : 1H  
F1 : 11.25 usec  
DW1 : 15.00000000 W  
  
P2 - Processing parameters  
SI : 65536  
SF : 500.1300125 MHz  
WDW : EM  
SSB : 0  
LB : 0.30 Hz  
GB : 0  
PC : 1.00

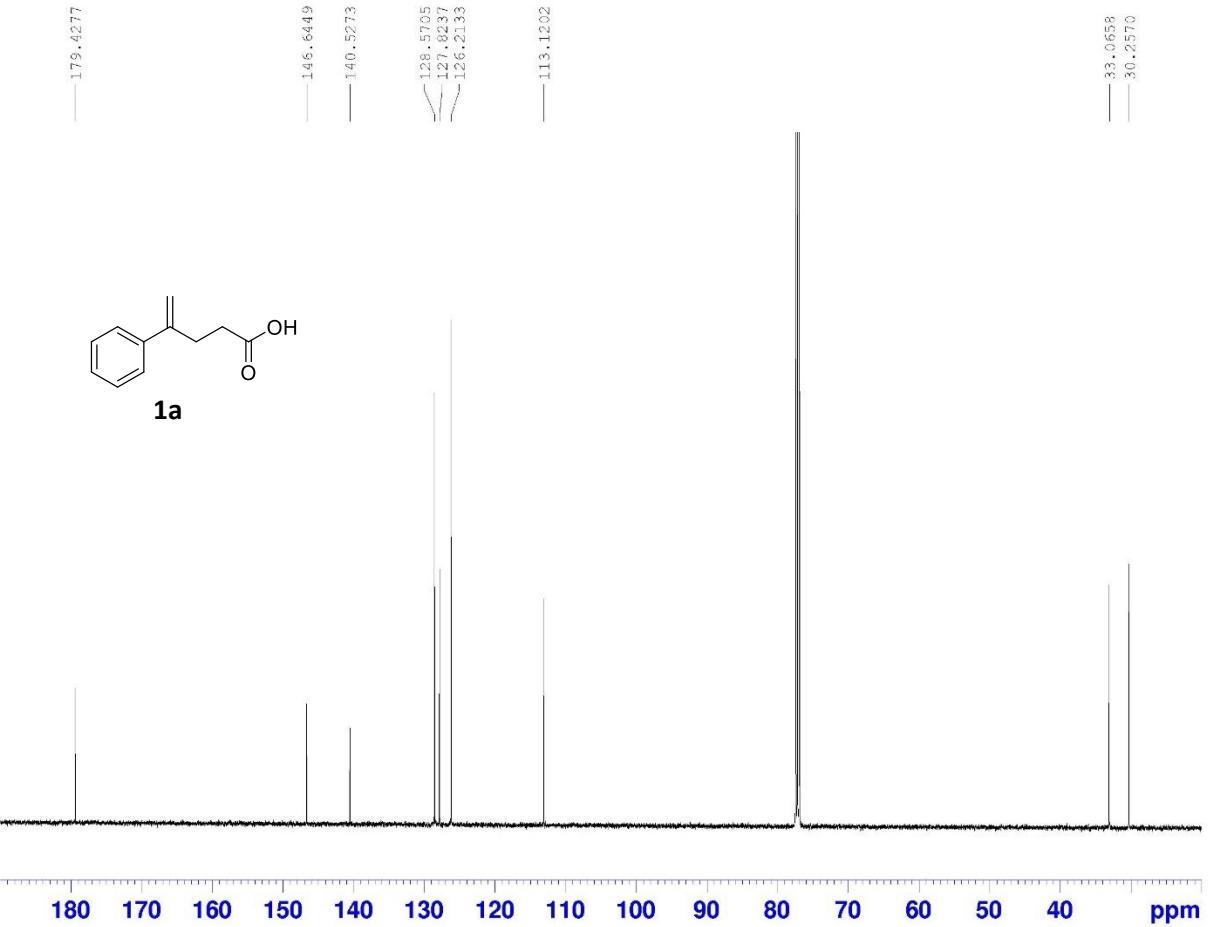


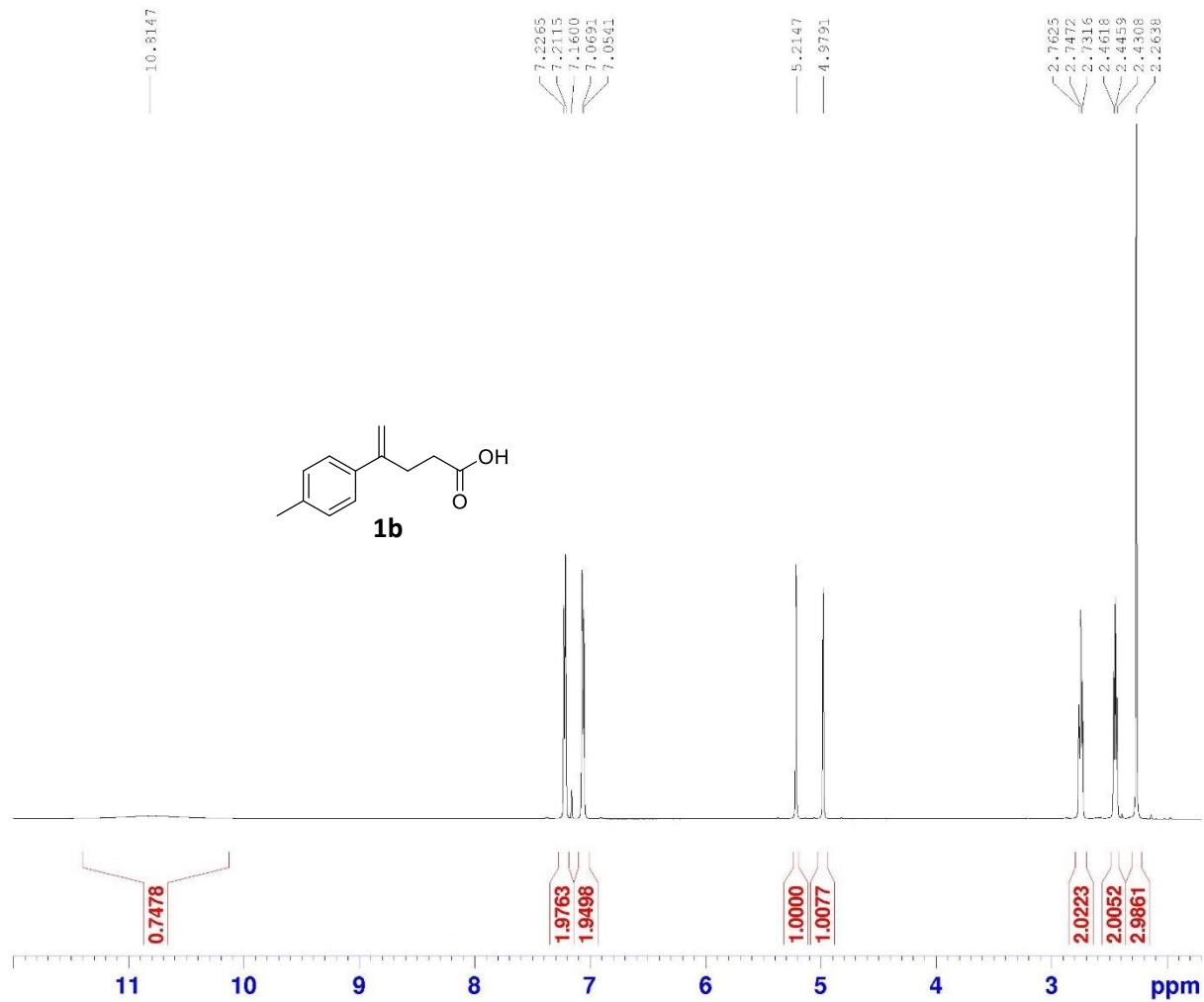


Current Data Parameters  
NAME JWC398 pm\_B.pure  
EXPNO 2  
PROCNO 1  
  
P2 - Acquisition Parameters  
Data\_2D 20190710  
Time 21.41 h  
INSTRUM spect  
PROBHD Z149001\_0010 {  
PULPROG zg30  
TD 65536  
T 10  
SOLVENT CDCl3  
NS 16  
DS 2  
SW0 10000.000 Hz  
FIDRES 0.305176 Hz  
AQ 3.2757999 sec  
RG 17.56  
DW 50.000 usec  
DE 10.00 usec  
TE 298.0 K  
D1 1.0000000 sec  
TD0 500.1330683 MHz  
RNUC 1H  
P1 11.25 usec  
FW1 15.0000000 W  
  
P2 - Processing parameters  
SI 65536  
SF 500.1300123 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

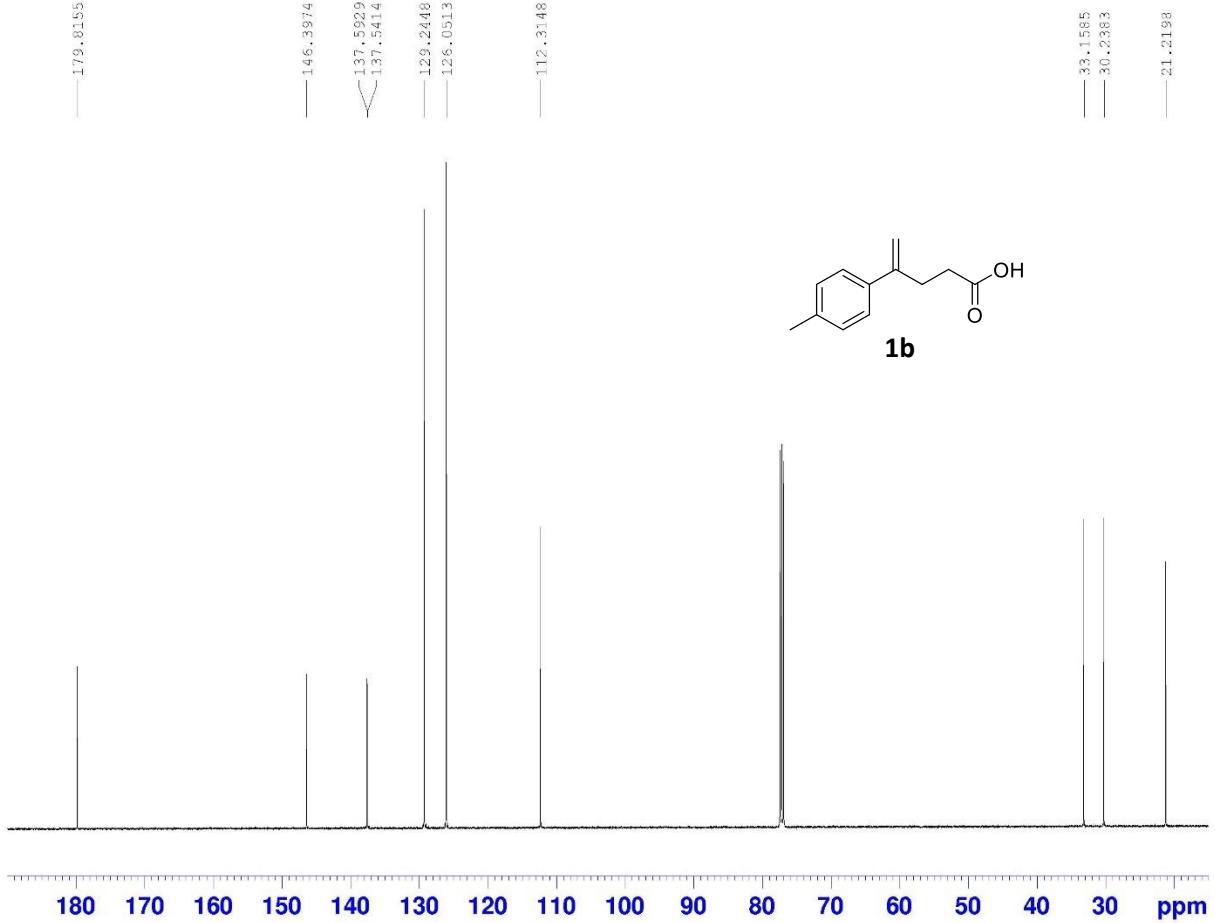


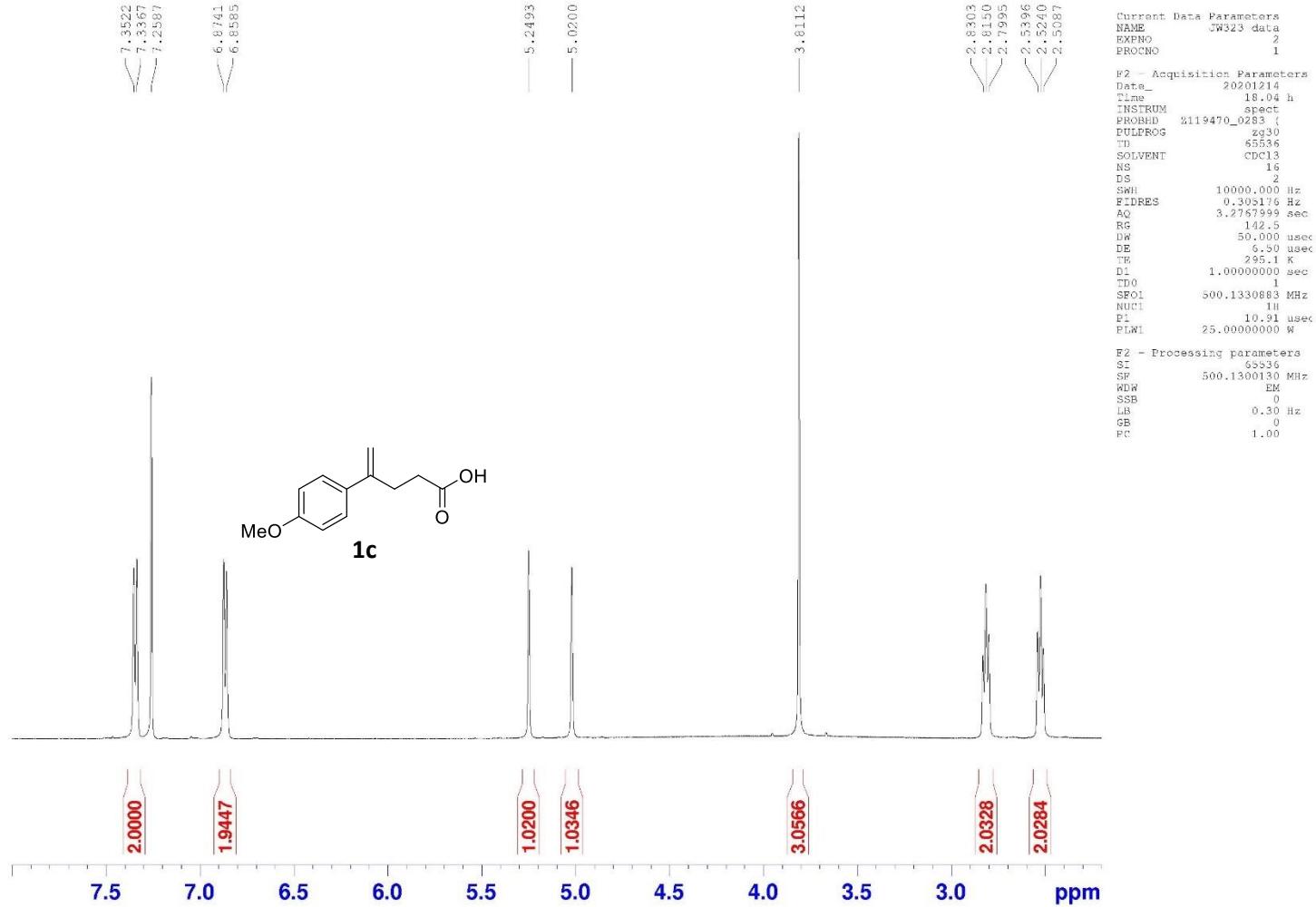


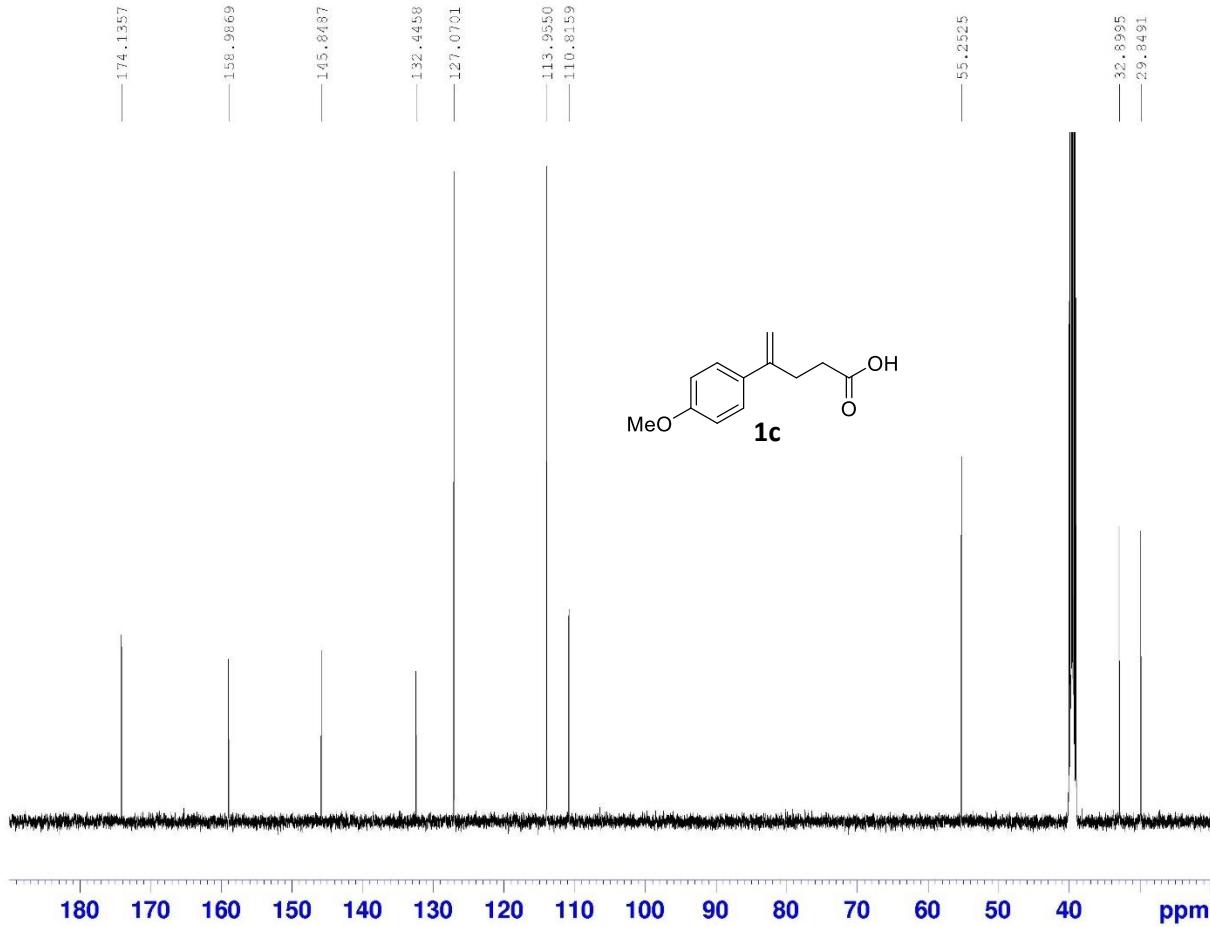




Current Data Parameters  
NAME JW344.dta  
EXPNO 1  
PROCNO 1  
  
P2 - Acquisition Parameters  
Data\_2D 20201210  
Time 23.37 h  
INSTRUM spect  
PROBHD Z119470\_0283\_1  
PULPROG zg30  
TD 65536  
T1 16  
TE 295.2 K  
TM 0.064  
SOLVENT CDCl3  
NS 16  
DS 2  
SW0 10000.000 Hz  
FIDRES 0.305176 Hz  
AQ 3.2757999 sec  
RG 50.6  
DW 50.000 usec  
DE 6.50 usec  
TE 295.2 K  
D1 1.0000000 sec  
TD0 500.1330625 MHz  
RNUC 1H  
P1 10.91 usec  
FW1 25.0000000 W  
  
P2 - Processing parameters  
SI 65536  
SF 500.1330625 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00







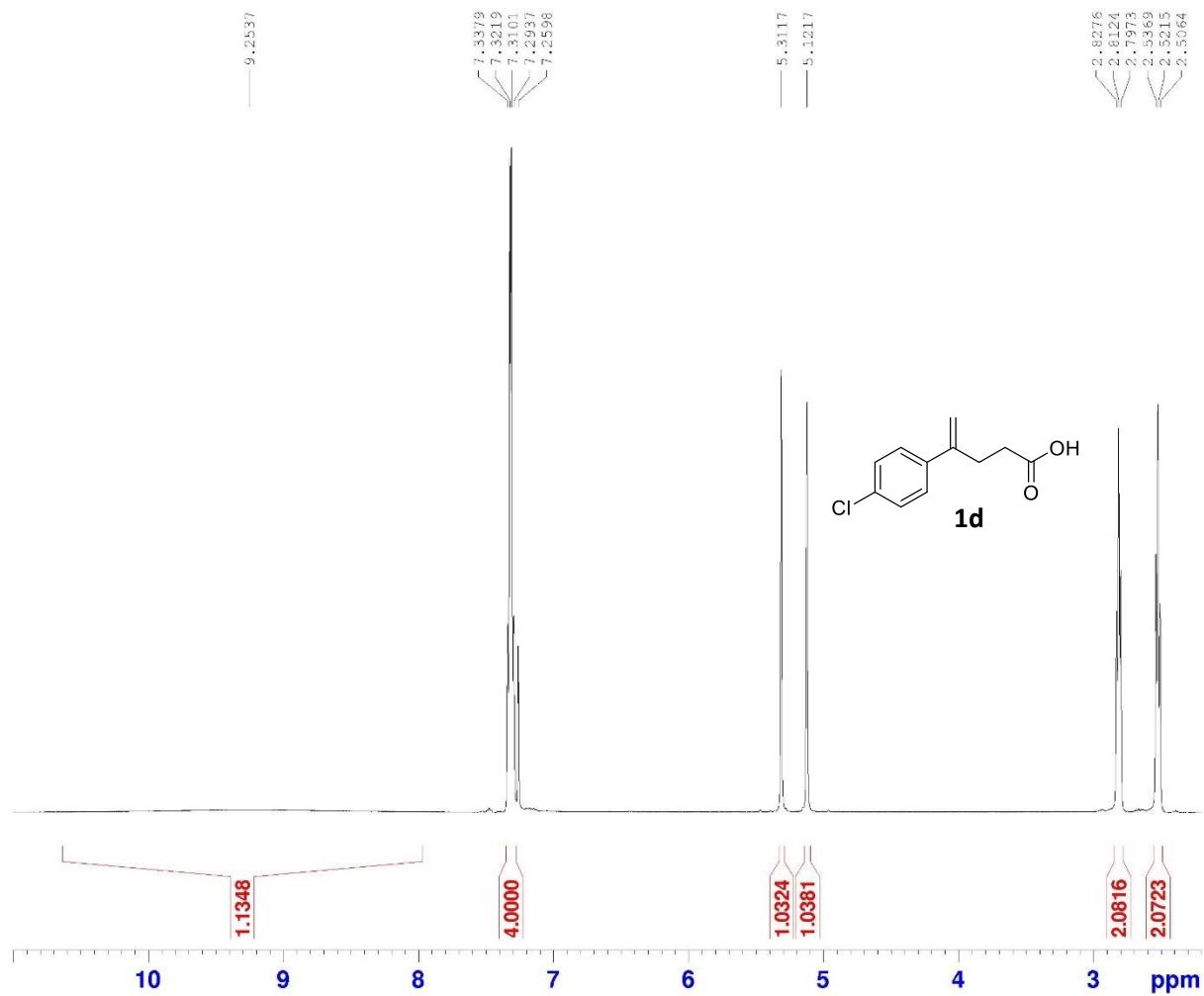
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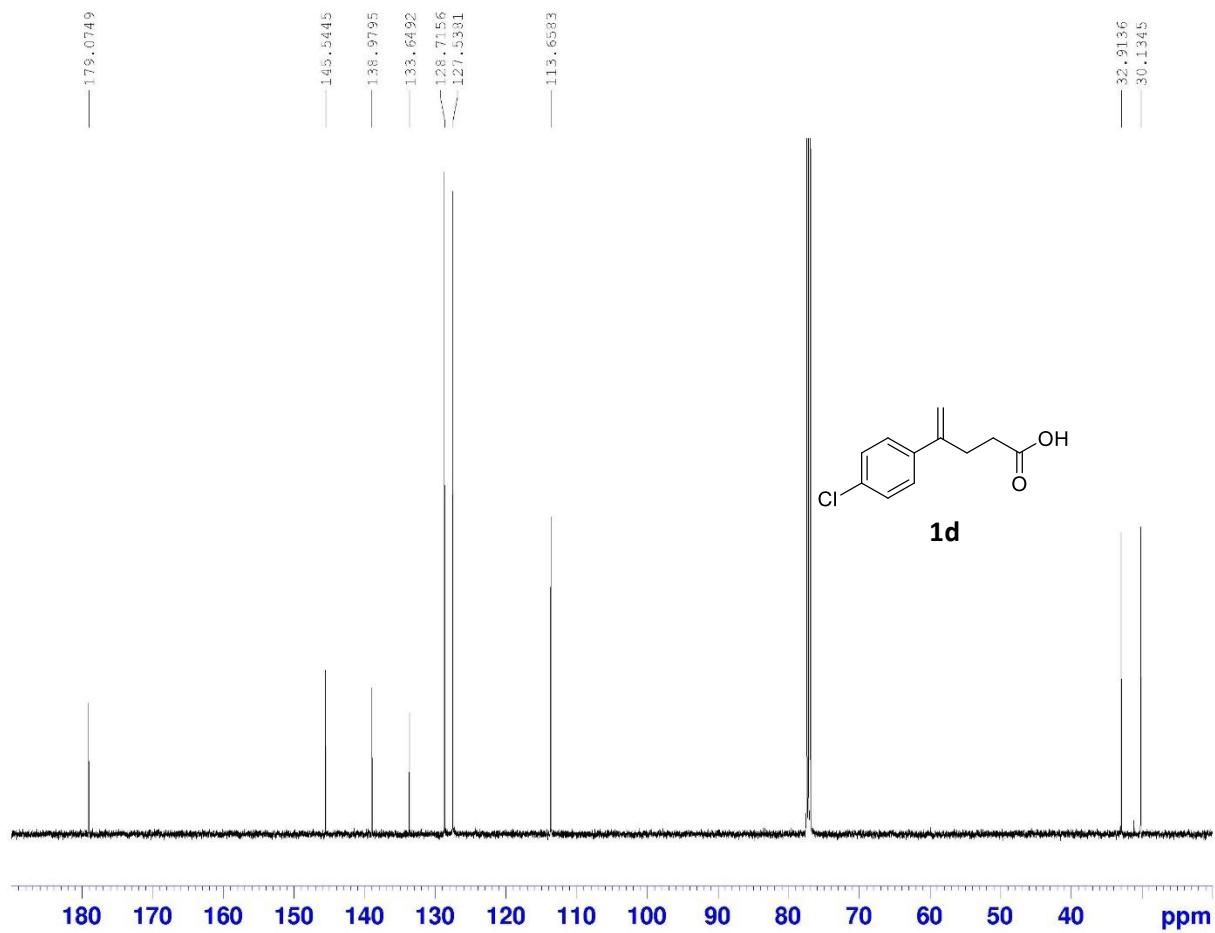
Current Data Parameters
NAME JW323 dmso data
EXPRO 1
PROCNO 1

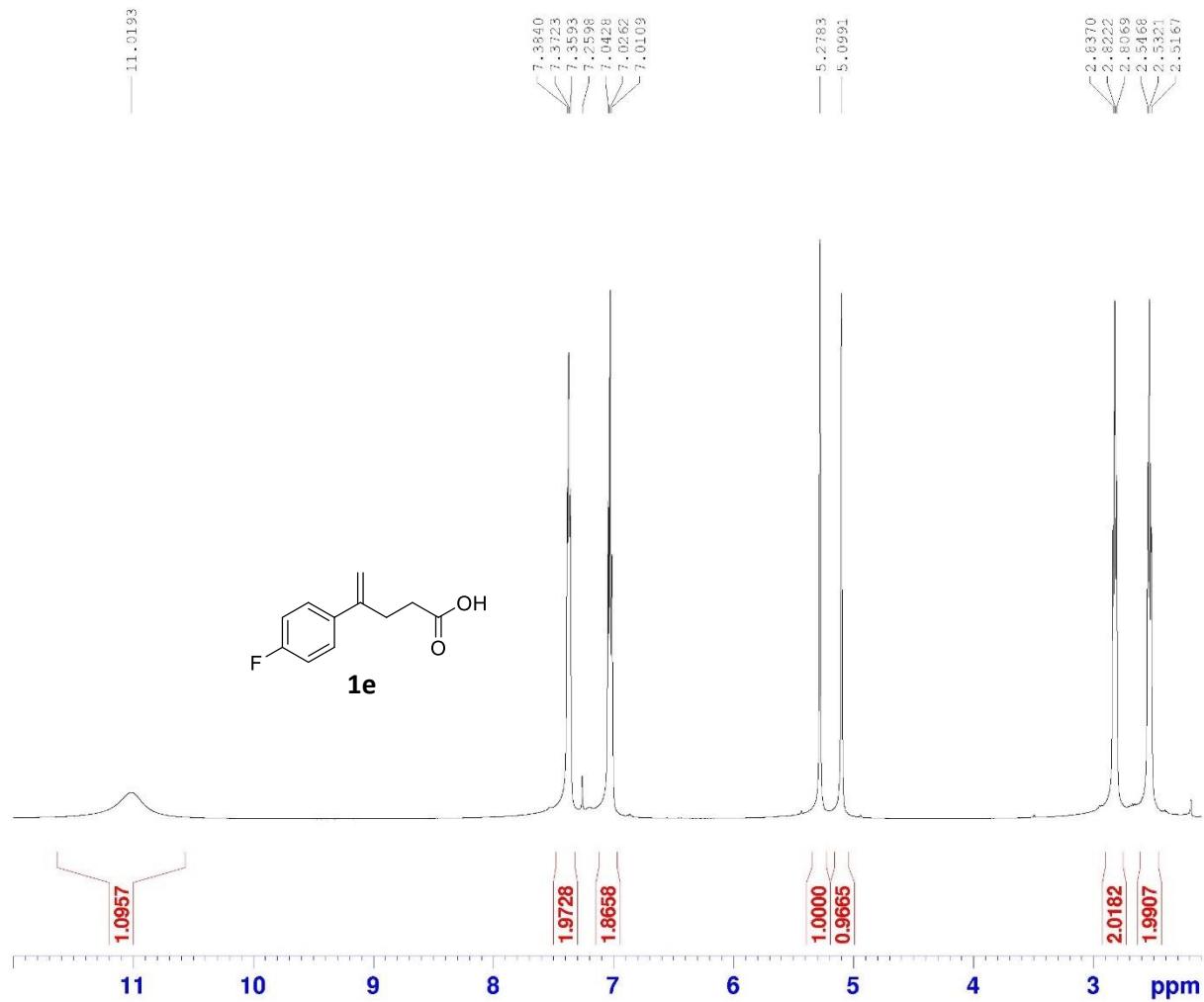
F2 - Acquisition Parameters
Date 20201215
Time 18.59 h
INSTRUM spect
PROBHD Z119470_0283
EQUILPROG zsp330
TD 65536
SOLVENT DMSO
NS 500
DS 4
SWH 29761.904 Hz
ETDRES 0.908261 Hz
AQ 1.1010048 sec
RG 205.72
DW 16.800 usec
DE 6.50 usec
TE 295.0 K
D1 2.0000000 sec
T1 0.0300000 sec
TD0 1
SFO1 125.7703433 MHz
NUC1 13C
P1 9.75 usec
PLW1 94.000000000 MHz
SF02 500.1320005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 8.00 usec
PLW2 25.000000000 W
PLW12 0.46495000 W
PLW13 0.23387000 W

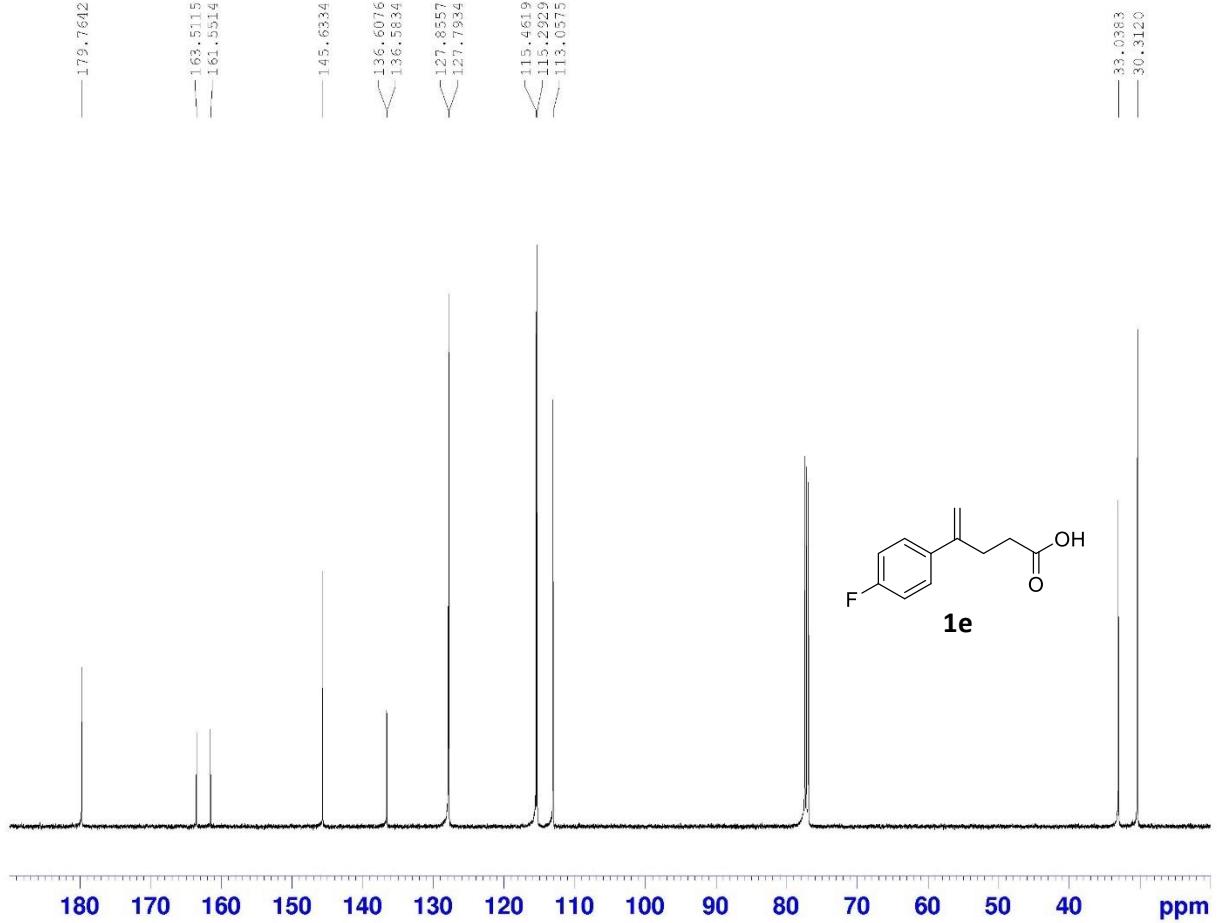
F2 - Processing parameters
SI 32768
SF 125.7578242 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

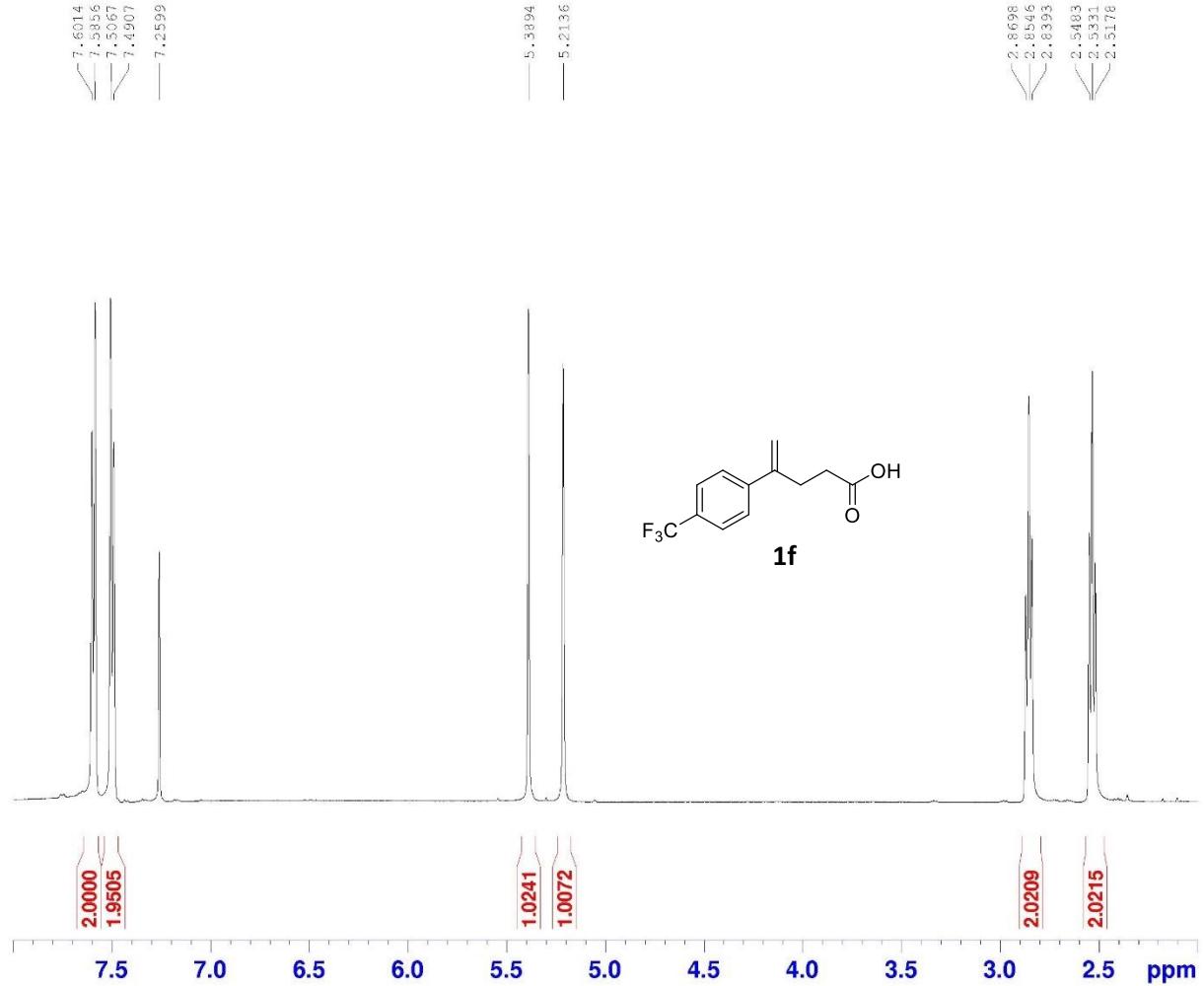
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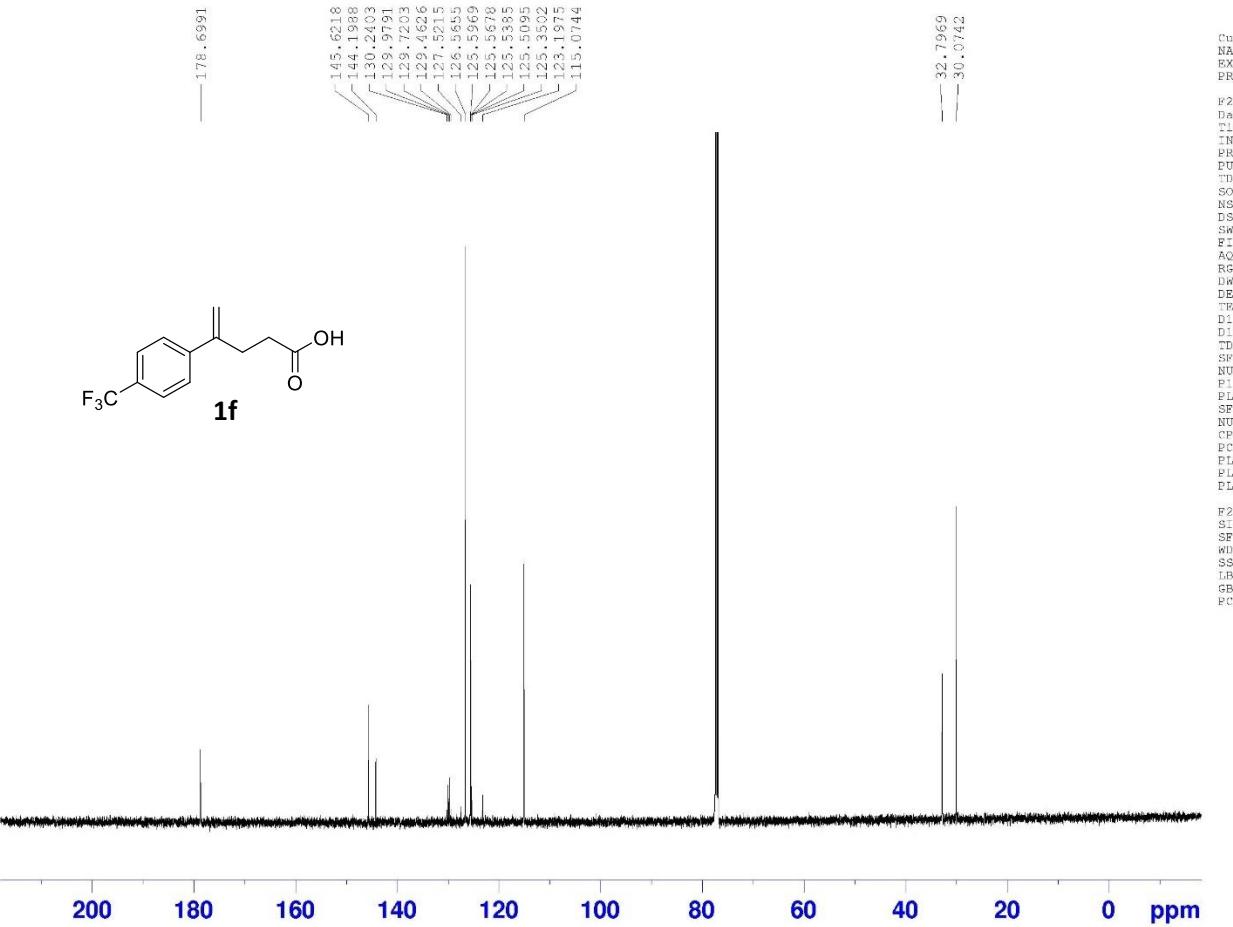


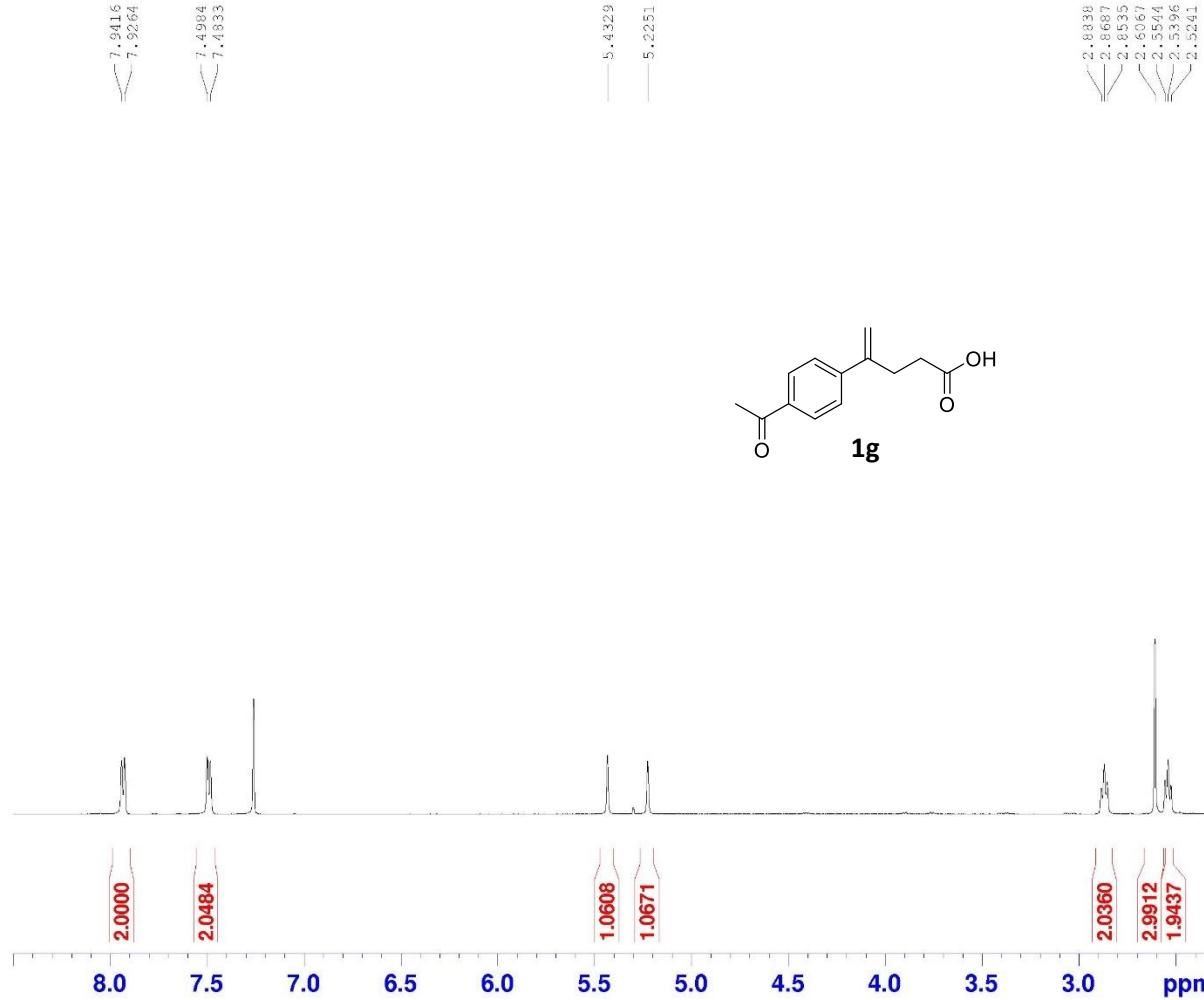




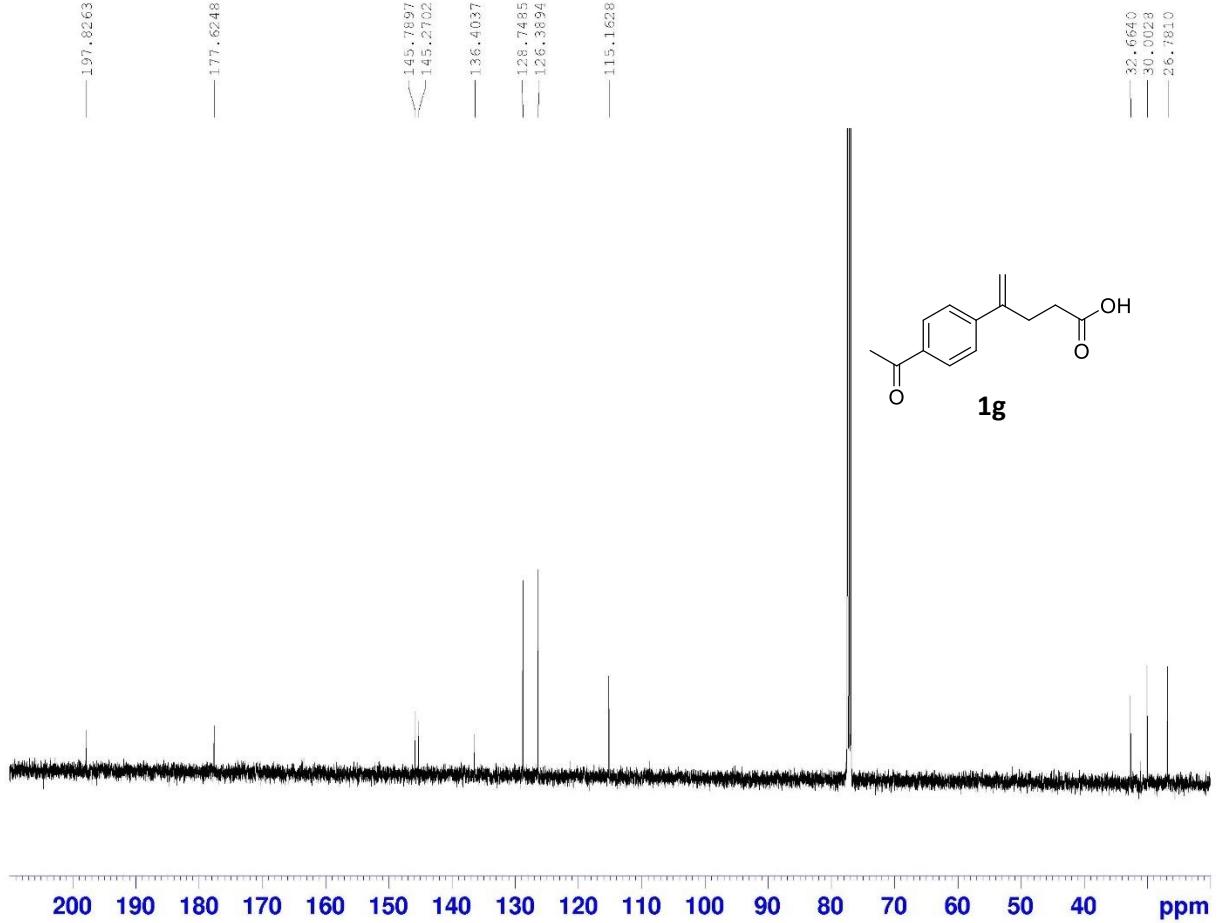


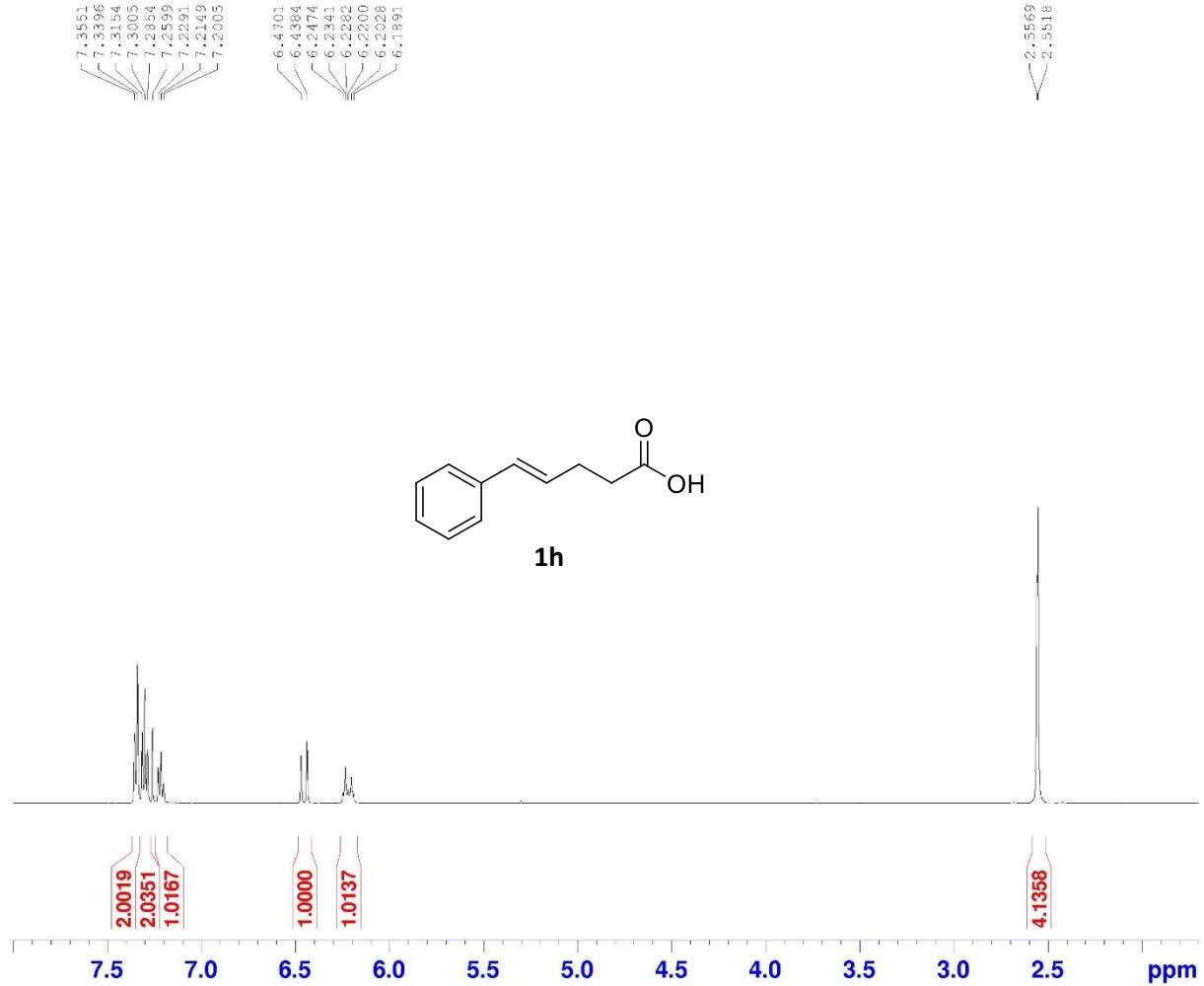
Current Data Parameters  
NAME CW287\_ptlc\_data  
EXPNO 2  
PROCNO 1  
  
P2 - Acquisition Parameters  
Data\_2D 20201223  
Time 22.29 h  
INSTRUM spect  
PROBHD Z119470\_0283\_1  
PULPROG zg30  
TD 65536  
T1 160  
SW1 55536  
SOLVENT CDCl3  
NS 16  
DS 2  
SW0 10000.000 Hz  
FIDRES 0.305176 Hz  
AQ 3.2757999 sec  
RG 142.5  
DW 50.000 usec  
DE 6.50 usec  
TE 295.2 K  
D1 1.0000000 sec  
TD0 500.1330683 MHz  
RNUC 1H  
P1 10.91 usec  
FW1 25.0000000 W  
  
P2 - Processing parameters  
SI 65536  
SF 500.1300121 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

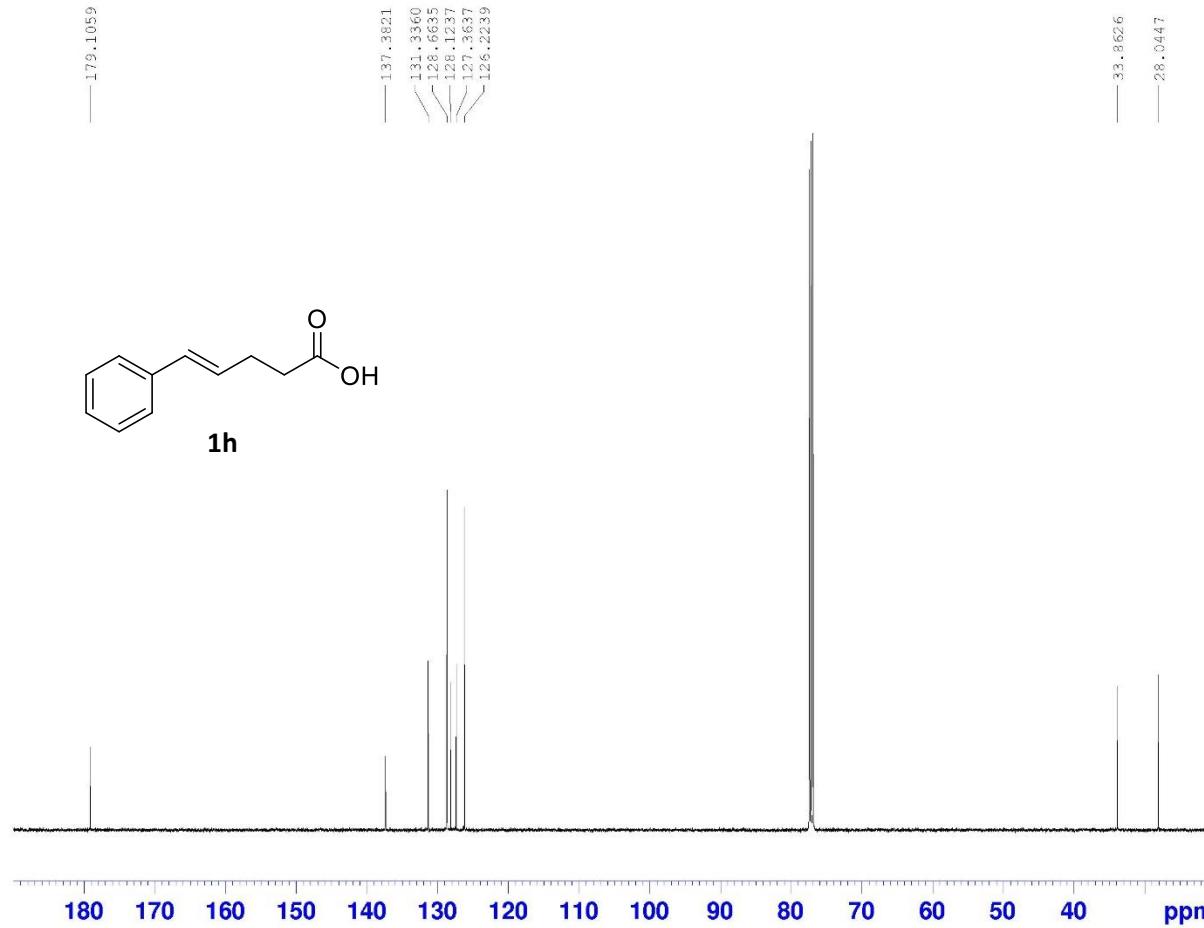
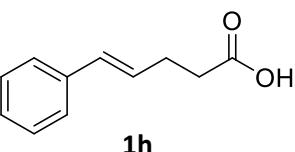




Current Data Parameters  
NAME CW289 recryst data  
EXPNO 1  
PROCNO 1  
  
P2 - Acquisition Parameters  
Date 20201215  
Time 21:56 h  
INSTRUM spect  
PROBHD Z119470\_0283  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 9  
DS 2  
SW0 10000.000 Hz  
FIDRES 0.305176 Hz  
AQ 3.2757999 sec  
RG 142.5  
DW 50.000 usec  
DE 6.50 usec  
TE 295.1 K  
D1 1.0000000 sec  
TD0 500.1330683 MHz  
RNUC 1H  
P1 10.91 usec  
FW1 25.0000000 W  
  
P2 - Processing parameters  
SI 65536  
SF 500.1300124 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



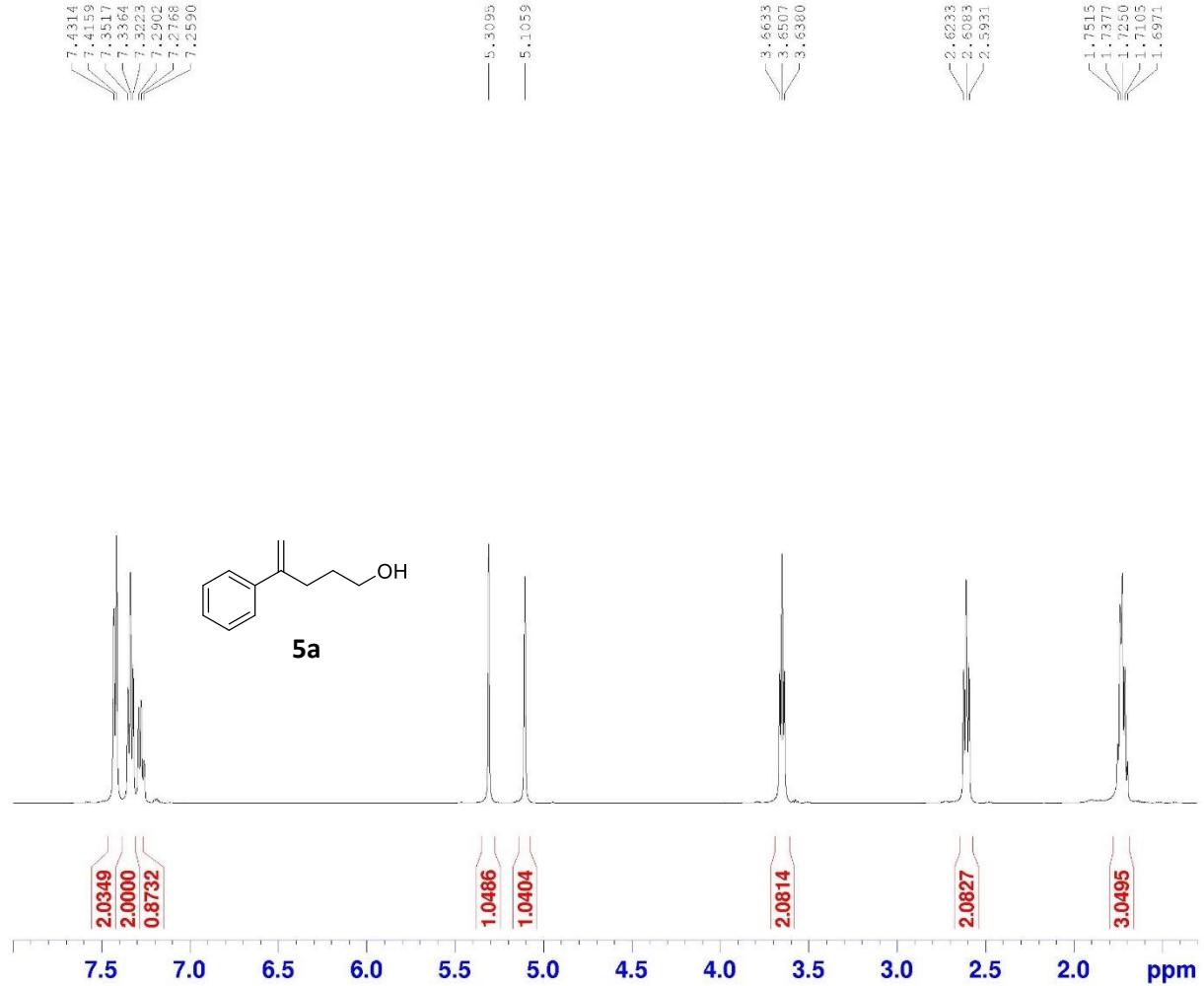




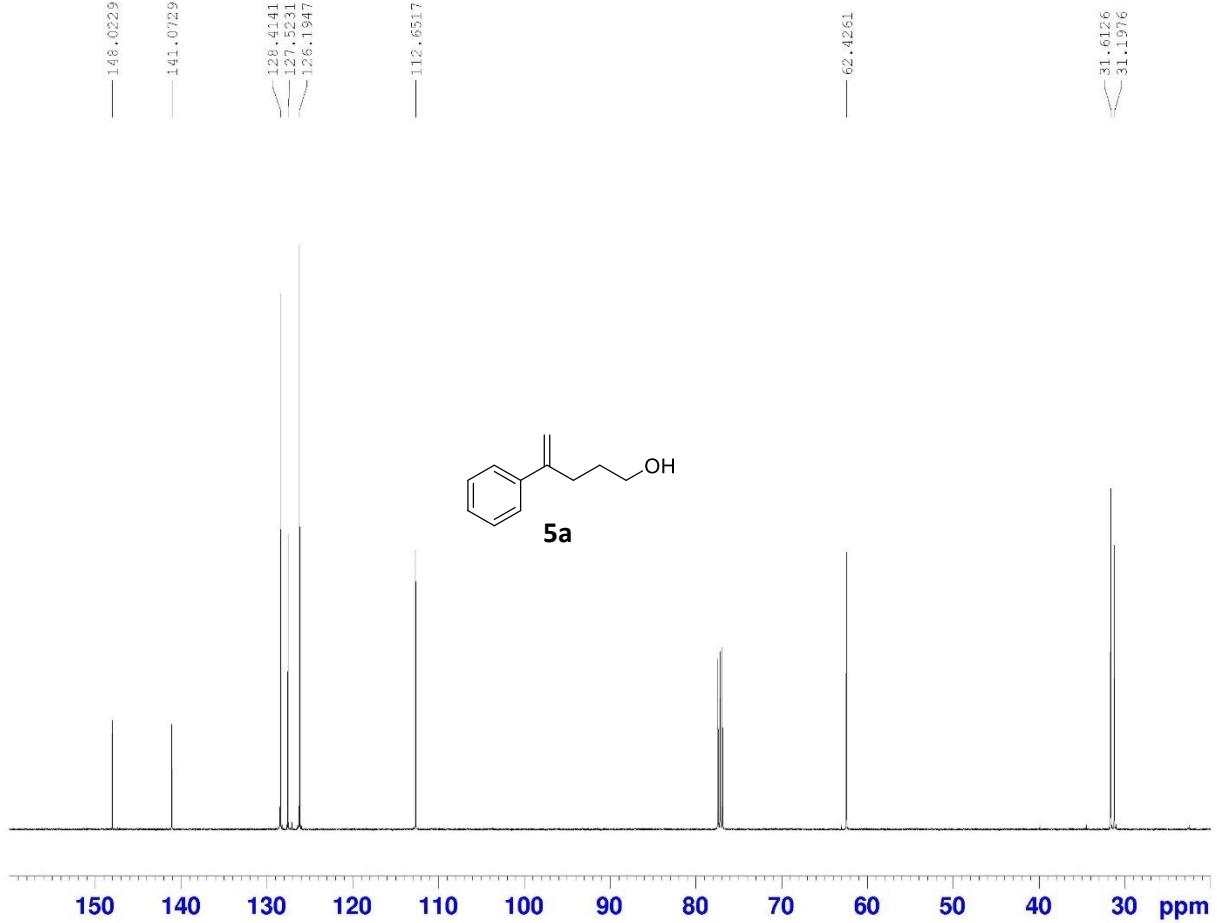
Current Data Parameters  
NAME JW312\_data  
EXPNO 3  
PROCNO 1

P2 - Acquisition Parameters  
Date\_ 20210113  
Time\_ 22.28 h  
INSTRUM spect  
PROBHD Z119470\_0283 {  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 1024  
DS 1  
SWH 29761.904 Hz  
ETDRES 0.998261 Hz  
AQ 1.1010048 sec  
RG 142.5  
DW 16.800 usec  
DE 6.50 usec  
TE 295.2 K  
D1 2.0000000 sec  
DL 0.0300000 sec  
TDO 125.7703643 MHz  
NUC1 13C  
PL 9.75 usec  
PLW1 94.00000000 W  
SF02 500.1320005 MHz  
NUC2 1H  
CPDPFG[2] waltz16  
PCPD2 80.00 usec  
PLW2 25.00000000 W  
PLW12 0.45495000 W  
PLW13 0.23387000 W

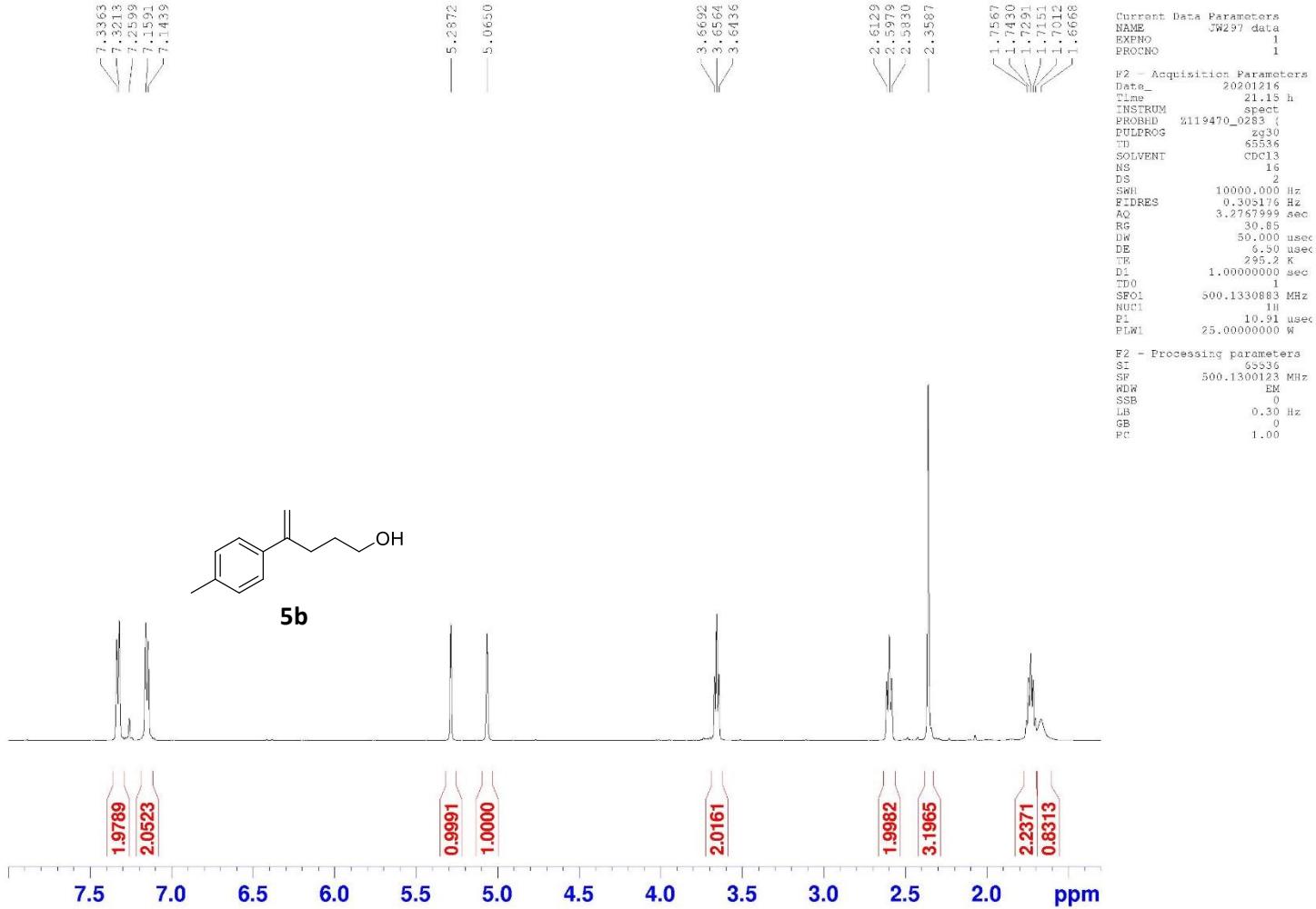
P2 - Processing parameters  
SI 32768  
SF 125.7577737 MHz  
WDW HM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

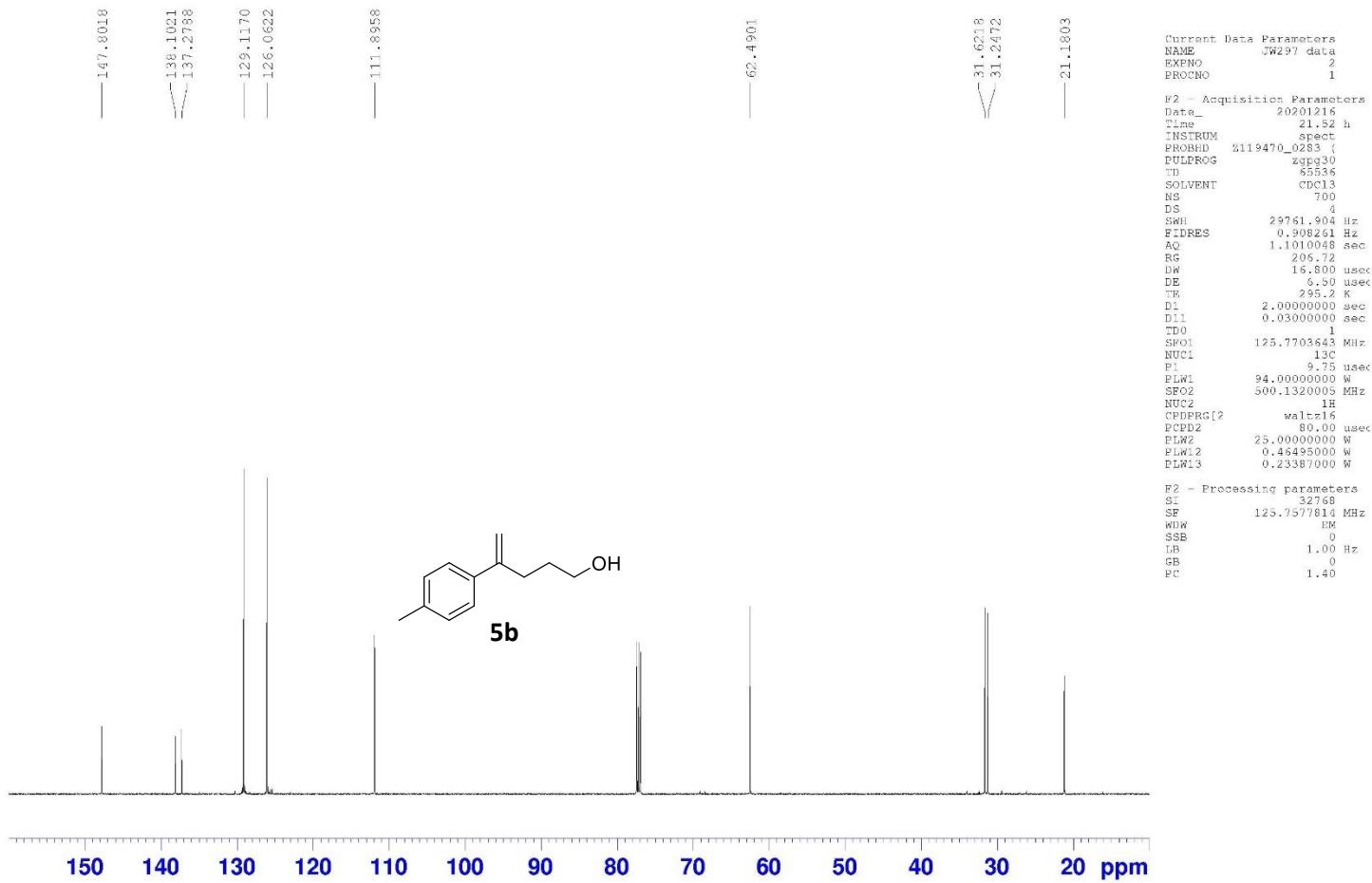


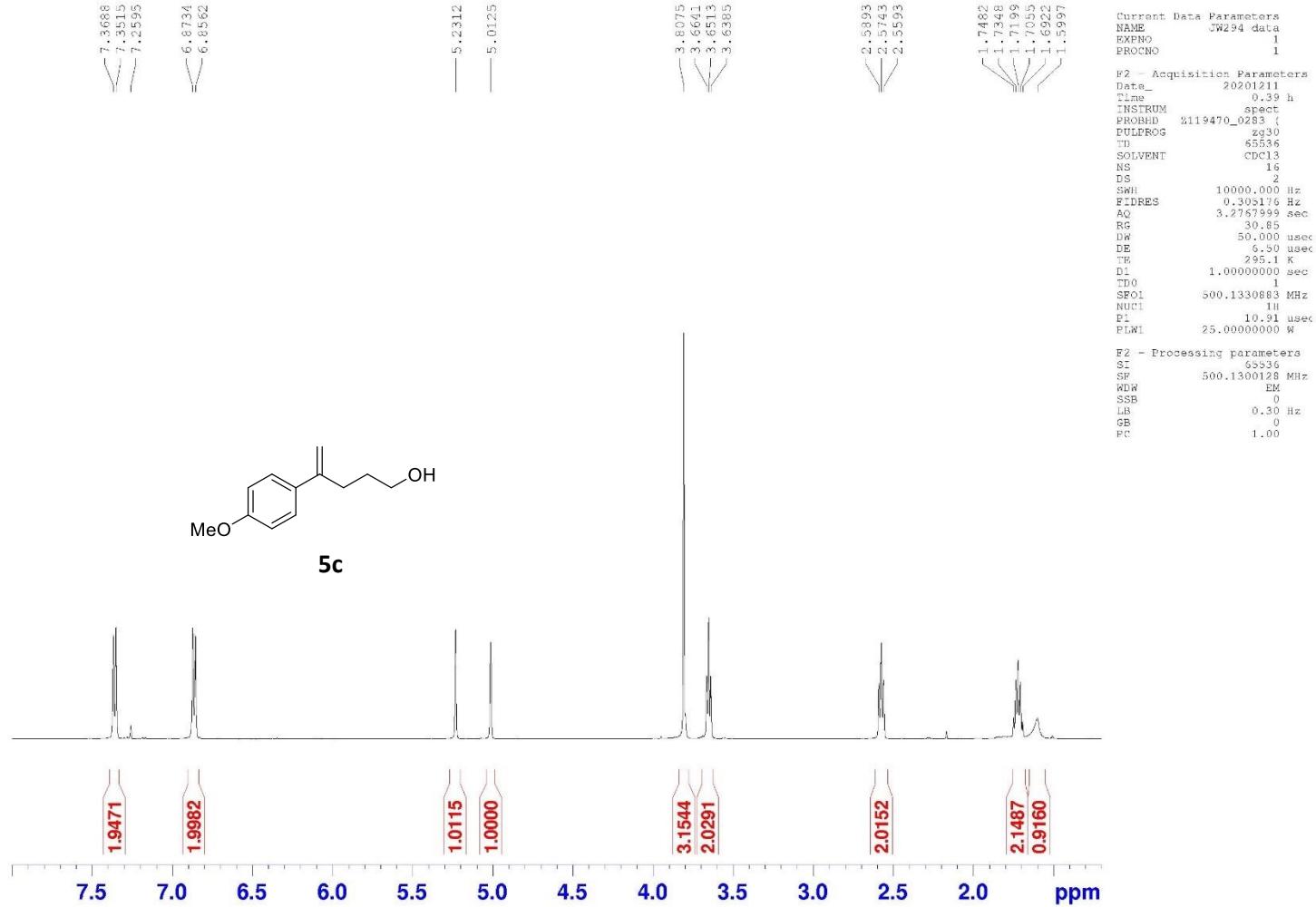
Current Data Parameters  
NAME JW340\_data  
EXPNO 1  
PROCNO 1  
  
P2 - Acquisition Parameters  
Data\_2D 20201202  
Time 20.34 h  
INSTRUM spect  
PROBHD Z119470\_0283\_1  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 10000.000 Hz  
FIDRES 0.305176 Hz  
AQ 3.2757999 sec  
RG 30.85  
DW 50.000 usec  
DE 6.50 usec  
TE 295.2 K  
D1 1.0000000 sec  
TD0 500.1330683 MHz  
RNUC 1H  
F1 10.91 usec  
FW1 25.0000000 W  
  
P2 - Processing parameters  
SI 65536  
SF 500.1300147 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

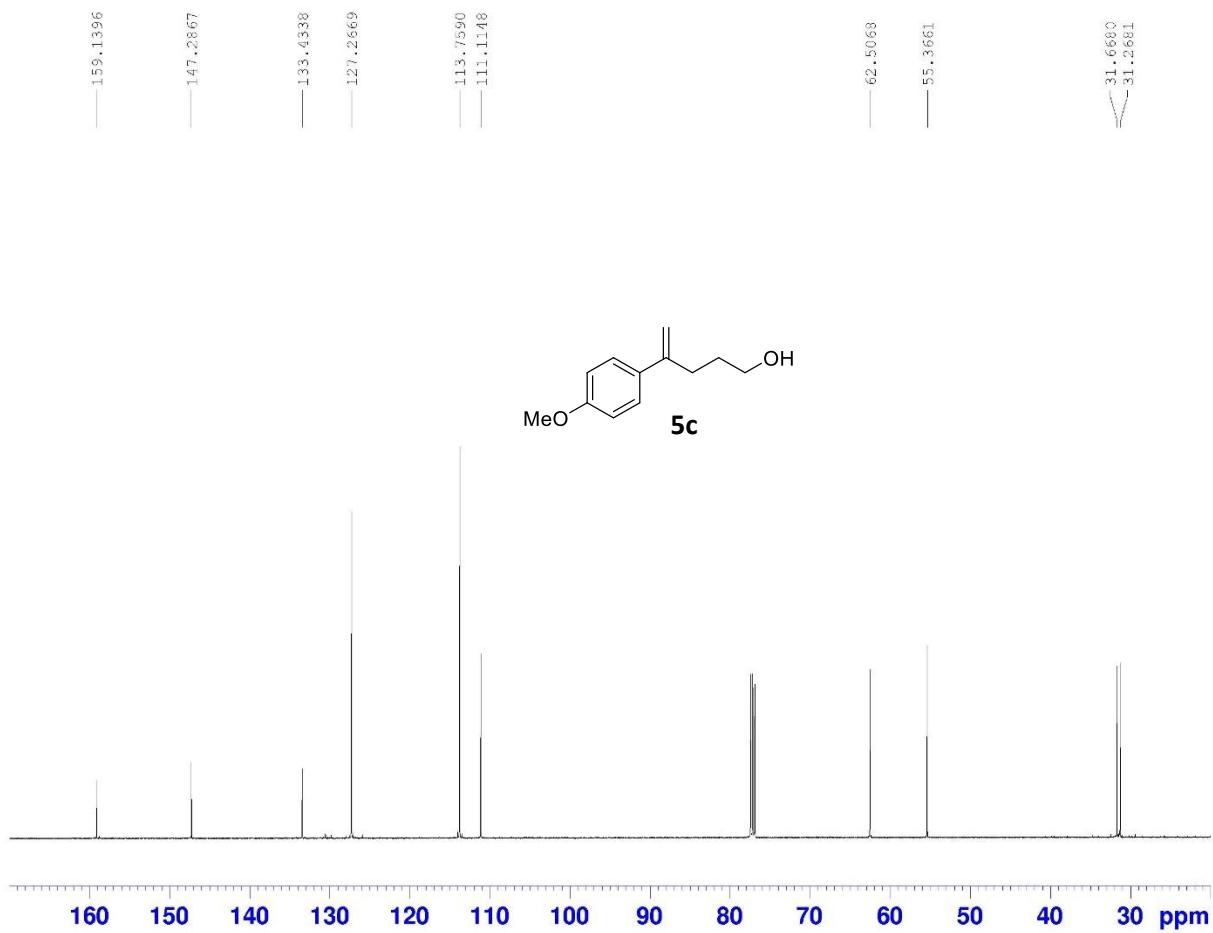


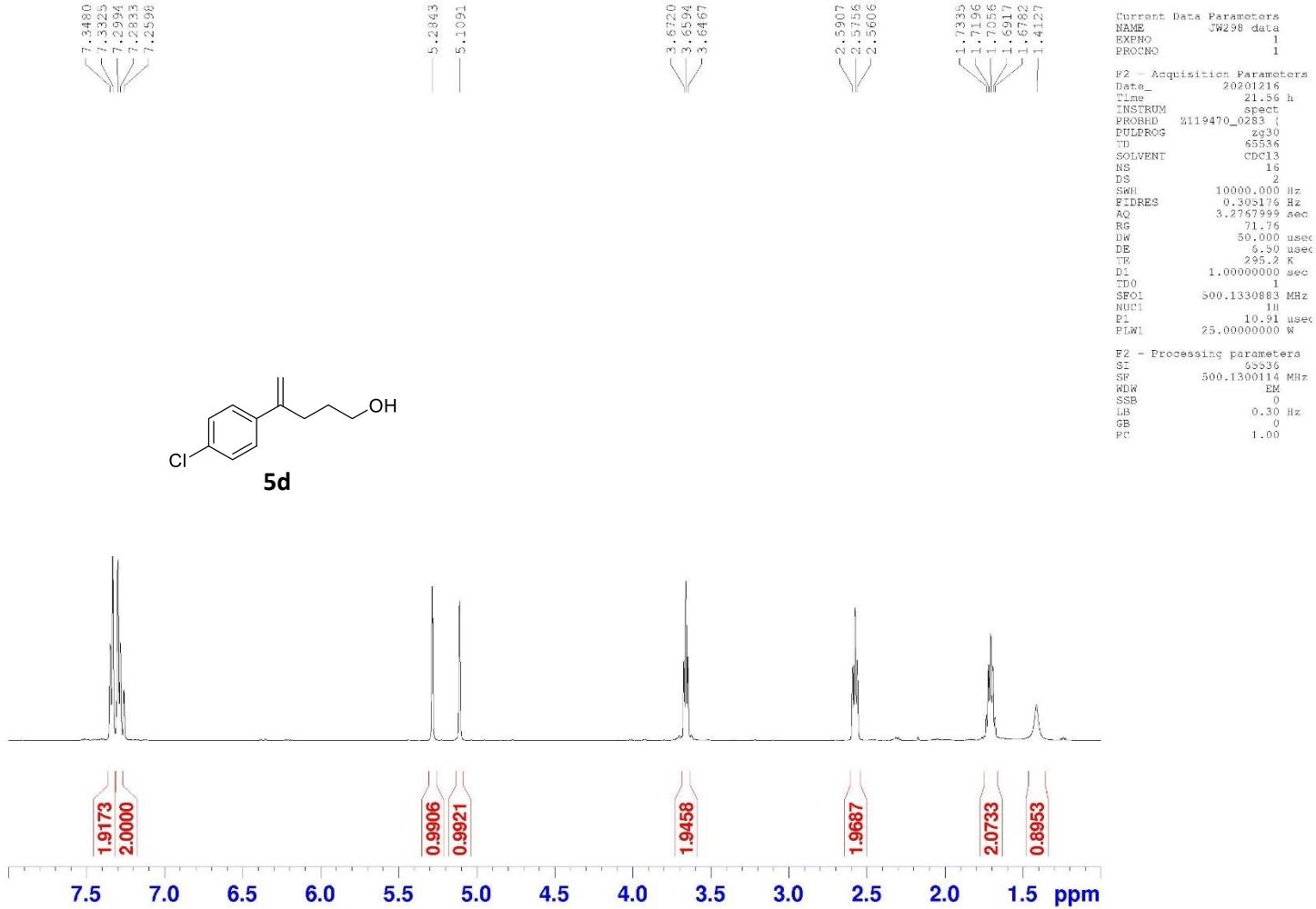
Current Data Parameters  
NAME JW340\_data  
EXPNO 2  
PROCNO 1  
  
P2 - Acquisition Parameters  
Data\_20201202  
Time 21.07 h  
INSTRUM spect  
PROBHD Z119470\_0283  
PULPROG zgpp30  
TD 65536  
T1 600  
SOLVENT CDCl3  
NS 600  
DS 8  
SW0 29761.904 Hz  
FIDRES 0.908261 Hz  
AQ 1.1010048 sec  
RG 205.72  
DW 16.800 usec  
DE 6.50 usec  
TE 295.1 K  
D1 2.0000000 sec  
DL 0.0300000 sec  
DP0  
SW1 125.7703643 MHz  
NUC1 13C  
P1 9.75 usec  
PLW1 94.00000000 w  
SF02 500.1320005 MHz  
NUC2 1H  
CPDPRG[2] waltz16  
PCPD2 80.00 usec  
PLW2 25.00000000 w  
PLW12 0.46495000 w  
PLW13 0.23387000 w  
  
P2 - Processing parameters  
SI 32768  
SF 125.7577826 MHz  
WDW MM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

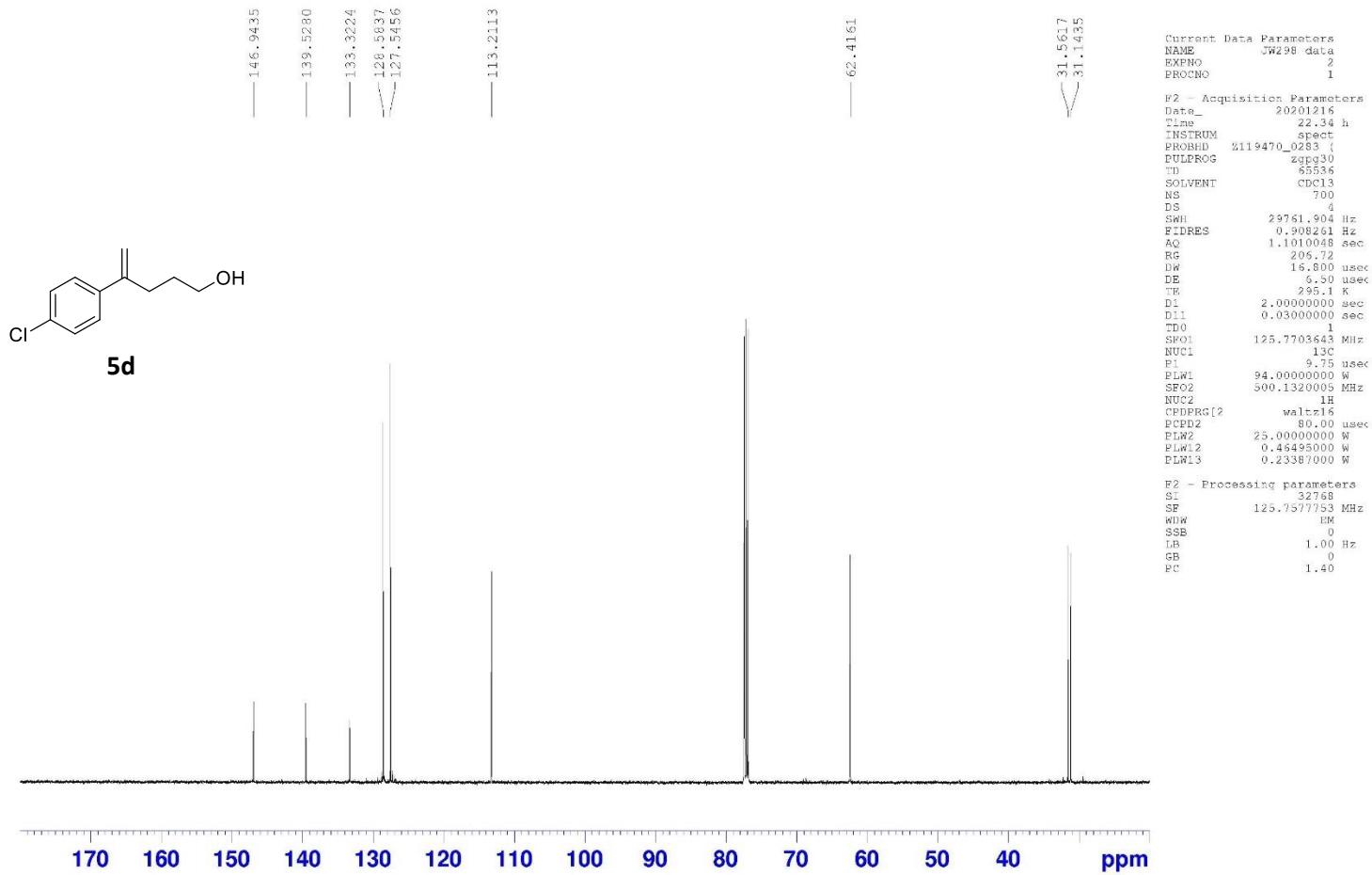












7.3805  
7.3681  
7.3553  
7.2600  
7.0199  
7.0039  
6.9877

5.2389  
5.0722

3.6555  
3.6429  
3.6302

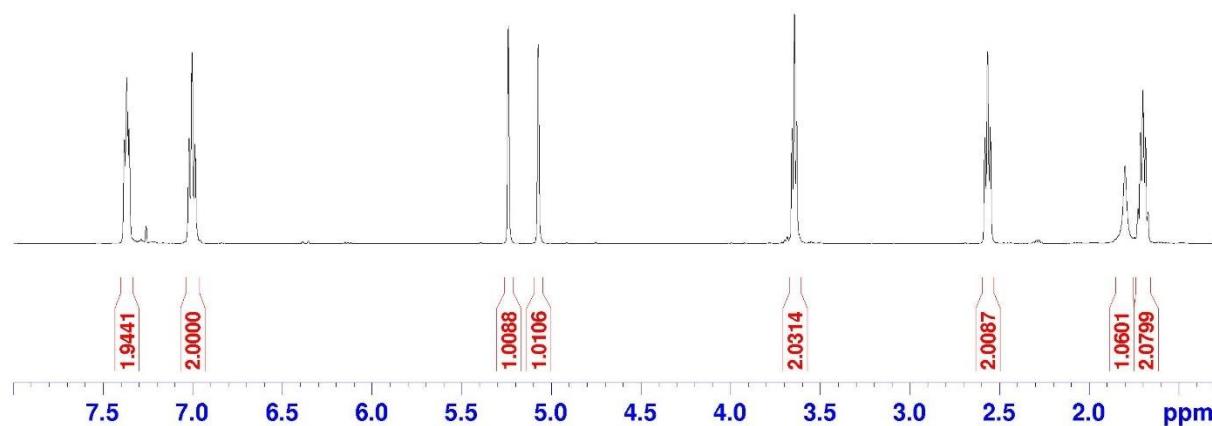
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2.5550  
2.5499

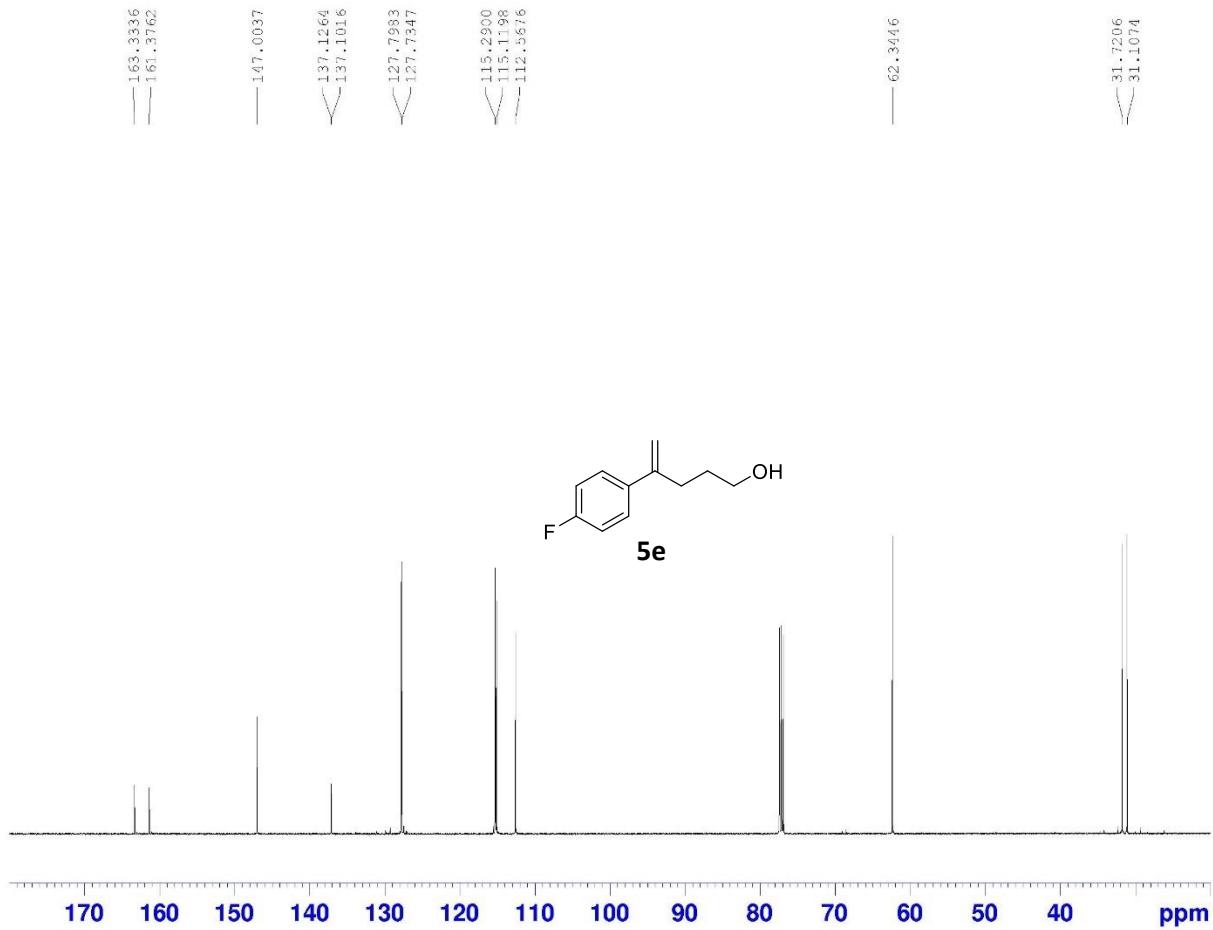
1.7988  
1.7261  
1.7122  
1.6982  
1.6844  
1.6708

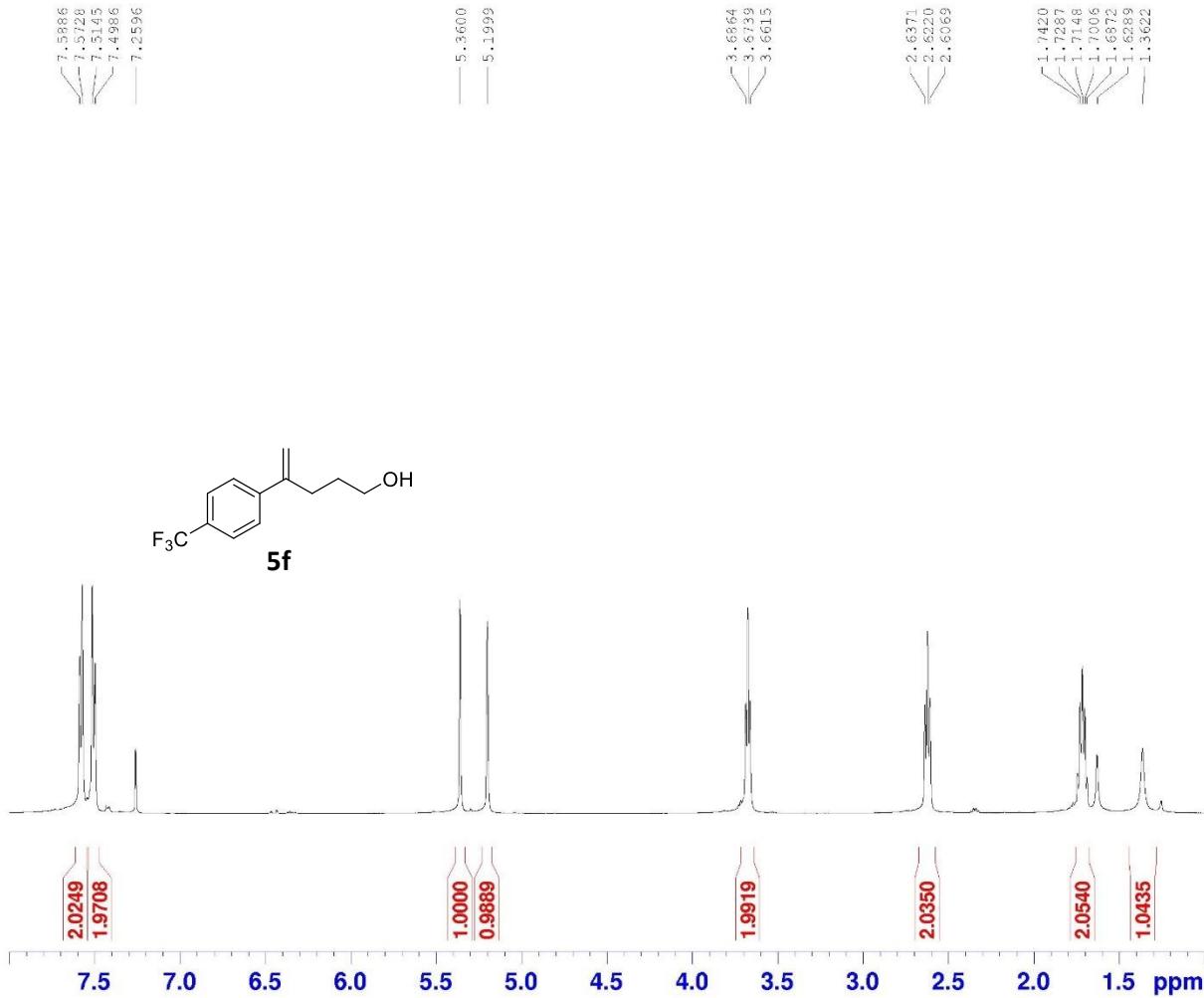
Current Data Parameters  
NAME JW295  
EXPNO 1  
PROCNO 1

P2 - Acquisition Parameters  
Date 20201216  
Time 20.33 h  
INSTRUM spect  
PROBHD Z119470\_0283  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SW0 10000.000 Hz  
FIDRES 0.305176 Hz  
AQ 3.2757999 sec  
RG 30.85  
DW 50.000 usec  
DE 6.50 usec  
TE 295.1 K  
D1 1.0000000 sec  
TD0 500.1330683 MHz  
RNUC 1H  
P1 10.91 usec  
FW1 25.0000000 W

P2 - Processing parameters  
SI 65536  
SF 500.1300120 MHz  
RDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00







Current Data Parameters  
NAME JW301A ptlc data  
EXPNO 1  
PROCNO 1

```

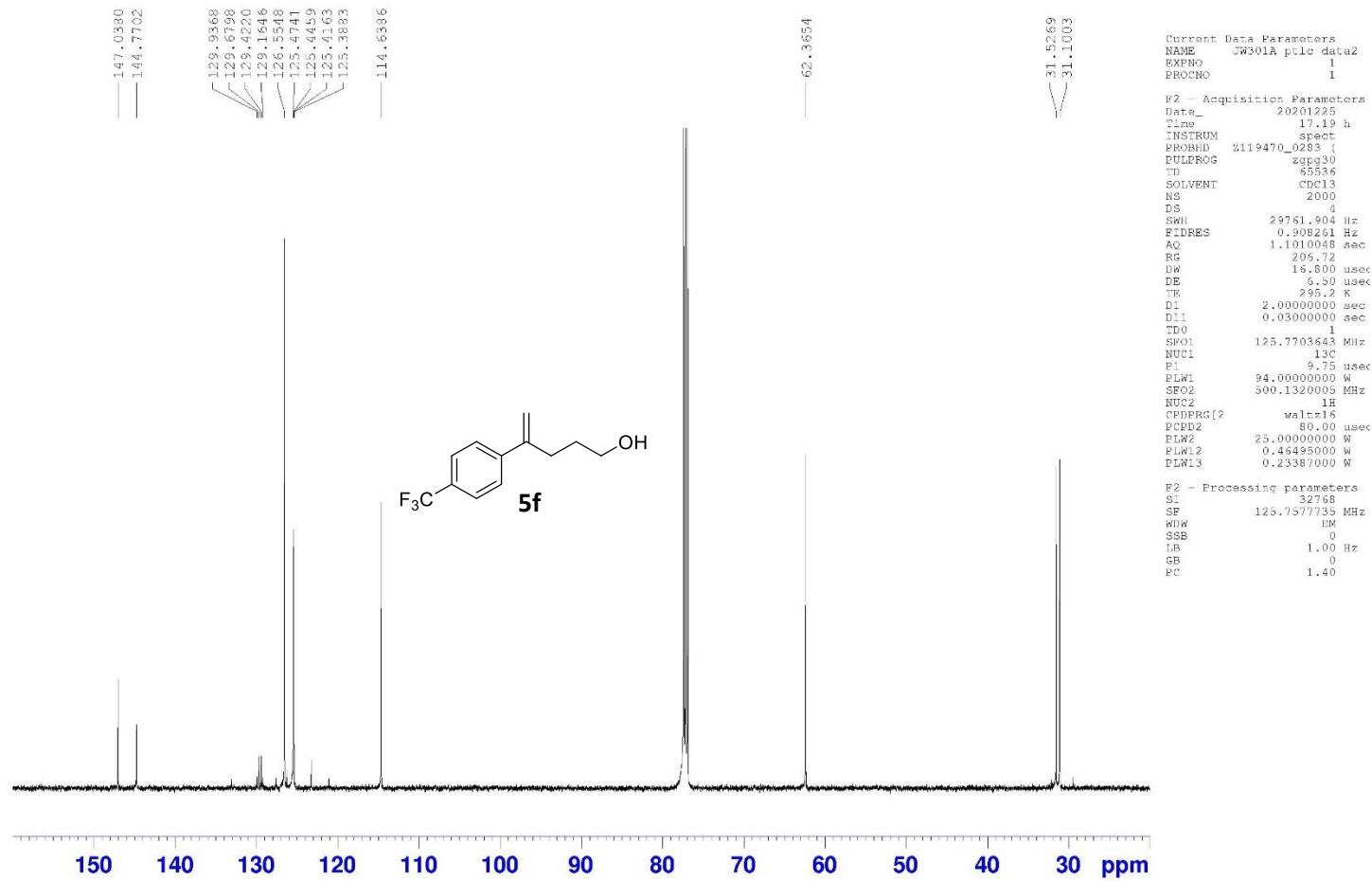
#2 Acquisition Parameters
Data_ 20201224
Time 2.39 h
INSTRUM spect
PROBHD Z119470_0283 (1
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.278799 sec
RG 93.28
DW 500.00 usec
DE 6.50 usec
TW 295.16
D1 1.0000000 sec
TDO 1
SFOL 500.1330883 MHz
NUC1 1H
PL 10.91 used
PLW1 25.00000000 W

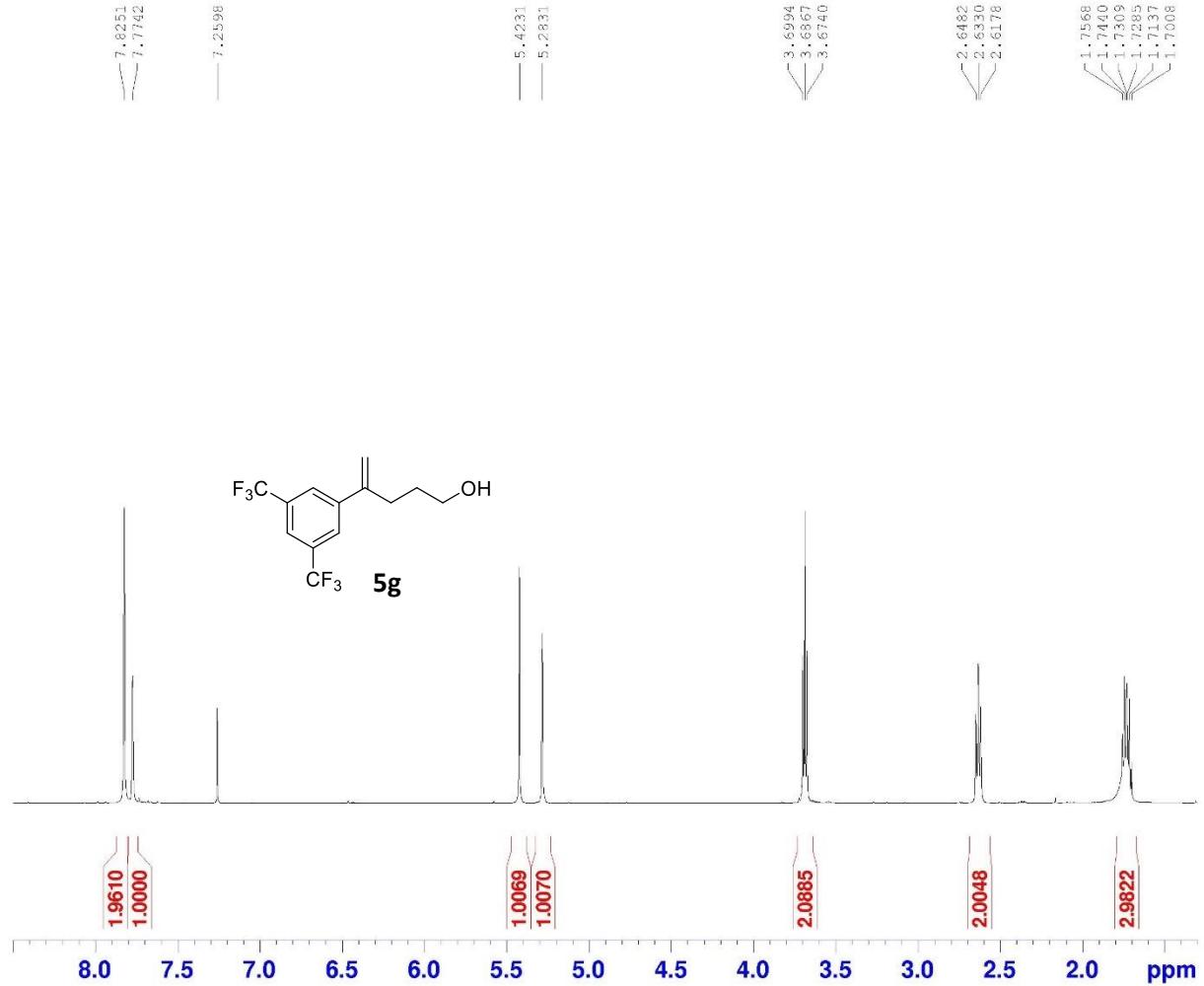
```

```

F2 - Processing parameters
SI      65536
SF      500.1300126 MHz
WDW     EM
SSB     0
LB      0.30 Hz
GB     0
PC      1.00

```





Current Data Parameters  
NAME JW301 C datax  
EXPNO 1  
PROCNO 1  
  
P2 - Acquisition Parameters  
Data\_20201207  
Time 21.14 h  
INSTRUM spect  
PROBHD Z119470\_0283\_1  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 10000.000 Hz  
FIDRES 0.305176 Hz  
AQ 3.2757999 sec  
RG 30.85  
DW 50.000 usec  
DE 6.50 usec  
TE 295.1 K  
D1 1.0000000 sec  
TD0 500.1330683 MHz  
RNUC 1H  
F1 10.91 usec  
FW1 25.0000000 W

P2 - Processing parameters  
SI 65536  
SF 500.1300123 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

