# **Supporting Information**

# Directed Ortho Borylation of Phenol Derivatives Catalyzed by a Silica-Supported Iridium Complex

Kenji Yamazaki, Soichiro Kawamorita, Hirohisa Ohmiya and Masaya Sawamura\*

Department of Chemistry, Faculty of Science, Hokkaido University, Sapporo 060-0810, Japan

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## **Instrumentation and Chemicals**

NMR spectra were recorded on a Varian Gemini 2000 spectrometer, operating at 300 MHz for <sup>1</sup>H NMR and 75.4 MHz for <sup>13</sup>C NMR. Chemical shift values for <sup>1</sup>H and <sup>13</sup>C are reference to Me<sub>4</sub>Si and the residual solvent resonances respectively. Chemical shifts are reported in  $\delta$  ppm. Elemental analysis was performed at the Center for Instrument Analysis, Hokkaido University. High-resolution mass spectra were recorded on a Thermo Scientific Exactive or JEOL JMS-T100GC mass spectrometer at the Center for Instrument Analysis, Hokkaido University. TLC analyses were performed on commercial glass plates bearing 0.25-mm layer of Merck Silica gel 60F<sub>254</sub>. Silica gel (Kanto Chemical Co., Silica gel 60 N, spherical, neutral) was used for column chromatography. Gas chromatographic (GC) analyses were conducted on a Shimadzu GC-14B equipped with a flame ionization detector. Gel permeation chromatography (GPC) was performed by LC-908 (Japan Analytical Industry Ltd., two in-line JAIGEL-2H, CHCl<sub>3</sub>, 3.5 mL/min, UV and RI detectors).

All reactions were carried out under nitrogen or argon atmosphere. Materials were obtained from commercial suppliers or prepared according to standard procedures unless otherwise noted. Silica-SMAP was prepared according to the reported procedure.<sup>1</sup> All solvents for catalytic reactions were degassed via four freeze–pump–thaw cycles before use. [Ir(OMe)(cod)]<sub>2</sub> was prepared according to the literature.<sup>2</sup> Pinacolatoborane and bis(pinacolato)diboron were purchased from

Aldrich and AllyChem Co., Ltd, respectively.

#### **Experimental Procedures**

## Typical Procedure for the Ortho-Borylation of Phenyl Diethylcarbamate (3aa) (Scheme 1).

In a glove box, Silica-SMAP (1, 0.064 mmol P  $g^{-1}$ , 40 mg, 0.0025 mmol), anhydrous, degassed hexane (1.1 mL), and [Ir(OMe)(cod)]<sub>2</sub> (0.8 mg, 0.00125 mmol) in hexane (0.4 mL) were placed in a 10 mL-glass tube containing a magnetic stirring bar, and the mixture was stirred for 1 min at 25 °C. **3aa** (196.5 mg, 1.0 mmol), and pinacolatoborane (**2**, 62.8 mg, 0.5 mmol) were added in the tube, which was then sealed with a screw cap. The tube was removed from the glove box. After the resulting mixture was stirred at 70 °C for 12 h, the mixture was filtered through a glass pipet equipped with a cotton filter. Solvent was removed under reduced pressure. An internal standard (1,1,2,2-tetrachloroethane) was added to the reaction mixture. The yield of the product was determined by <sup>1</sup>H NMR. The crude material was purified by GPC to give the borylation product **4aa** (100.5 mg, 0.32 mmol) in 64% isolated yield.

Suzuki–Miyaura Cross-Coupling/Deprotection of Carbamate (Scheme 2, upper side). 2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl *N*,*N*-diethylcarbamate (**4aa**) (96.6 mg, 0.30 mmol), 2-bromothiophene (62.9 mg, 0.36 mmol), Na<sub>2</sub>CO<sub>3</sub> (256.0 mg, 2.4 mmol) and Pd(PPh<sub>3</sub>)<sub>4</sub> (18.1 mg, 0.015 mmol) in a mixed solvent consisting of DME (1.0 mL) and H<sub>2</sub>O (0.1 mL) were placed in 10 mL-glass tube containing a magnetic stirring bar. The tube was then sealed with a screw cap in argon. After being stirred at 90 °C for 24 h, the glass tube was cooled to rt. The reaction mixture was washed with water and brine, and was dried over MgSO<sub>4</sub>. Solvent was removed under reduced pressure. The crude material was dissolved in Et<sub>2</sub>O (10.0 mL) in a two-neck round-bottom flask equipped with a reflux condenser. After being stirred at reflux for 7 h, the flask was cooled to room temperature, and water (5.0 mL) and 1M HCl aq (5.0 mL) were in turn added to the reaction mixture at 0 °C. The mixture was extracted with EtOAc (2 × 20 mL). The combined organic layers were washed with brine and dried over MgSO<sub>4</sub>. The solvent was evaporated. Flash silica gel column chromatography (hexane/EtOAc 90:10) of the crude product provided **6** (36.2 mg, 0.20 mmol) in 68% yield. Spectral data match those previously reported.<sup>3</sup>

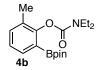
Suzuki–Miyaura Cross-Coupling/Ni-Catalyzed Cross-Coupling (Scheme 2, bottom side). 2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl *N*,*N*-diethylcarbamate (4aa) (96.1 mg, 0.30 mmol), 2-bromobenzene (58.1 mg, 0.37 mmol), Na<sub>2</sub>CO<sub>3</sub> (258.1 mg, 2.4 mmol), Pd(PPh<sub>3</sub>)<sub>4</sub> (17.7 mg, 0.015 mmol) in a mix solvent consisting of DME (1.0 ml) and H<sub>2</sub>O (0.1 ml) were placed in a 10

mL-glass tube containing a magnetic stirring bar. The tube was then sealed with a screw cap under argon. After being stirred at 90 °C for 24 h, the glass tube was cooled to rt. The reaction mixture was washed with water and brine, and was dried over MgSO<sub>4</sub>. After filtration, the solvent was evaporated. Flash silica gel column chromatography (hexane/EtOAc 95:5) of the crude product provided **7** (65.4 mg, 0.24 mmol) in 81% yield. In a glove box, **7** (65.4 mg, 0.24 mmol), Ni(acac)<sub>2</sub> (3.4 mg, 0.013 mmol), 1-[2-(diphenylphosphino)phenyl]ethanol (3.7 mg, 0.012 mmol) in Et<sub>2</sub>O (0.6 mL) and anhydrous, degassed Et<sub>2</sub>O (0.9 mL) were placed sequentially in a 10 mL-glass tube containing a magnetic stirring bar, which was then sealed with a screw cap. The tube was removed from the glove box, and 4-methoxyphenylmagnesium bromide (480  $\mu$ L, 0.48 mmol, 1.0 M in Et<sub>2</sub>O) was added to the tube. After being stirred at room temperature for 41 h, sat. NH<sub>4</sub>Cl was added to the reaction mixture. The mixture was extracted with EtOAc (2 × 20 mL). The combined organic layers were washed with brine and dried over MgSO<sub>4</sub>. The solvent was evaporated. Flash silica gel column chromatography (hexane/EtOAc 95:5) of the crude product provided **8** (50.8 mg, 0.20 mmol) in 80% yield. Spectral data match those previously reported.<sup>4</sup>

# **Compounds Characterization**

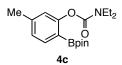
The phenol derivatives **3ab**, **3ac**, **3ad**, **3ae**, **3af** and **3ah** are known compounds. The Starting materials **3aa**, <sup>5</sup> **3ag**, <sup>6</sup> **3b**, <sup>7</sup> **3c**, <sup>8</sup> **3d**, <sup>8</sup> **3g**, <sup>9</sup> **3i**<sup>5</sup>, **3l**<sup>10</sup> and **3m**<sup>9</sup> shown in Scheme 1 and Table 1 are known compounds. Compound **4aa** is found in the literature.<sup>11</sup> The borylation products **4aa** and **4b–4s** were purified by GPC.

## 2-Methyl-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl N,N-Diethylcarbamate (4b)



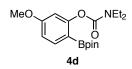
Coloress oil. <sup>1</sup>**H NMR** (CDCl<sub>3</sub>)  $\delta$  1.18–1.33 (m, 6H), 1.30 (s, 12H), 2.21 (s, 3H), 3.39 (q, *J* = 7.2 Hz, 2H), 3.52 (q, *J* = 7.2 Hz, 2H), 7.10 (t, *J* = 7.2 Hz, 1H), 7.28 (d, *J* = 7.2 Hz, 1H), 7.60 (d, *J* = 7.2 Hz, 1H). <sup>13</sup>**C NMR** (CDCl<sub>3</sub>)  $\delta$  13.32, 14.05, 16.16, 24.70, 41.53, 41.90, 83.32, 124.92, 130.56, 133.84, 133.88, 154.29, 154.69. A signal for the carbon directly attached to the boron atom was not observed. **Anal.** Calcd for C<sub>18</sub>H<sub>28</sub>BNO<sub>4</sub>: C, 64.88%; H, 8.47%; N, 4.20%. Found: C, 64.65; H, 8.55%; N, 4.22%.

5-Methyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl N,N-Diethylcarbamate (4c)



Coloress oil. <sup>1</sup>**H NMR** (CDCl<sub>3</sub>)  $\delta$  1.17–1.33 (m, 6H), 1.29 (s, 12H), 2.34 (s, 3H), 3.38 (q, *J* = 6.9 Hz, 2H), 3.50 (q, *J* = 7.2 Hz, 2H), 6.90 (s, 1H), 7.01 (d, *J* = 7.5 Hz, 1H), 7.66 (d, *J* = 7.5 Hz, 1H). <sup>13</sup>**C NMR** (CDCl<sub>3</sub>)  $\delta$  13.20, 13.79, 21.16, 24.60, 41.40, 41.72, 83.16, 122.87, 125.65, 136.11, 142.77, 154.87, 156.33. A signal for the carbon directly attached to the boron atom was not observed. **Anal.** Calcd for C<sub>18</sub>H<sub>28</sub>BNO<sub>4</sub>: C, 64.88%; H, 8.47%; N, 4.20%. Found: C, 64.60; H, 8.54%; N, 4.21%.

5-Methoxy-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl *N,N*-Diethylcarbamate (4d)

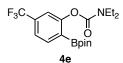


Colorress oil. <sup>1</sup>**H** NMR (CDCl<sub>3</sub>)  $\delta$  1.18–1.31 (m, 6H), 1.29 (s, 12H), 3.38 (q, *J* = 7.2, Hz, 2H), 3.51 (q, *J* = 7.2 Hz, 2H), 3.81 (s, 3H), 6.63 (d, *J* = 2.4 Hz, 1H), 6.75 (dd, *J* = 8.4, 2.4 Hz, 1H), 7.70 (d, *J* = 8.4 Hz, 1H). <sup>13</sup>**C** NMR (CDCl<sub>3</sub>)  $\delta$  13.25, 13.86, 24.68, 41.48, 41.79, 55.18, 83.13, 108.02, 111.11, 137.31, 154.70, 157.92, 163.09. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (*m*/*z*): [M+Na]<sup>+</sup> calcd for C<sub>18</sub>H<sub>28</sub>O<sub>5</sub>NBNa, 372.19527; found, 372.19570.

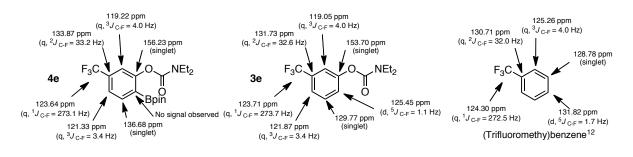
#### **3-Trifluoromethylphenyl** *N*,*N*-Diethylcarbamate (3e)

Coloress oil. <sup>1</sup>**H NMR** (CDCl<sub>3</sub>)  $\delta$  1.19–1.29 (m, 6H), 3,36–3.49 (m, 4H), 7.34 (m, 1H), 7.40 (s, 1H), 7.44–7.51 (m, 2H). <sup>13</sup>**C NMR** (CDCl<sub>3</sub>)  $\delta$  13.14, 14.06, 41.88, 42.29, 119.05 (q, *J* = 4.0 Hz), 121.87 (q, *J* = 3.4 Hz), 123.71 (q, *J* = 273.7 Hz), 125.45 (d, *J* = 1.1 Hz), 129.77, 131.73 (q, *J* = 32.6 Hz), 151.72, 153.70, 156.23. **Anal.** Calcd for C<sub>12</sub>H<sub>14</sub>F<sub>3</sub>NO<sub>2</sub>: C, 55.17%; H, 5.40%; N, 5.36%. Found: C, 55.07%; H, 5.32%; N, 5.34%.

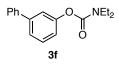
2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-5-trifluoromethylphenyl *N*,*N*-Diethylcarbamate (4e)



Coloress oil. <sup>1</sup>**H NMR** (CDCl<sub>3</sub>)  $\delta$  1.18–1.31 (m, 6H), 1.31 (s, 12H), 3.39 (q, *J* = 7.2 Hz, 2H), 3.49 (q, *J* = 7.2 Hz, 2H), 7.33 (s, 1H), 7.44 (d, *J* = 7.8 Hz, 1H), 7.88 (d, *J* = 7.8 Hz, 1H). <sup>13</sup>**C NMR** (CDCl<sub>3</sub>)  $\delta$  13.20, 13.84, 24.70, 41.59, 41.97, 83.92, 119.23 (q, *J* = 4.0 Hz), 121.36 (q, *J* = 3.4 Hz), 123.65 (q, *J* = 273.1 Hz), 133.89 (q, *J* = 33.2 Hz), 136.68, 154.41, 156.23. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (*m*/*z*): [M+Na]<sup>+</sup> calcd for C<sub>18</sub>H<sub>25</sub> O<sub>4</sub>BF<sub>3</sub>NNa, 410.17209; found, 410.17247. The regioselectivity was assigned on the basis of the *J*<sub>C–F</sub> values in the <sup>13</sup>C NMR spectrum.

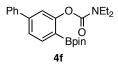


### (1,1'-Biphenyl)-3-yl N,N-Diethylcarbamate (3f)



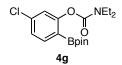
Coloress oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>)  $\delta$  1.20–1.30 (m, 6H), 3.38–3.50 (m, 4H), 7.12 (m, 1H), 7.31–7.37 (m, 2H), 7.40–7.45 (m, 4H), 7.58–7.61 (m, 2H). <sup>13</sup>C NMR (CDCl<sub>3</sub>)  $\delta$  13.16, 14.03, 41.72, 42.07, 120.51, 120.55, 123.79, 127.16, 127.46, 128.69, 129.44, 140.43, 142.57, 151.94, 154.20. Anal. Calcd for C<sub>17</sub>H<sub>19</sub>NO<sub>2</sub>: C, 75.81%; H, 7.11%; N, 5.20%. Found: C, 75.85%; H, 7.17%; N, 5.21%.

#### 5-Phenyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl N,N-Diethylcarbamate (4f)

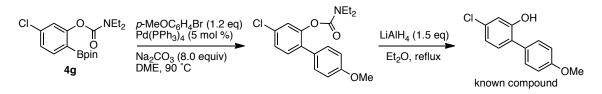


Colorress oil. <sup>1</sup>**H** NMR (CDCl<sub>3</sub>)  $\delta$  1.19–1.32 (m, 6H), 1.32 (s, 12H), 3.40 (q, *J* = 6.9, Hz, 2H), 3.53 (q, *J* = 7.2 Hz, 2H), 7.31 (d, *J* = 1.8 Hz, 1H), 7.36 (dm, *J* = 7.2 Hz, 1H), 7.40 (m, 1H), 7.61 (dm, *J* = 7.2 Hz, 2H), 7.84 (d, *J* = 7.8 Hz, 1H). <sup>13</sup>**C** NMR (CDCl<sub>3</sub>)  $\delta$  13.26, 13.88, 24.69, 41.51, 41.82, 83.41, 120.90, 123.44, 127.19, 127.73, 128.70, 136.64, 140.22, 145.28, 154.82, 156.72. A signal for the carbon directly attached to the boron atom was not observed. HRMS–ESI (*m*/*z*): [M+Na]<sup>+</sup> calcd for C<sub>23</sub>H<sub>30</sub>O<sub>4</sub>NBNa, 418.21601; found, 418.21634.

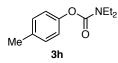
5-Chloro-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl N,N-Diethylcarbamate (4g)



Colorress oil. <sup>1</sup>**H** NMR (CDCl<sub>3</sub>)  $\delta$  1.17–1.33 (m, 6H), 1.30 (s, 12H), 3.38 (q, *J* = 7.2, Hz, 2H), 3.49 (q, *J* = 7.2 Hz, 2H), 7.11 (d, *J* = 1.8 Hz, 1H), 7.18 (dd, *J* = 8.1, 1.8 Hz, 1H), 7.70 (d, *J* = 8.1 Hz, 1H). <sup>13</sup>**C** NMR (CDCl<sub>3</sub>)  $\delta$  13.18, 13.81, 24.65, 41.52, 41.89, 83.59, 122.85, 125.11, 137.00, 137.49, 154.33, 156.79. A signal for the carbon directly attached to the boron atom was not observed. Anal. Calcd for C<sub>17</sub>H<sub>25</sub>BClNO<sub>4</sub>: C, 57.74%; H, 7.13%; N, 3.96%. Found: C, 57.49; H, 7.10%; N, 3.88%. The synthesis of known compound 4-chloro-4'-methoxy-[1,1'-biphenyl]-2-ol<sup>13</sup> from 4g by Suzuki-Miyaura coupling followed by deprotection of the carbamate moiety confirmed the assignment for 4g.

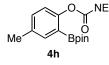


## 4-Methylphenyl Diethylcarbamate (3h)



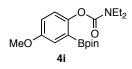
Coloress oil. <sup>1</sup>**H NMR** (CDCl<sub>3</sub>)  $\delta$  1.20–1.24 (m, 6H), 2.33 (s, 3H), 3.37–3.44 (m, 4H), 6.99 (dm, J = 8.1 Hz, 2H), 7.15 (d, J = 8.1 Hz, 2H). <sup>13</sup>**C NMR** (CDCl<sub>3</sub>)  $\delta$  13.13, 13.93, 20.52, 41.58, 41.93, 121.37, 129.60, 134.45, 149.27, 154.42. **Anal.** Calcd for C<sub>12</sub>H<sub>17</sub>NO<sub>2</sub>: C, 69.54%; H, 8.27%; N, 6.76%. Found: C, 69.52%; H, 8.40%; N, 6.75%.

#### 4-Methyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl N,N-Diethylcarbamate (4h)



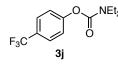
Colorress oil. <sup>1</sup>**H NMR** (CDCl<sub>3</sub>)  $\delta$  1.17–1.30 (m, 6H), 1.30 (s, 12H), 2.32 (s, 3H), 3.37 (q, *J* = 7.2, Hz, 2H), 3.50 (q, *J* = 7.2 Hz, 2H), 6.95 (d, *J* = 8.1 Hz, 1H), 7.23 (dd, *J* = 8.1, 2.4 Hz, 1H), 7.57 (d, *J* = 2.4 Hz, 1H). <sup>13</sup>**C NMR** (CDCl<sub>3</sub>)  $\delta$  13.18, 13.79, 20.37, 24.59, 41.34, 41.66, 83.25, 121.90, 132.71, 134.01, 136.48, 154.07, 154.95. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (*m*/*z*): [M+H]<sup>+</sup> calcd for C<sub>18</sub>H<sub>29</sub>BNO<sub>4</sub>, 334.21896; found, 334.21924.

4-Methoxy-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl *N*,*N*-Diethylcarbamate (4i)



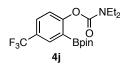
Coloress oil. <sup>1</sup>**H NMR** (CDCl<sub>3</sub>)  $\delta$  1.17–1.30 (m, 6H), 1.30 (s, 12H), 3.37 (q, *J* = 6.9 Hz, 2H), 3.49 (q, *J* = 6.9 Hz, 2H), 3.81 (s, 3H), 6.96-6.97 (m, 2H), 7.27 (m, 1H). <sup>13</sup>**C NMR** (CDCl<sub>3</sub>)  $\delta$  13.27, 13.87, 24.69, 41.42, 41.78, 55.55, 83.47, 118.23, 119.75, 123.19, 149.90, 155.24, 156.44. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (*m/z*): [M+Na]<sup>+</sup> calcd for C<sub>18</sub>H<sub>28</sub>BNO<sub>5</sub>Na, 372.19527; found, 372.19552.

### 4-Trifluoromethylphenyl N,N-Diethylcarbamate (3j)

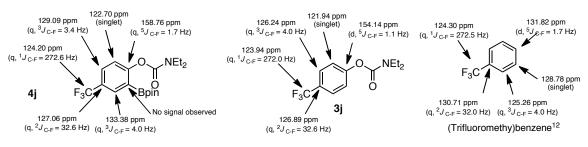


Coloress oil. <sup>1</sup>**H NMR** (CDCl<sub>3</sub>)  $\delta$  1.19–1.29 (m, 6H), 3.36–3.49 (m, 4H), 7.25 (d, J = 8.4 Hz, 2H), 7.63 (d, J = 8.4 Hz, 2H). <sup>13</sup>**C NMR** (CDCl<sub>3</sub>)  $\delta$  12.72, 13.63, 41.62, 42.01, 121.94, 123.94 (q, J = 272.0 Hz), 126.24 (q, J = 4.0 Hz), 126.89 (q, J = 32.6 Hz), 153.25, 154.14 (d, J = 1.1 Hz). **Anal.** Calcd for C<sub>12</sub>H<sub>14</sub>F<sub>3</sub>NO<sub>2</sub>: C, 55.17%; H, 5.40%; N, 5.36%. Found: C, 55.13%; H, 5.36%; N, 5.36%.

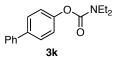
# 2-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-4-trifluoromethylphenyl *N*,*N*-Diethylcarbamate (4j)



White solid. <sup>1</sup>**H** NMR (CDCl<sub>3</sub>)  $\delta$  1.18–1.32 (m, 6H), 1.30 (s, 12H), 3.39 (q, J = 7.2, Hz, 2H), 3.51 (q, J = 7.2 Hz, 2H), 7.19 (d, J = 8.4 Hz, 1H), 7.68 (dd, J = 8.4, 2.1 Hz, 1H), 8.04 (d, J = 2.1 Hz, 1H). <sup>13</sup>C NMR (CDCl<sub>3</sub>)  $\delta$  13.18, 13.84, 24.70, 41.59, 41.95, 83.93, 122.70, 124.20 (q, J = 272.6 Hz), 127.06 (q, J = 32.6 Hz), 129.09 (q, J = 3.4 Hz), 133.38 (q, J = 4.0 Hz), 154.24, 158.76 (q, J = 1.7 Hz). A signal for the carbon directly attached to the boron atom was not observed. Anal. Calcd for C<sub>18</sub>H<sub>25</sub>F<sub>3</sub>NO<sub>4</sub>: C, 55.83%; H, 6.51%; N, 3.62%. Found: C, 55.47%; H, 6.43%; N, 3.57%. m.p. 63.5–64.5 °C. The regioselectivity was assigned on the basis of the  $J_{C-F}$  values in the <sup>13</sup>C NMR spectrum.

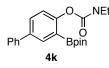


(1,1'-Biphenyl)-4-yl N,N-Diethylcarbamate (3k)

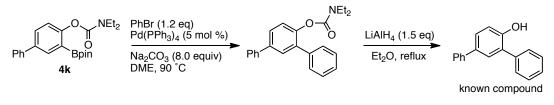


Coloress oil. <sup>1</sup>**H NMR** (CDCl<sub>3</sub>)  $\delta$  1.20–1.30 (m, 6H), 3.40–3.48 (m, 4H), 7.26 (dm, J = 8.4 Hz, 2H), 7.34 (tt, J = 7.2, 1.5 Hz, 1H), 7.44 (tm, J = 7.2 Hz, 2H), 7.55–7.59 (m, 4H). <sup>13</sup>**C NMR** (CDCl<sub>3</sub>)  $\delta$  13.16, 14.01, 41.72, 42.08, 121.99, 127.05, 127.12, 127.91, 128.71, 138.15, 140.60, 151.02, 154.22. **Anal.** Calcd for C<sub>17</sub>H<sub>19</sub>NO<sub>2</sub>: C, 75.81%; H, 7.11%; N, 5.20%. Found: C, 75.63; H, 7.15%; N, 5.22%.

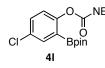
4-Phenyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl N,N-Diethylcarbamate (4k)



White solid. <sup>1</sup>**H** NMR (CDCl<sub>3</sub>)  $\delta$  1.19–1.32 (m, 6H), 1.32 (s, 12H), 3.40 (q, J = 7.2, Hz, 2H), 3.53 (q, J = 7.2 Hz, 2H), 7.15 (d, J = 8.4 Hz, 1H) , 7.32 (tt, J = 7.5, 1.8 Hz, 1H), 7.42 (t, J = 7.5 Hz, 2H), 7.60 (dd, J = 7.5, 1.8 Hz, 2H), 7.64 (dd, J = 8.4, 2.4 Hz, 1H), 7.99 (d, J = 2.4 Hz, 1H). <sup>13</sup>C NMR (CDCl<sub>3</sub>)  $\delta$  13.20, 13.82, 24.64, 41.44, 41.77, 83.41, 122.51, 126.99, 127.14, 128.58, 130.78, 134.94, 137.78, 140.63, 154.80, 155.73. A signal for the carbon directly attached to the boron atom was not observed. Anal. Calcd for C<sub>23</sub>H<sub>30</sub>BNO<sub>4</sub>: C, 69.88%; H, 7.65%; N, 3.54%. Found: C, 69.76; H, 7.75%; N, 3.55%. m.p. 154.7–156.0 °C. The synthesis of known compound [1,1':3',1"-terphenyl]-4'-ol<sup>14</sup> from **4k** by Suzuki–Miyaura coupling followed by deprotection of the carbomate moiety confirmed the assignment for **4k**.



#### 4-Chloro-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl N,N-Diethylcarbamate (41)

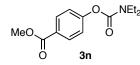


Coloress oil. <sup>1</sup>**H** NMR (CDCl<sub>3</sub>)  $\delta$  1.17–1.36 (m, 6H), 1.30 (s, 12H), 3.37 (q, *J* = 7.5 Hz, 2H), 3.49 (q, *J* = 7.2 Hz, 2H), 7.01 (d, *J* = 8.4 Hz, 1H), 7.38 (dd, *J* = 8.4, 2.7 Hz, 1H), 7.73 (d, *J* = 2.7 Hz, 1H). <sup>13</sup>**C** NMR (CDCl<sub>3</sub>)  $\delta$  13.26, 13.89, 24.74, 41.54, 41.92, 83.82, 123.77, 130.47, 131.98, 135.76, 154.62, 154.69. A signal for the carbon directly attached to the boron atom was not observed. HRMS–ESI (*m*/*z*): [M+H]<sup>+</sup> calcd for C<sub>17</sub>H<sub>26</sub>BCINO<sub>4</sub>, 354.16434; found, 354.16429.

5-Bromo-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl *N*,*N*-Diethylcarbamate (4m)

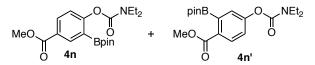
Wthie solid. <sup>1</sup>**H NMR** (CDCl<sub>3</sub>)  $\delta$  1.17–1.33 (m, 6H), 1.29 (s, 12H), 3.37 (q, J = 7.2, Hz, 2H), 3.48 (q, J = 7.2 Hz, 2H), 7.27 (d, J = 1.8 Hz, 1H) , 7.34 (dd, J = 8.1, 1.8 Hz, 1H), 7.63 (d, J = 8.1 Hz, 1H). <sup>13</sup>**C NMR** (CDCl<sub>3</sub>)  $\delta$  13.22, 13.84, 24.69, 41.55, 41.93, 83.64, 125.65, 125.74, 128.07, 137.19, 154.36, 156.72. A signal for the carbon directly attached to the boron atom was not observed. **Anal.** Calcd for C<sub>17</sub>H<sub>25</sub>BBrNO<sub>4</sub>: C, 51.29%; H, 6.33%; N, 3.52%. Found: C, 50.97%; H, 6.28%; N, 3.43%. m.p. 84.5–85.4 °C.

# 4-(Methoxycarbonyl)phenyl N,N-Diethylcarbamate (3n)



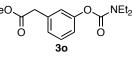
Colorress oil. <sup>1</sup>**H NMR** (CDCl<sub>3</sub>)  $\delta$  1.19–1.28 (m, 6H), 3.36–3.48 (m, 4H), 3.91 (s, 3H), 7.21 (dm, J = 8.7 Hz, 2H), 8.23 (dm, J = 8.7 Hz, 2H). <sup>13</sup>**C NMR** (CDCl<sub>3</sub>)  $\delta$  12.79. 13.71, 41.54, 41.88, 51.55, 121.26, 126.46, 130.62, 153.08, 155.10, 166.11. **HRMS–ESI** (*m*/*z*): [M+Na]<sup>+</sup> calcd for C<sub>13</sub>H<sub>17</sub>NO<sub>4</sub>Na, 274.10498; found, 274.10497.

4-(Methoxycarbonyl)-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl N,N-Diethylcarbamate (4n) and 4-(Methoxycarbonyl)-3-(4,4,5,5-tetramethyl-1,3,2-dioxa borolan-2-yl)phenyl N,N-Diethylcarbamate (4n')



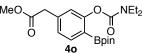
Colorress oil. <sup>1</sup>**H NMR** (CDCl<sub>3</sub>) **4n**;  $\delta$  1.18–1.32 (m, 6H), 1.32 (s, 12H), 3.39 (q, *J* = 7.2 Hz, 2H), 3.51 (q, *J* = 7.2 Hz, 2H), 3.91 (s, 3H), 7.15 (d, *J* = 8.4 Hz, 1H), 8.12 (dd, *J* = 8.4, 2.4 Hz, 1H), 8.45 (d, *J* = 2.4 Hz, 1H). **4n**';  $\delta$  3.90 (s, 3H), 7.21–7.23 (m, 2H), 7.95 (d, *J* = 9.3 Hz, 1H) (only observed peaks). <sup>13</sup>**C NMR** (CDCl<sub>3</sub>) **4n**+**4n**';  $\delta$  13.15, 13.20, 13.86, 14.07, 41.56, 41.84, 41.90, 42.15, 83.76, 84.08, 122.15, 122.34, 125.02, 126.67, 129.96, 130.36, 133.65, 137.94, 154.23, 154.57, 159.87, 166.73 (only observed peaks). **HRMS–ESI** (*m*/*z*): [M+Na]<sup>+</sup> calcd for C<sub>19</sub>H<sub>28</sub>NO<sub>6</sub>BNa, 400.19014; found, 400.19046. The position of the boron atom was determined by comparison of the <sup>1</sup>H NMR chemical shifts with those of the isomer **4n**': the aromatic proton of **4n** at C3 position was observed at a significantly lower magnetic field than the aromatic proton of **4n**' at C2 position, which indicates that the **4n** is a borylated at the ortho position of the carbamate moiety.

### 3-(2-Methoxy-2-oxoethyl)phenyl N,N-Diethylcarbamate (30)



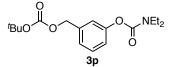
Colorress oil. <sup>1</sup>**H** NMR (CDCl<sub>3</sub>)  $\delta$  1.18–1.27 (m, 6H), 3.37–3.45 (m, 4H), 3.63 (s, 2H), 3.70 (s, 3H), 7.04-7.13 (m, 3H), 7.31 (t, J = 7.5 Hz, 1H). <sup>13</sup>**C** NMR (CDCl<sub>3</sub>)  $\delta$  13.13, 13.98, 40.68, 41.69, 42.03, 51.87, 120.54, 122.63, 125.96, 129.22, 135.15, 151.62, 154.10, 171.71. Anal. Calcd for C<sub>14</sub>H<sub>19</sub>NO<sub>4</sub>: C, 63.38%; H, 7.22%; N, 5.28%. Found: C, 63.02%; H, 7.21%; N, 5.24%.

# 5-(2-Methoxy-2-oxoethyl)-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl *N*,*N*-Diethylcarbamate (40)



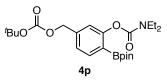
Colorress oil. <sup>1</sup>**H NMR** (CDCl<sub>3</sub>)  $\delta$  1.17–1.35 (m, 6H), 1.29 (s, 12H), 3.38 (q, *J* = 7.2 Hz, 2H), 3.50 (q, *J* = 7.2 Hz, 2H), 3.62 (s, 2H), 3.67 (s, 3H), 7.02 (s, 1H), 7.12 (d, *J* = 8.1 Hz, 1H), 7.73 (d, *J* = 8.1 Hz, 1H). <sup>13</sup>**C NMR** (CDCl<sub>3</sub>)  $\delta$  13.28, 13.87, 24.70, 40.93, 41.51, 41.83, 51.99, 83.42, 123.16, 125.80, 136.44, 138.31, 154.78, 156.41, 171.56. **Anal.** Calcd for C<sub>20</sub>H<sub>30</sub>BNO<sub>6</sub>: C, 61.39%; H, 7.73%; N, 3.58%. Found: C, 61.06%; H, 7.65%; N, 3.59%.

**3-**[{(*tert*-Butoxycarbonyl)oxy}methyl]phenyl *N*,*N*-Diethylcarbamate (3p)

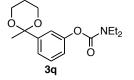


Colorress oil. <sup>1</sup>**H** NMR (CDCl<sub>3</sub>)  $\delta$  1.18–1.29 (m, 6H), 1.49 (s, 9H), 3.35–3.47 (m, 4H), 5.08 (s, 2H), 7.10 (dm, J = 7.8 Hz, 1H), 7.15 (s, 1H), 7.20 (d, J = 7.8 Hz, 1H), 7.35 (t, J = 7.8 Hz, 1H). <sup>13</sup>**C** NMR (CDCl<sub>3</sub>)  $\delta$  13.08, 13.94, 27.48 41.65, 42.00, 67.91, 82.12, 121.39, 121.64, 124.63, 129.24, 136.93, 151.60, 153.31, 153.98. Anal. Calcd for C<sub>17</sub>H<sub>25</sub>NO<sub>5</sub>: C, 63.14%; H, 7.79%; N, 4.33%. Found: C, 62.84; H, 7.80%; N, 4.37%.

5-[{(*tert*-Butoxycarbonyl)oxy}methyl]-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl *N*,*N*-Diethylcarbamate (4p)

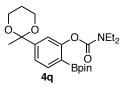


Coloress oil. <sup>1</sup>**H** NMR (CDCl<sub>3</sub>)  $\delta$  1.17–1.32 (m, 6H), 1.30 (s, 12H), 1.49 (s, 9H), 3.37 (q, *J* = 7.2 Hz, 2H), 3.50 (q, *J* = 6.9 Hz, 2H), 5.08 (s, 2H), 7.09 (d, *J* = 1.2 Hz, 1H), 7.19 (dd, *J* = 7.8, 1.2 Hz, 1H) 7.76 (d, *J* = 7.8 Hz, 1H). <sup>13</sup>**C** NMR (CDCl<sub>3</sub>)  $\delta$  13.24, 13.85, 24.69, 27.60, 41.50, 41.83, 67.81, 82.28, 83.47, 121.57, 124.08, 136.42, 140.07, 153.39, 154.73, 156.40. A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (*m*/*z*): [M+Na]<sup>+</sup> calcd for C<sub>23</sub>H<sub>36</sub>BO<sub>7</sub>NNa, 472.24770; found, 472.24743.



Colorress oil. <sup>1</sup>**H NMR** (CDCl<sub>3</sub>)  $\delta$  1.19–1.29 (m, 7H), 1.52 (s, 3H), 2.12 (m, 1H), 3.37–3.47 (m, 4H), 3.79–3.87 (m, 4H), 7.09 (ddd, J = 7.8, 2.4, 1.5 Hz, 1H), 7.18 (t, J = 1.5 Hz, 1H), 7.27 (m, 1H), 7.39 (t, J = 7.8 Hz, 1H). <sup>13</sup>**C NMR** (CDCl<sub>3</sub>)  $\delta$  13.18, 14.05, 25.14, 32.12, 41.72, 42.06, 61.18, 100.15, 120.38, 121.00, 123.44, 129.49, 142.84, 152.07, 154.18. **Anal.** Calcd for C<sub>16</sub>H<sub>23</sub>NO<sub>4</sub>: C, 65.51%; H, 7.90%; N, 4.77%. Found: C, 65.20%; H, 7.94%; N, 4.76%.

5-(2-Methyl-1,3-dioxan-2-yl)-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl *N*,*N*-Diethylcarbamate (4q)



White solid. <sup>1</sup>**H NMR** (CDCl<sub>3</sub>)  $\delta$  1.18–1.34 (m, 7H), 1.31 (s, 12H), 1.49 (s, 3H), 2.11 (m, 1H), 3.39 (q, J = 7.2 Hz, 2H), 3.52 (q, J = 7.2 Hz, 2H), 3.77–3.89 (m, 4H), 7.13 (d, J = 1.5 Hz, 1H), 7.27 (m, 1H), 7.82 (d, J = 8.1 Hz, 1H). <sup>13</sup>**C NMR** (CDCl<sub>3</sub>)  $\delta$  13.21, 13.85, 24.66, 25.12, 32.01, 41.43, 41.76, 61.27, 83.42, 100.16, 120.86, 123.19, 136.66, 145.95, 154.74, 156.82. **Anal.** Calcd for C<sub>22</sub>H<sub>34</sub>BNO<sub>6</sub>: C, 63.02%; H, 8.17%; N, 3.34%. Found: C, 62.74%; H, 8.30%; N, 3.32%. m.p. 105.5–106.5 °C.

# 5-Fluoro-2-methylphenyl Diethylcarbamate (3r)

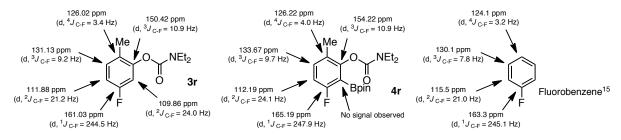


Colorress oil. <sup>1</sup>**H** NMR (CDCl<sub>3</sub>)  $\delta$  1.19–1.29 (m, 6H), 2.17 (s, 3H), 3.36–3.50 (m, 4H), 6.80–6.88 (m, 2H), 7.14 (tm, J = 7.2 Hz, 1H). <sup>13</sup>**C** NMR (CDCl<sub>3</sub>)  $\delta$  13.01, 13.89, 15.34, 41.69, 42.07, 109.86 (d, J = 24.0 Hz), 111.88 (d, J = 21.2 Hz), 126.02 (d, J = 3.4 Hz), 131.13 (d, J = 9.2 Hz), 150.42 (d, J = 10.9 Hz), 153.32, 161.03 (d, J = 244.5 Hz). Anal. Calcd for C<sub>12</sub>H<sub>16</sub>FNO<sub>2</sub>: C, 63.98%; H, 7.16%; N, 6.22%. Found: C, 63.87%; H, 7.18%; N, 6.26%.

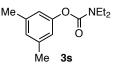
5-Fluoro-2-methyl-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl *N*,*N*-Diethylcarbamate (4r)



Coloress oil. <sup>1</sup>**H NMR** (CDCl<sub>3</sub>)  $\delta$  1.18–1.35 (m, 6H), 1.33 (s, 12H), 2.15 (s, 3H), 3.39 (q, J = 7.2 Hz, 2H), 3.49 (q, J = 7.2 Hz, 2H), 6.82 (t, J = 8.4 Hz, 1H), 7.21 (m, 1H). <sup>13</sup>**C NMR** (CDCl<sub>3</sub>)  $\delta$  13.24, 14.05, 15.72, 24.64, 41.55, 41.93, 83.48, 112.19 (d, J = 24.1 Hz), 126.22 (d, J = 4.0 Hz), 133.67 (d, J = 9.7 Hz), 153.80, 154.22 (d, J = 10.9 Hz), 165.19 (d, J = 247.9 Hz). A signal for the carbon directly attached to the boron atom was not observed. **HRMS–ESI** (m/z): [M+H]<sup>+</sup> calcd for C<sub>18</sub>H<sub>28</sub>O<sub>4</sub>NBF, 352.20954; found, 352.21046. m.p. 112.6–115.2 °C. The regioselectivity was assigned on the basis of the  $J_{C-F}$  values in the <sup>13</sup>C NMR spectrum.

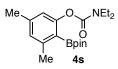


# 3,5-Dimethylphenyl Diethylcarbamate (3s)



Colorress oil. <sup>1</sup>**H NMR** (CDCl<sub>3</sub>)  $\delta$  1.17–1.26 (m, 6H), 2.30 (s, 6H), 3.35–3.45 (m, 4H), 6.74 (t, J = 0.9 Hz, 2H), 6.82 (d, J = 0.9 Hz, 1H). <sup>13</sup>**C NMR** (CDCl<sub>3</sub>)  $\delta$  13.01, 13.83, 20.81, 41.51, 41.84, 119.22, 126.62, 138.75, 151.30, 154.32. **Anal.** Calcd for C<sub>13</sub>H<sub>19</sub>NO<sub>2</sub>: C, 70.56%; H, 8.65%; N, 6.33%. Found: C, 70.47; H, 8.74%; N, 6.26%.

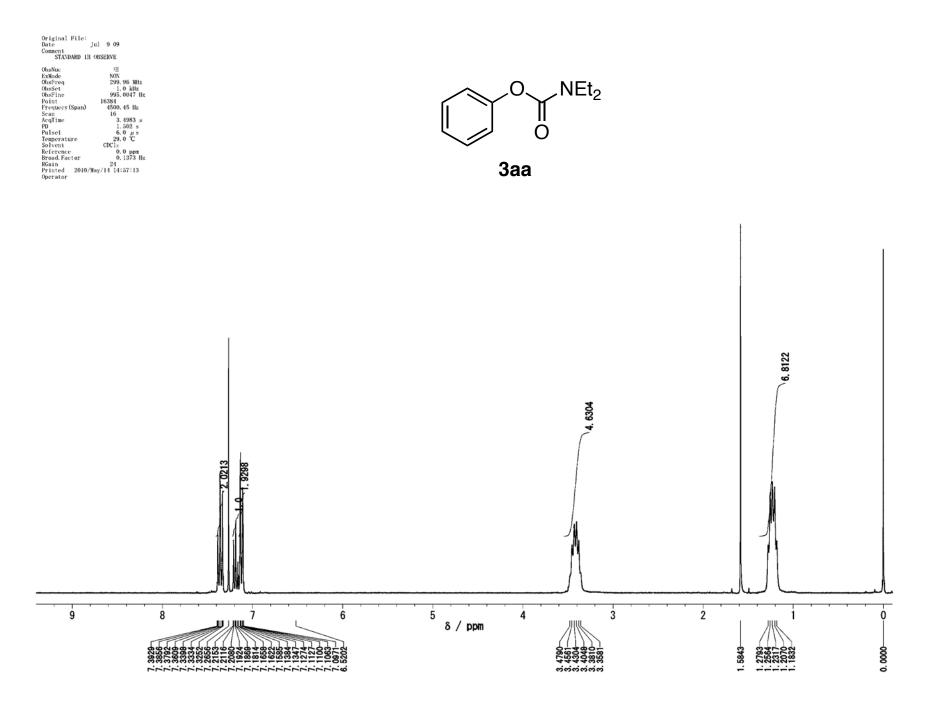
# 3,5-Dimethyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl *N,N*-Diethylcarbamate (4s)

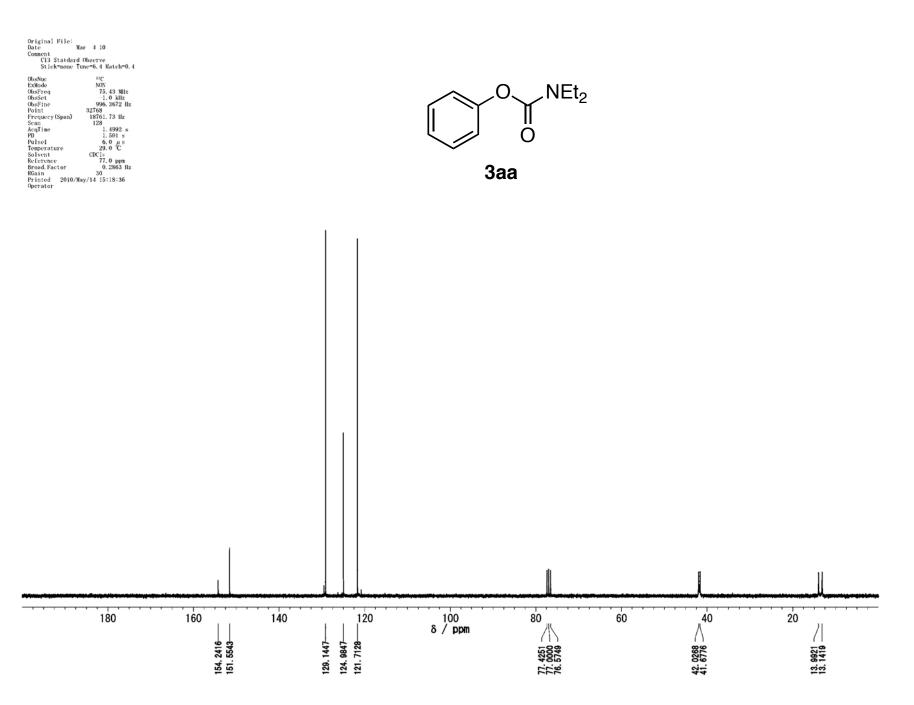


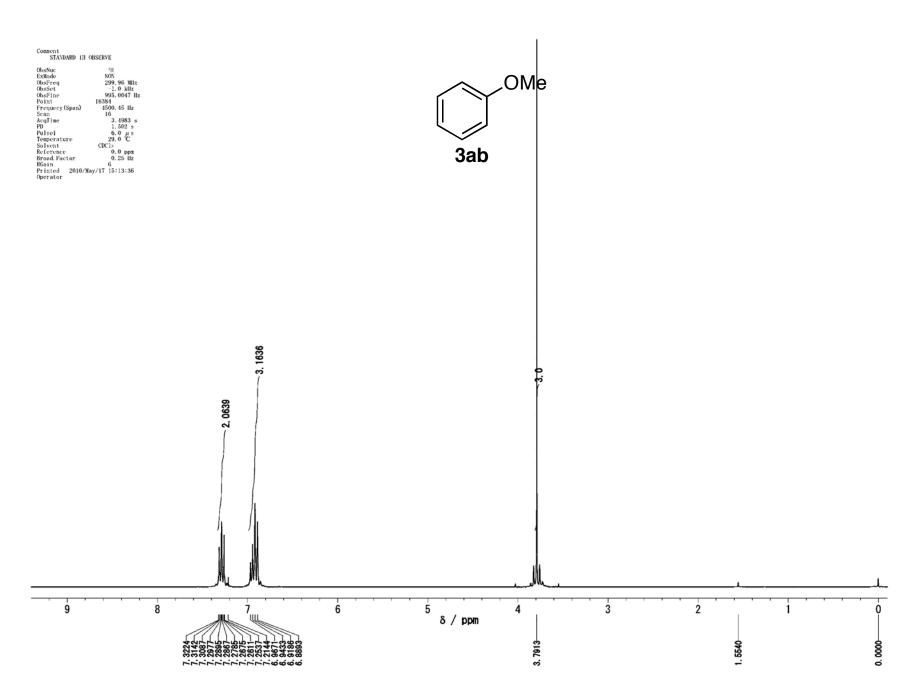
White solid. <sup>1</sup>**H** NMR (CDCl<sub>3</sub>)  $\delta$  1.16–1.35 (m, 6H), 1.31 (s, 12H), 2.27 (s, 3H), 2.44 (s, 3H), 3.37 (q, *J* = 7.2 Hz, 2H), 3.46 (q, *J* = 7.2 Hz, 2H), 6.68 (s, 1H), 6.82 (s, 1H). <sup>13</sup>**C** NMR (CDCl<sub>3</sub>)  $\delta$  13.27, 13.97, 21.01, 22.16, 24.74, 41.46, 41.73, 83.13, 119.99, 127.98, 141.09, 144.90, 155.02, 156.11. Anal. Calcd for C<sub>19</sub>H<sub>30</sub>BNO<sub>4</sub>: C, 65.72; H, 8.71%; N, 4.03%. Found: C, 65.49; H, 8.79%; 3.92%. m.p. 71.5–72.4 °C.

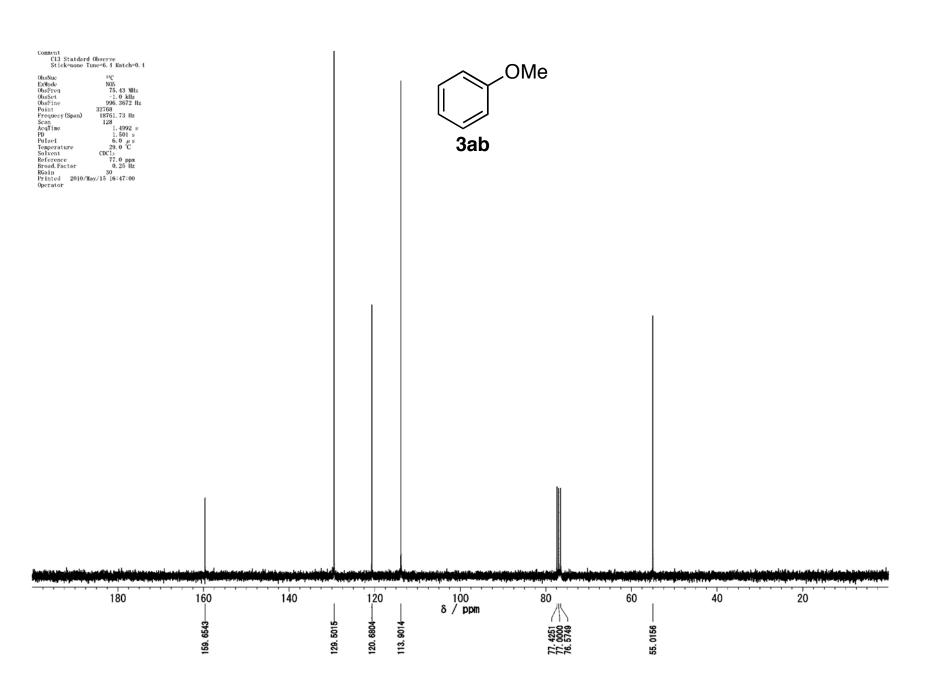
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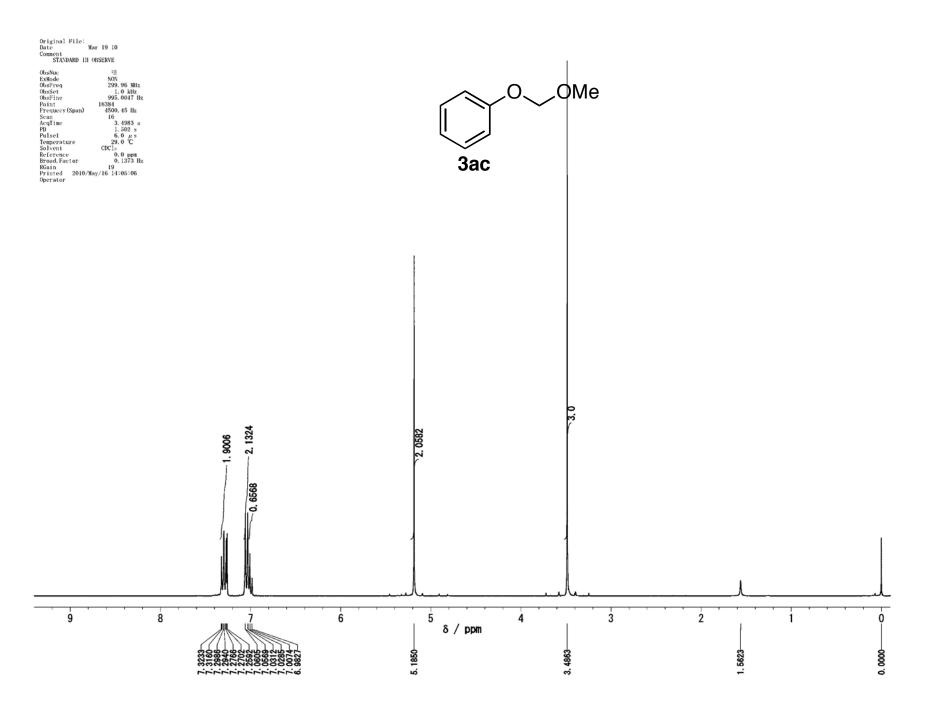
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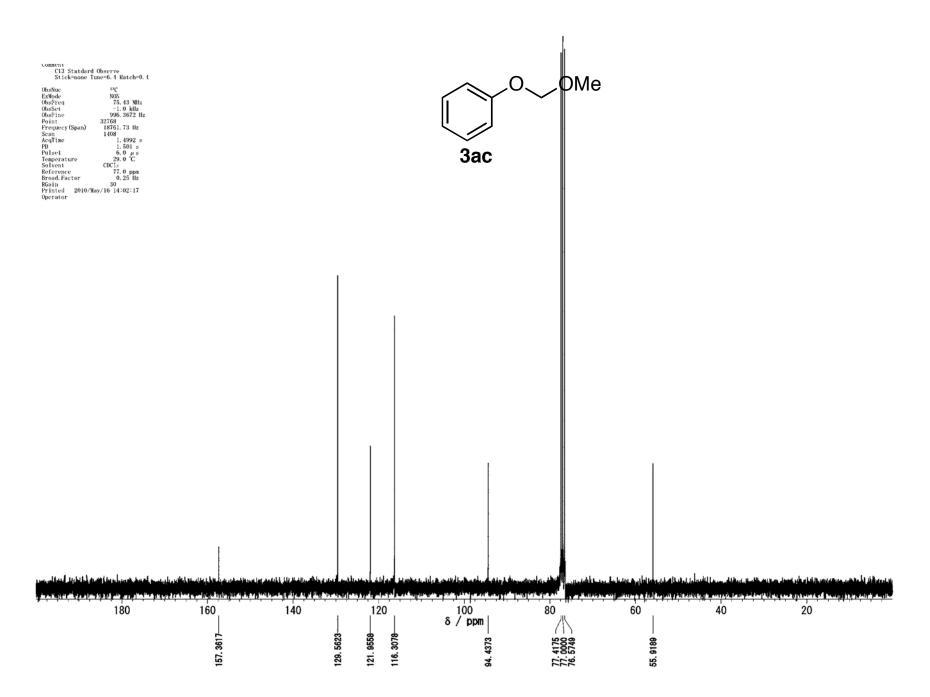


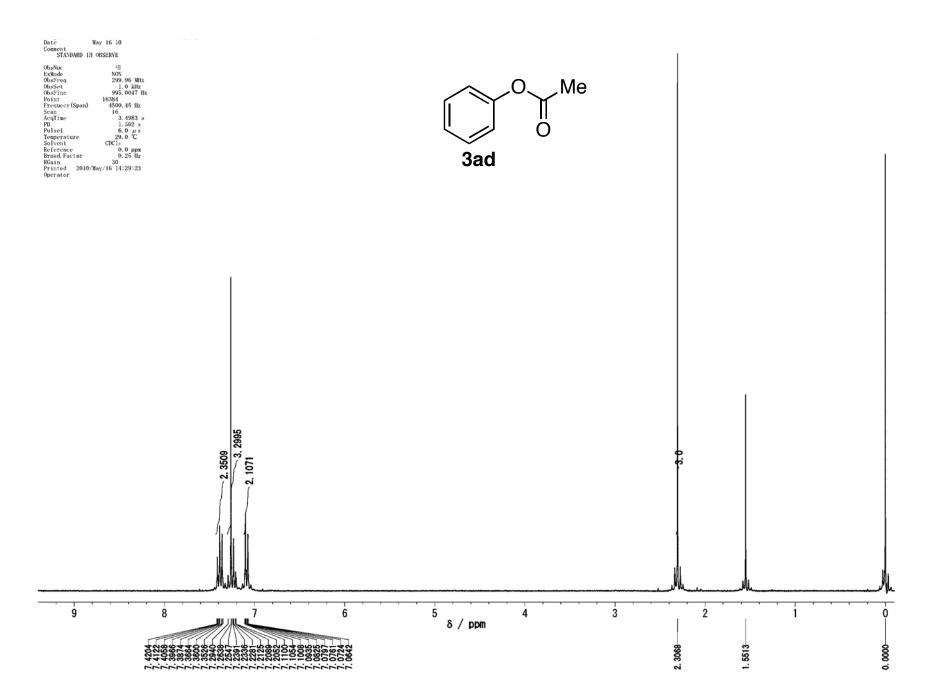


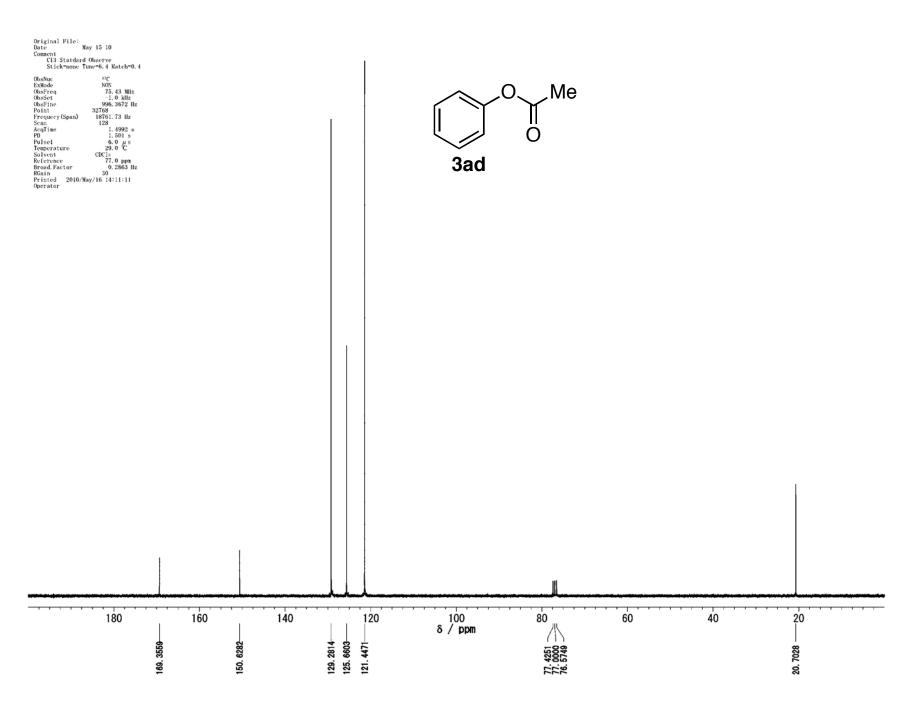


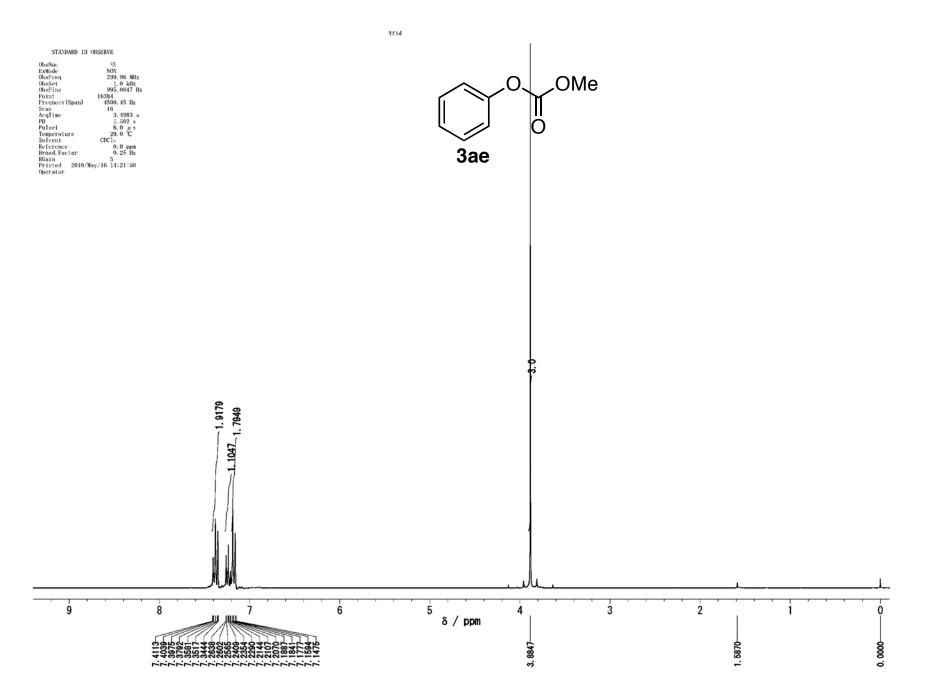


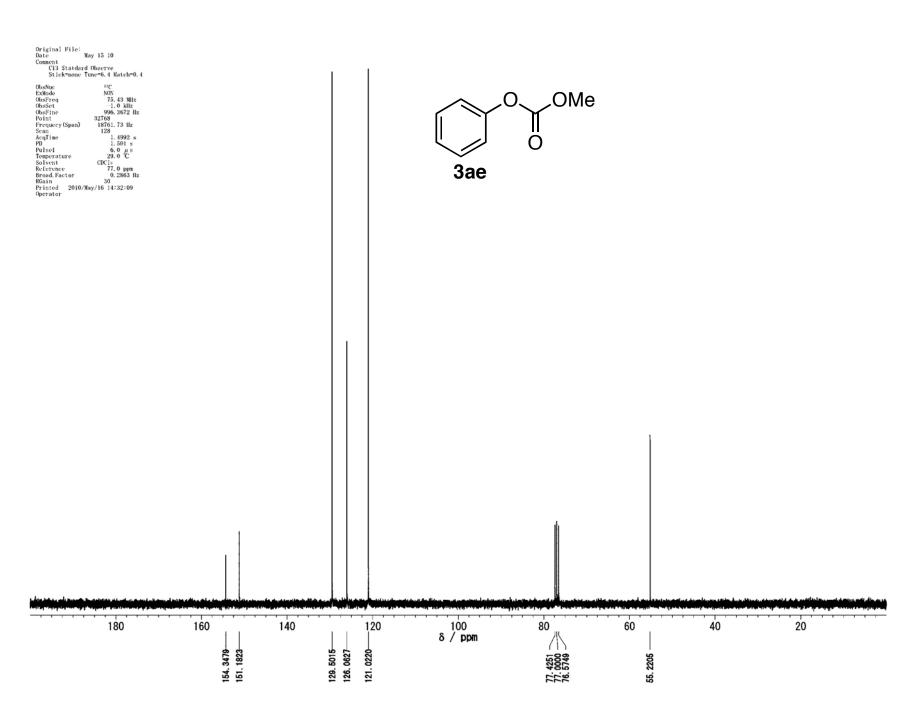


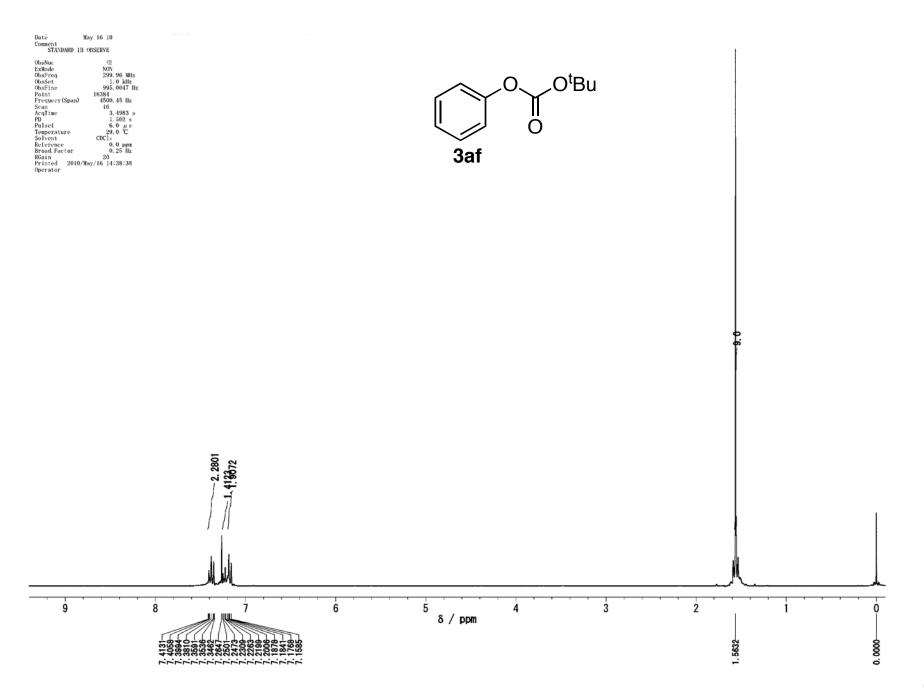


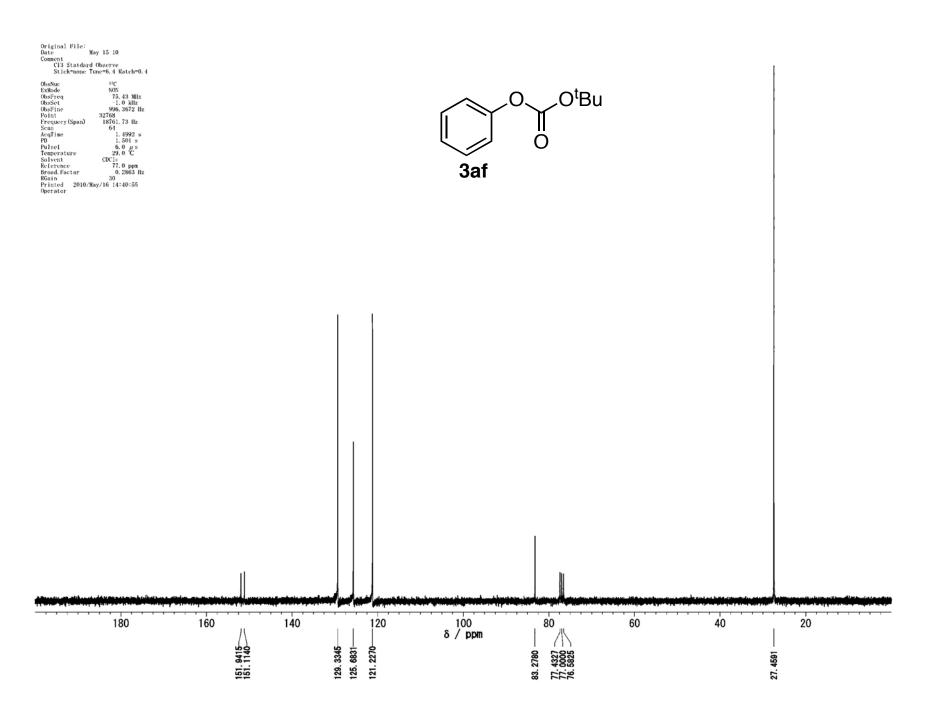


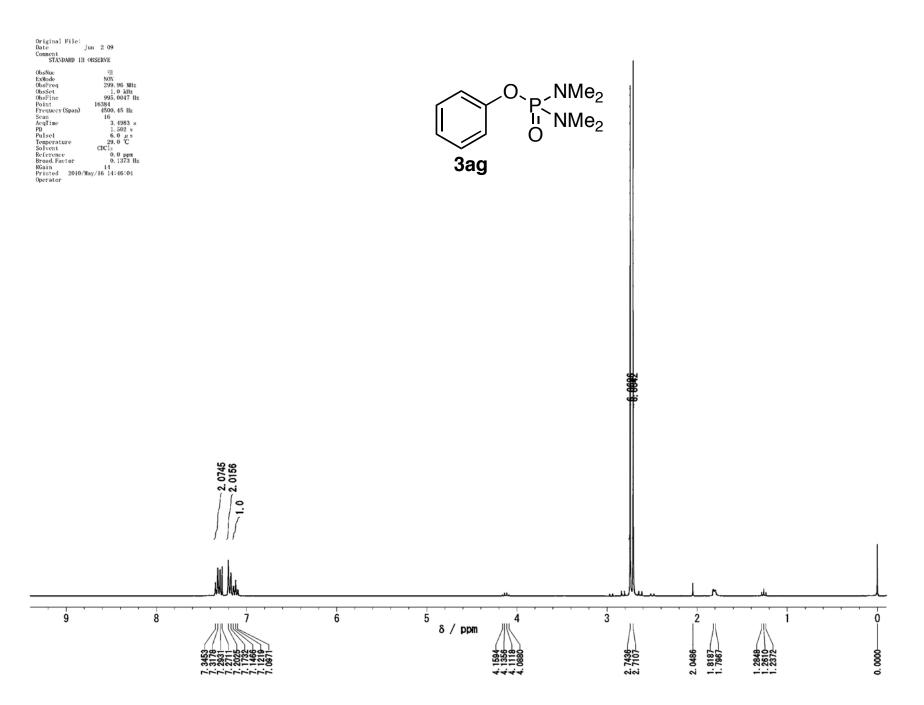


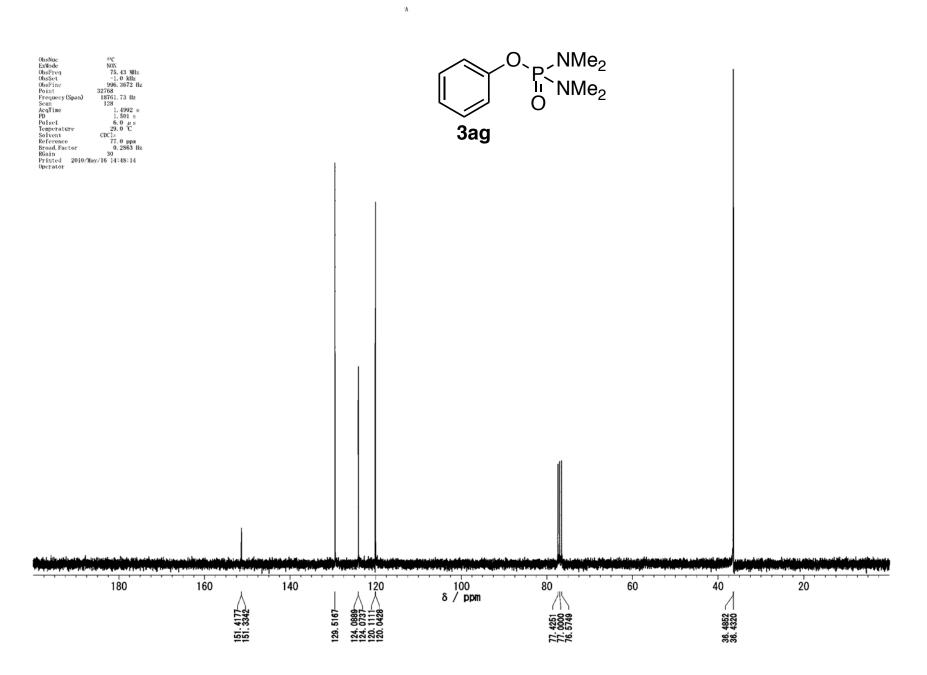


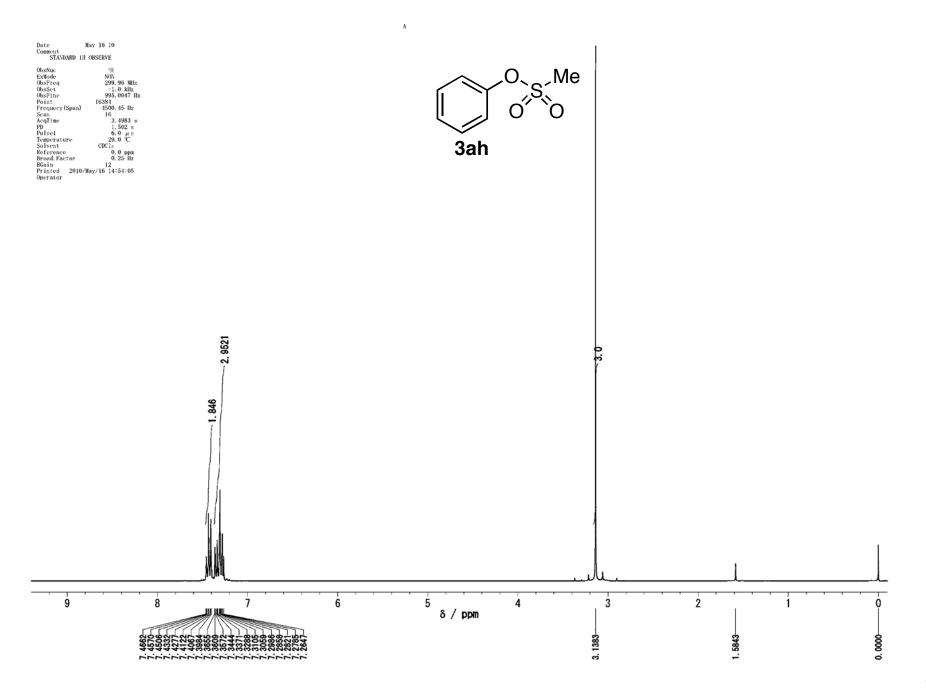


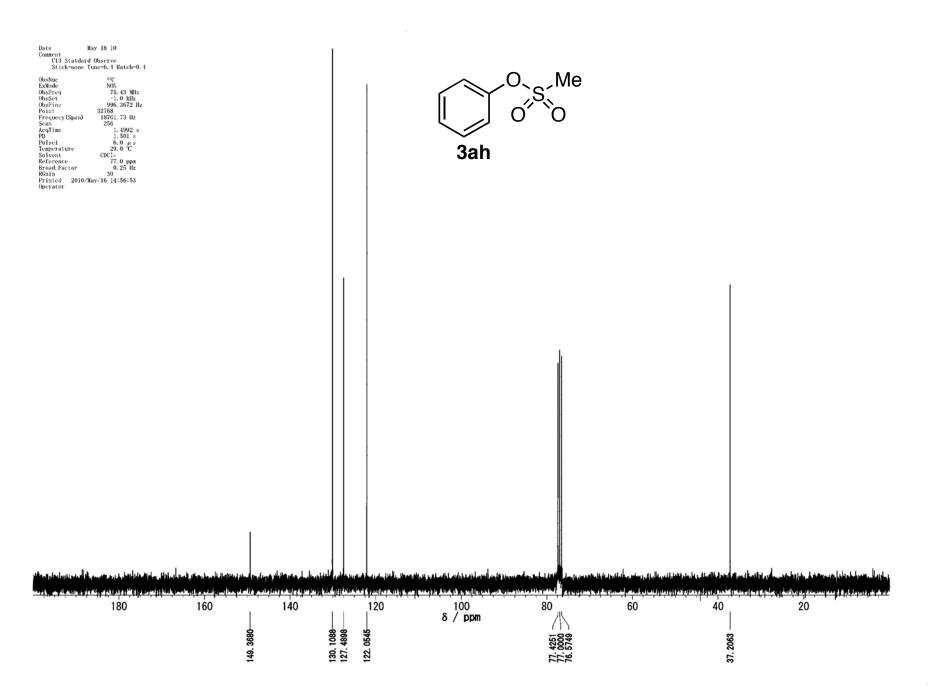


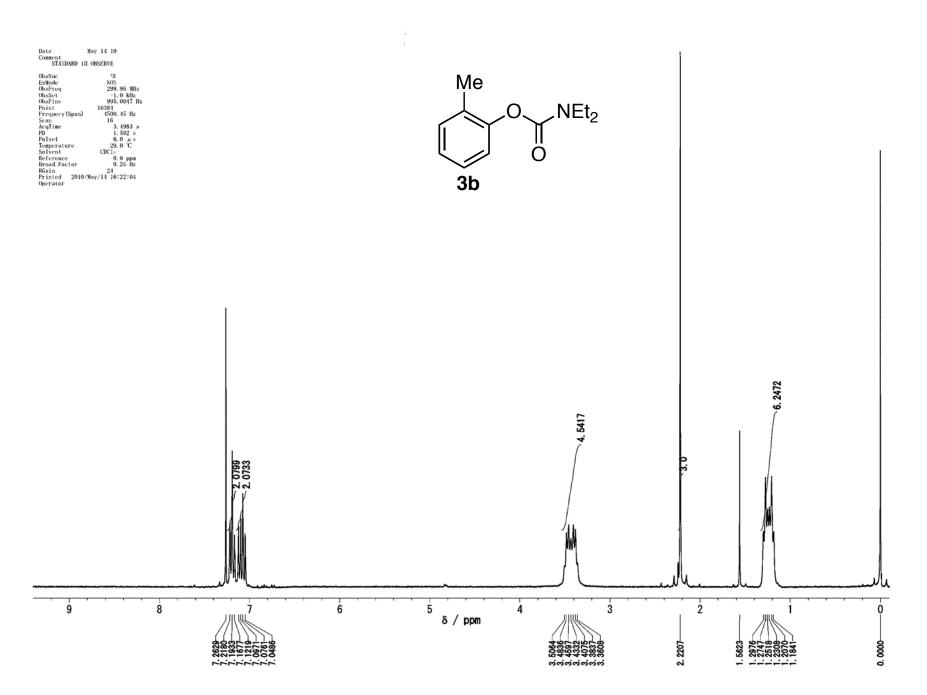


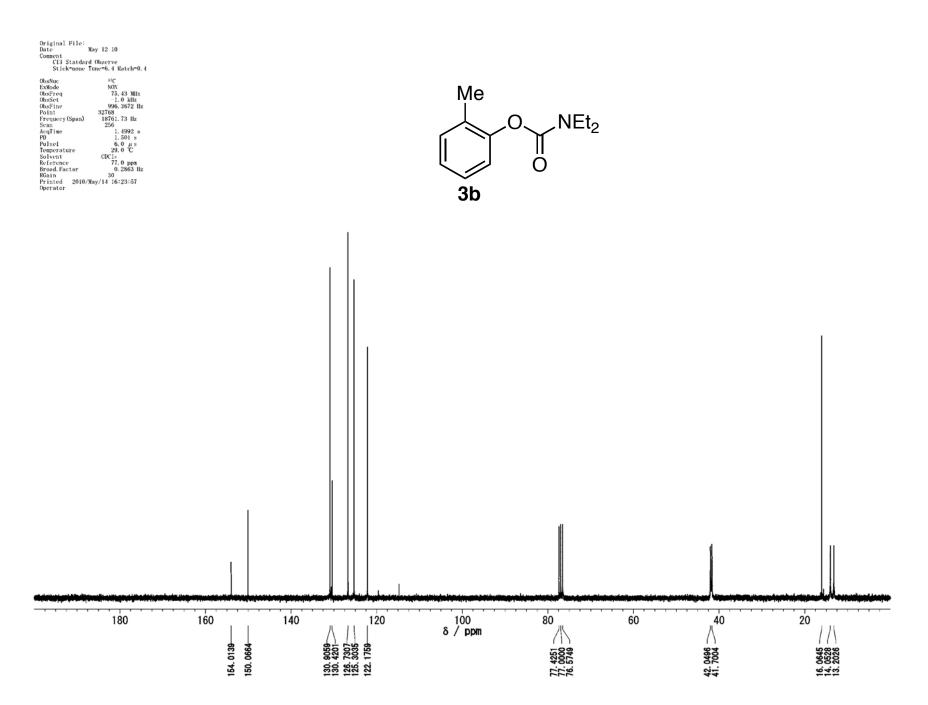


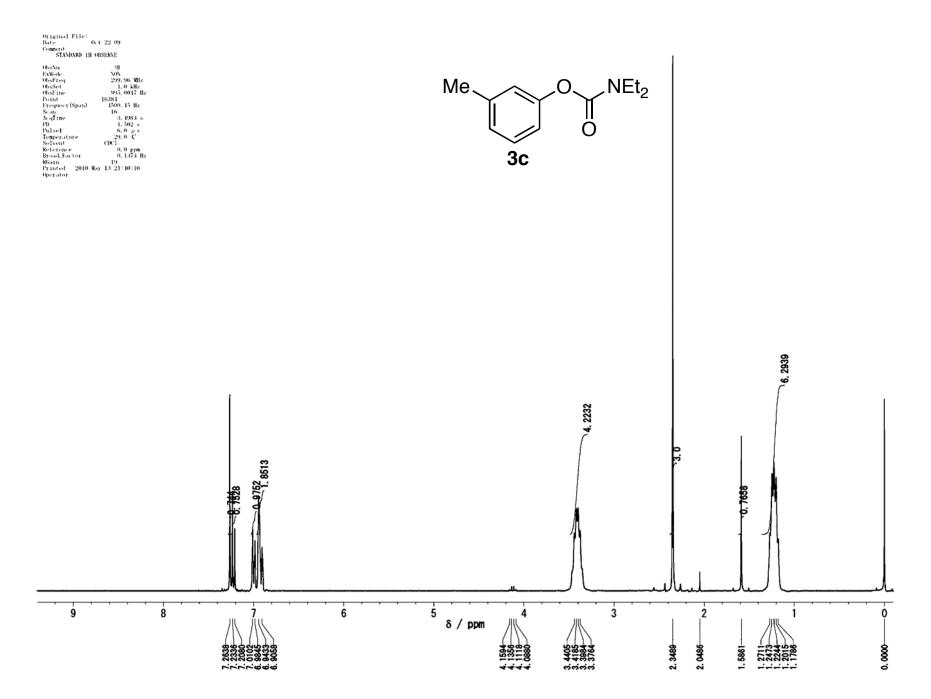


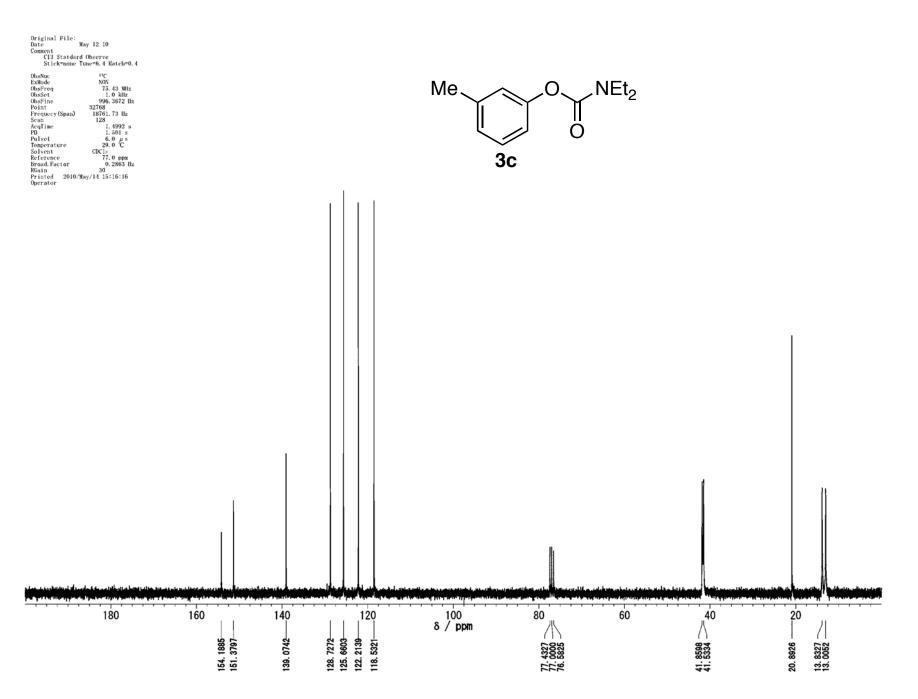


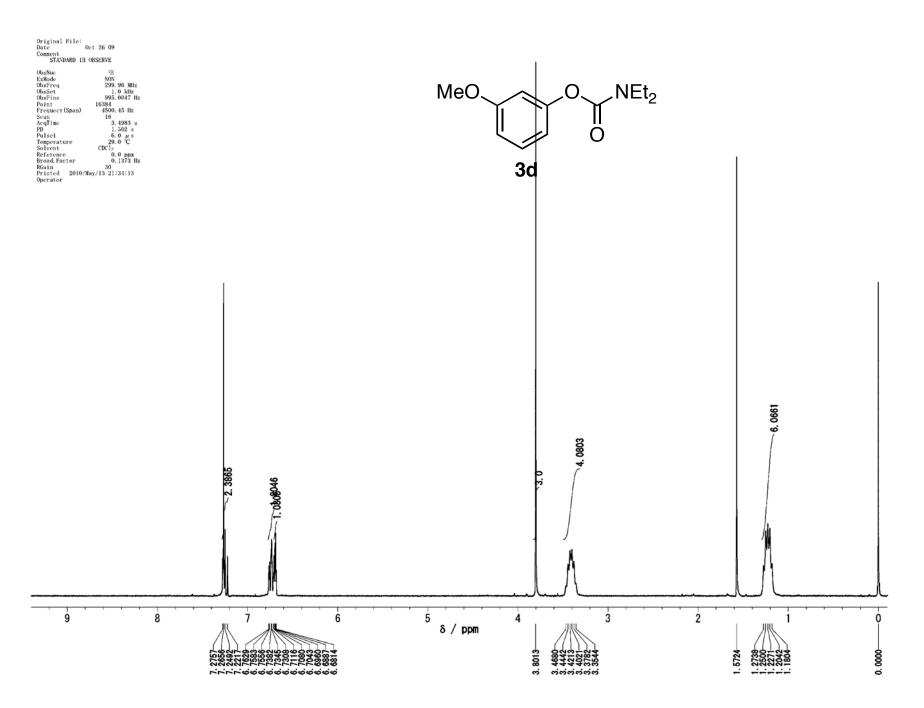


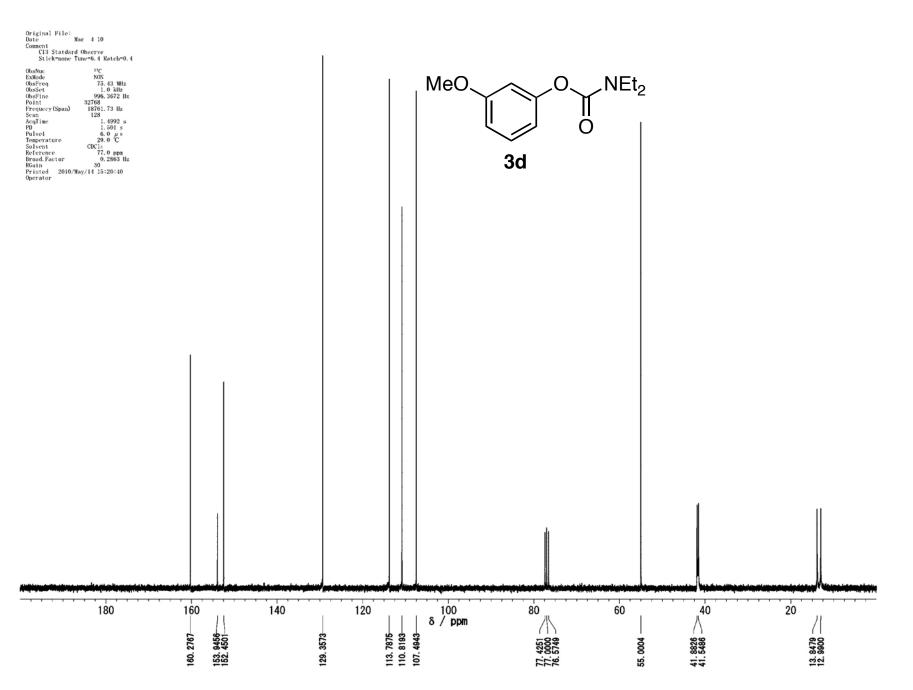


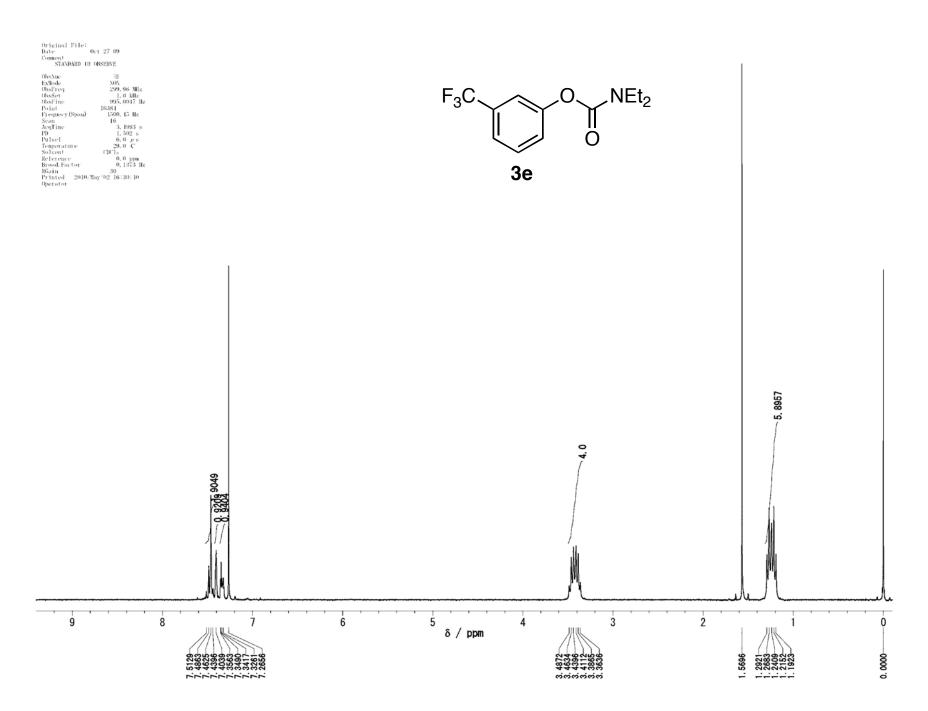


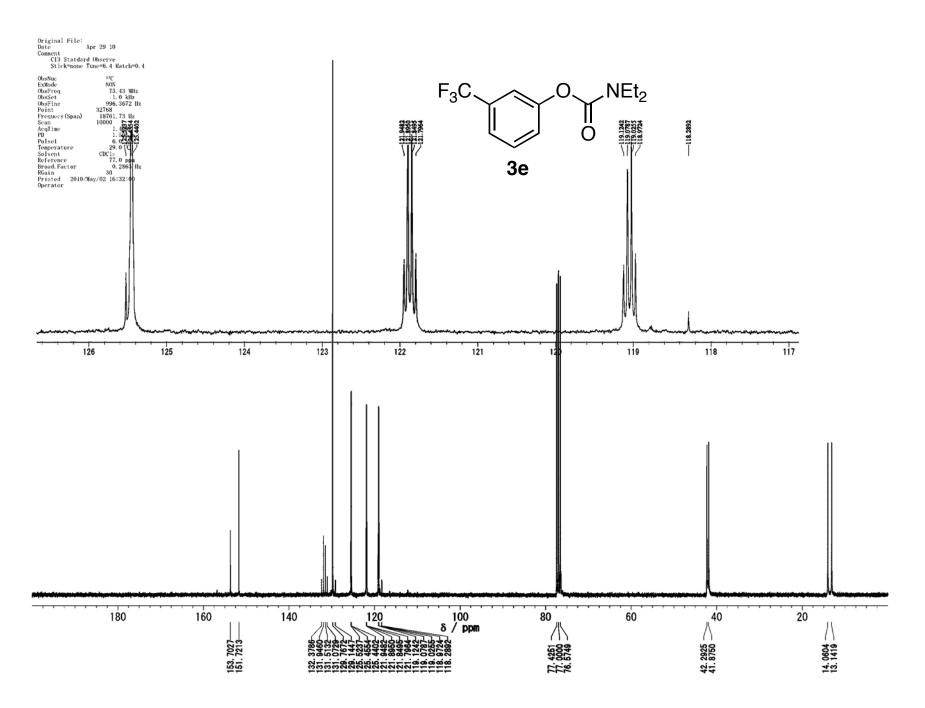


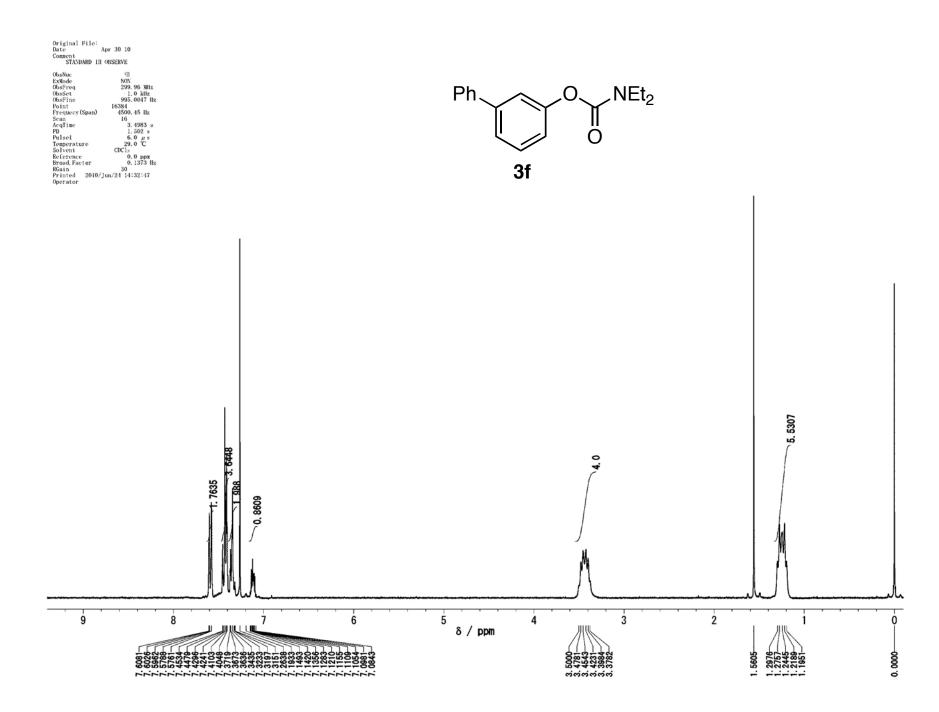


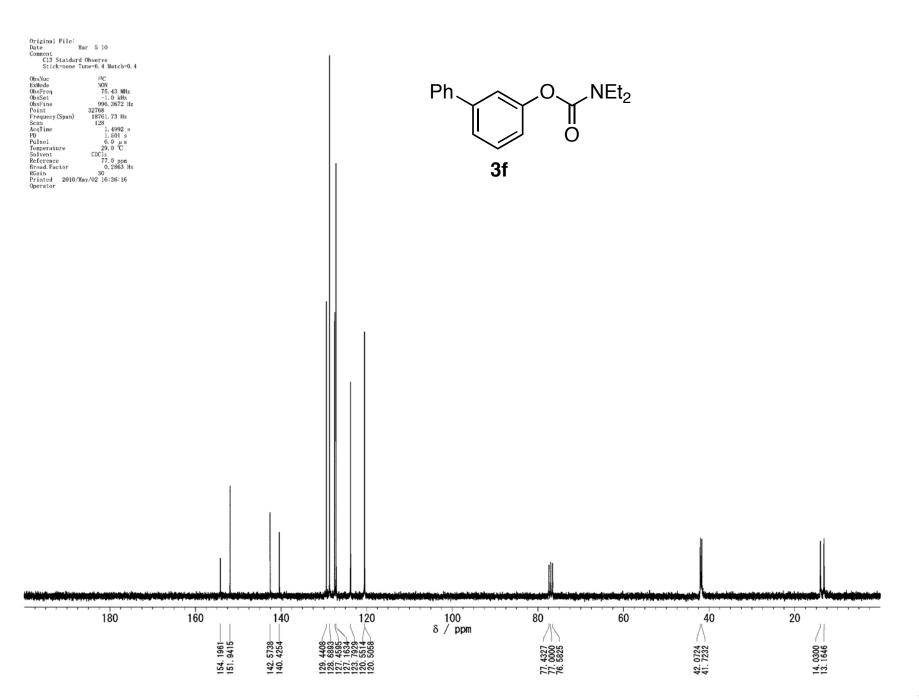






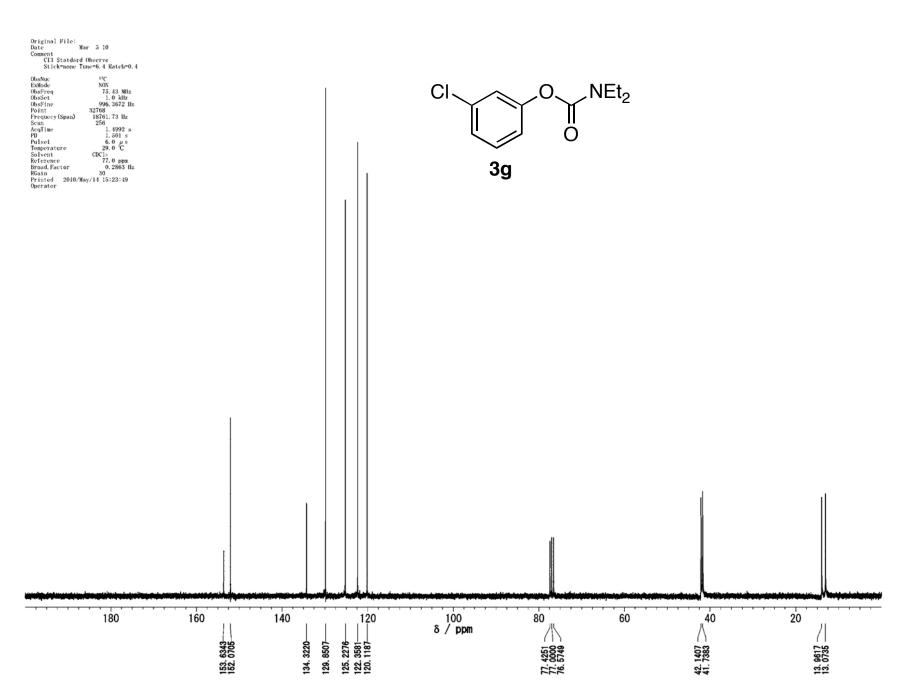




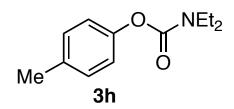


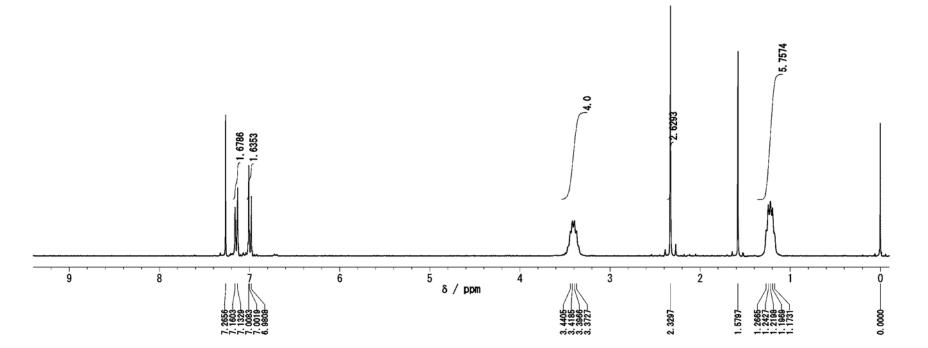
9 9 ( 1955	ObsNuc    Hi      EXMode    NON      ObsStrq    299,96 Miz      ObsStrq    1.0 Alt      ObsStre    995,0047 Hz      Point    1534      Promotion    1534      Promotion    1534      Promotion    1534      Promotion    1500,45 Hz      Stem    1      Point    1.502 ±      Pulsel    6.0 µ ±      Temporature    29,0 °C      Solvent    CDC1      Reference    0.0 ppm      Broad, Factor    0.1373 Hz      Roint    21      Operator    21
4. 435	2625
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$-\frac{1}{9} + \frac{1}{8} + \frac{1}{6} + \frac{1}{5} + \frac{1}{8} + \frac{1}{9} + \frac{1}{1} + 1$	9 $8$ $7$ $6$ $5$ $5$ / ppm $4$ $3$ $2$ $1$

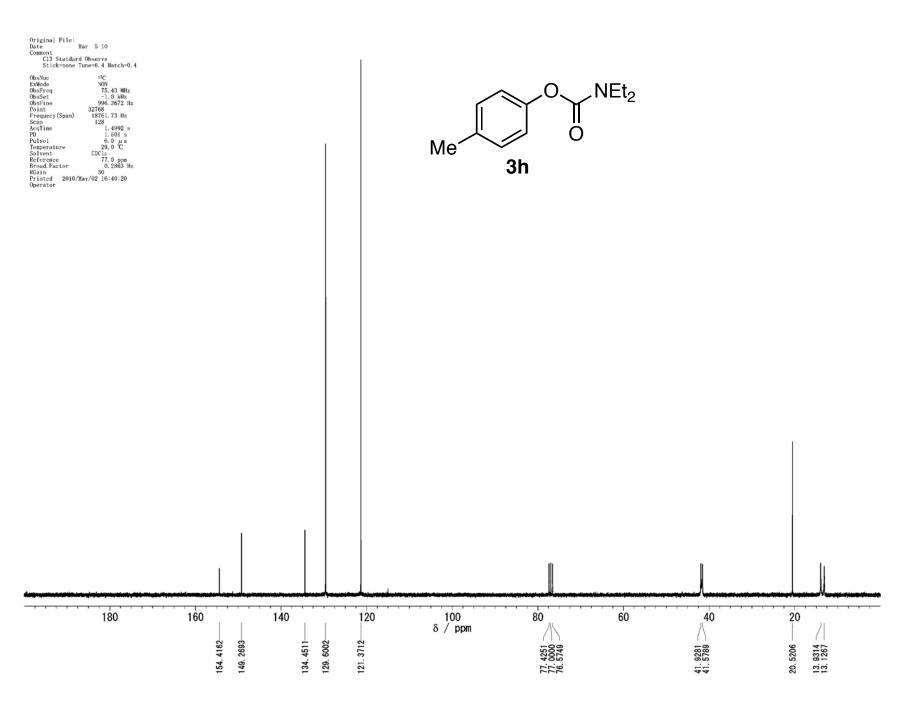
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P	oint	16384
F	requecy (Span)	4500, 45 Hz
S	can	16
A	cqTime	3.4983
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s	olvent	CDC1 <sub>3</sub>
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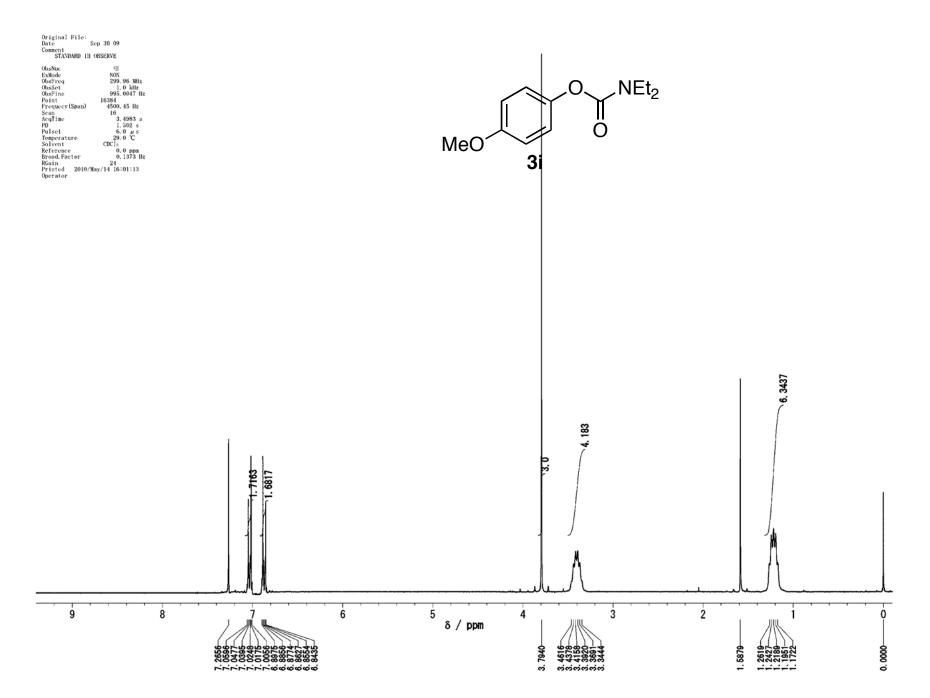


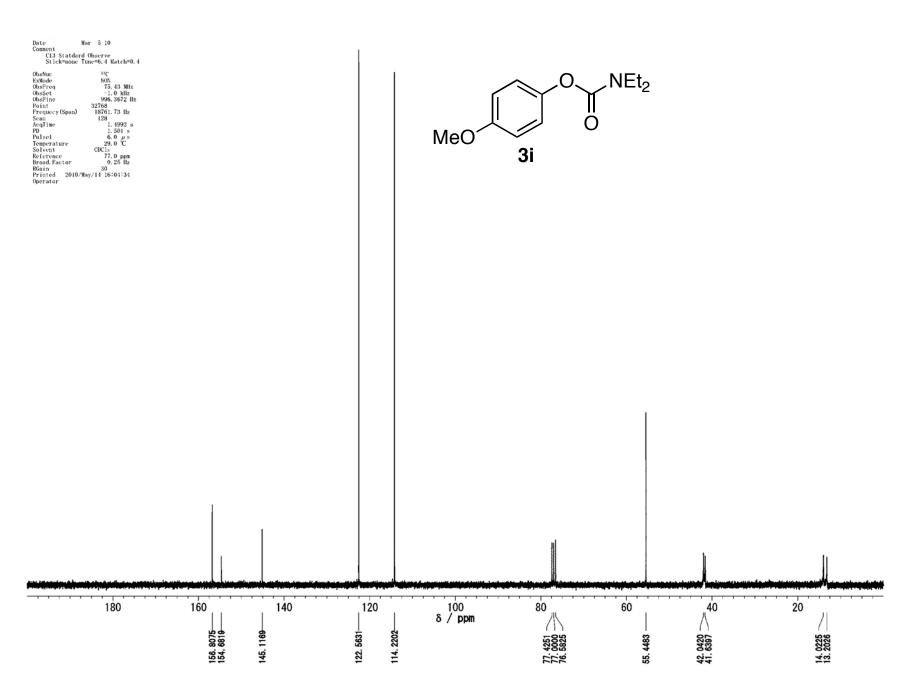
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Point	16384		
Frequecy (Span)	4500, 45 IIz		
Scan	16		
AcqTime	3.4983 s		
PD	1.502 s		
Pulsel	6.0 µ s		
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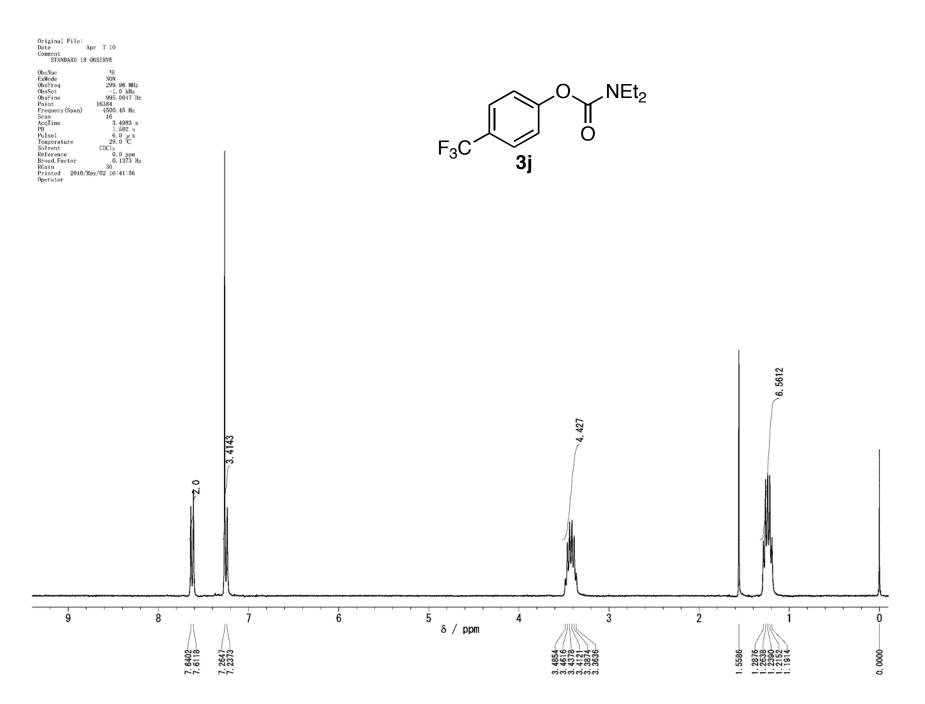


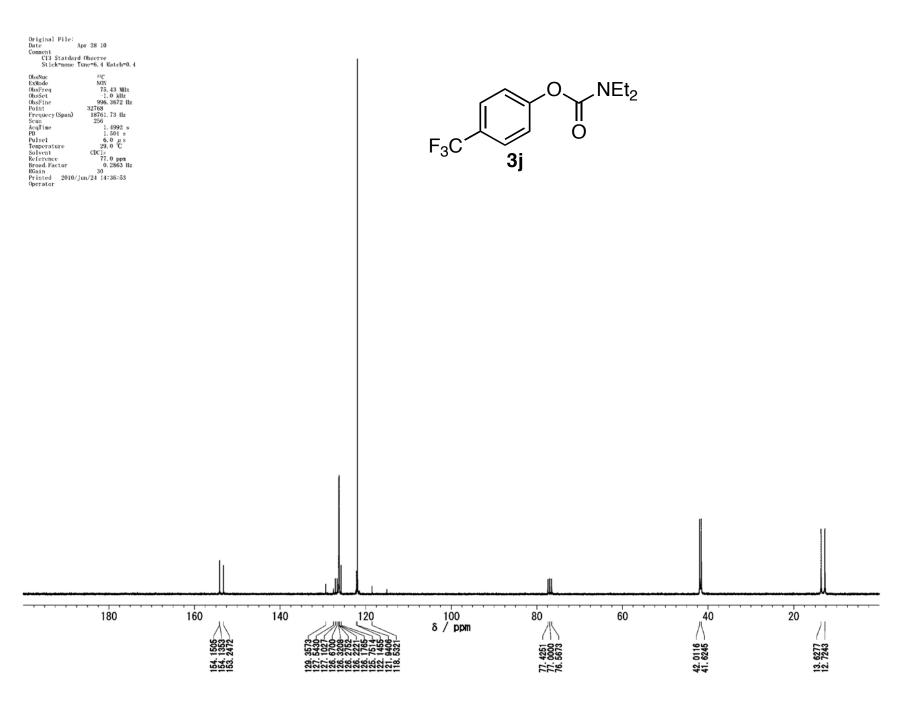


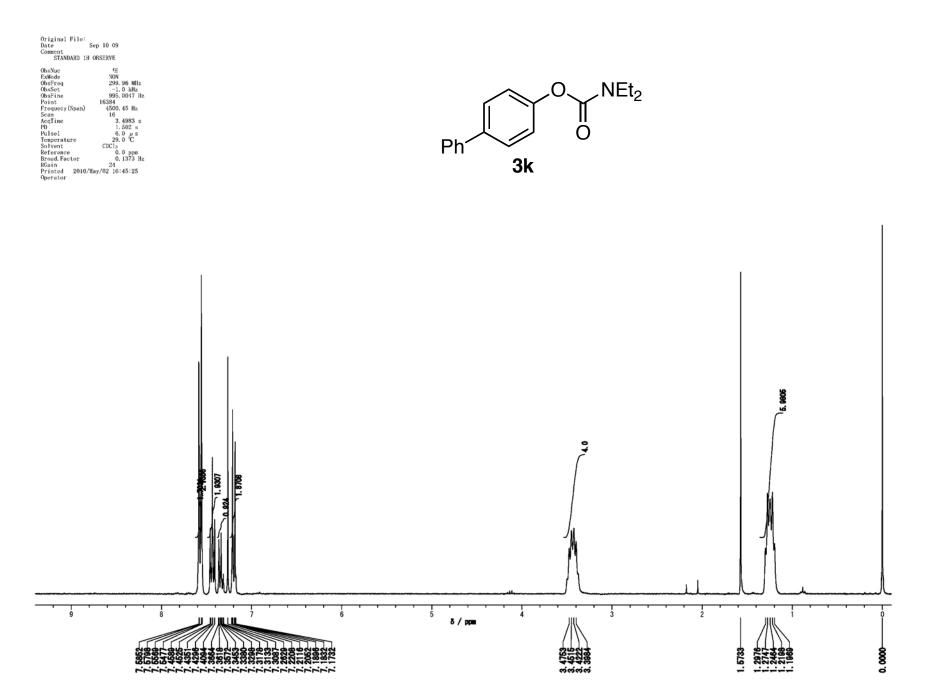


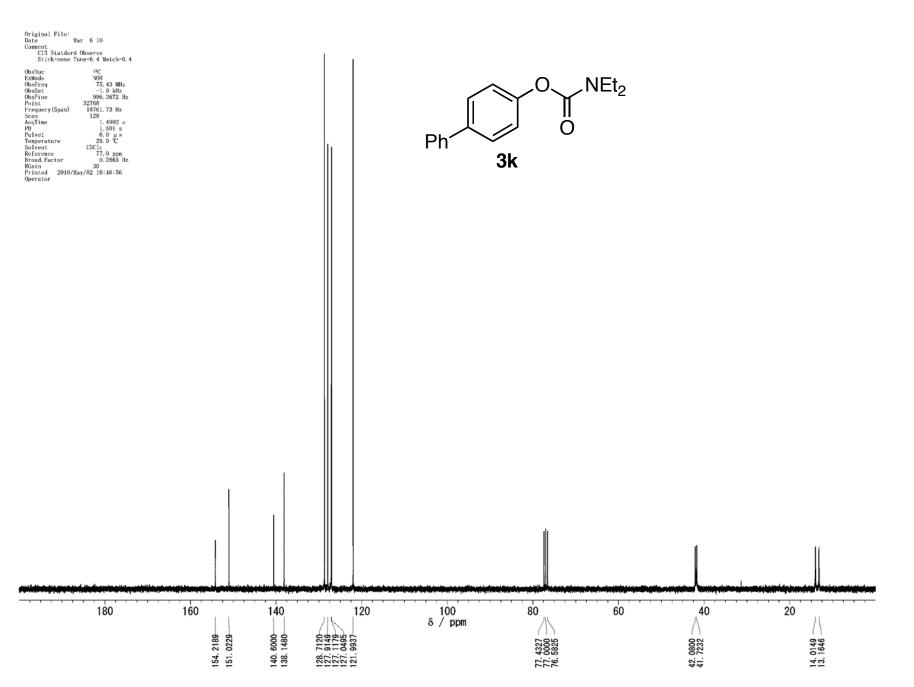




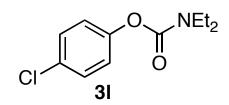


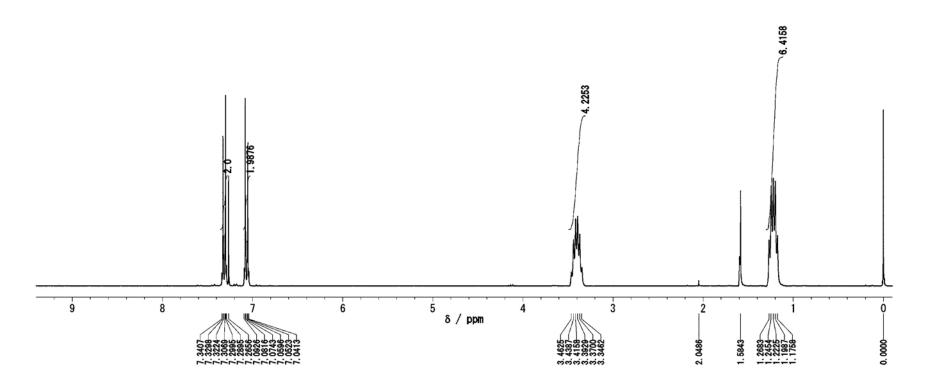


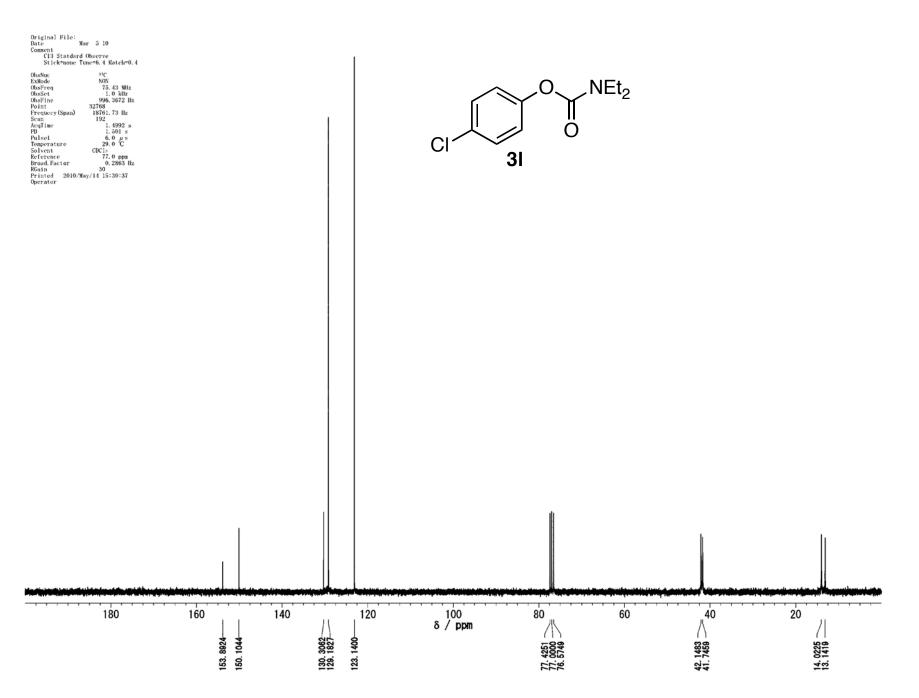


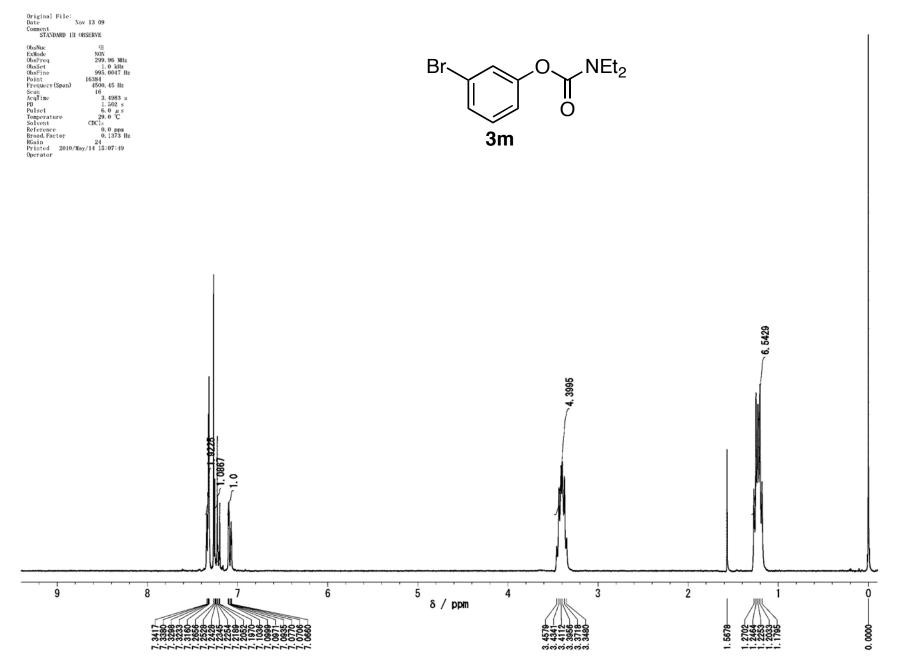


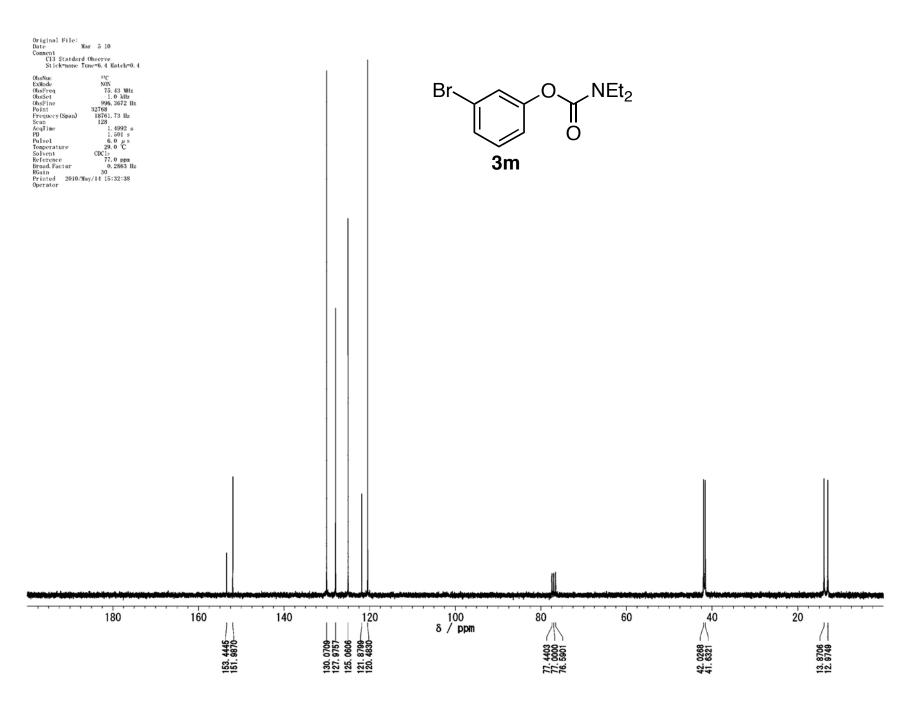
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ObeNuc ExMode ObsFreq ObsFreq ObsFreq ObsFreq ObsFreq Sean AcqTime PD Pulsel Temperature Solvent Reference Broad, Factor Resin Printed 2010/May, Operator	$\begin{array}{c} 11\\ \text{NON}\\ 239, 96 \ \text{MHz}\\ 1.0 \ \text{KHZ}\\ 995, 0047 \ \text{Hz}\\ 995, 0047 \ \text{Hz}\\ 16384 \ \text{Hz}\\ 164983 \ \text{Hz}\\ 1.502 \ \text{Hz}\\ 29, 0 \ \text{CCC}\\ 1.0 \ \text{Hz}\\ 29, 0 \ \text{Hz}\\ 29, 0 \ \text{Hz}\\ 1373 \ \text{Hz}\\ 19\\ 19\\ 1415:02:58\end{array}$

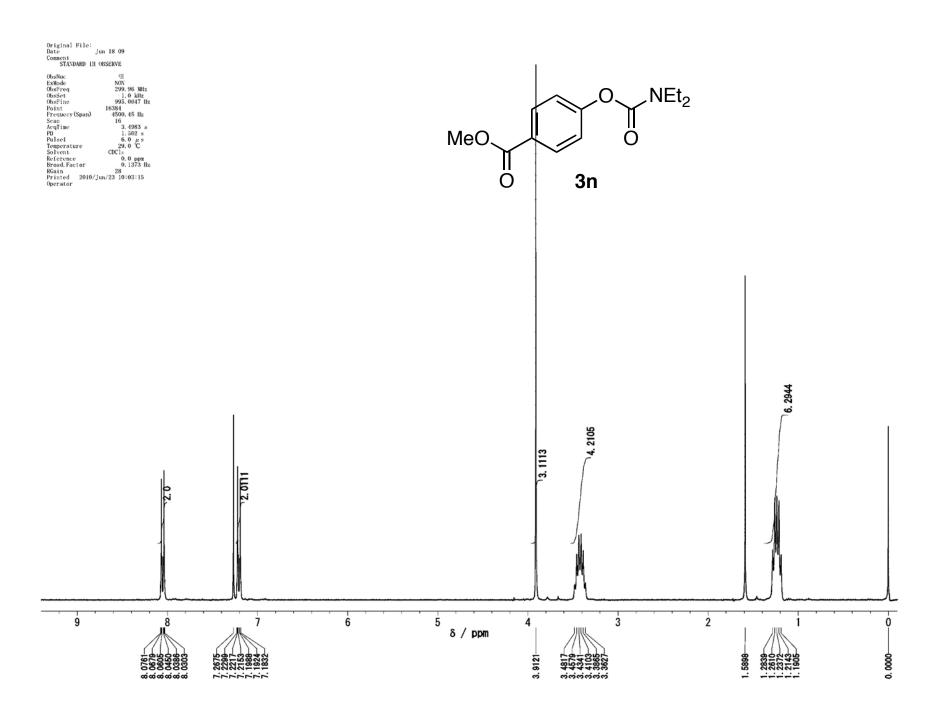


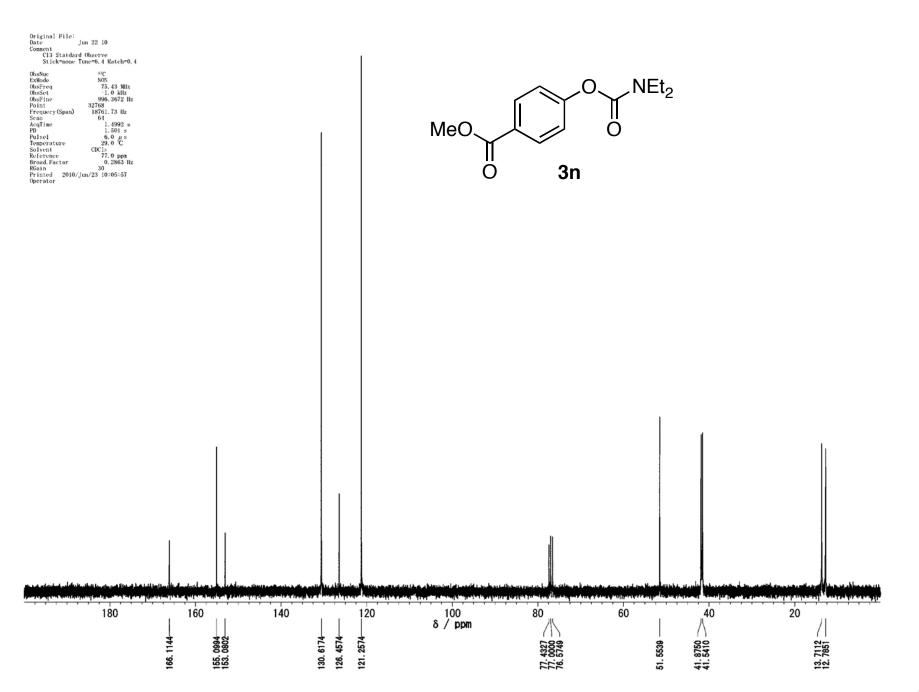




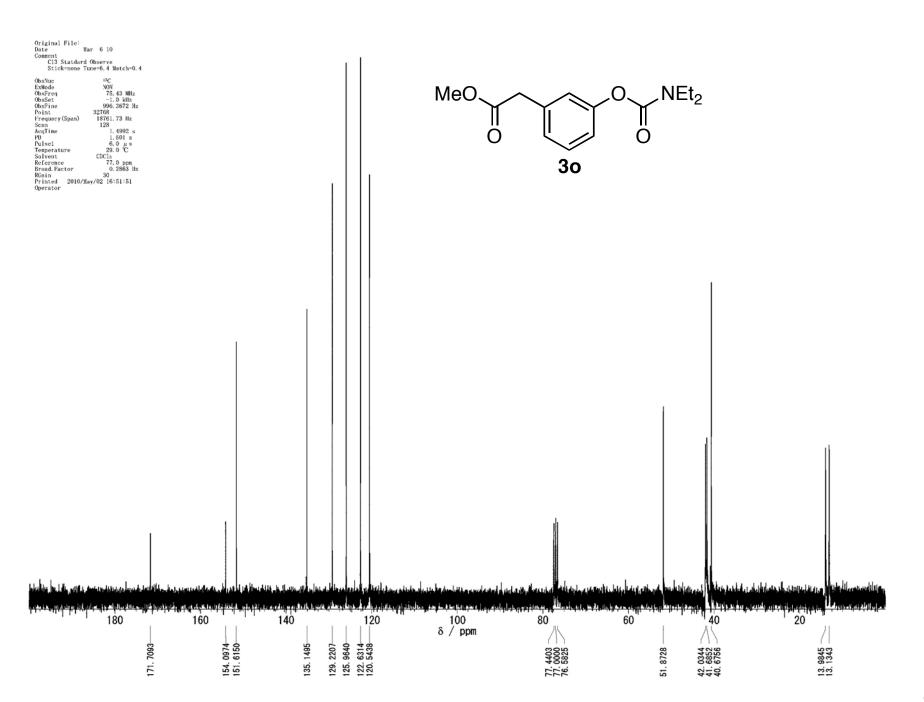


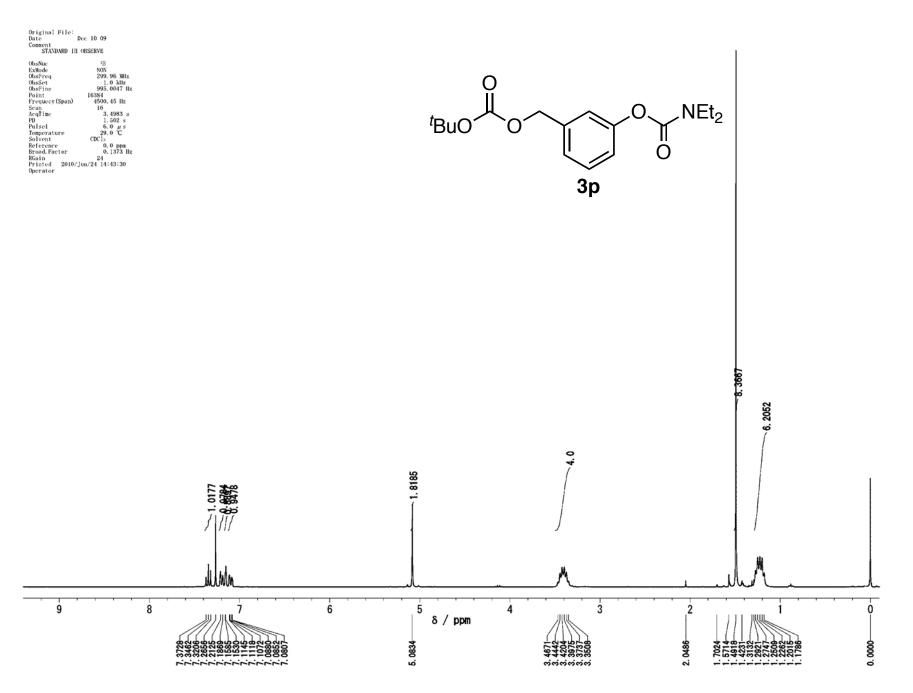


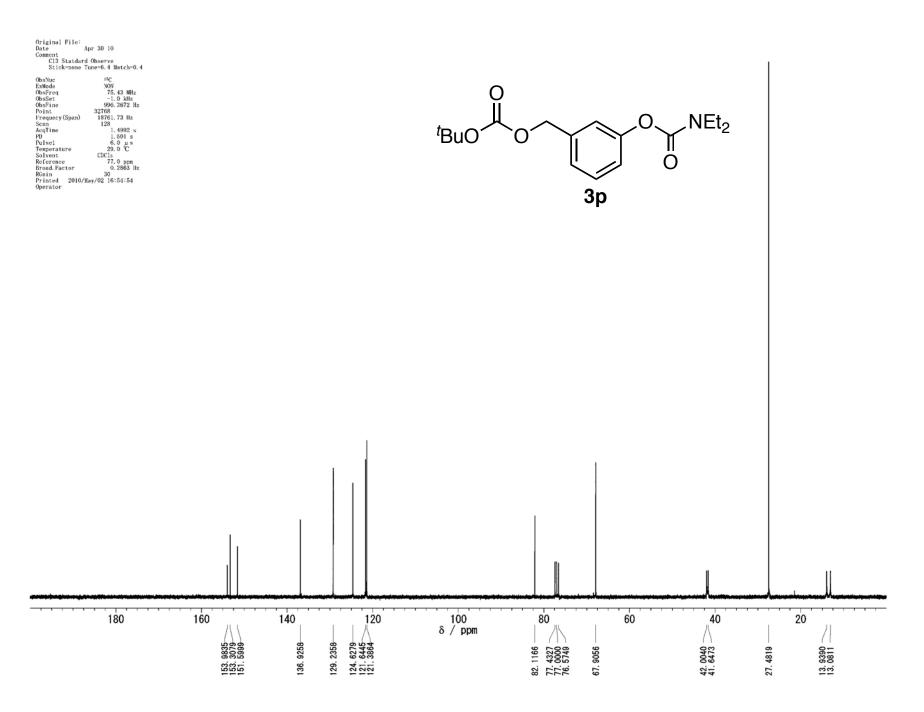


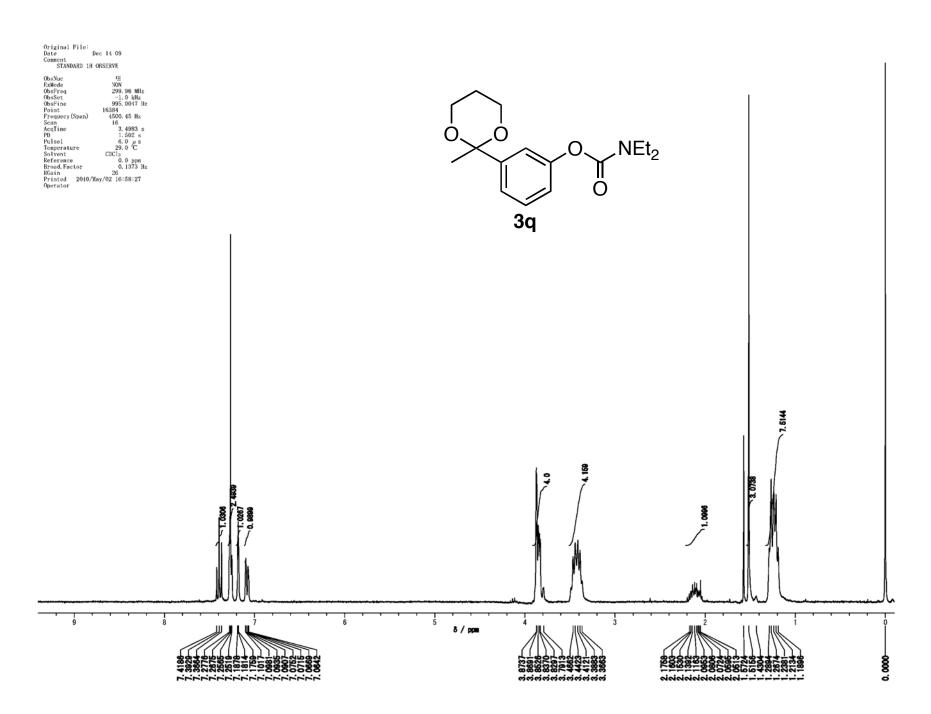


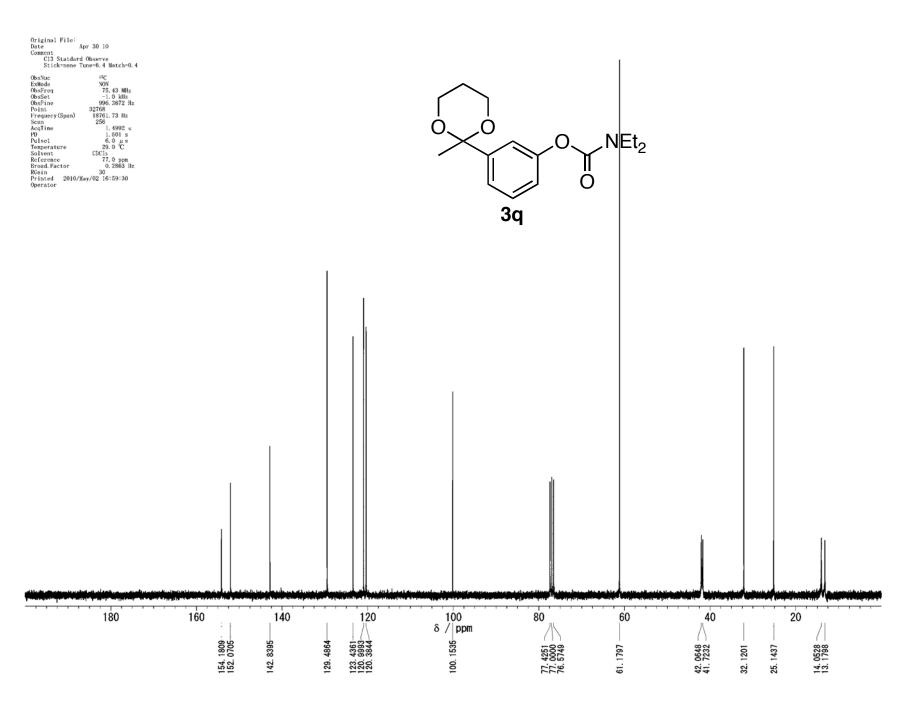


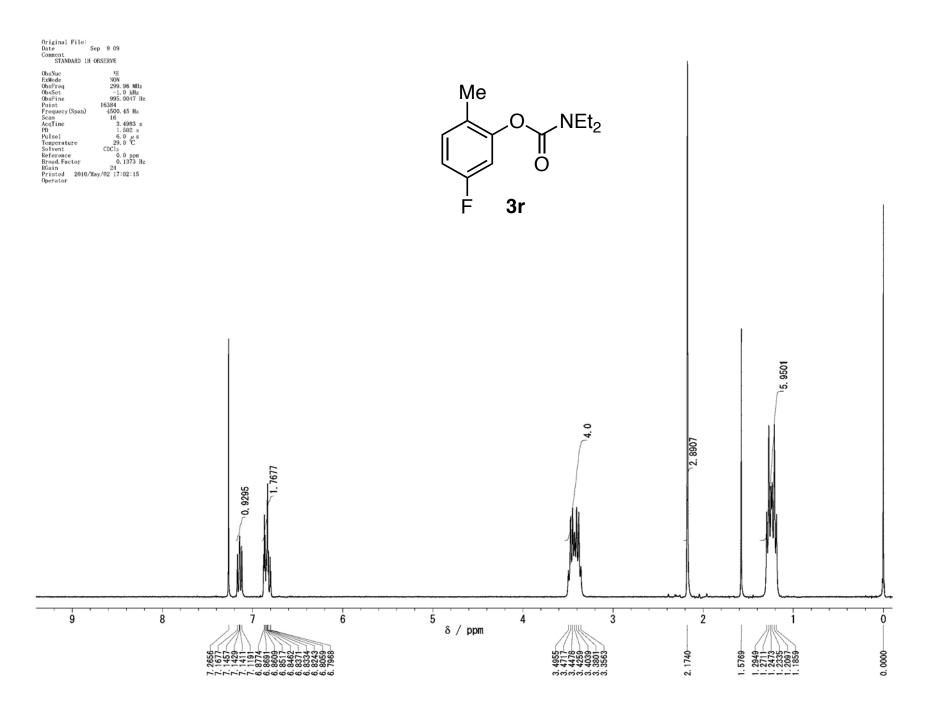


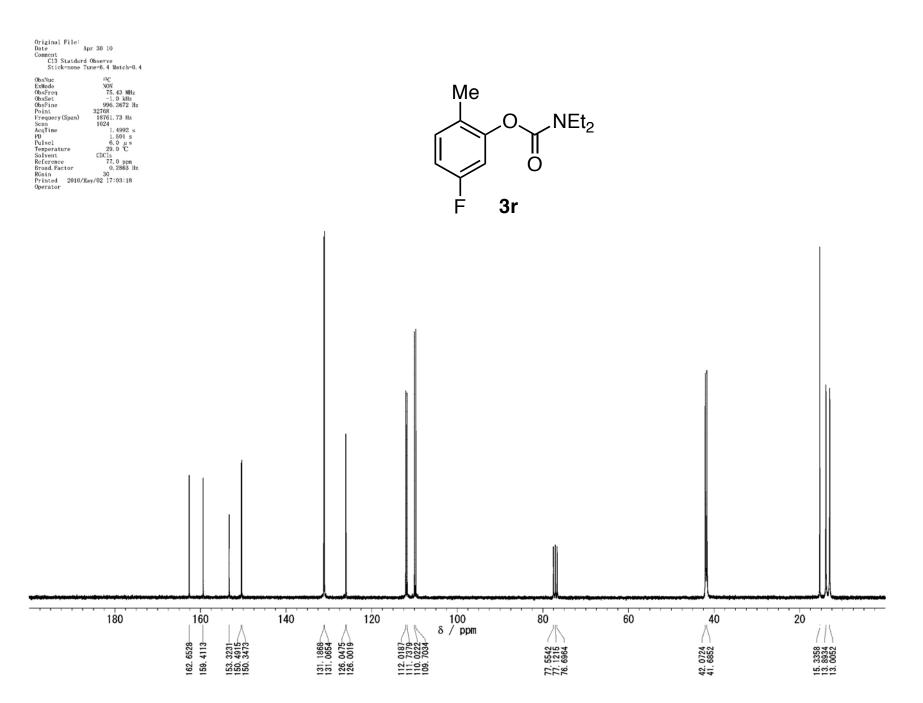


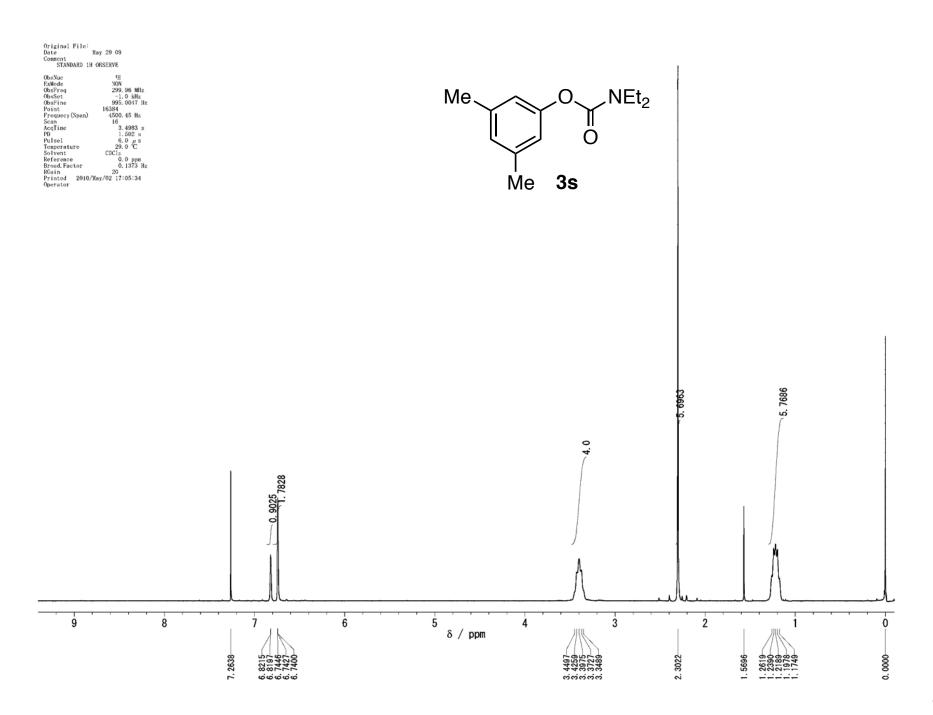


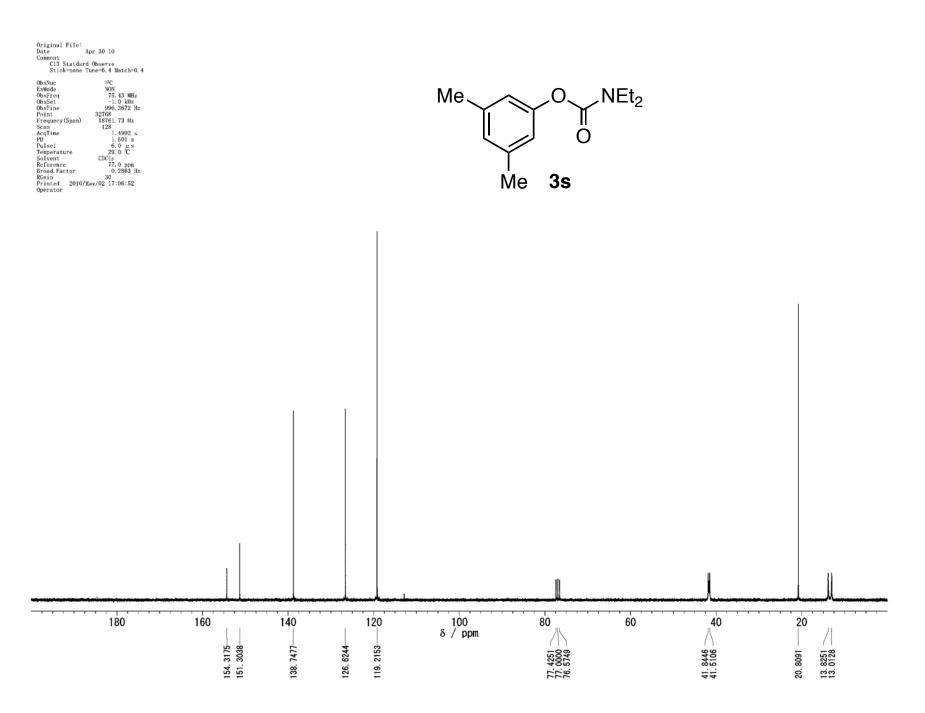


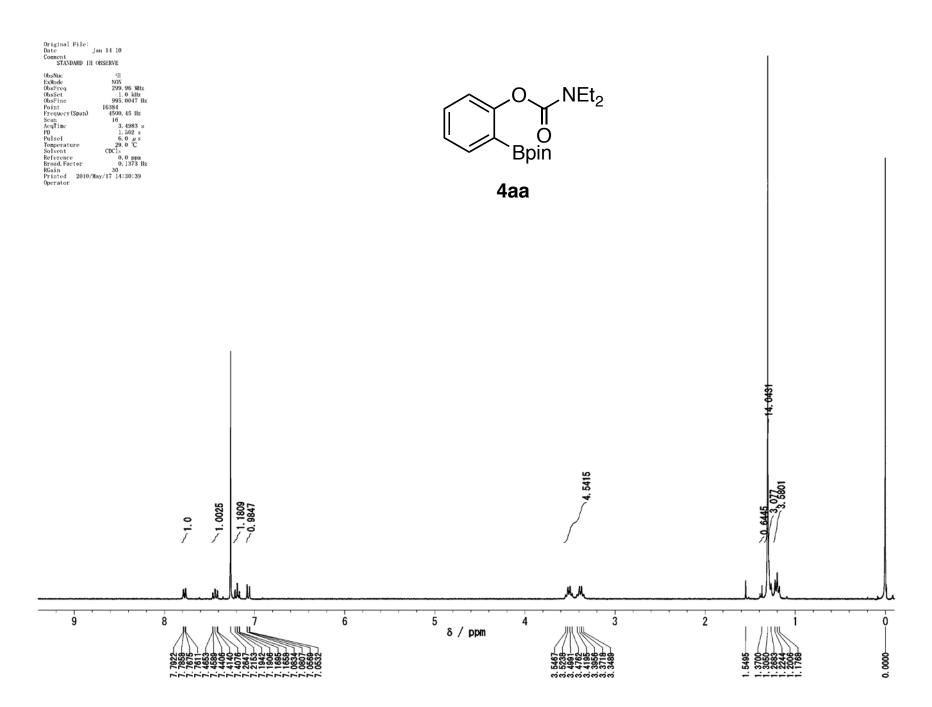


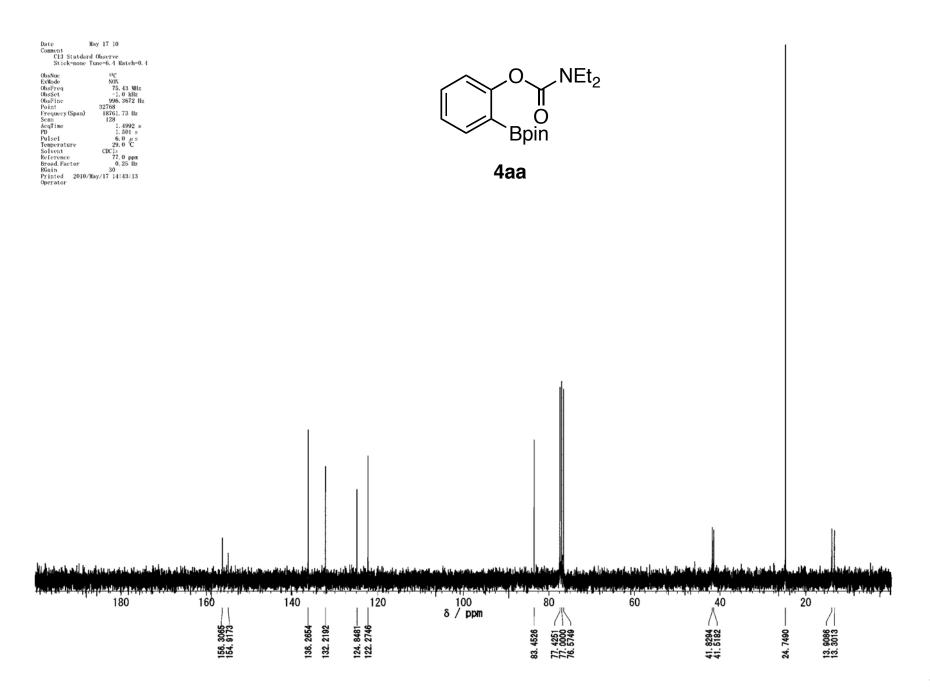


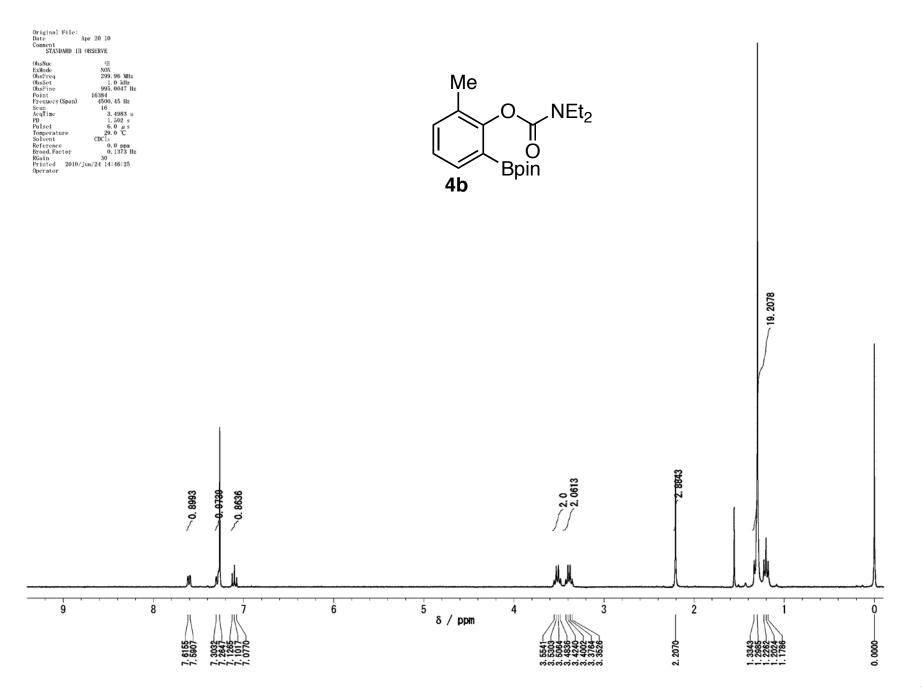


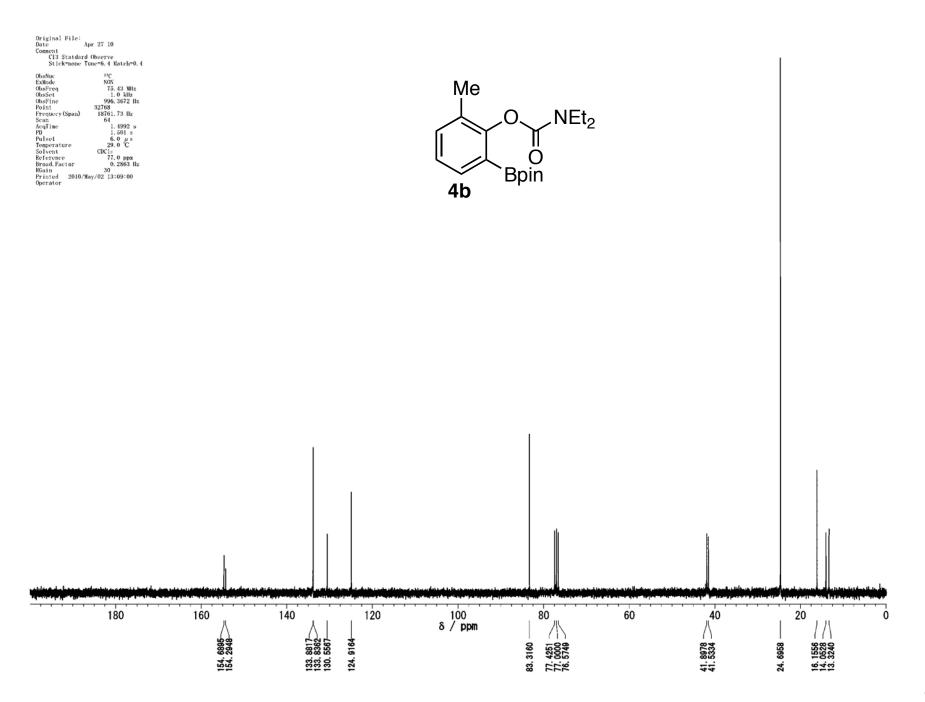


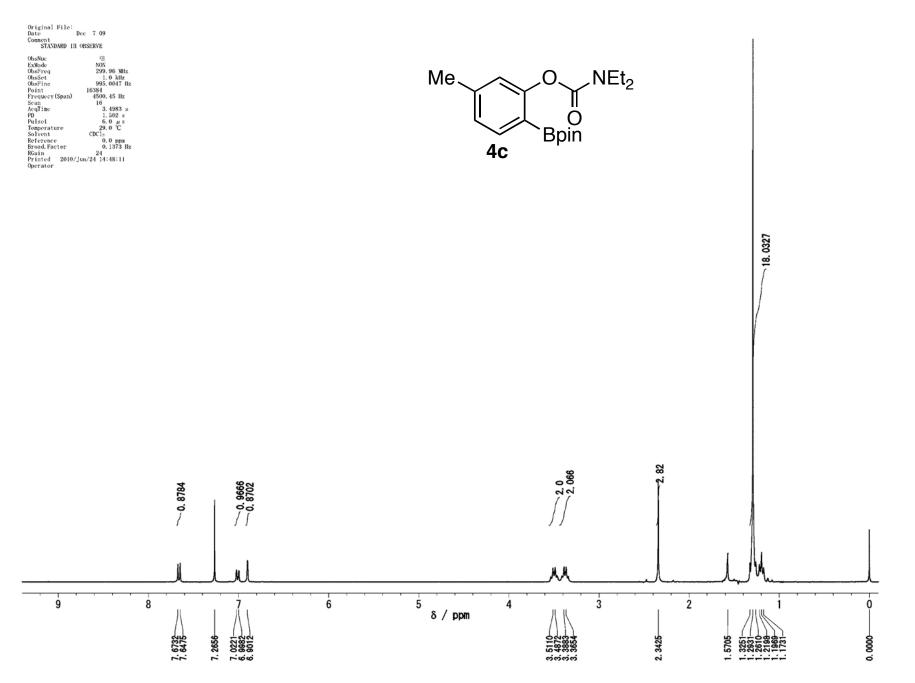


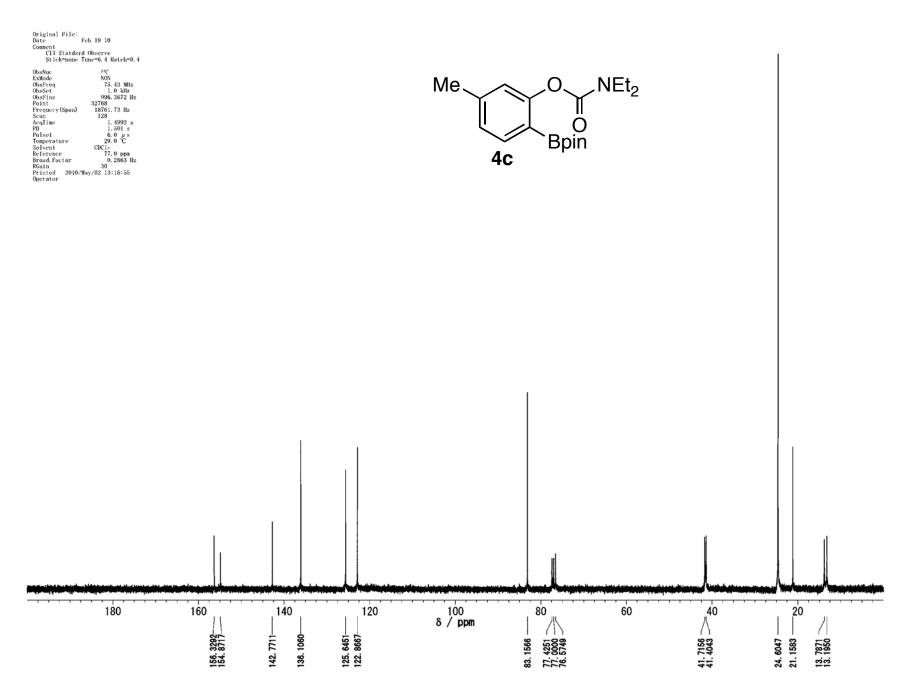


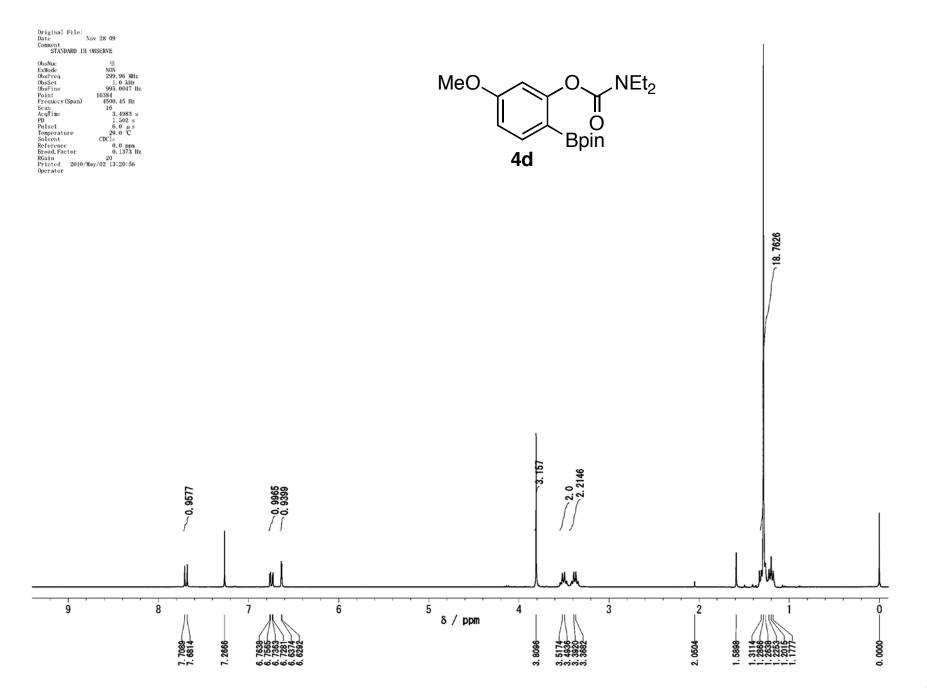


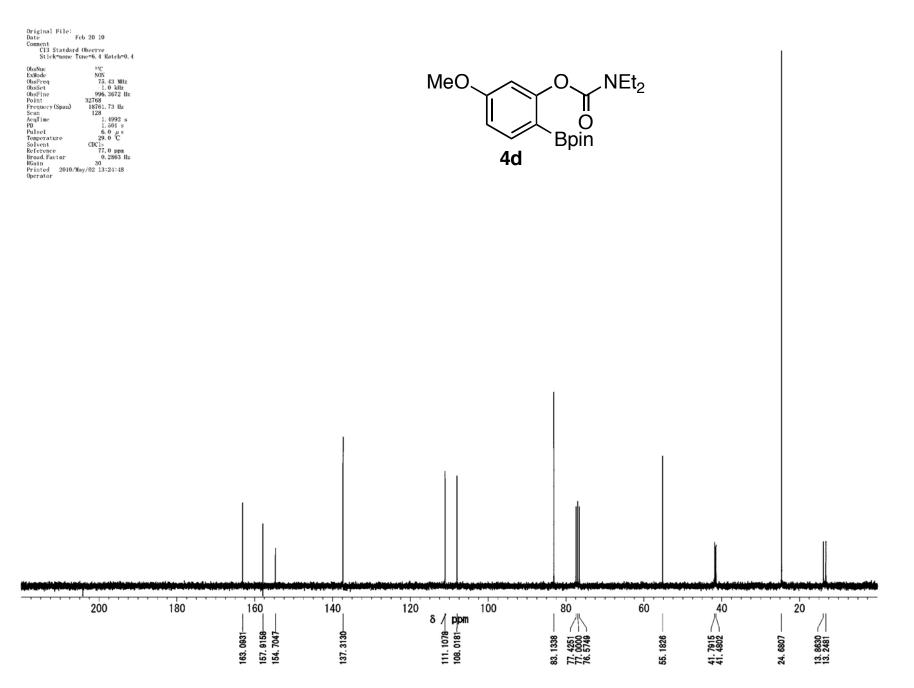




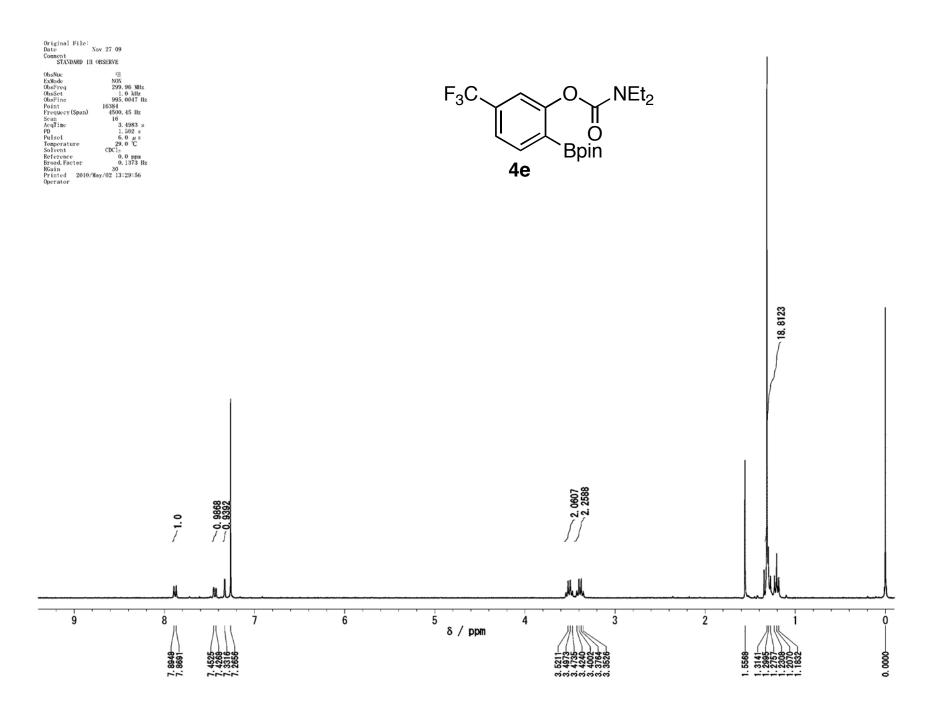


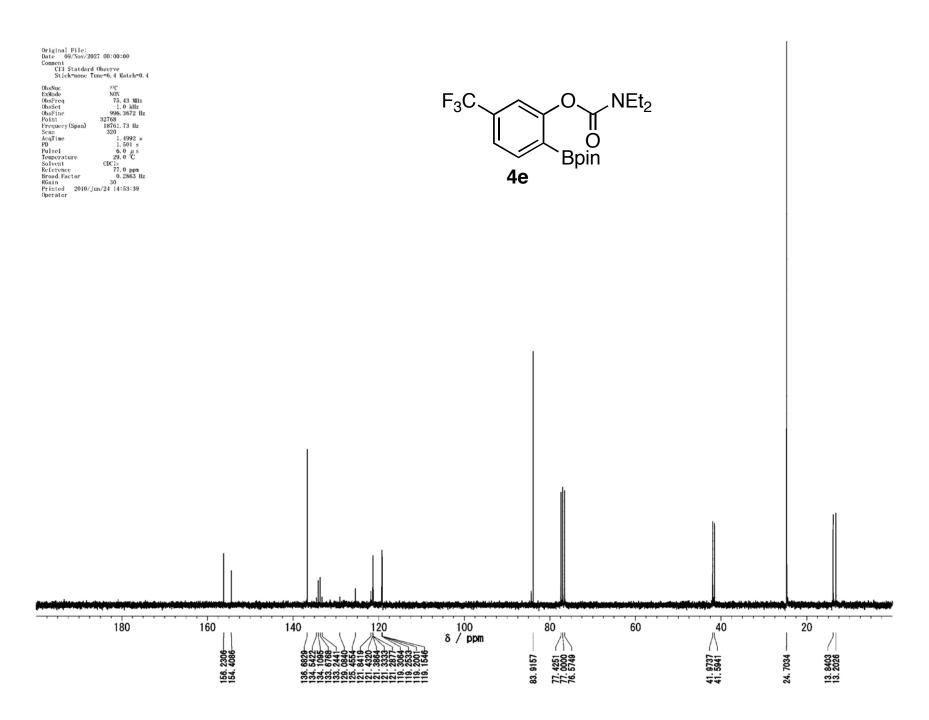


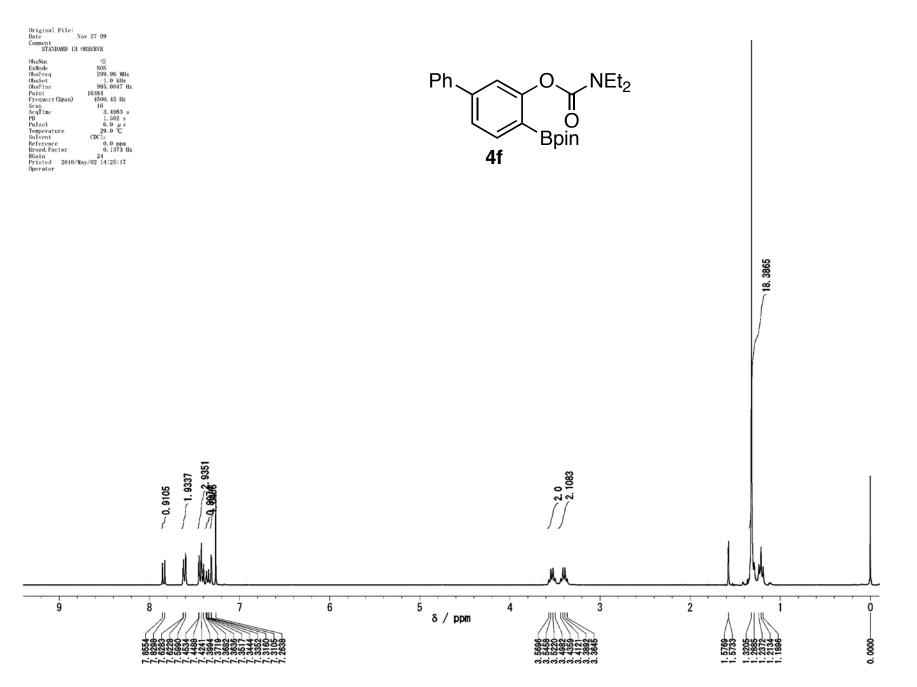


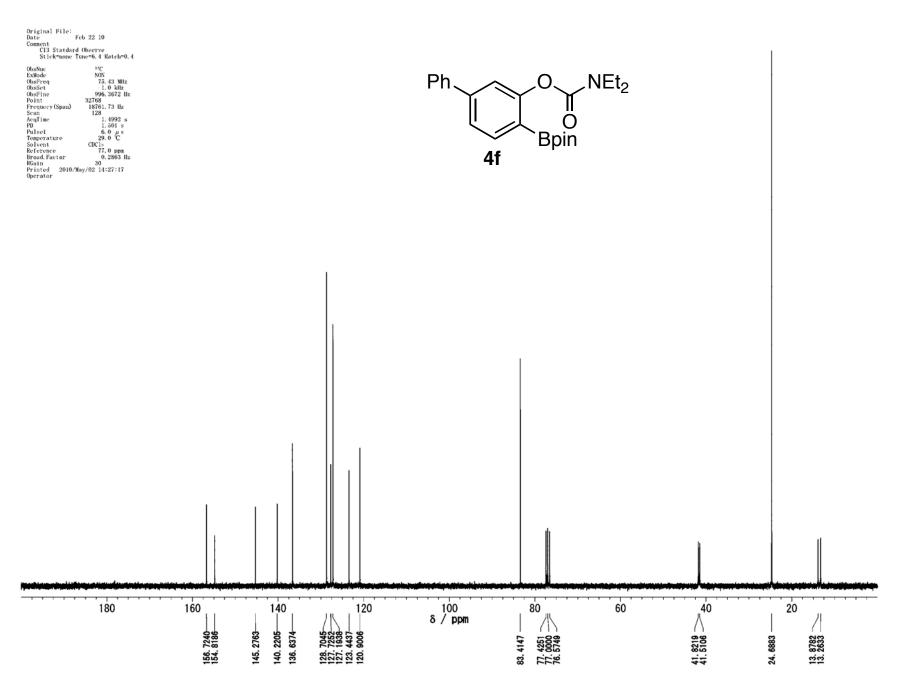


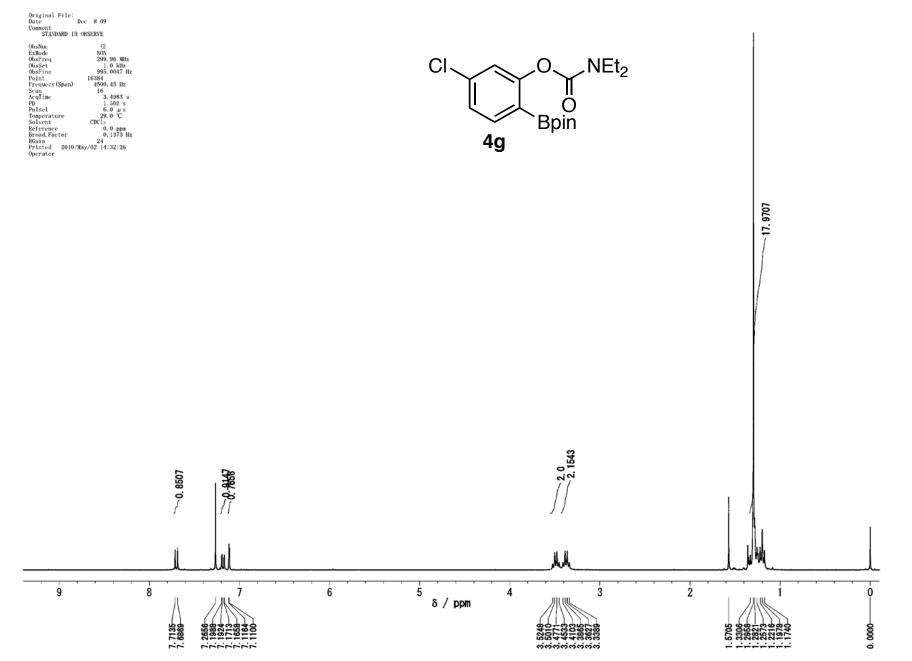
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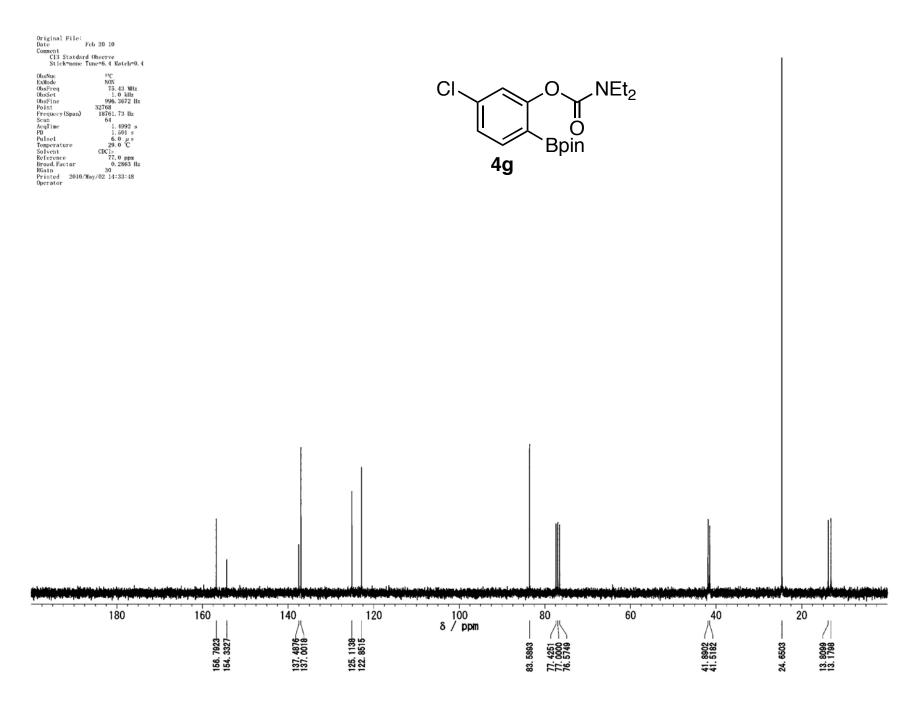




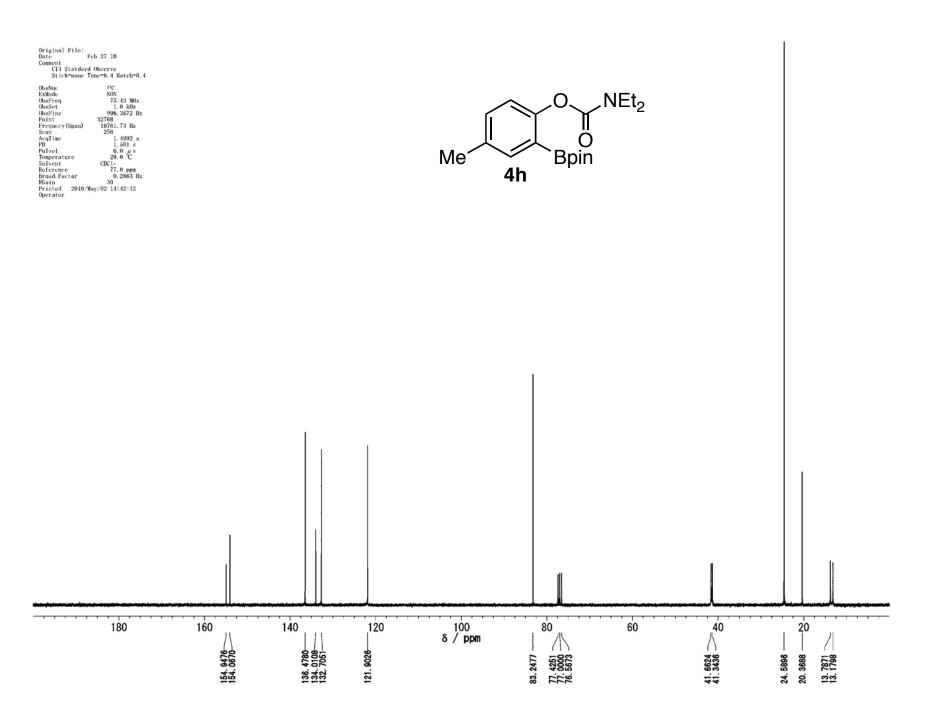


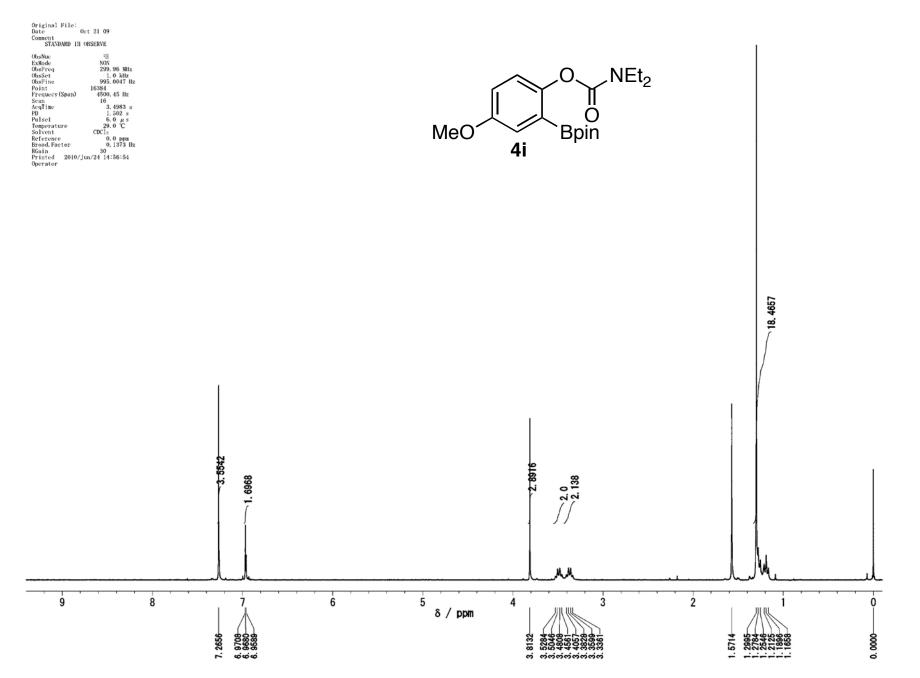


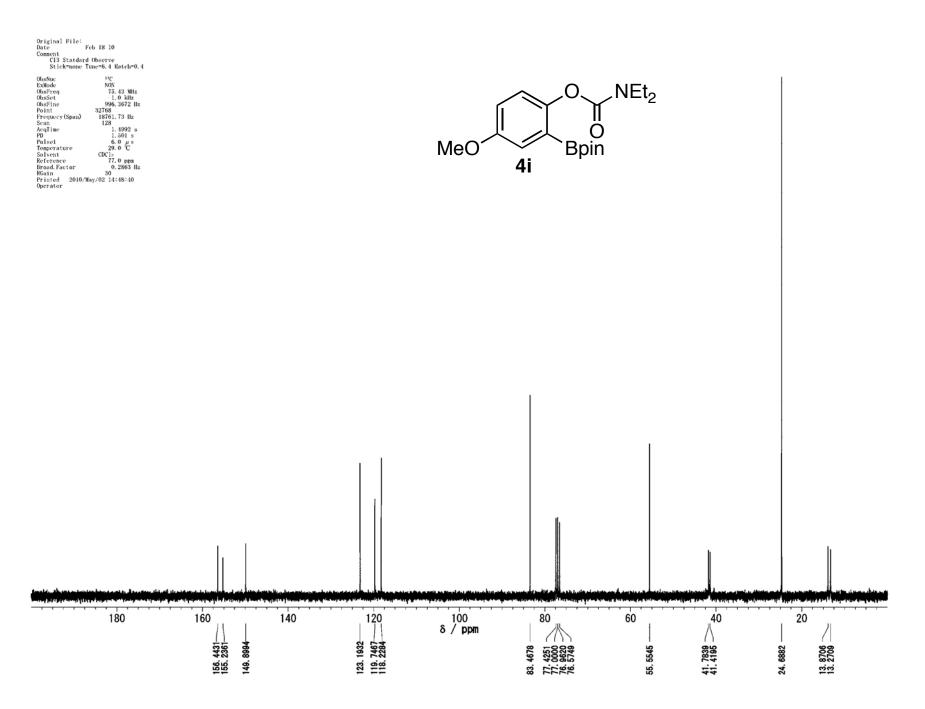


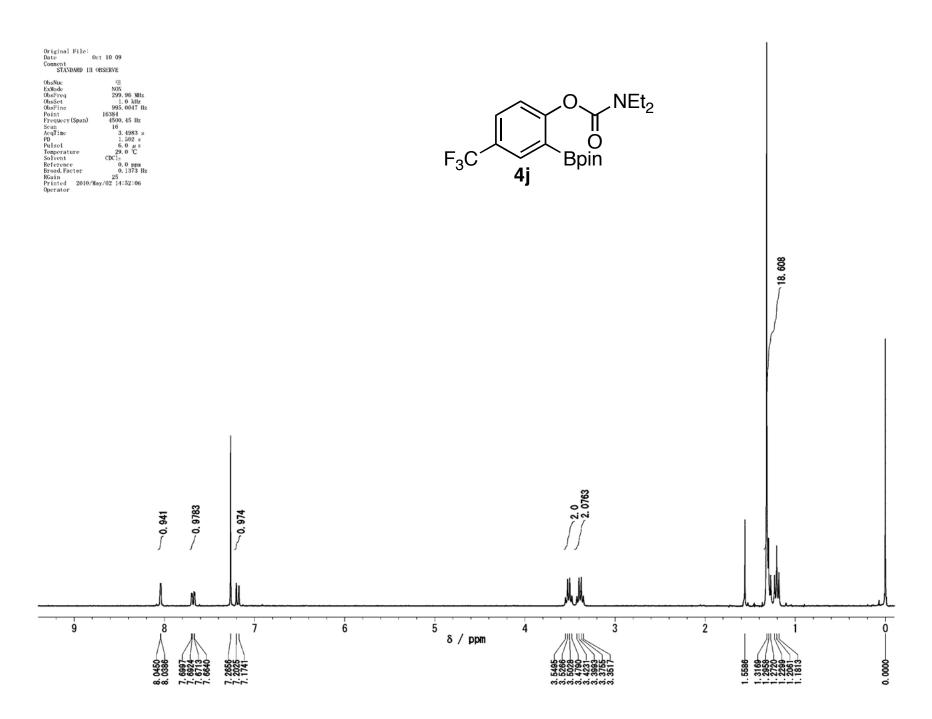


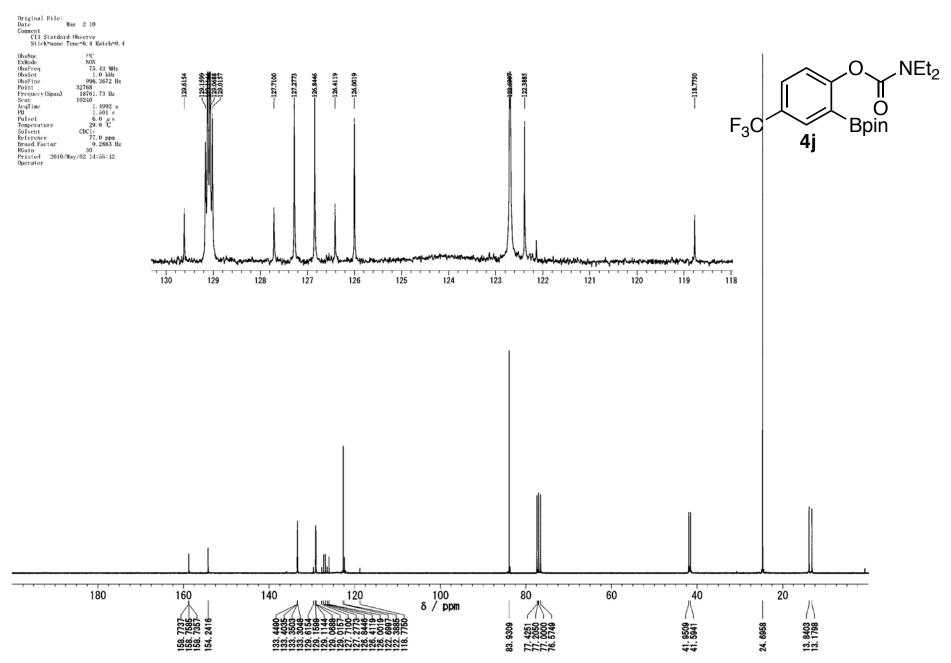


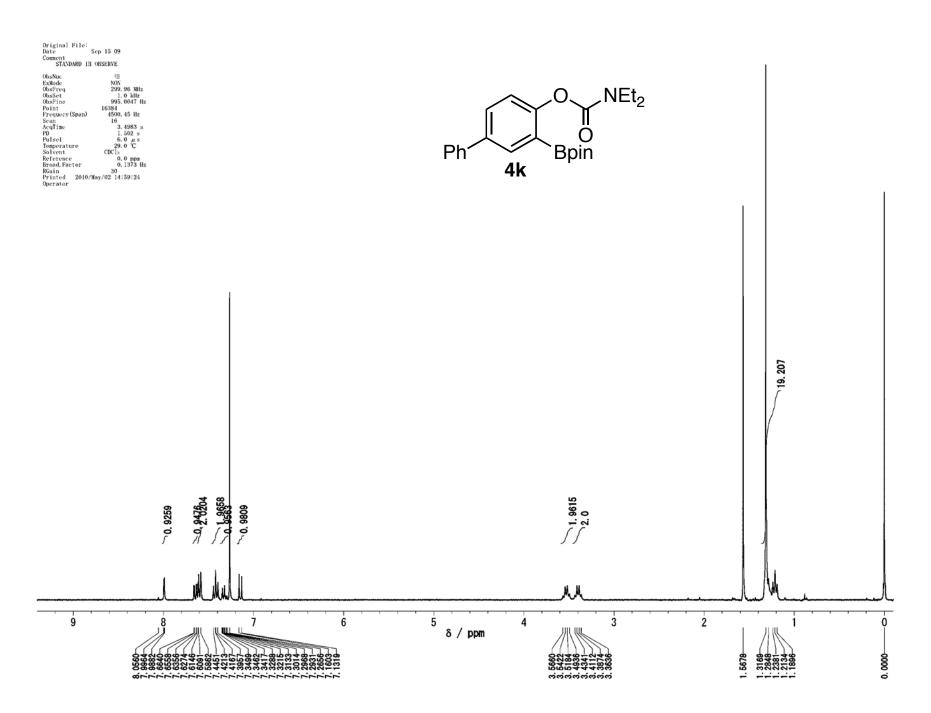


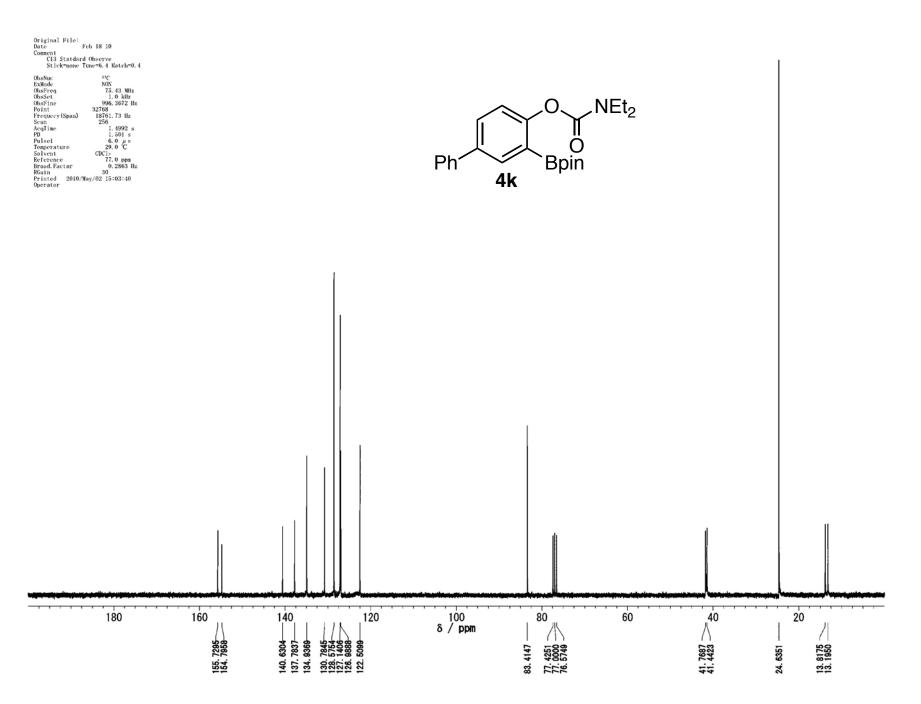


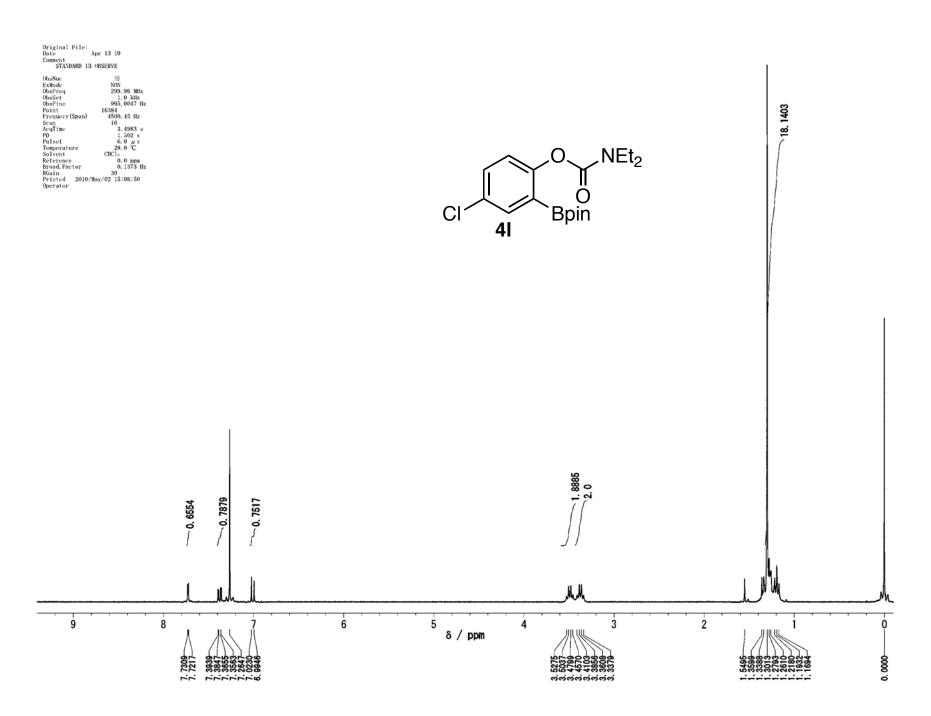


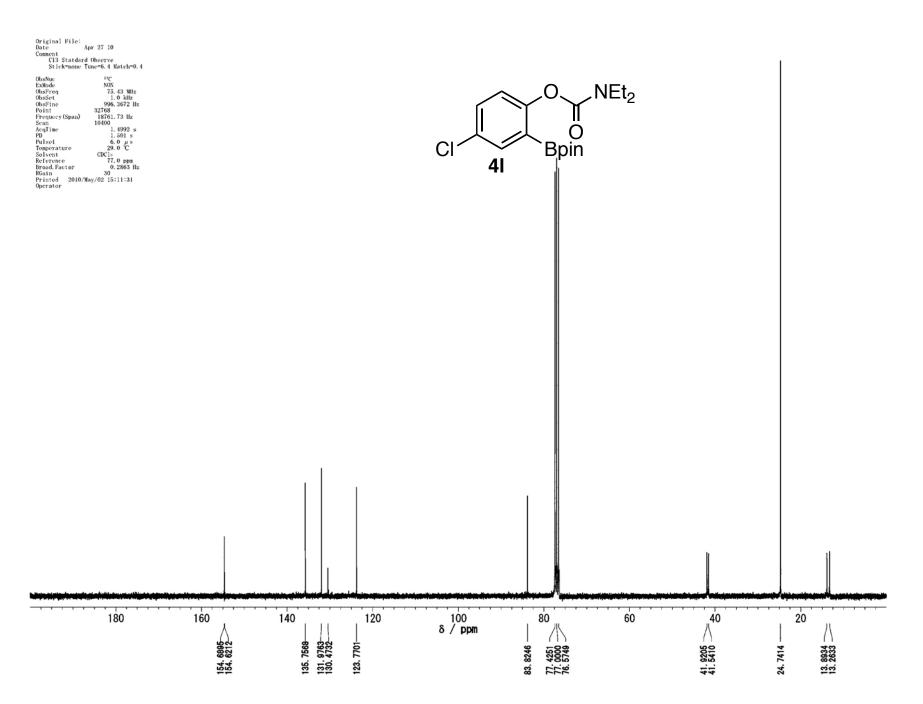


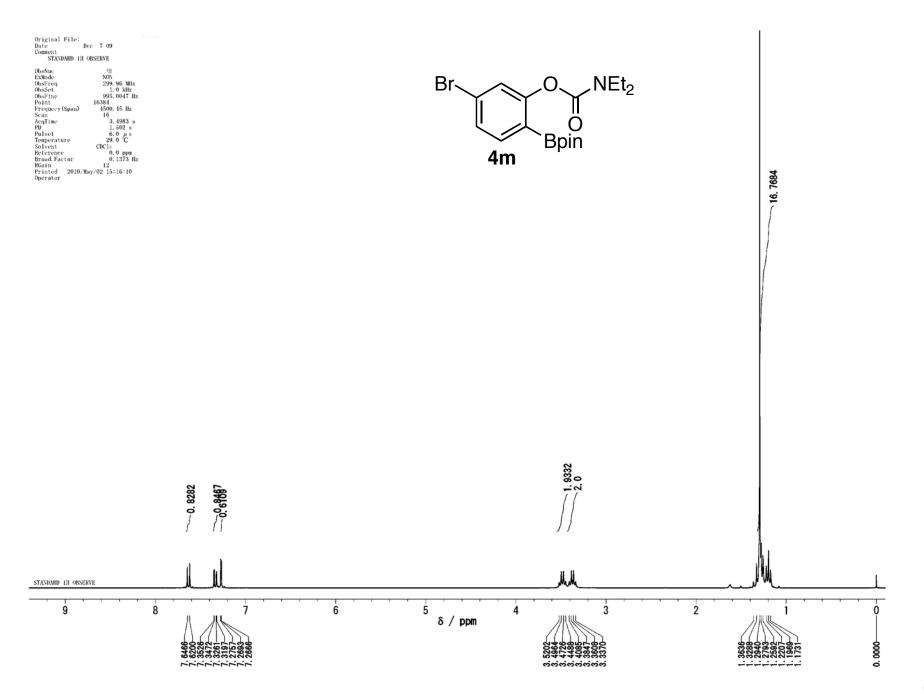


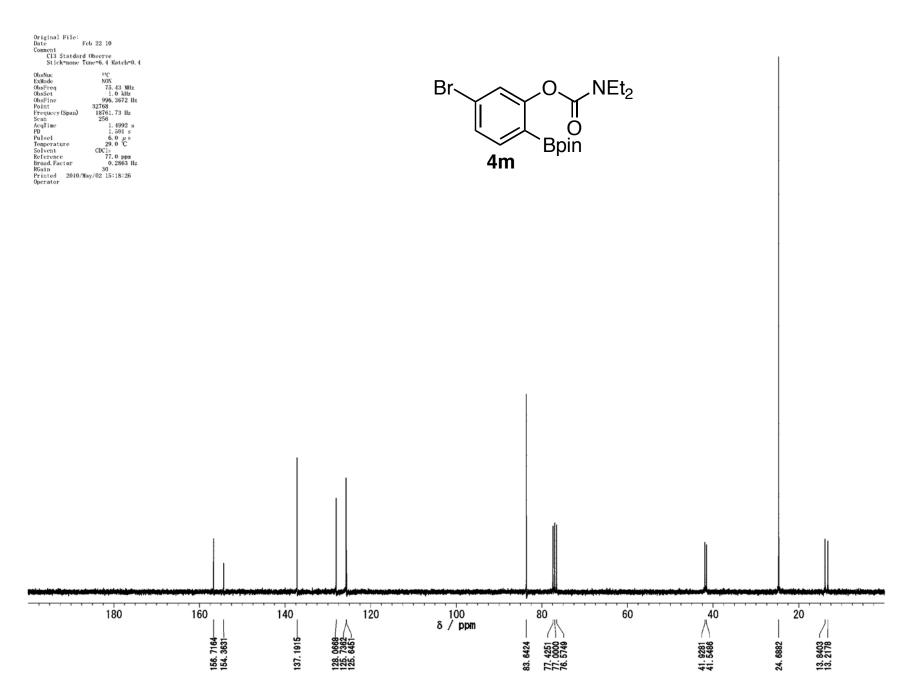


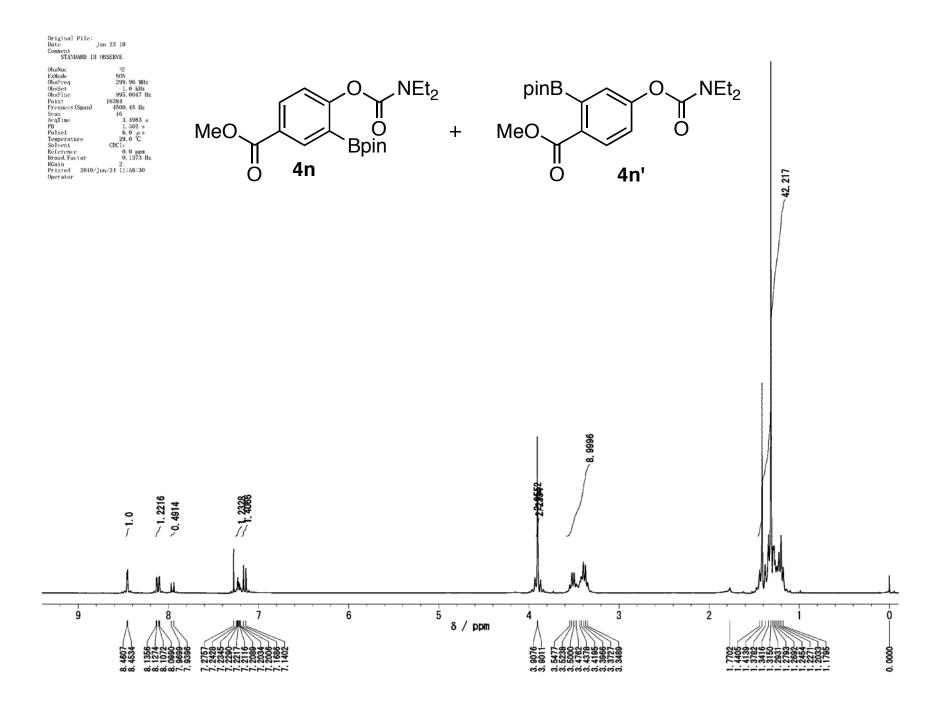


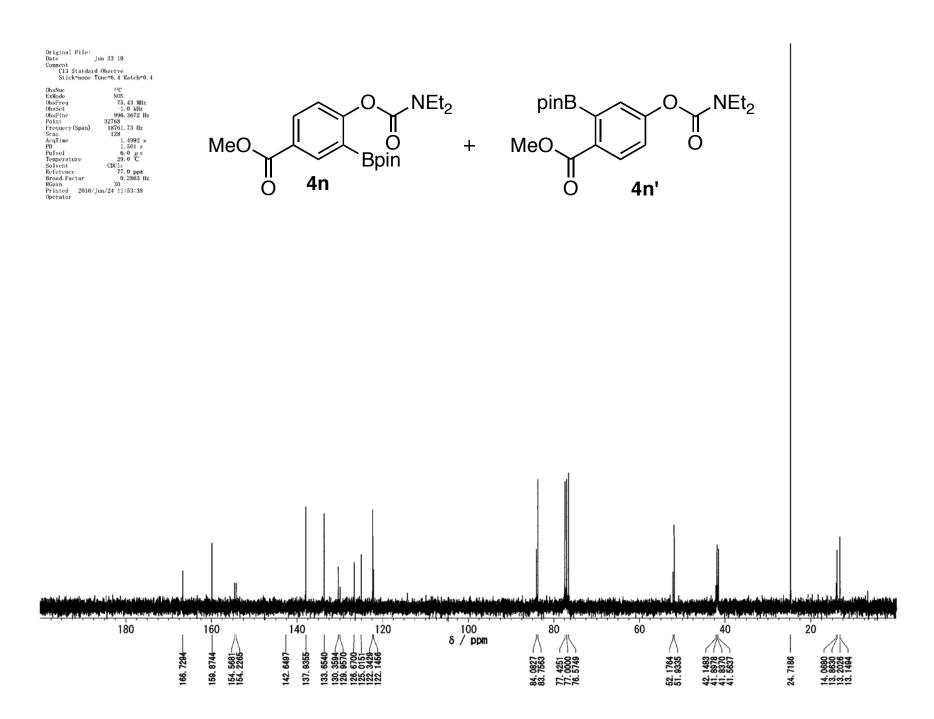


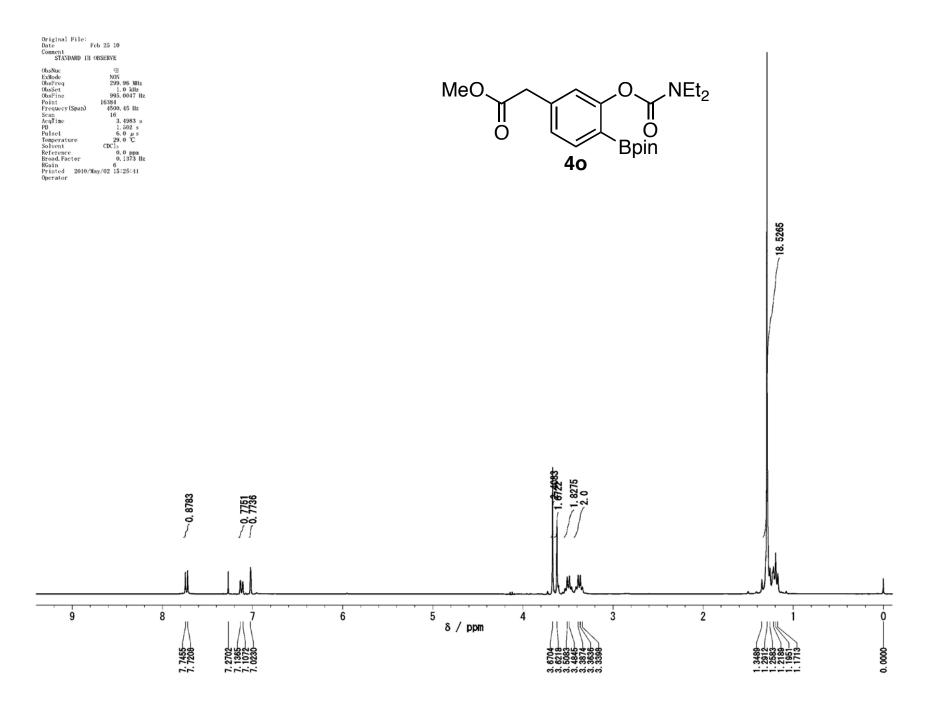


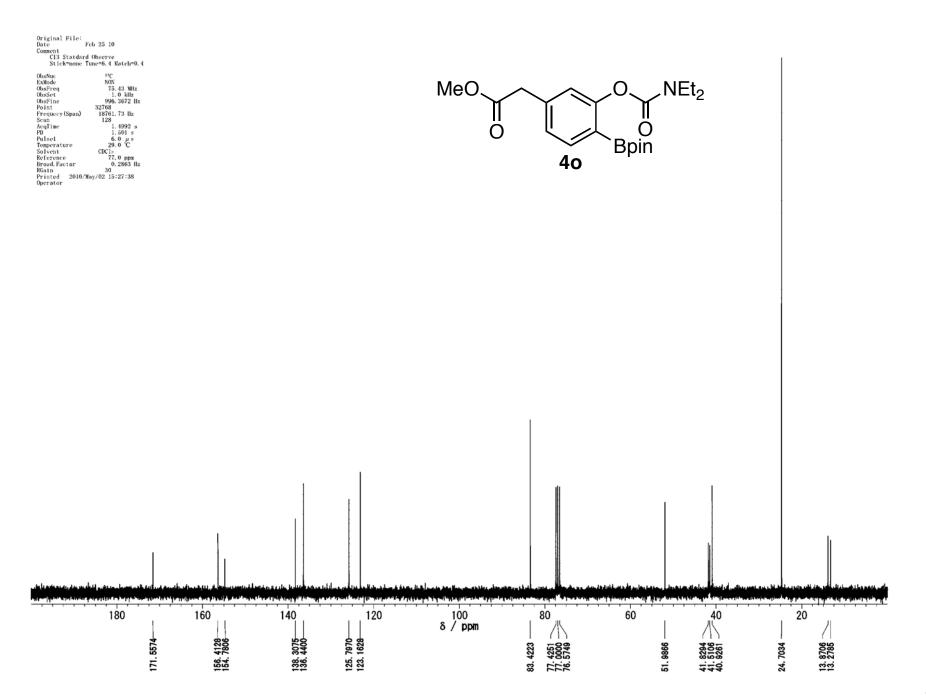


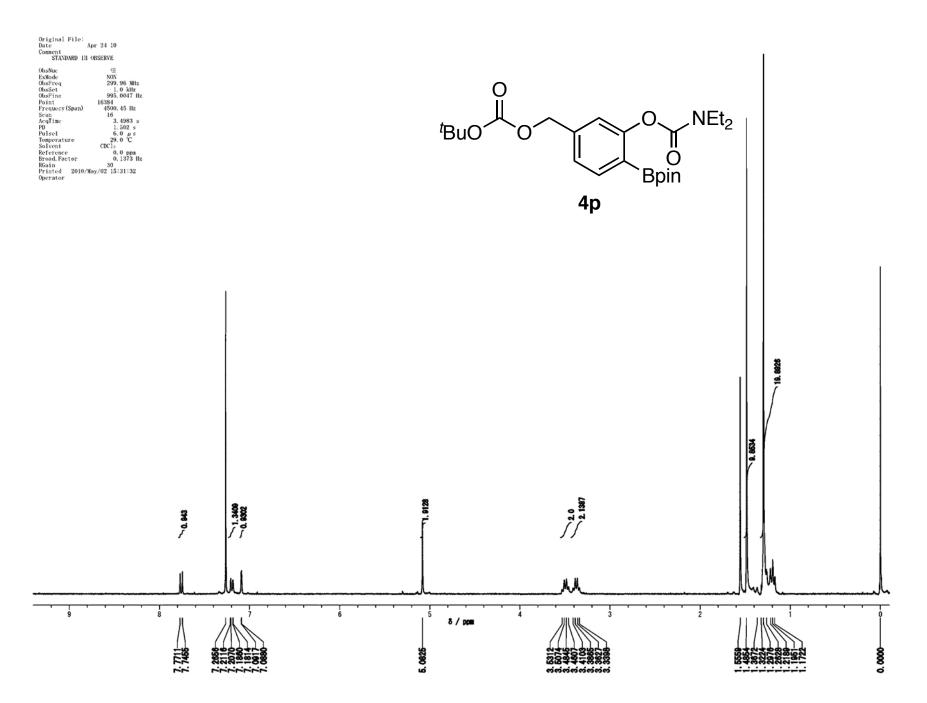


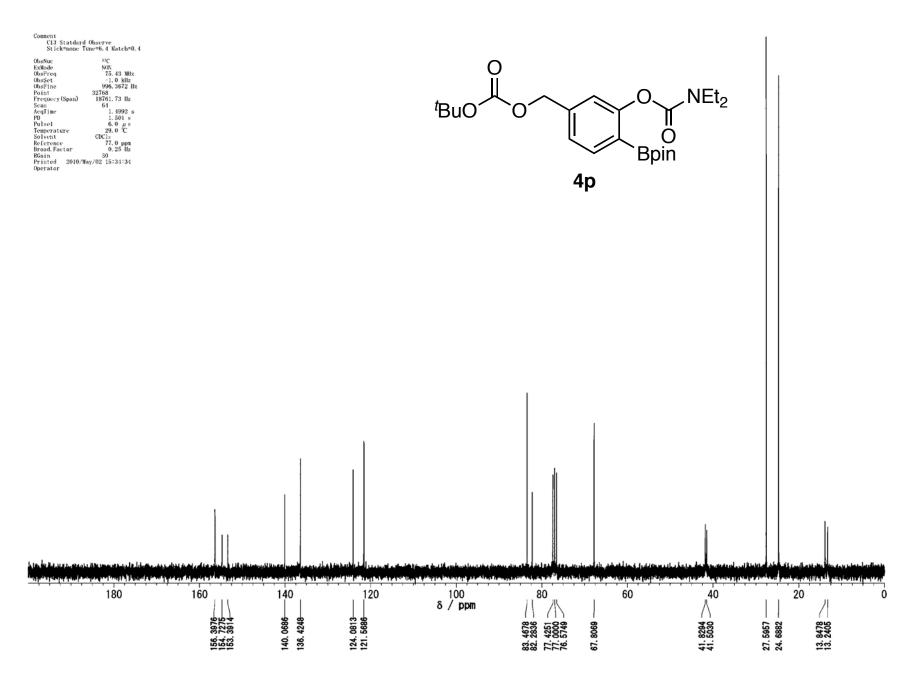


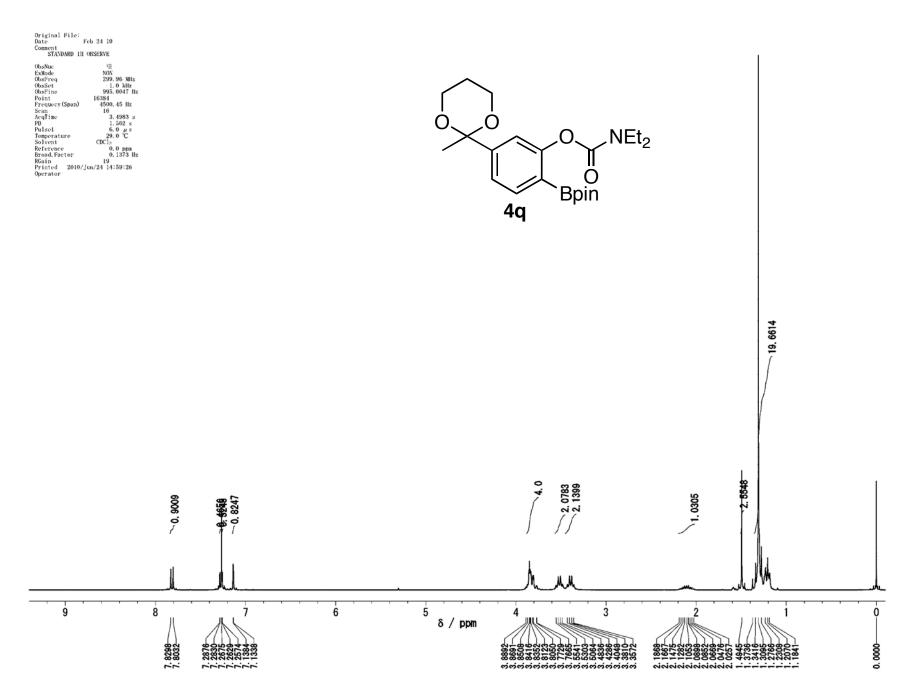


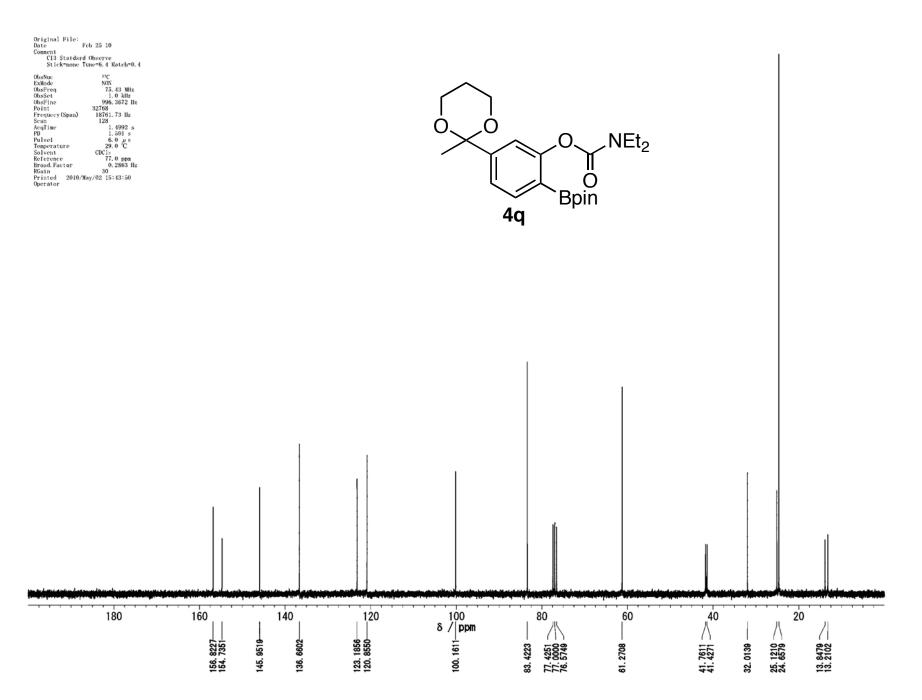


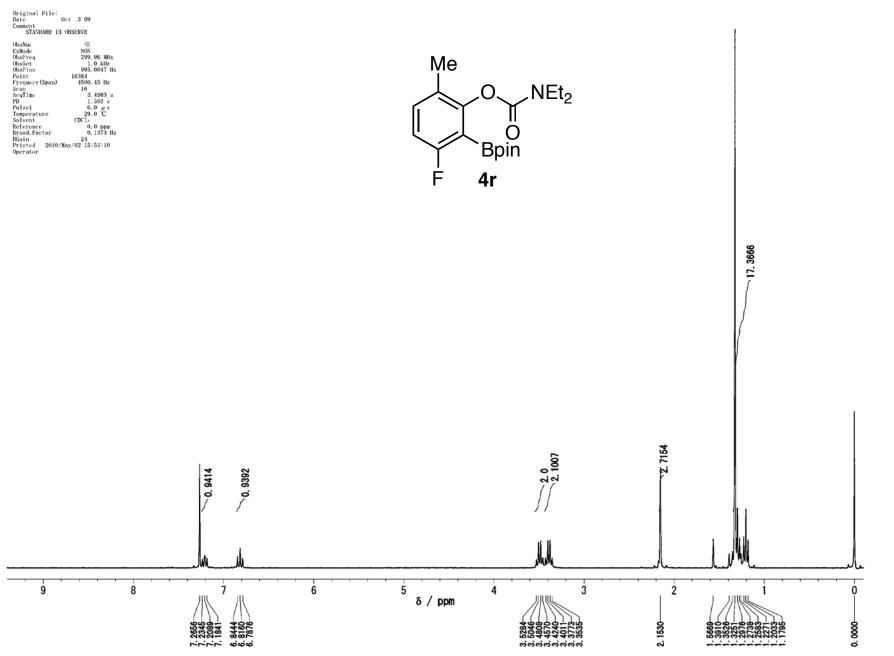


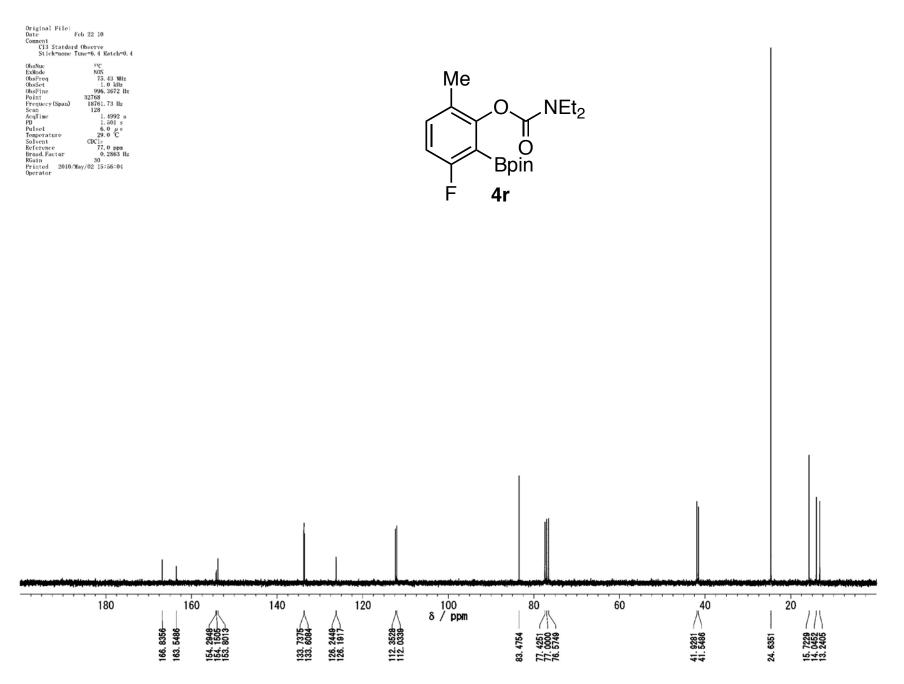


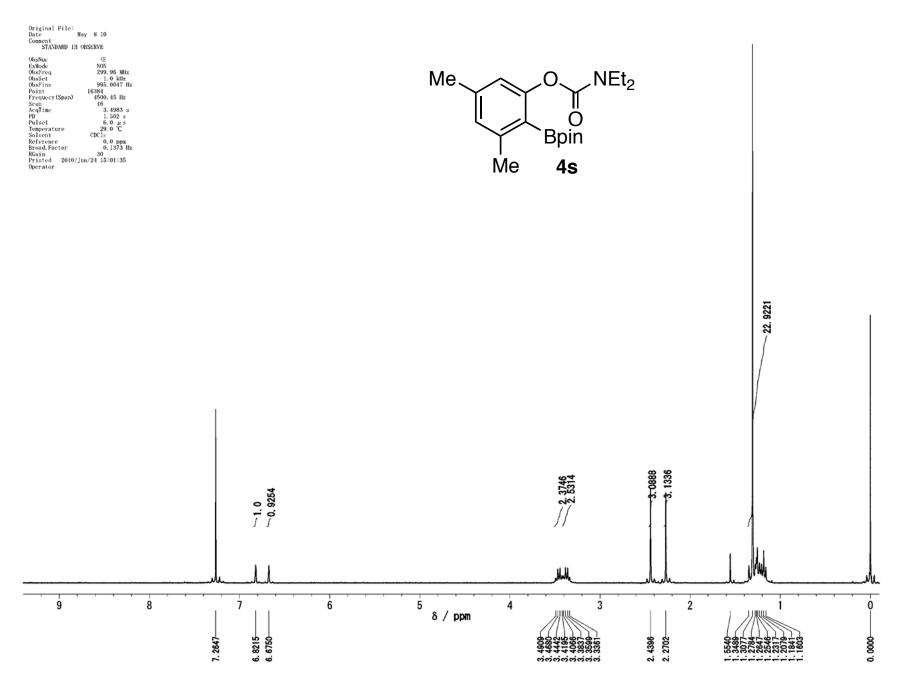


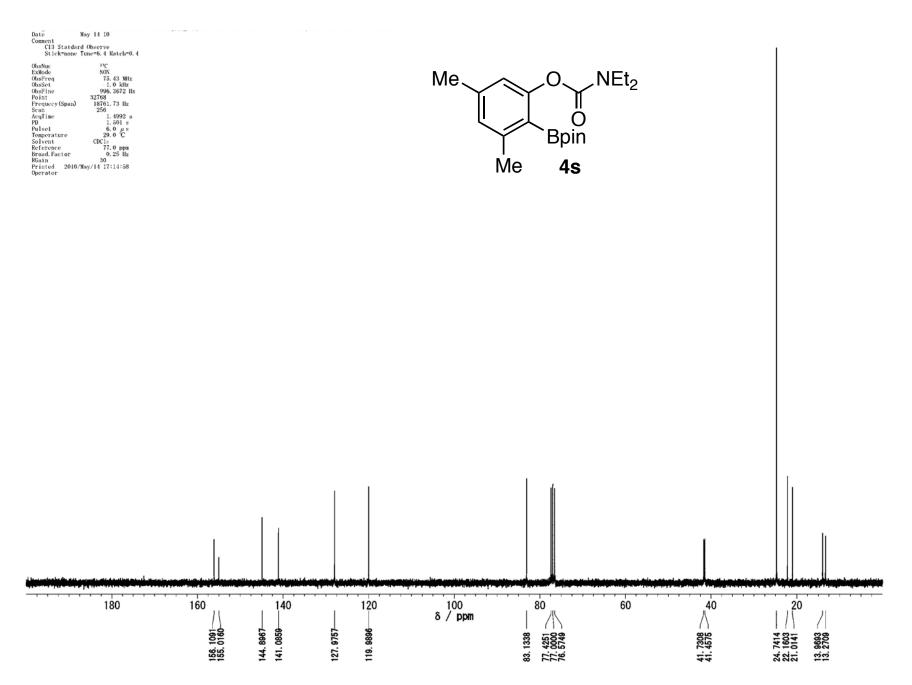


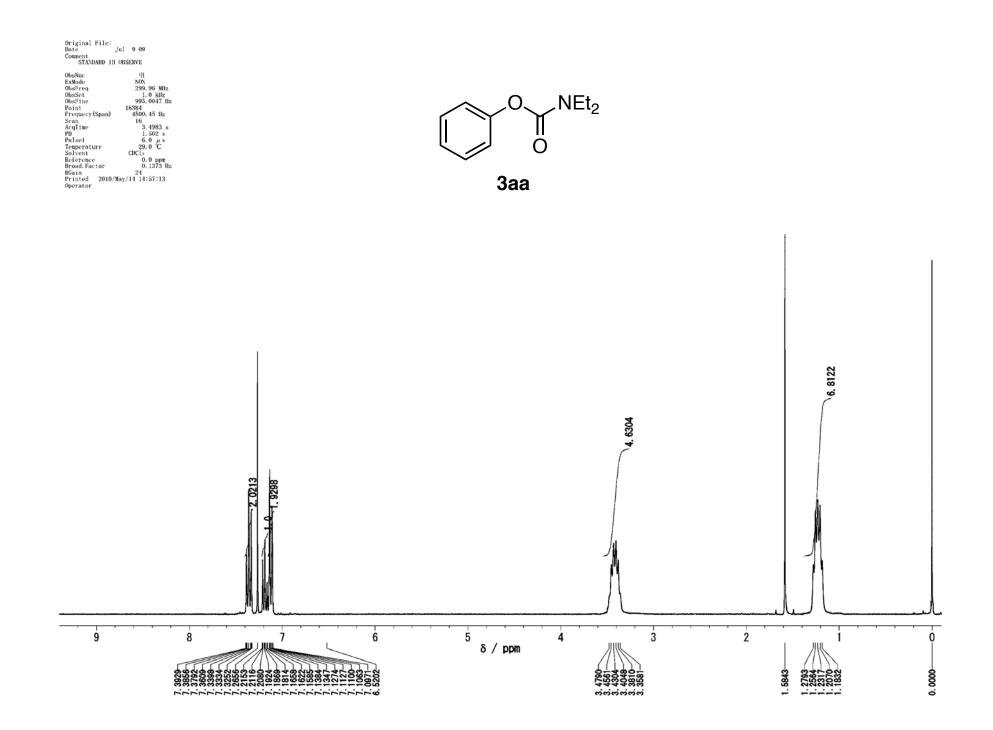




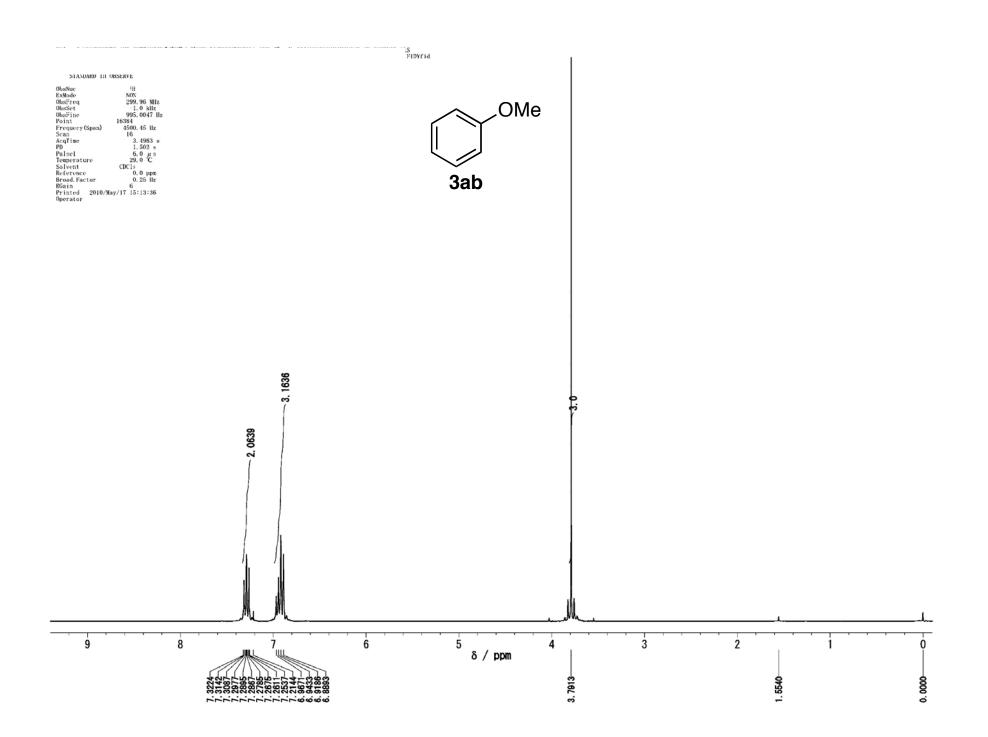


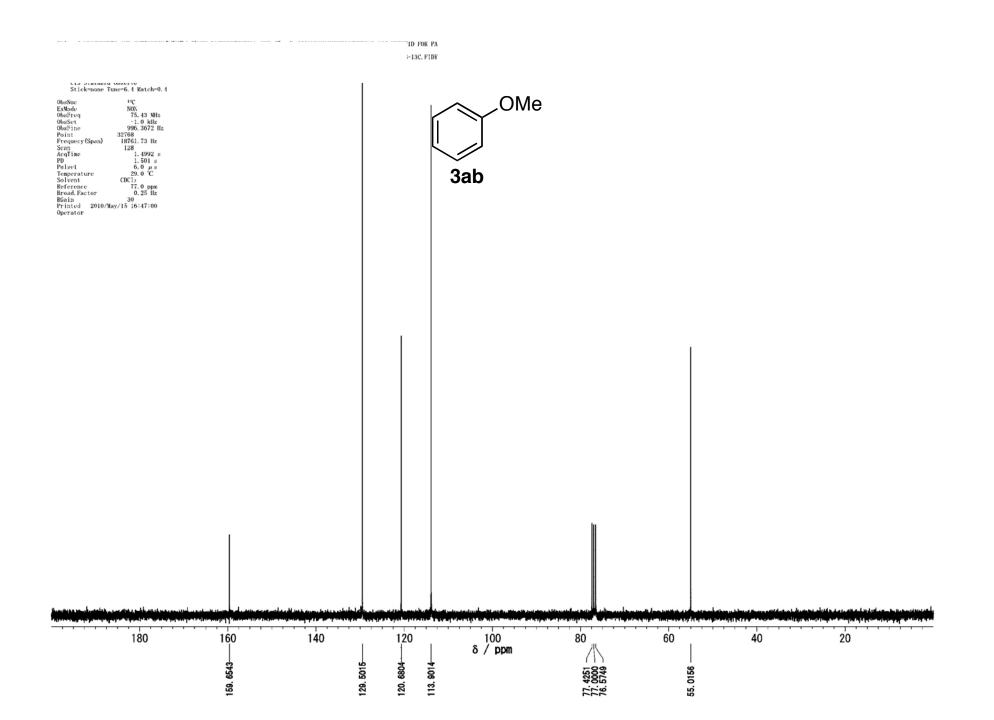


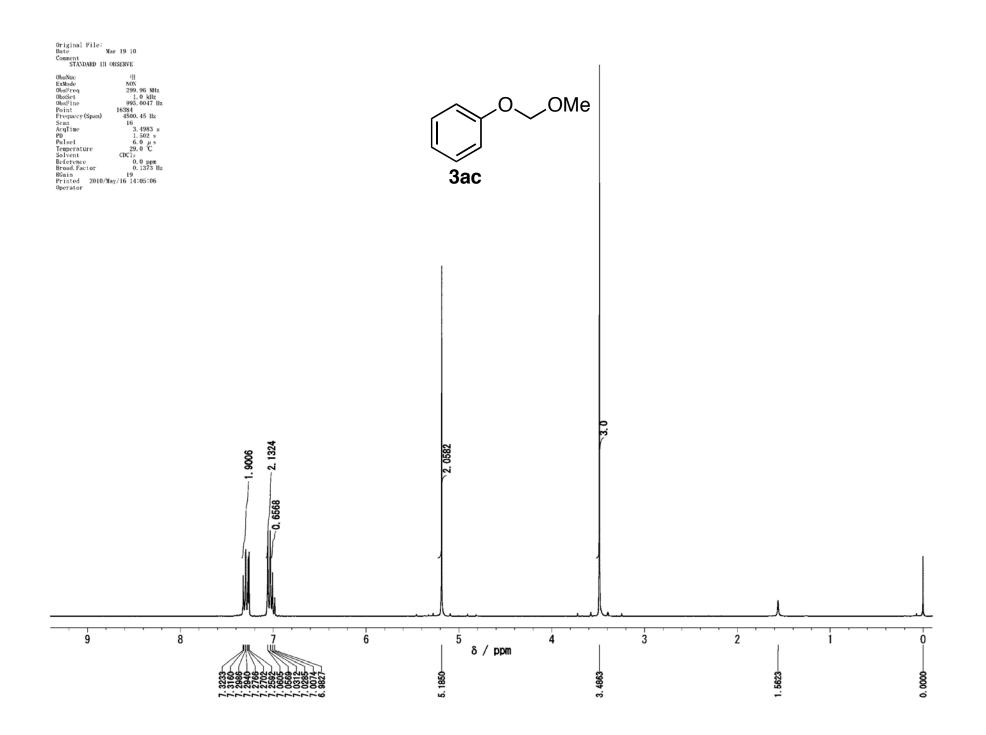


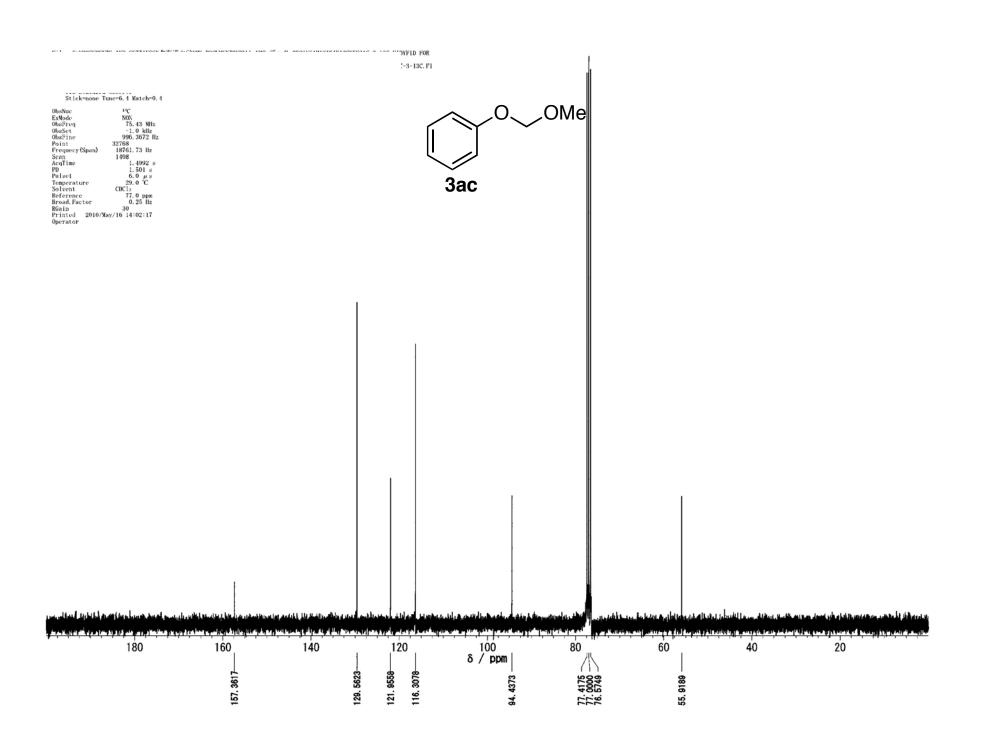


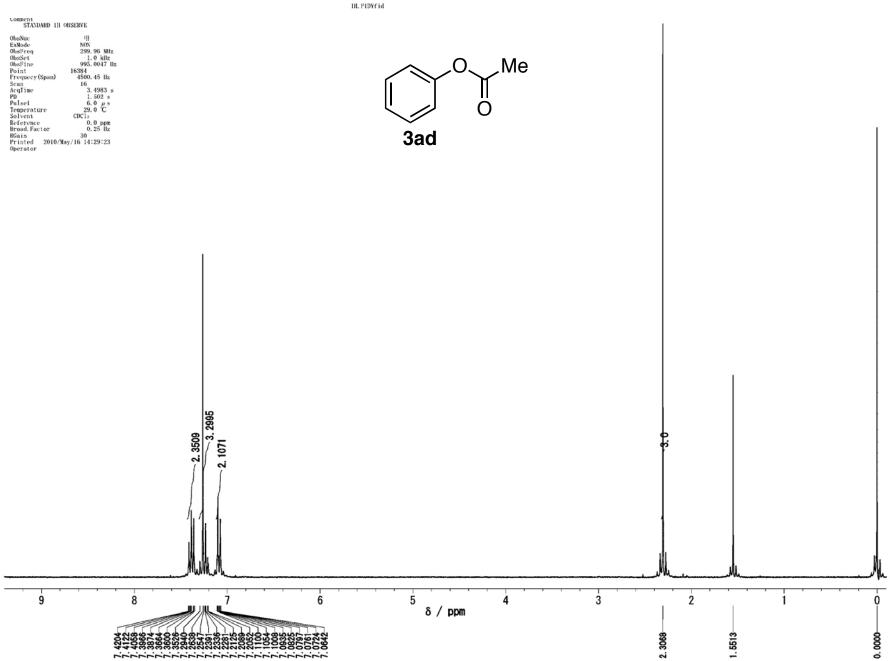
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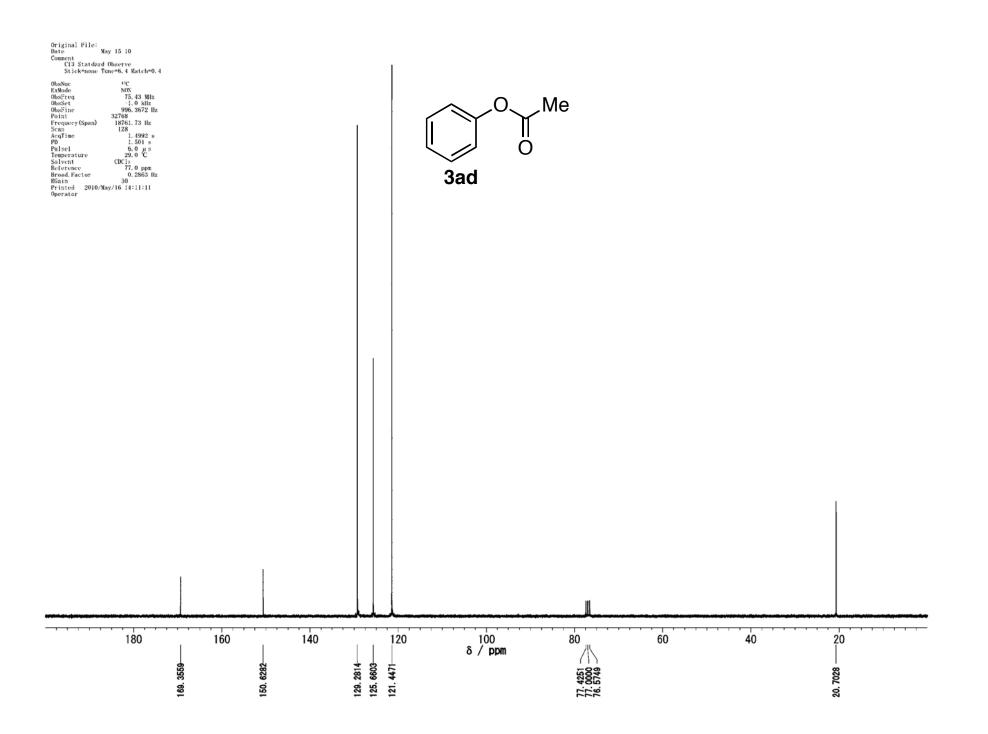


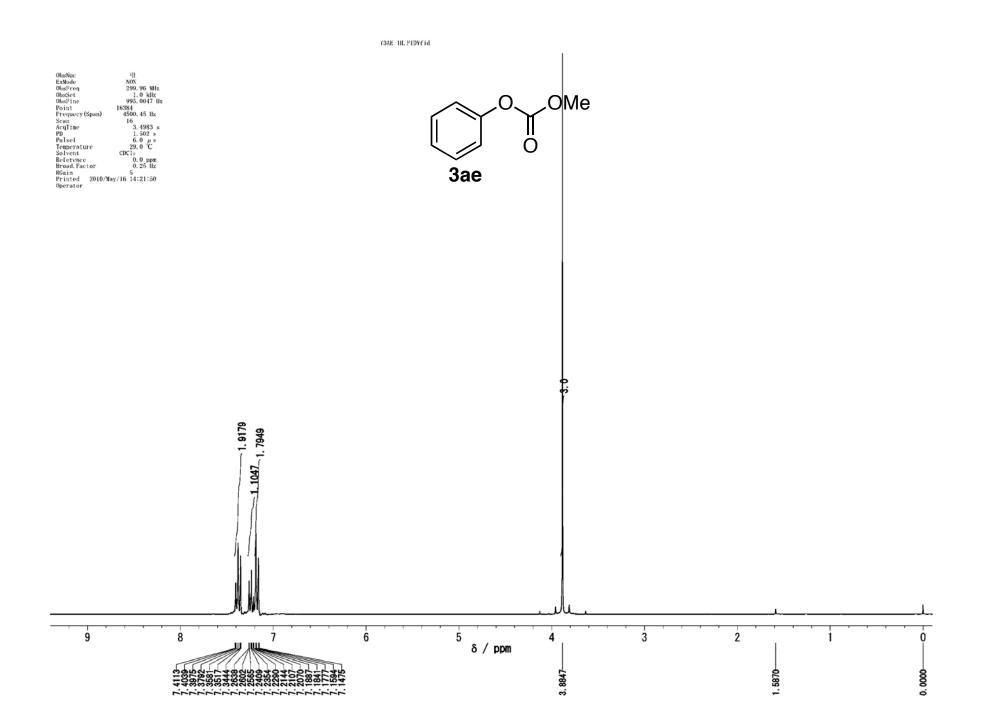


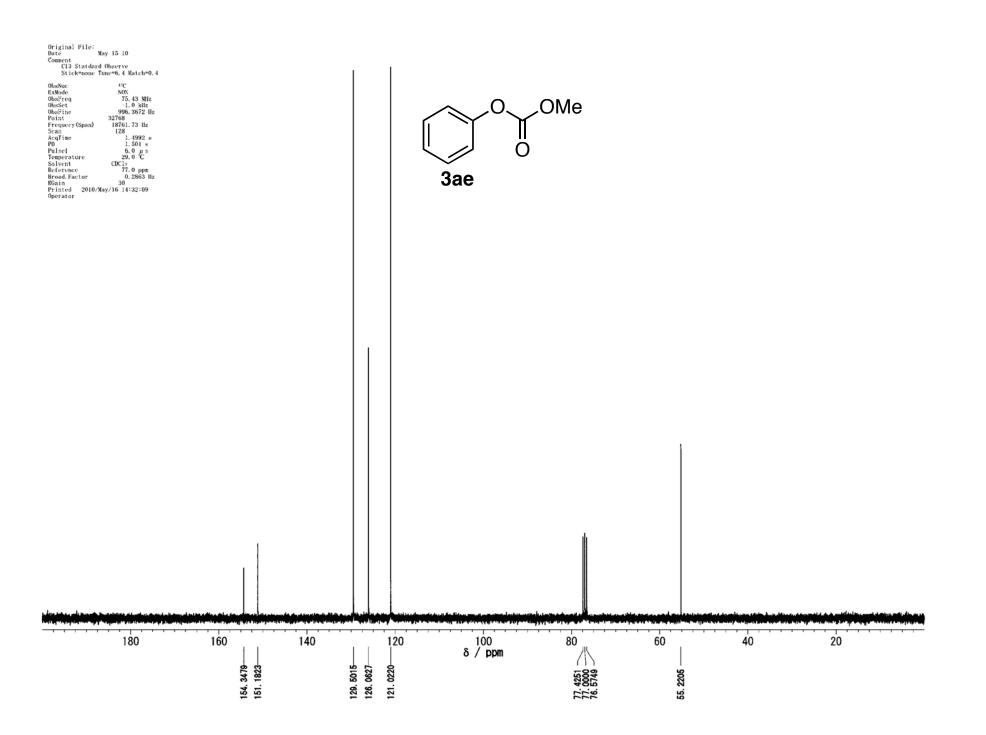


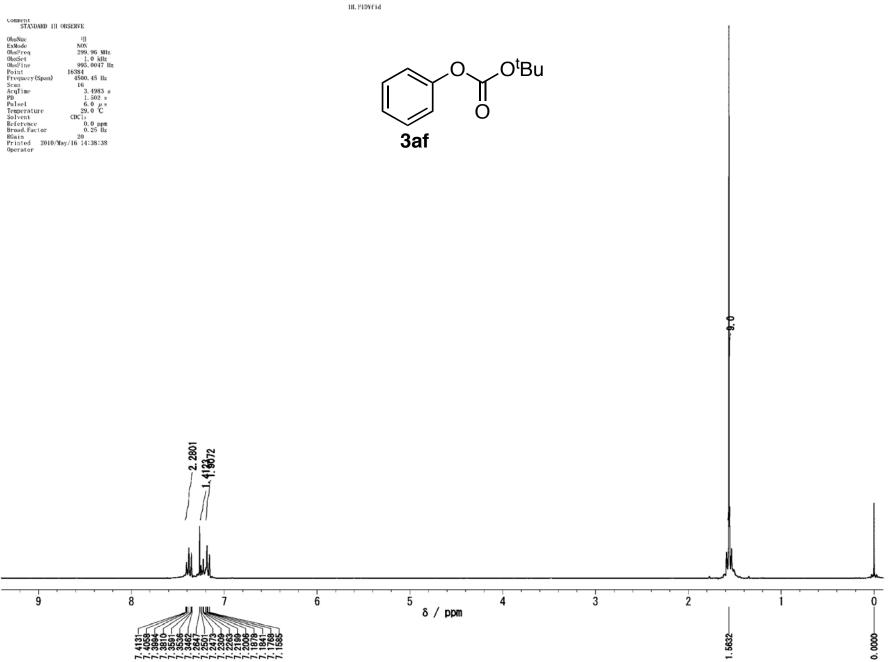
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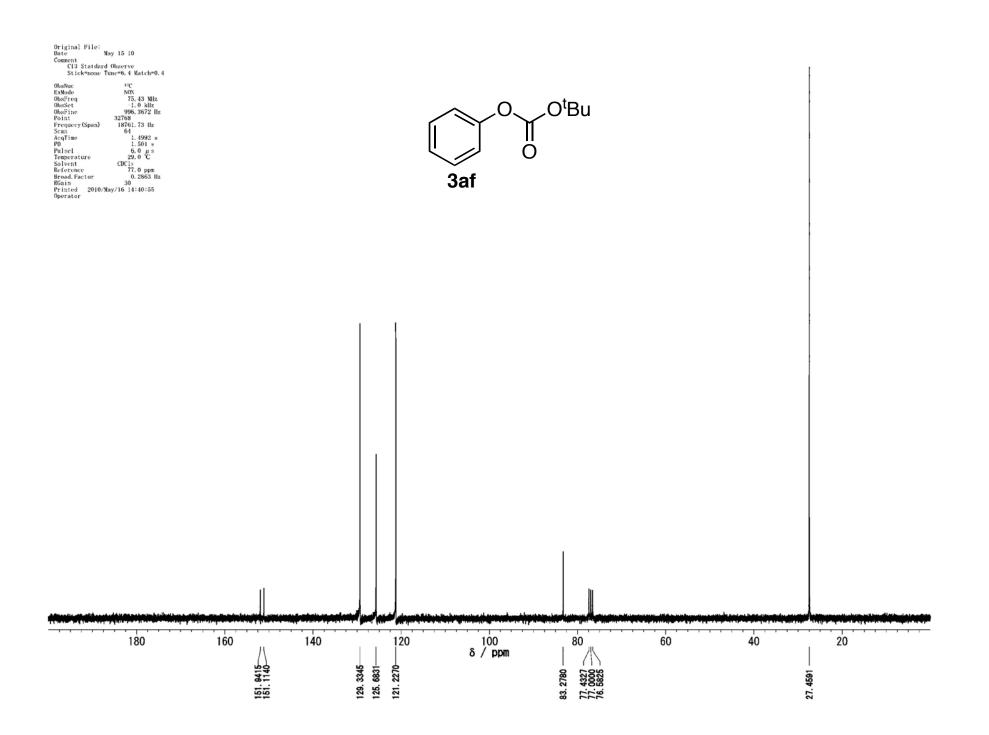
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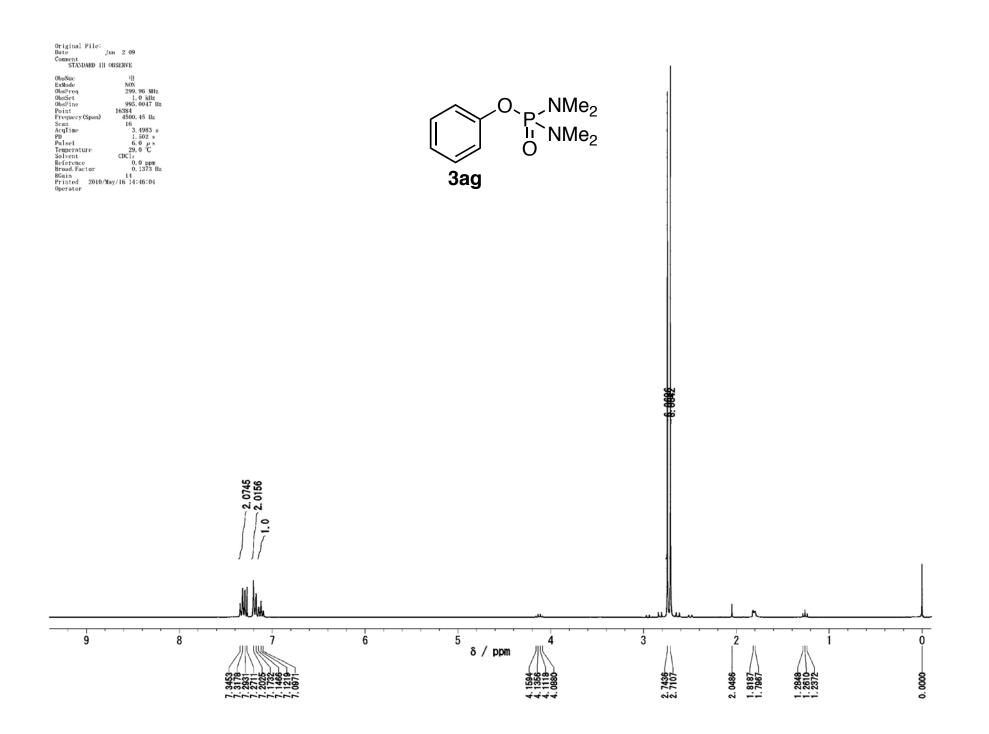


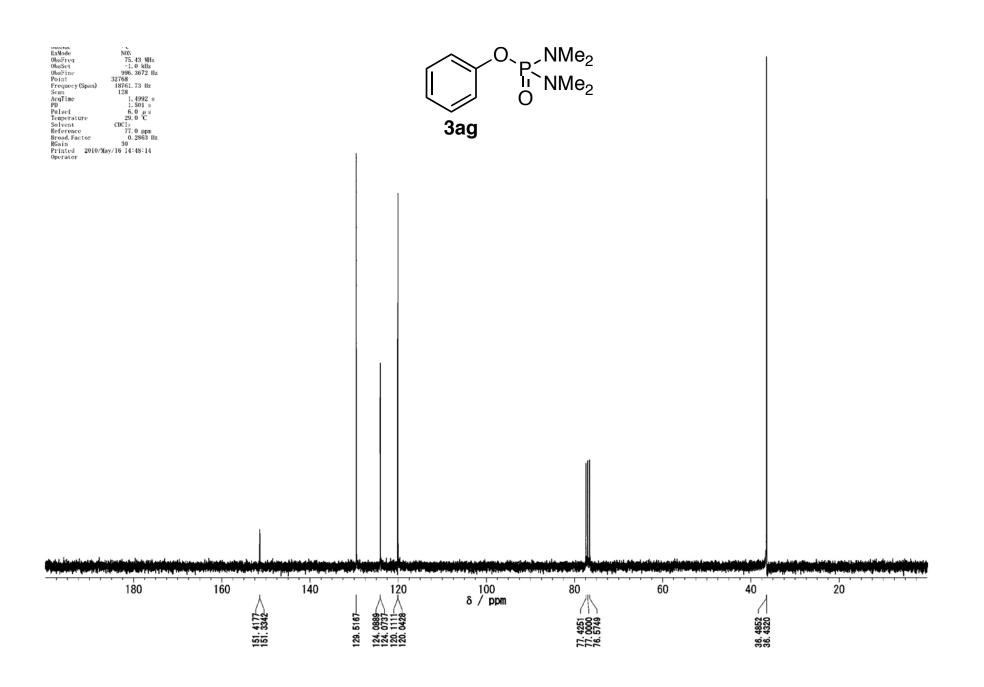


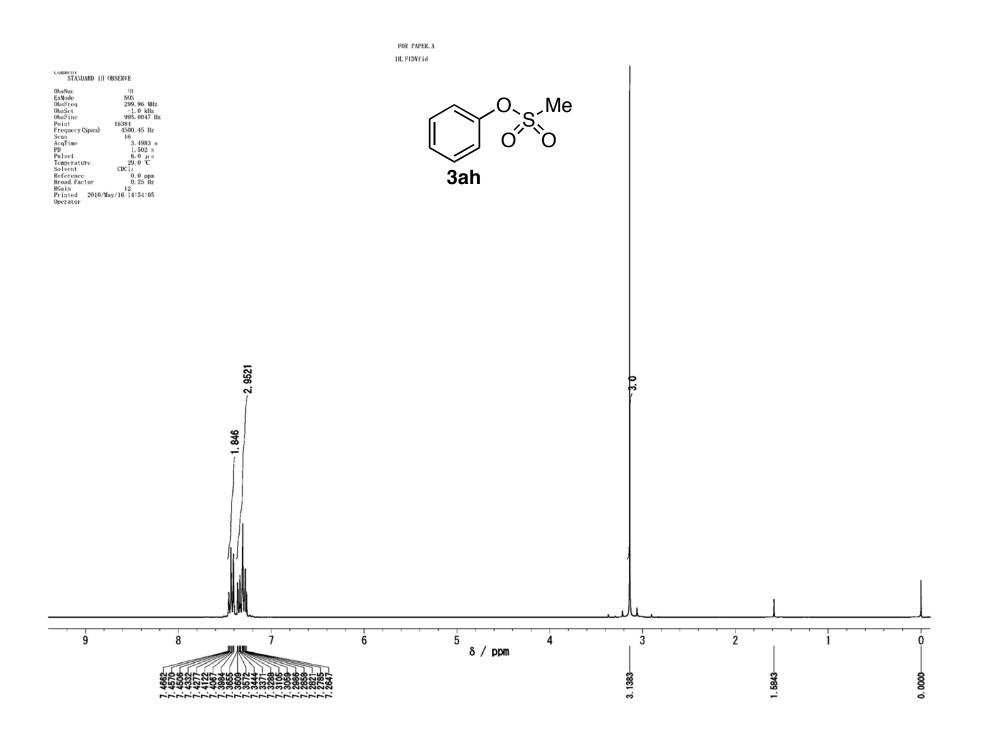


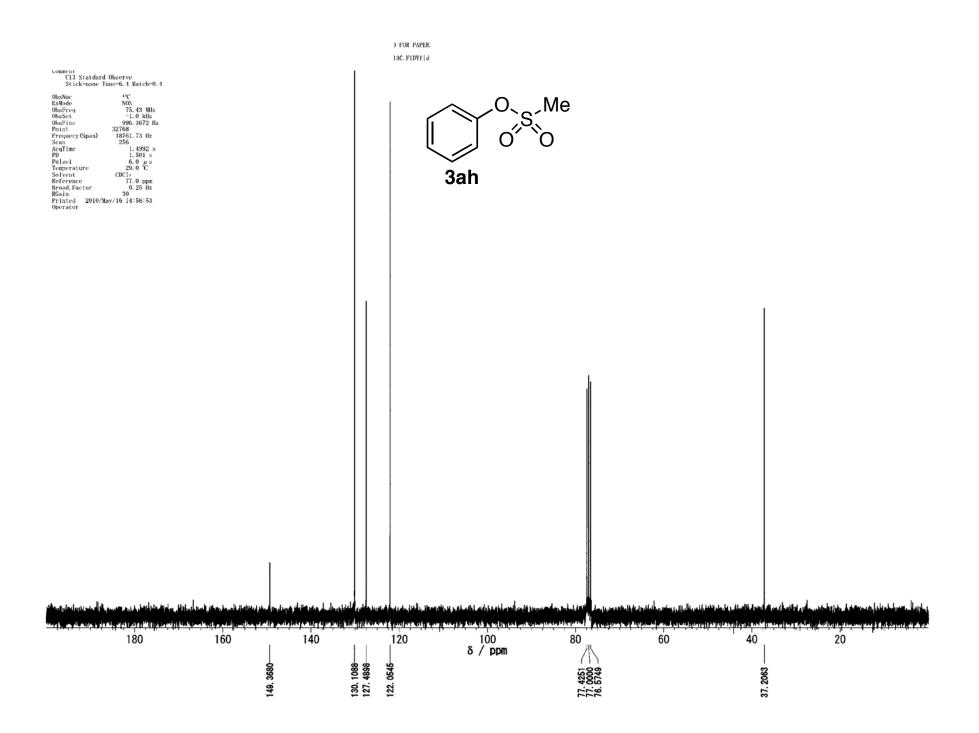


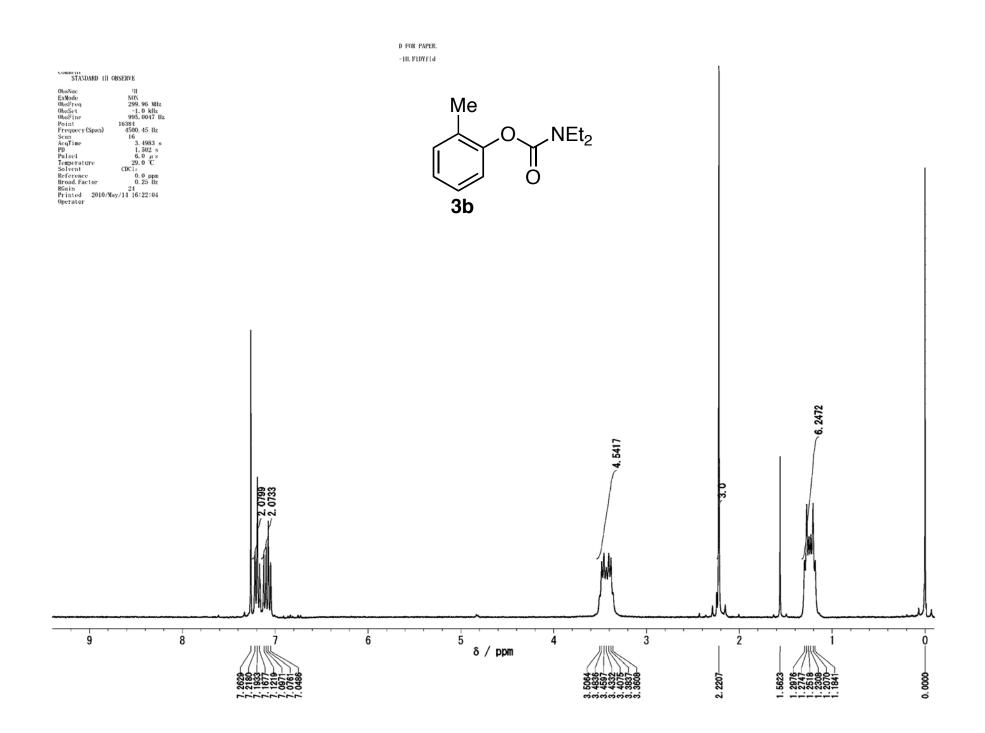


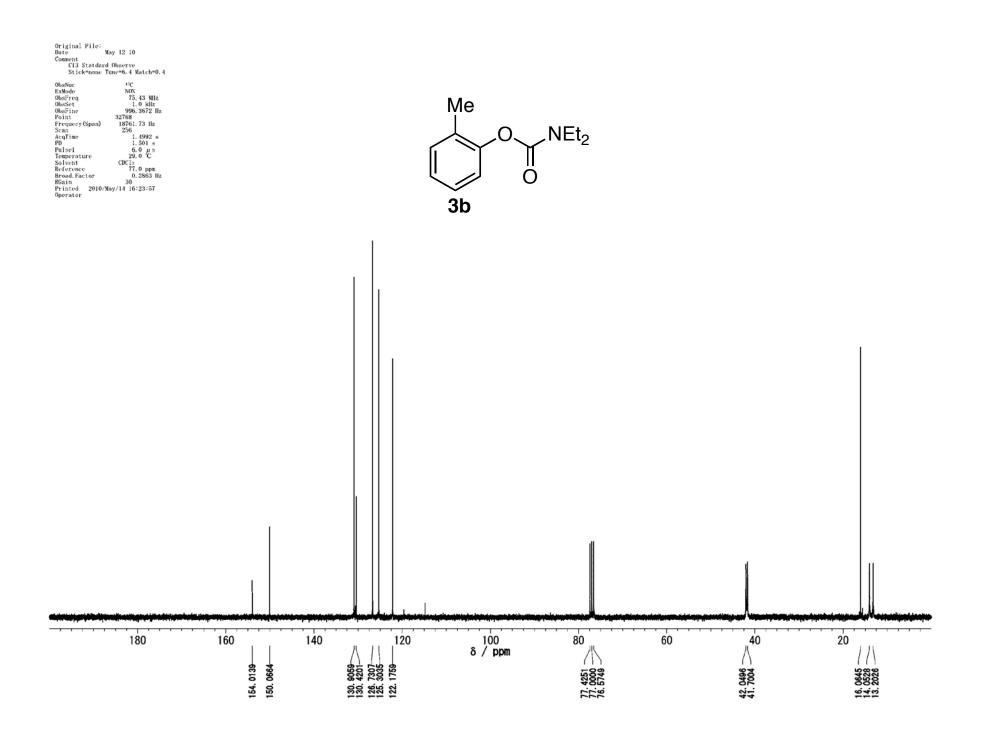


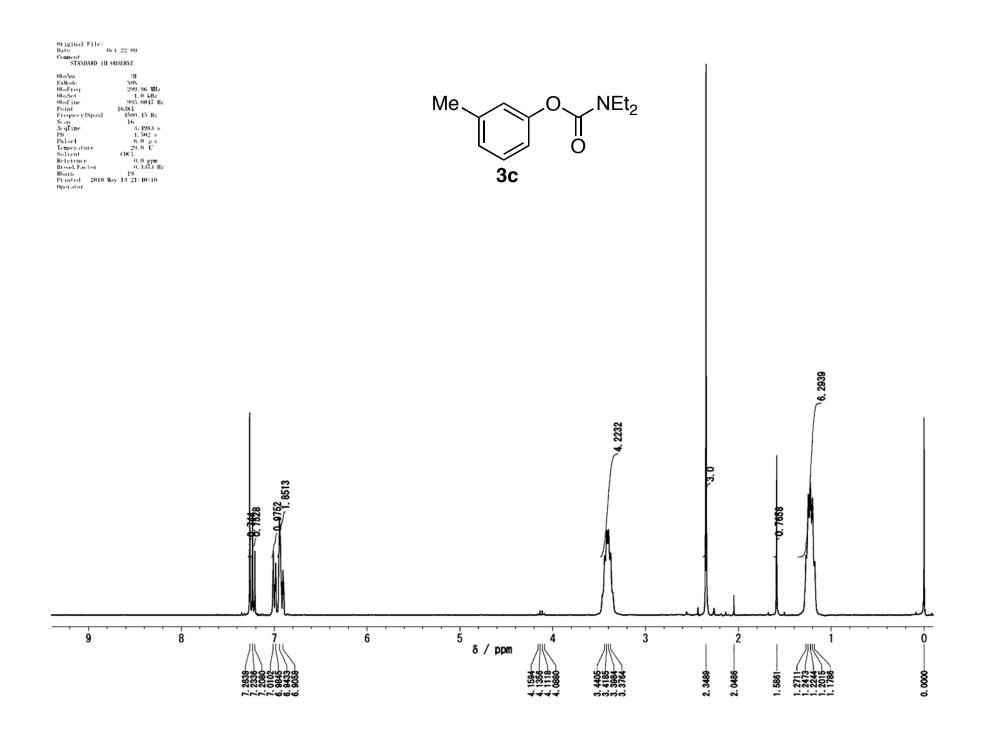


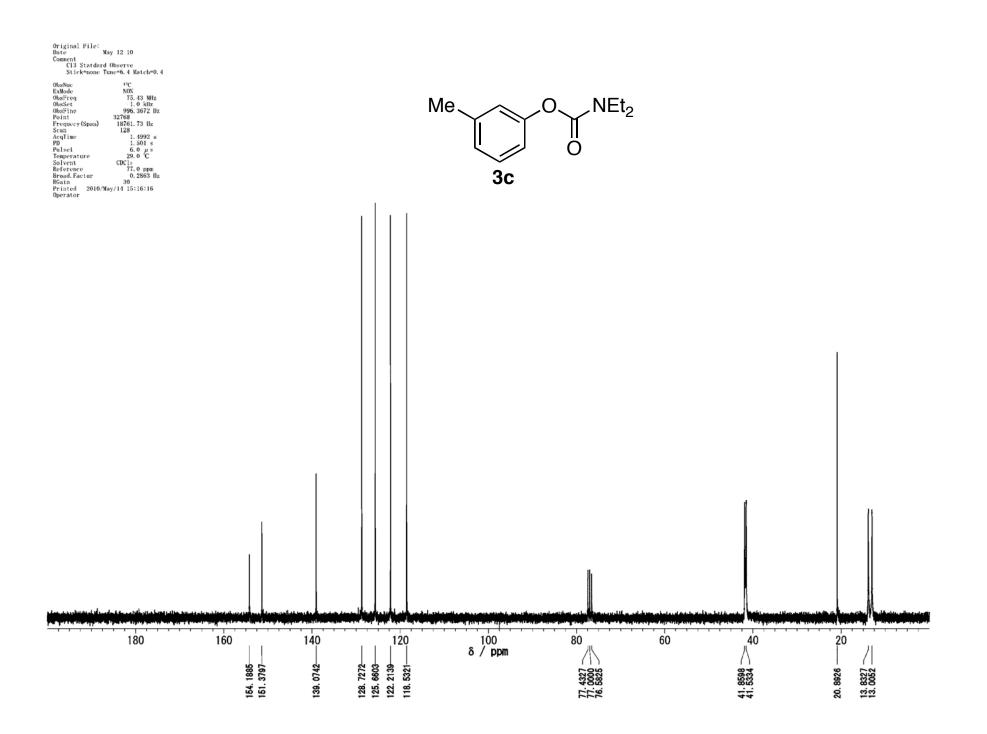


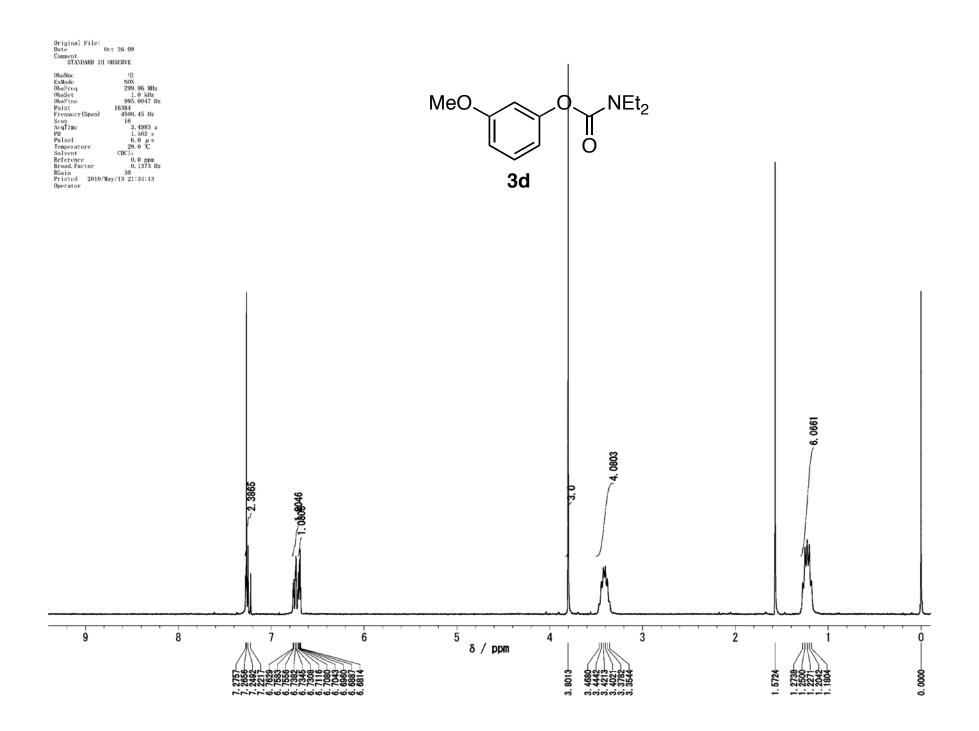


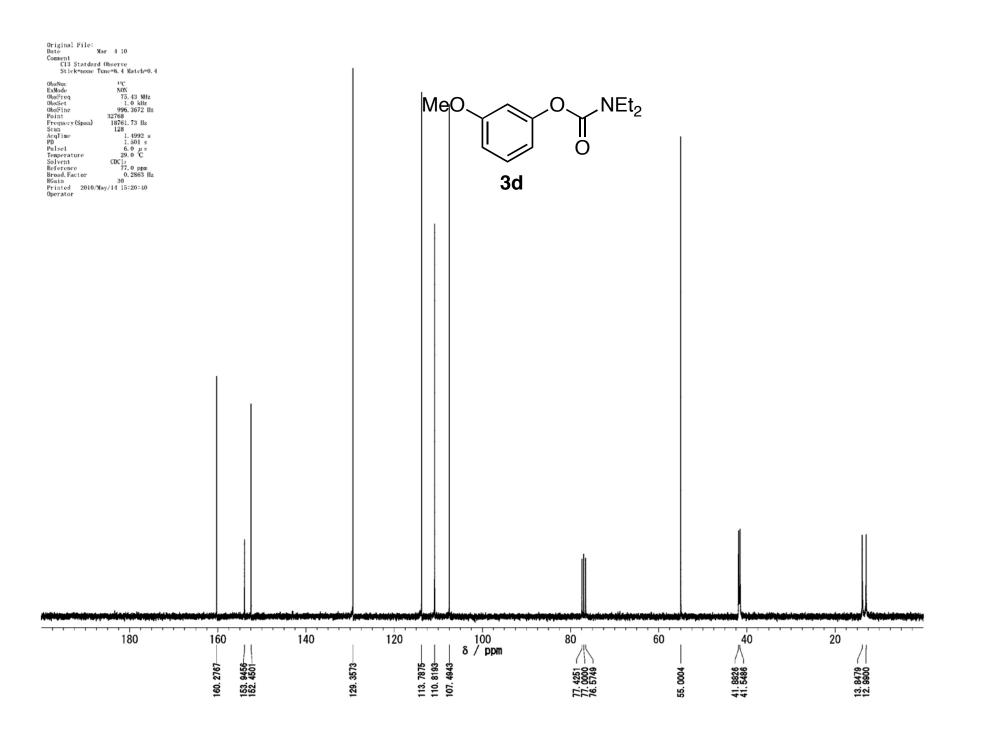


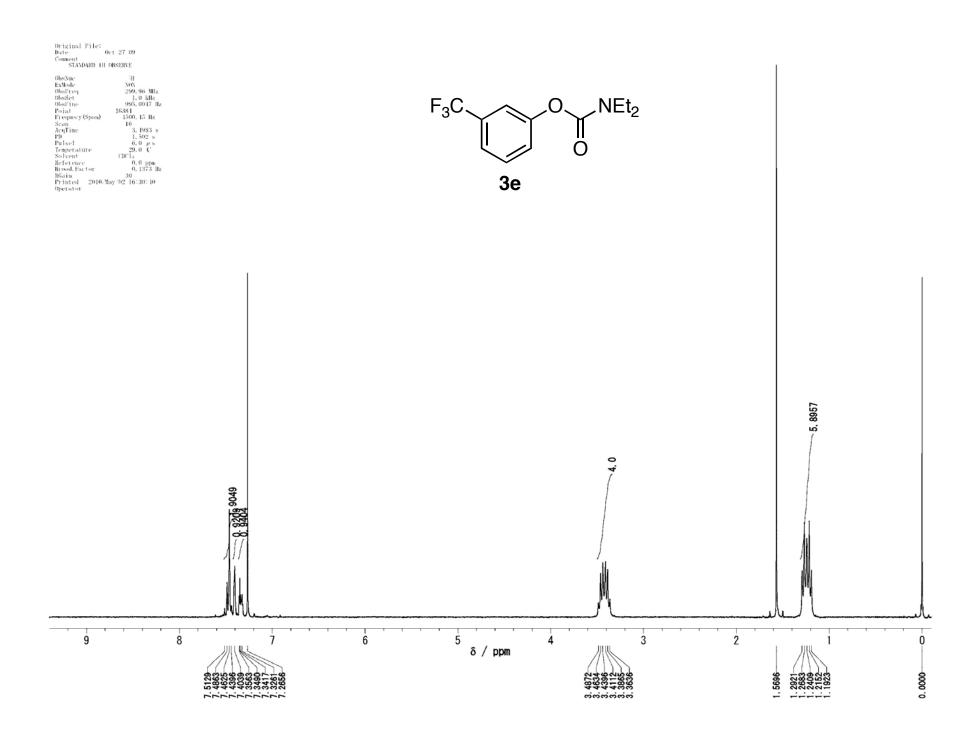


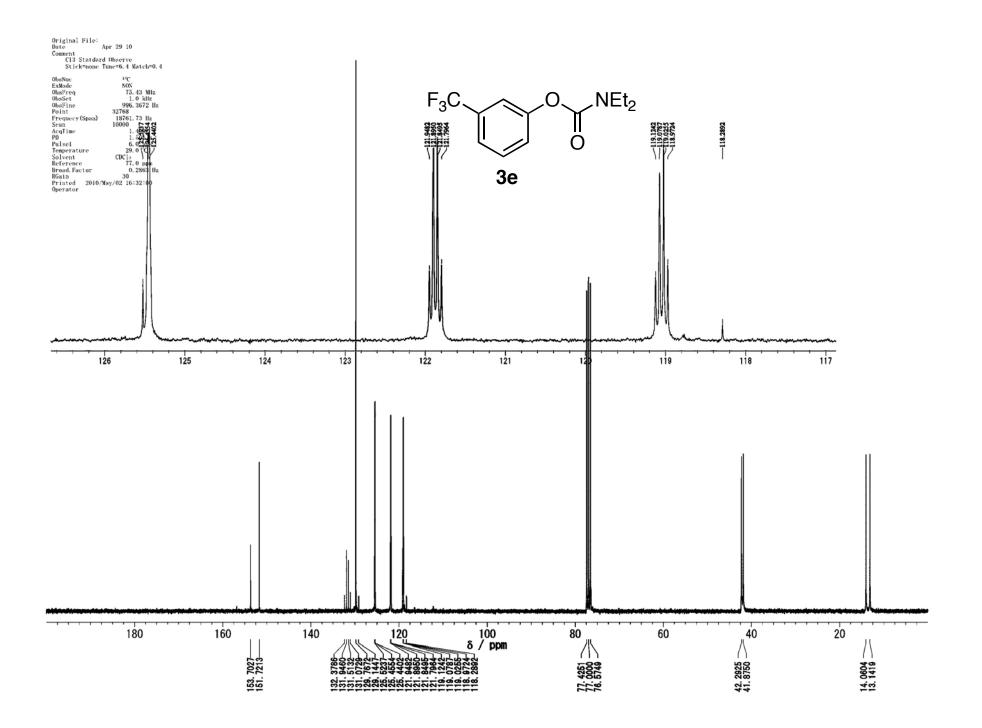


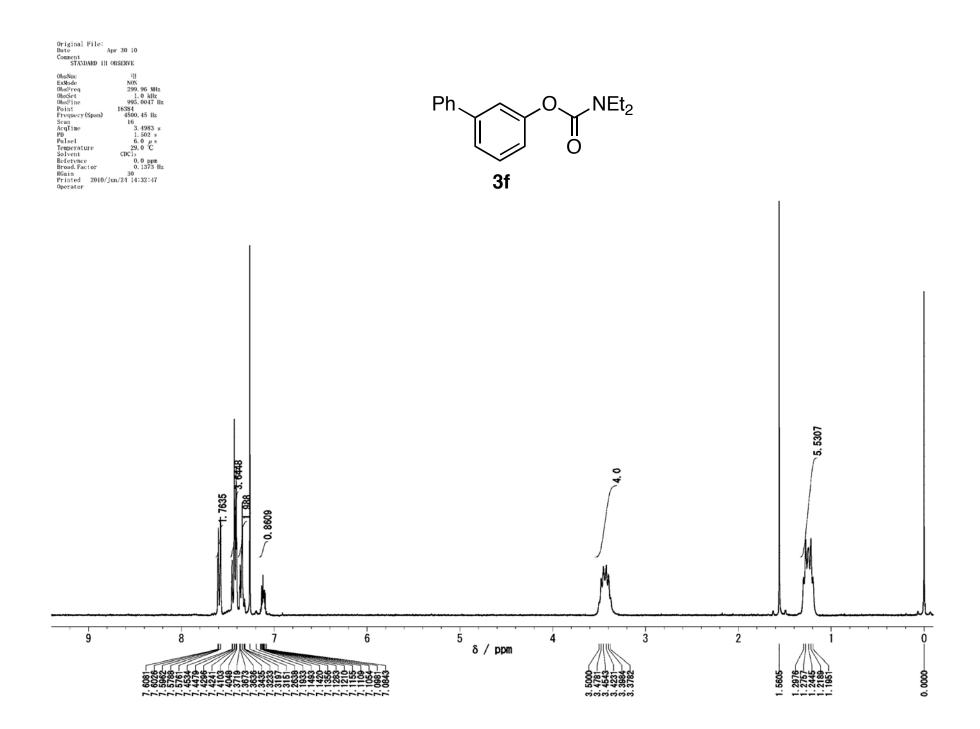


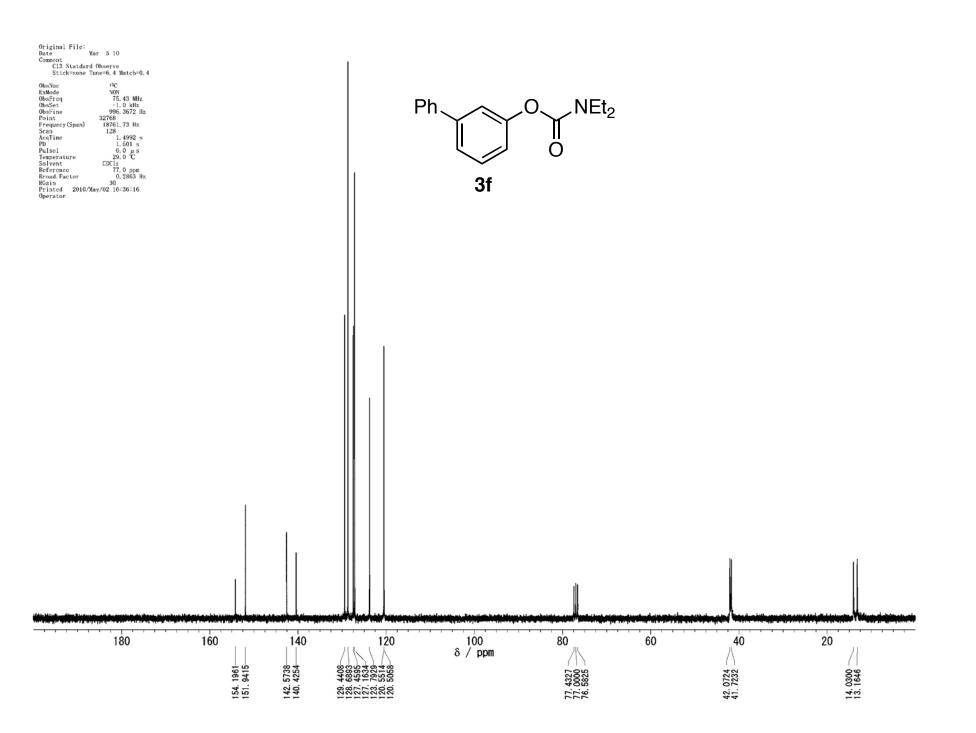




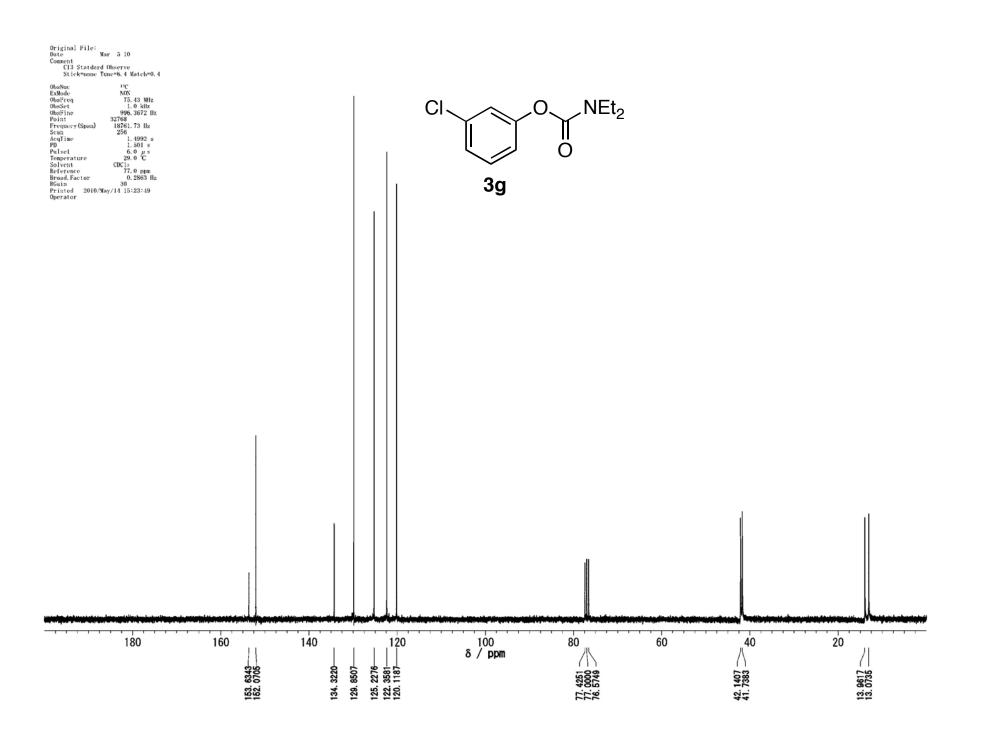




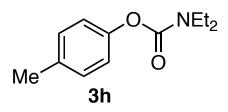


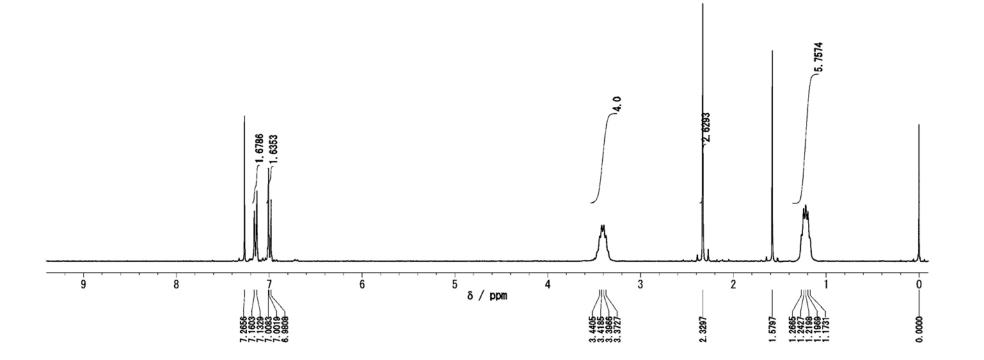


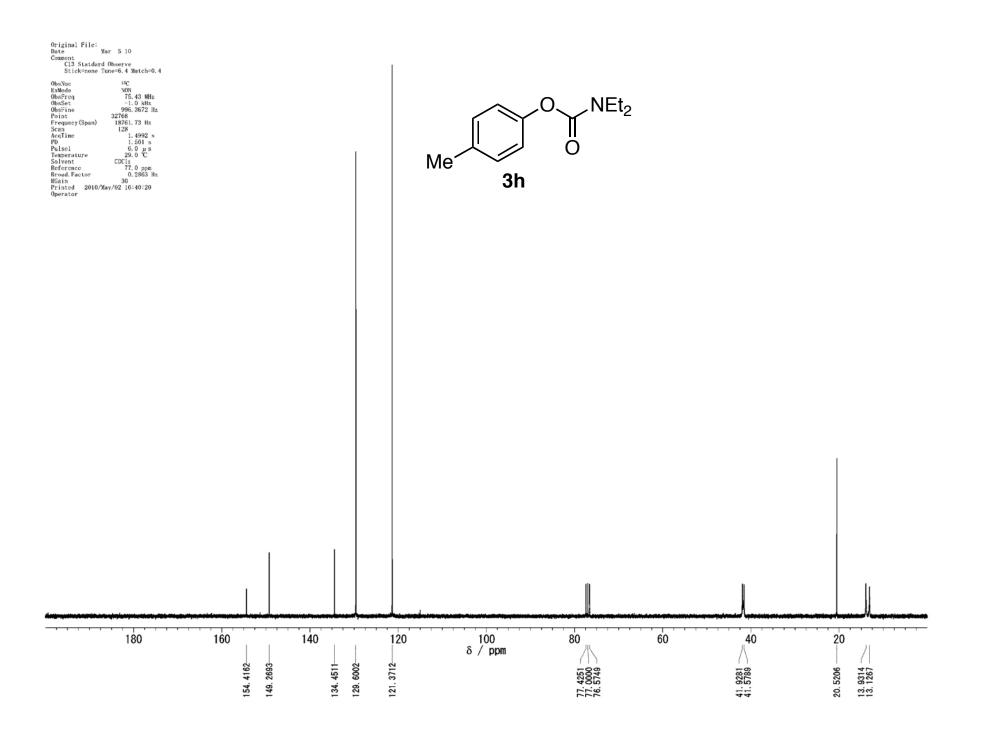
$\begin{array}{c} \text{Comment} \\ \text{STADDARD III OBSERVE} \\ \hline \\ \text{ObsNuc} & \text{III} \\ \text{ExMode} & \text{MUE} \\ \text{GbsNuc} & 299, 96 & \text{MHz} \\ \text{ObsSer} & 299, 96 & \text{MHz} \\ \text{ObsSer} & 0.10 & \text{MHz} \\ \text{ObsNuc} & 16334 & \text{Od-17 IIIz} \\ \text{ObsNuc} & 16334 & \text{Od-17 IIIz} \\ \text{Point} & 16334 & \text{Od-17 IIIz} \\ \text{Point} & 16334 & \text{Od-17 IIIz} \\ \text{Frequecy}(\text{Span}) & 16334 & \text{Span} \\ \text{Point} & 16334 & \text{Span} \\ \text{AcqTime} & 364983 & \text{s} \\ \text{Point} & 16334 & \text{Od-17 IIIz} \\ \text{Scan} & 16 & \text{Od-18 IIIz} \\ \text{Scan} & 16 & \text{Od-18 IIIz} \\ \text{Scan} & 16 & \text{Od-18 IIIz} \\ \text{Point} & 16334 & \text{Od-18 IIIz} \\ \text{Point} & 2900 & \text{C} \\ \text{Solvent} & \text{CDC1s} \\ \text{Reference} & 0.0 & \text{pm} \\ \text{Broad, Factor} & 0.1373 & \text{Hz} \\ \text{KGain} & 2010/May/13 & 21:57:58 \\ \text{Operator} \end{array}$	CI O NEt <sub>2</sub> 3g	
		6. 5925
	 4. 4435	
 	5 δ / ppm	2. 0486 2. 0486 1. 15660 1. 12000 0. 0000 0. 0000 0

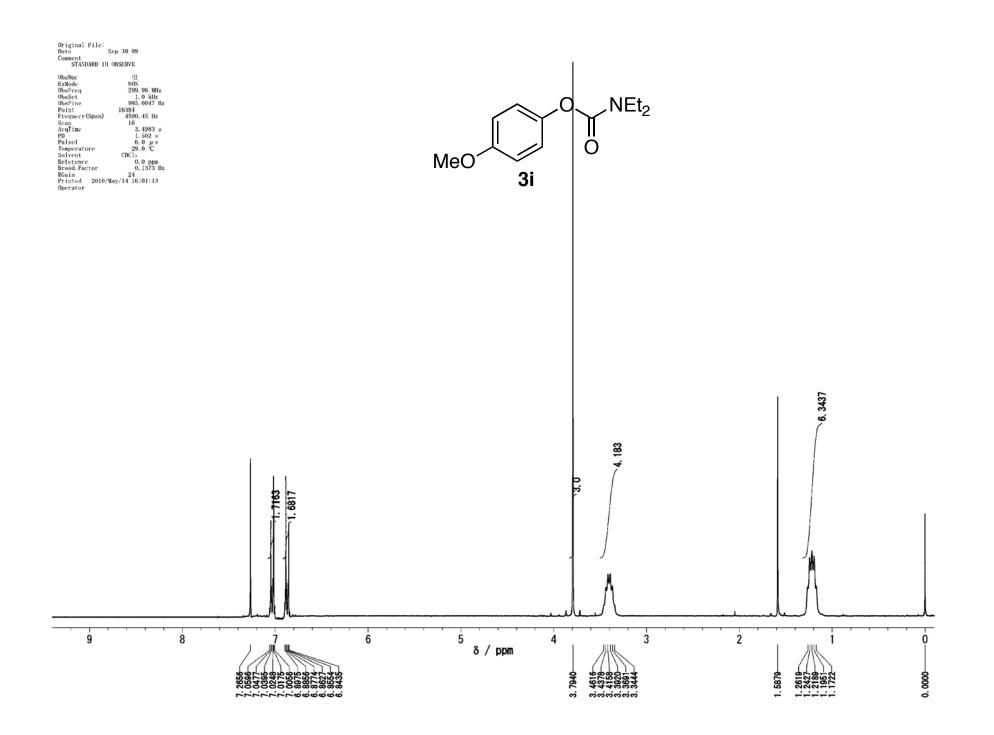


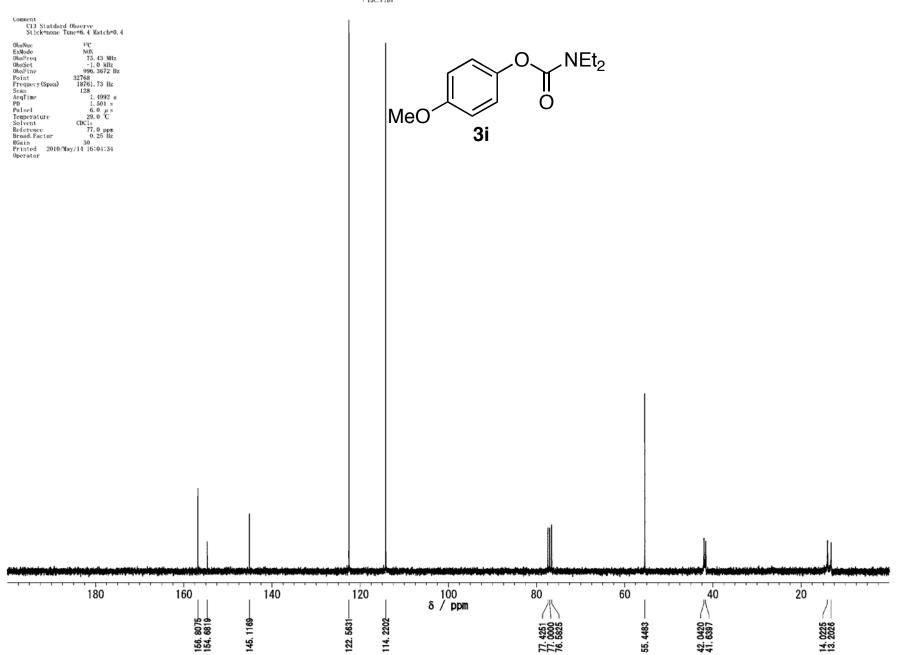
$\begin{array}{llllllllllllllllllllllllllllllllllll$	Original File: Date Se Comment STANDARD III 0	p 26 09 BSERVE
	ExMode ObsFreq ObsFreq ObsFine Point Prequecy(Span) Scan AcqTime PD Pulsel Temperature Temperature Solvent Reference Broad, Factor RGain Printed 2010/ju	NOX 299.96 MHz -1.0 kHz 995.0047 Hz 18384 4500.45 Hz 6.0 μs 29.0 °C CDC1a 0.1373 Hz 24



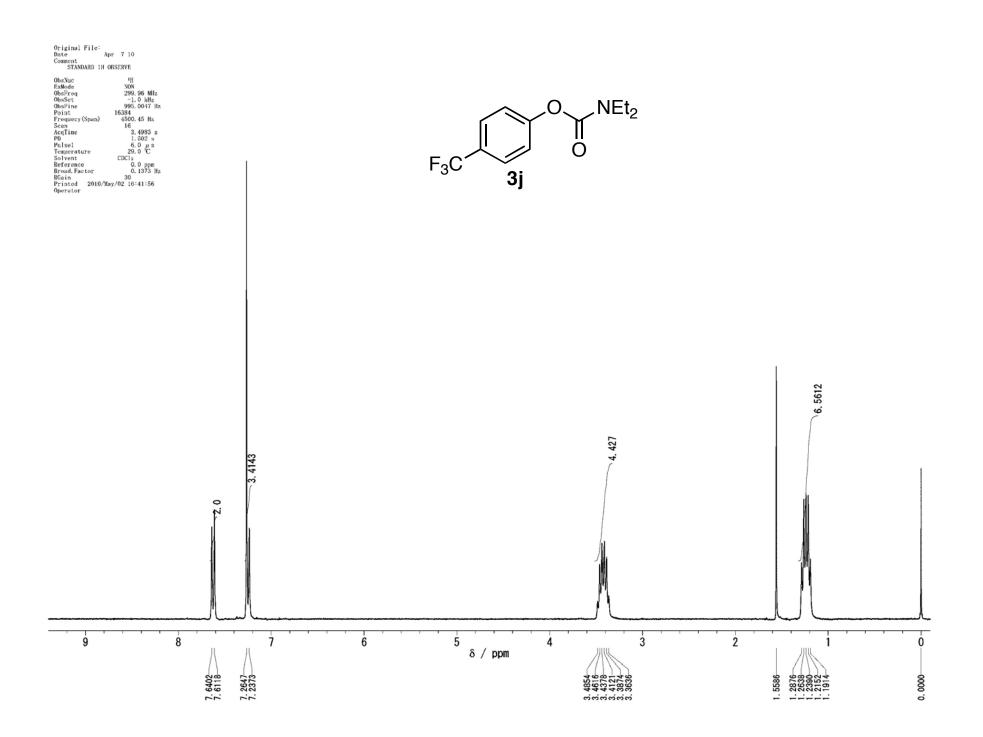


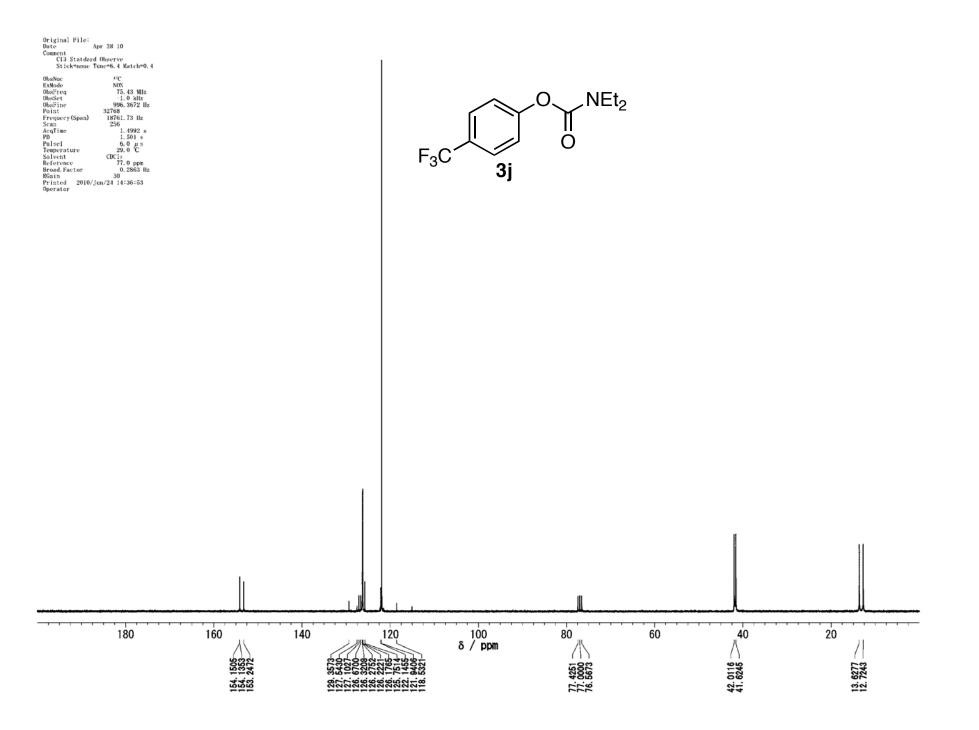


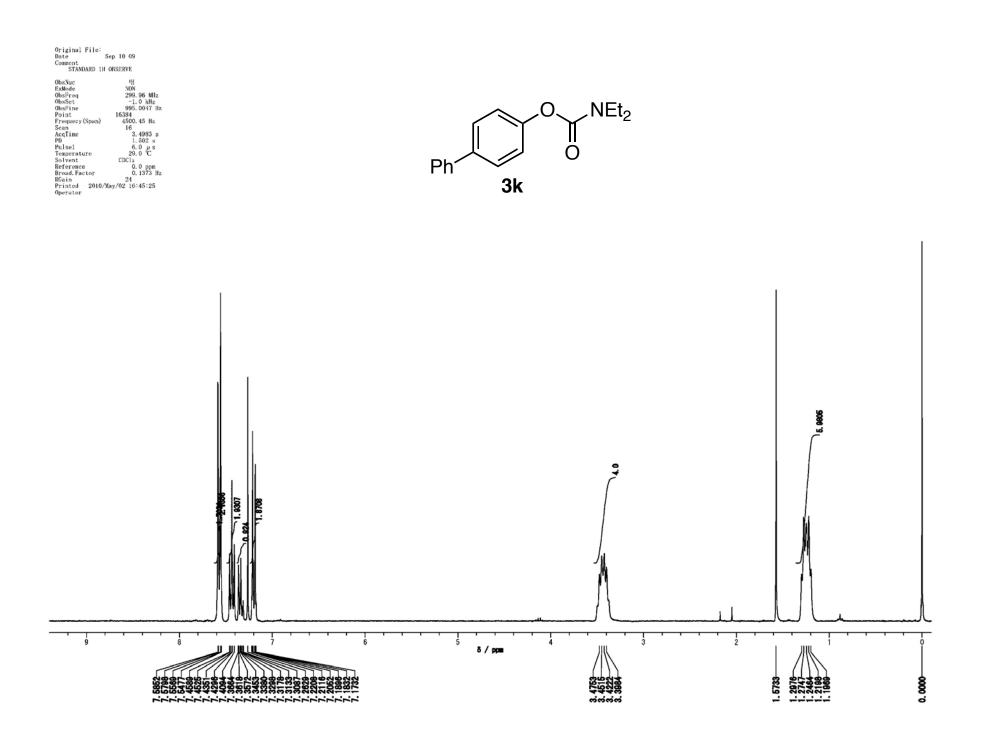


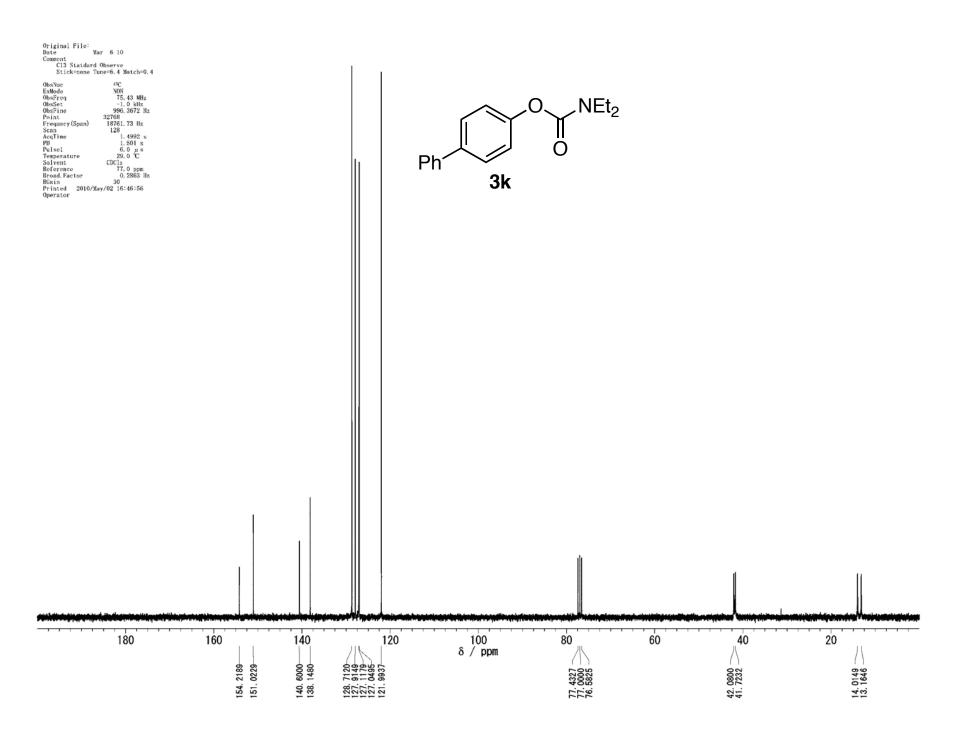


13C. F1DY

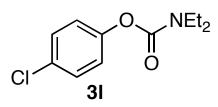


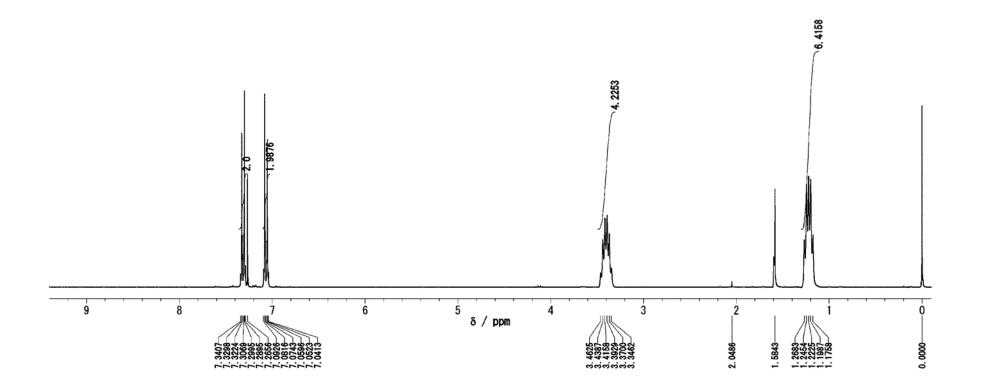


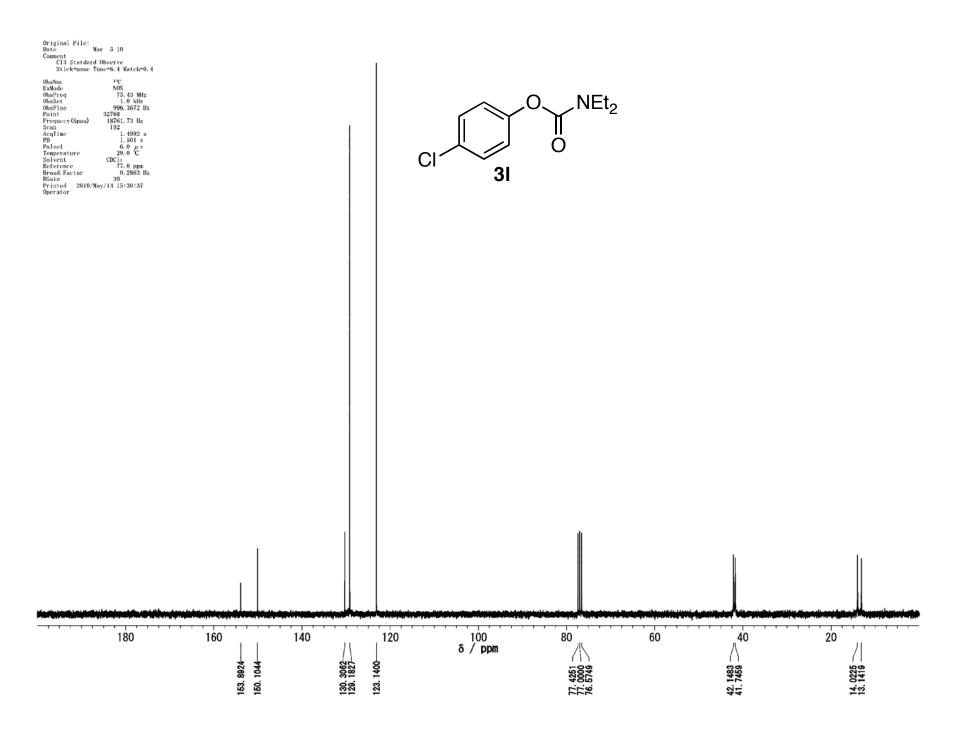


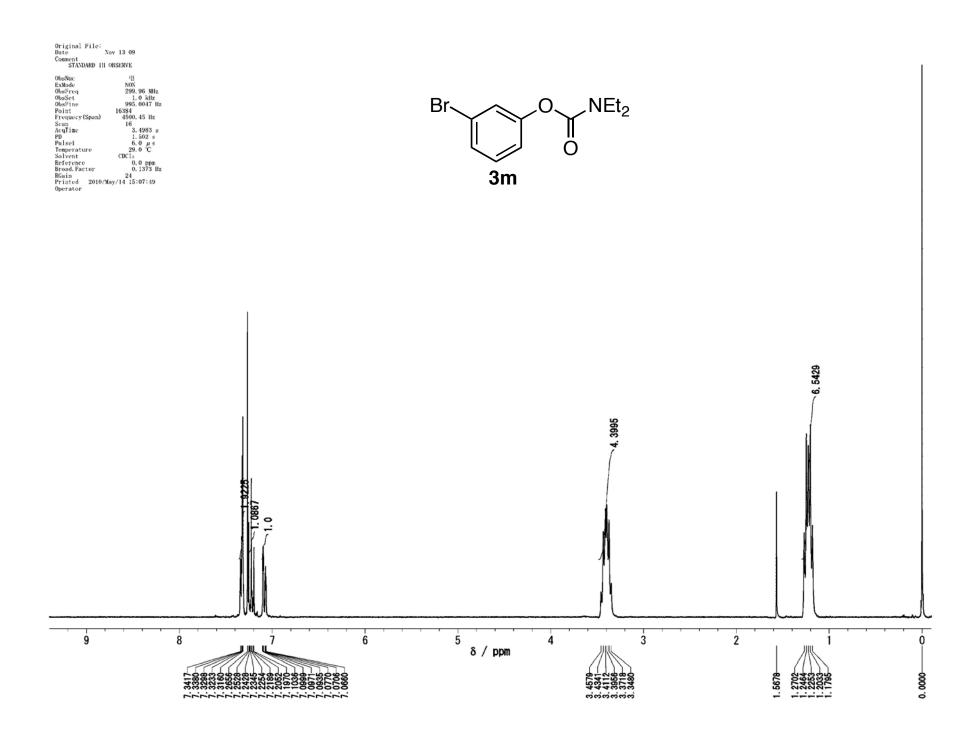


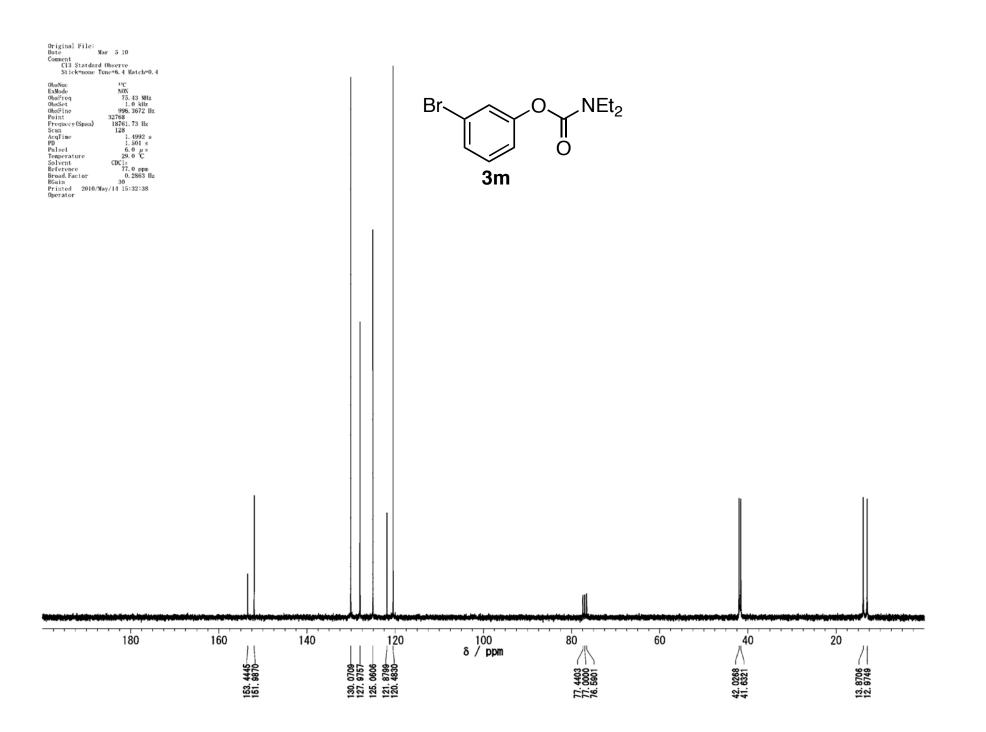
01Nu. 10	Original File: Date Jun 6-09 Comment STANDARD IN OBSERVE	
Doswut      Tit        Dustreq      299.96 MHz        ObsFreq      299.96 MHz        ObsFreq      1.0 kHz        Scan      16385.0047 Hz        Scan      16383 s        AcqTime      3.4983 s        Philsel      1.6 0 s        Solvent      CDC1 s        Solvent      CDC1 s        Shraad, Factor      0.1373 Hz        Goard      2010/May/14 15:02:58        Operator      2010/May/14 15:02:58	ÖbsFreq      299, 96 Wills        ObsSet      -1.0 Altr        ObsSet      -1.0 Altr        ObsFine      995,0047 Hz        Prequecy(Span)      4500,45 Hz        Scan      16        AcqT1mc      3,4983 x        P0      1.562 s        Pulsel      6,0 µ s        Temperature      29,0 C        Solvent      CDC1x        Reference      0,0 pm        Broad, Factor      0,1373 Hz        Gain      19        Printed      2010/May/14 15:02:58	

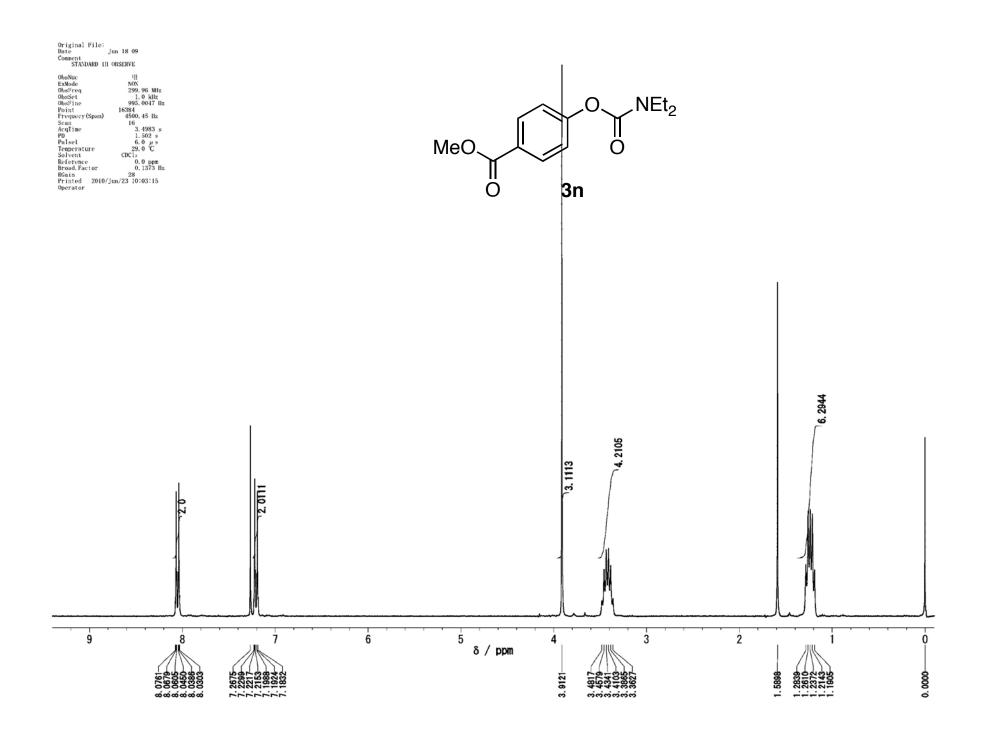


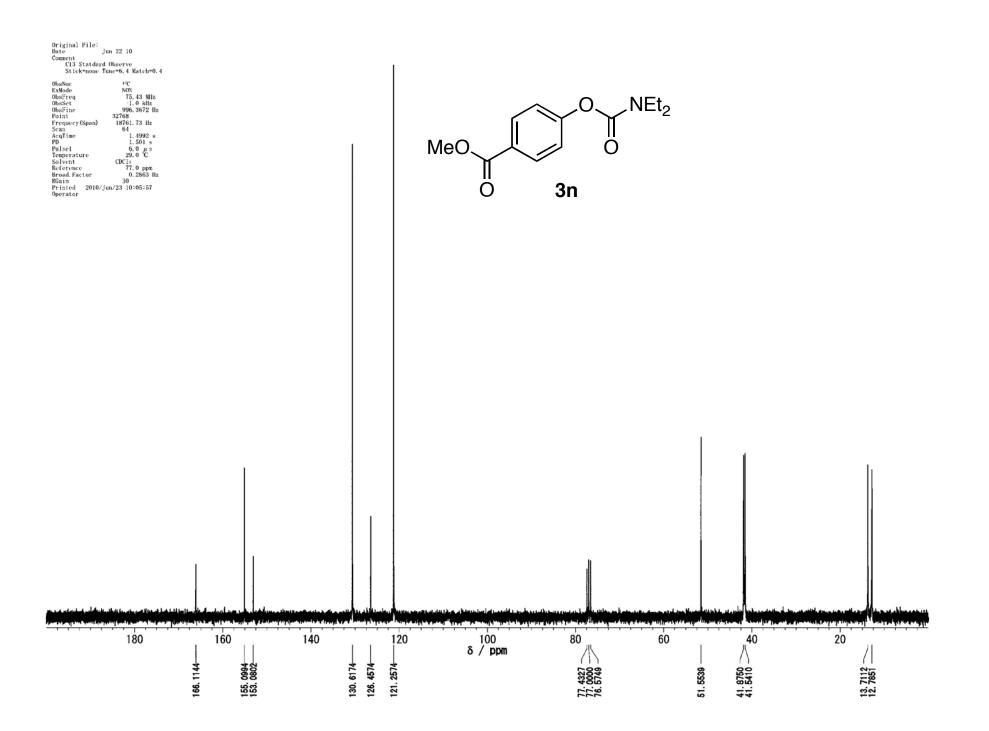


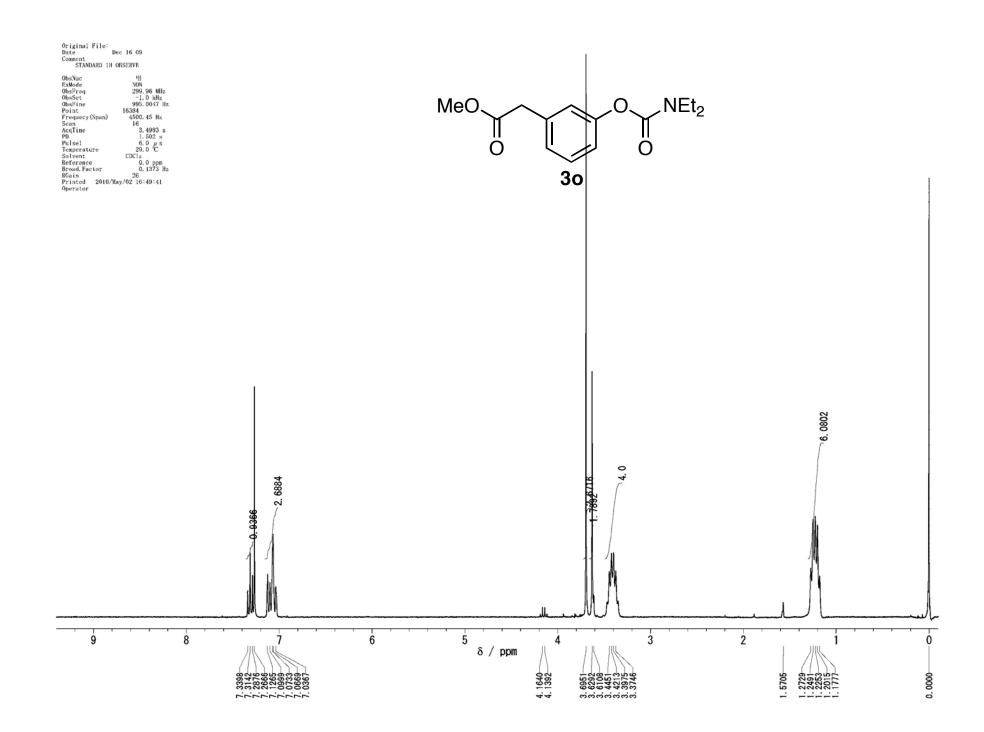


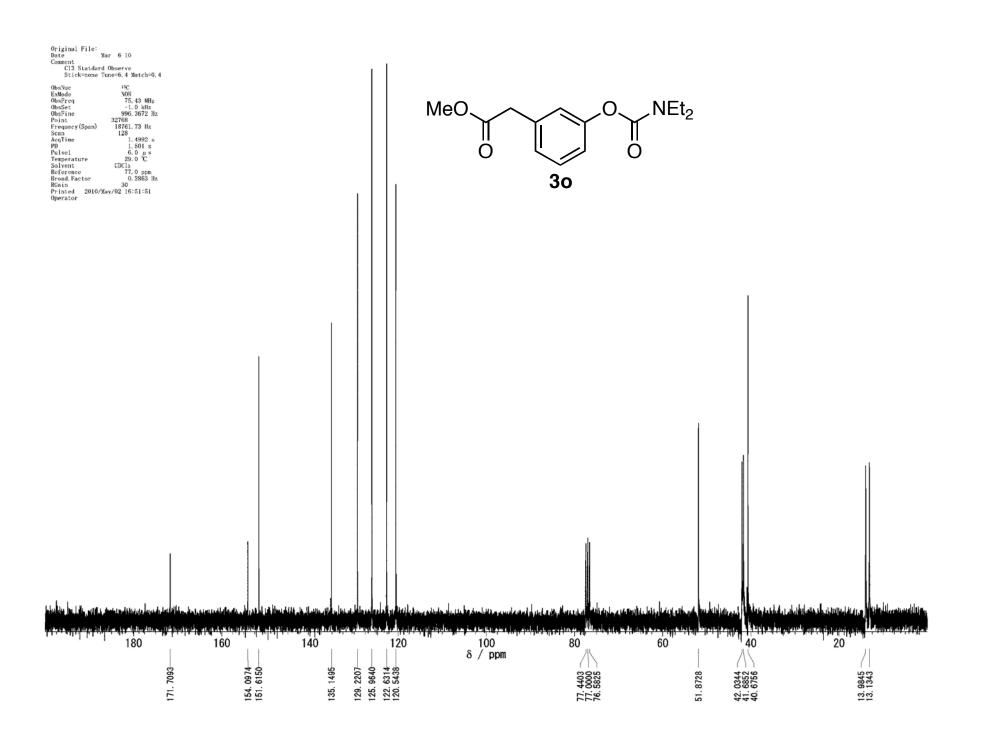


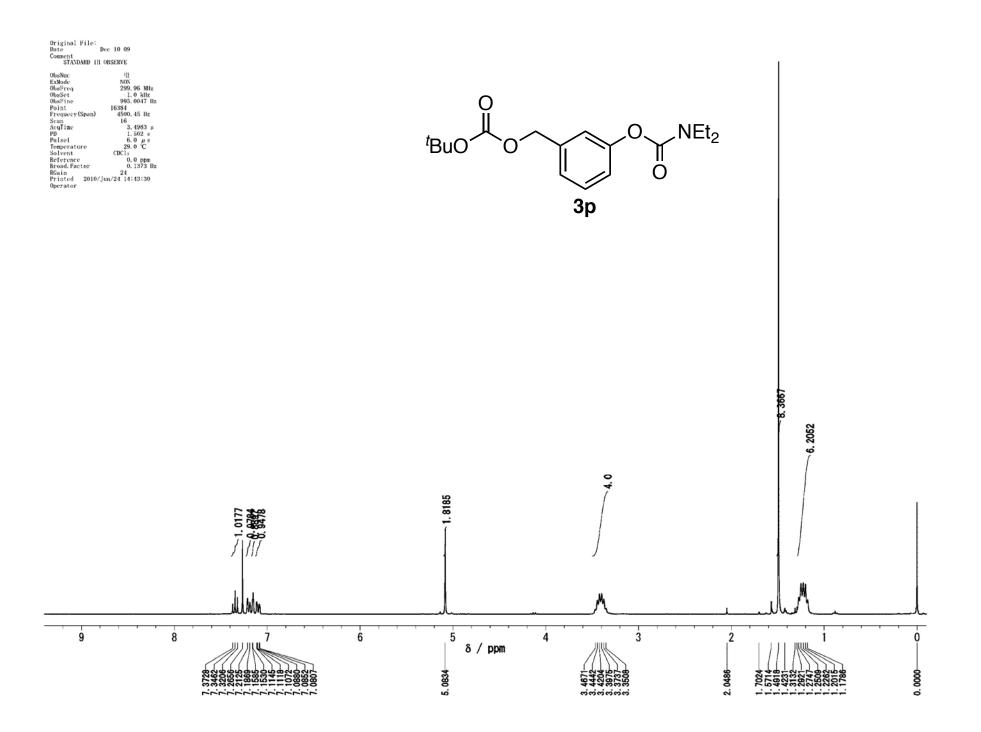


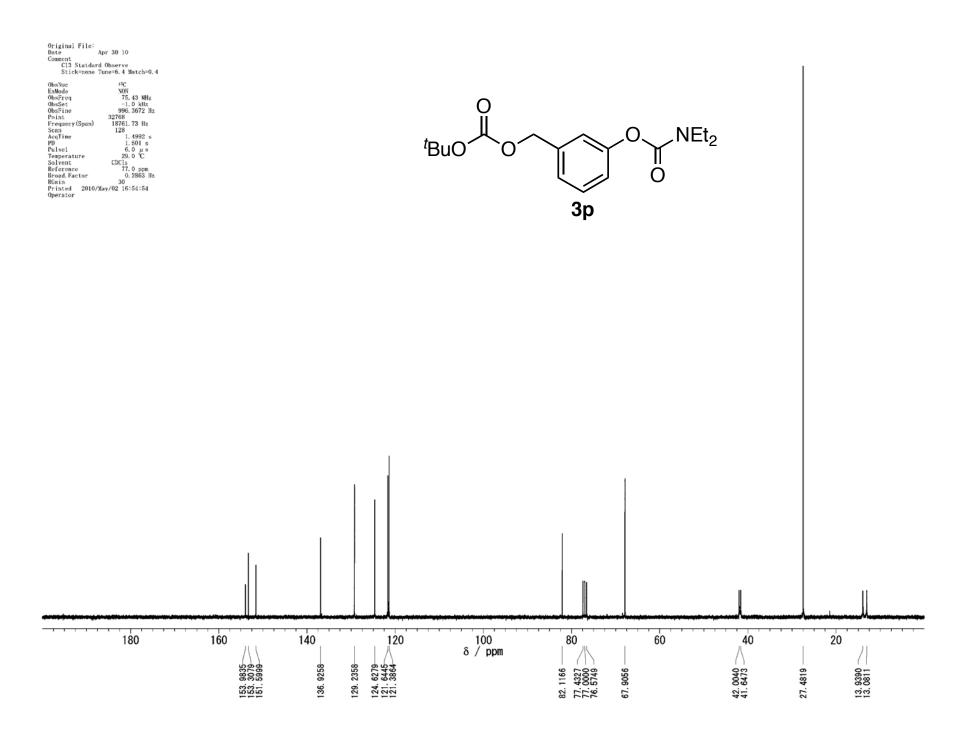


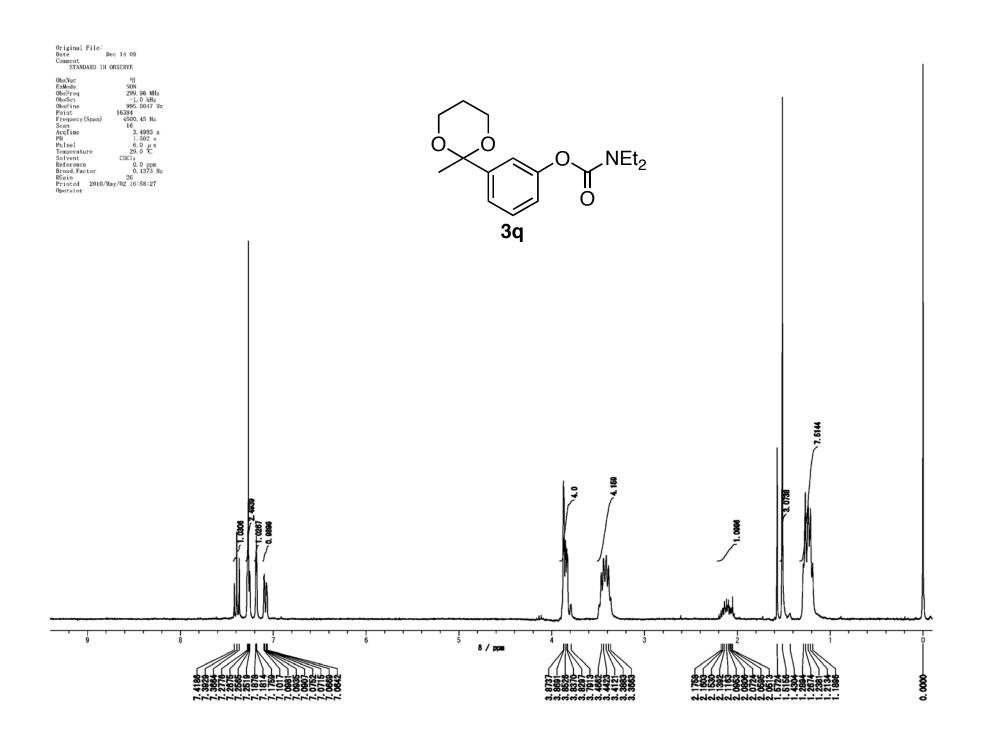


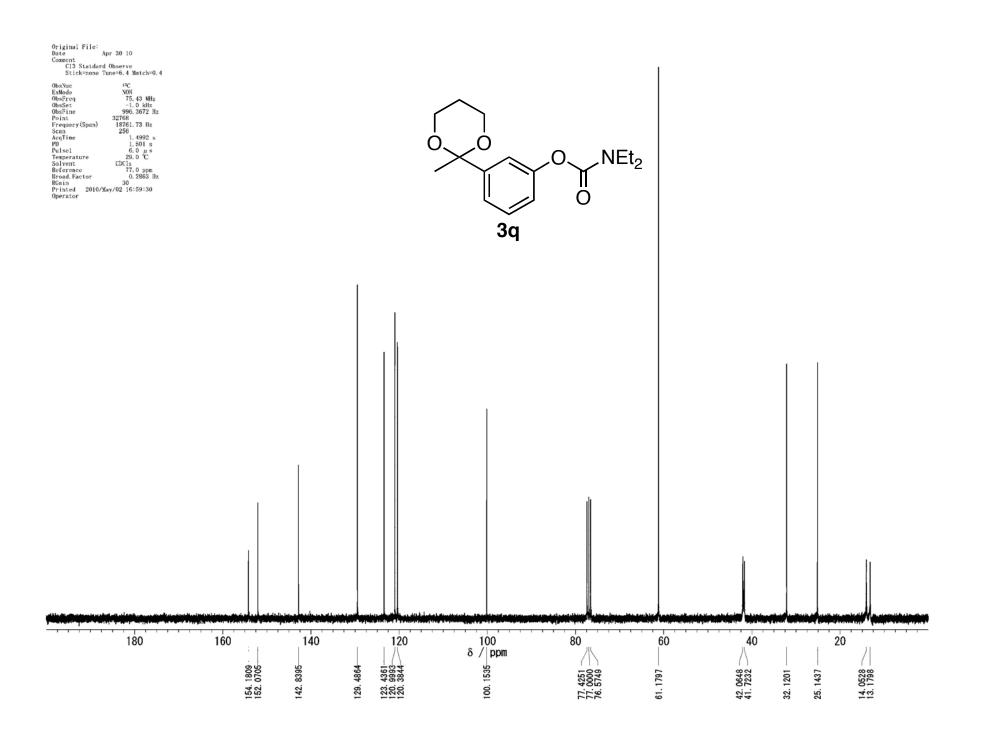


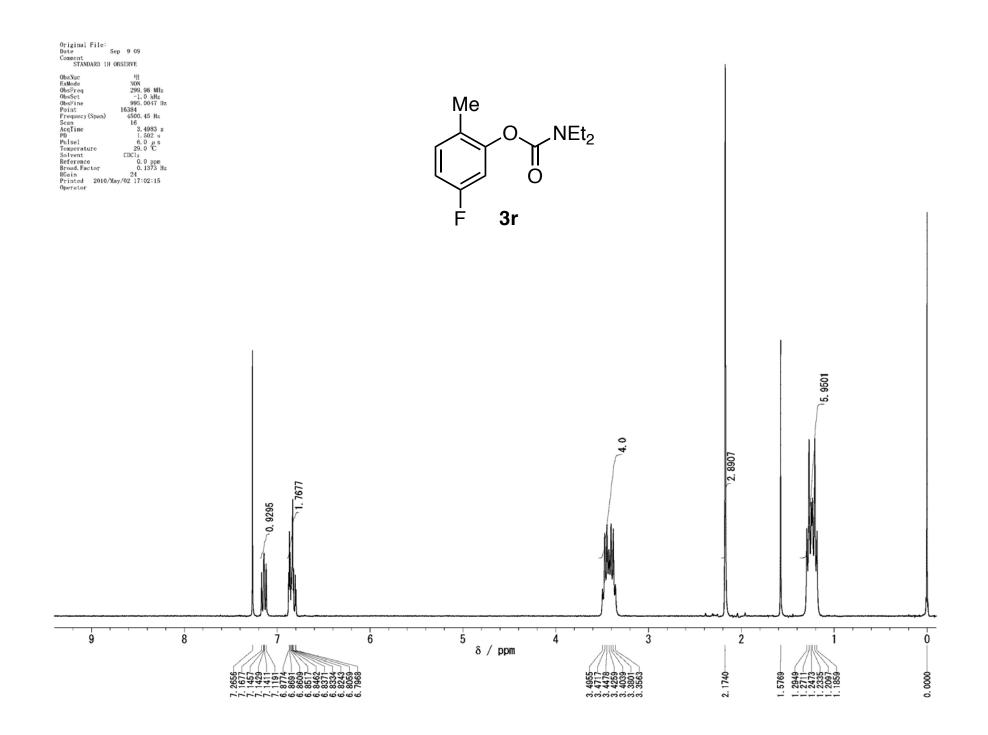


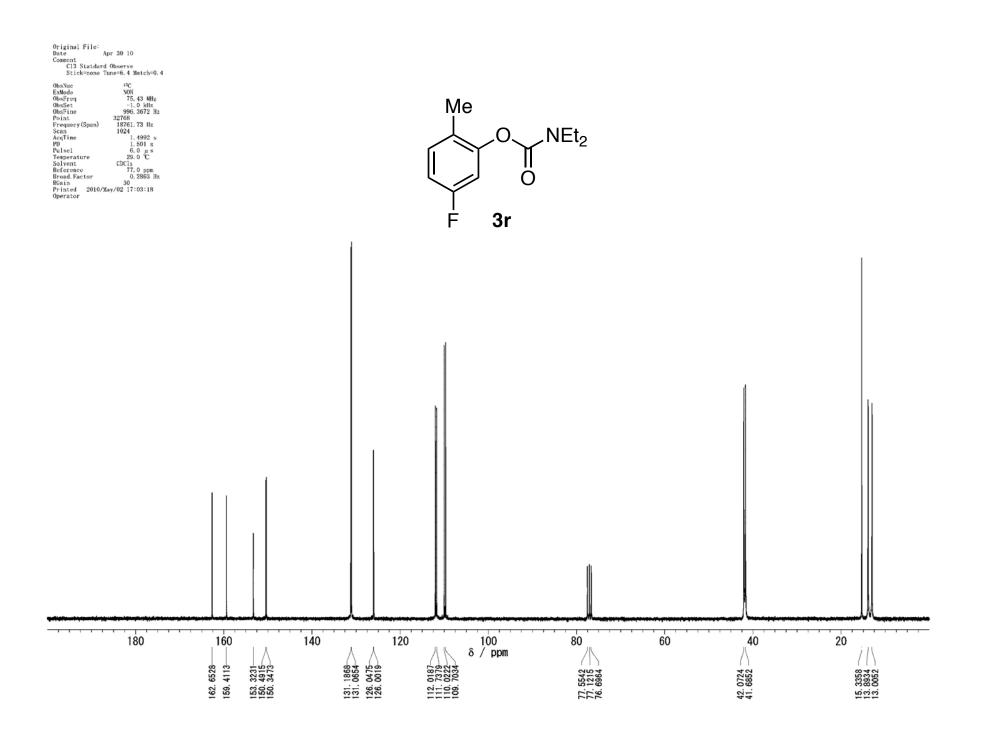


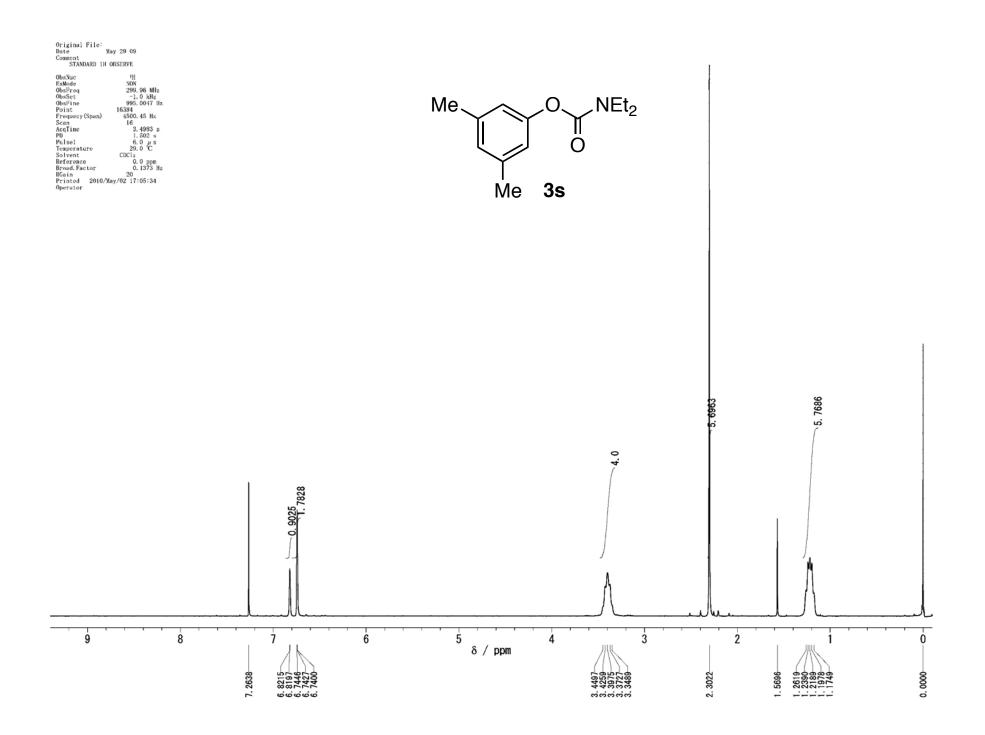


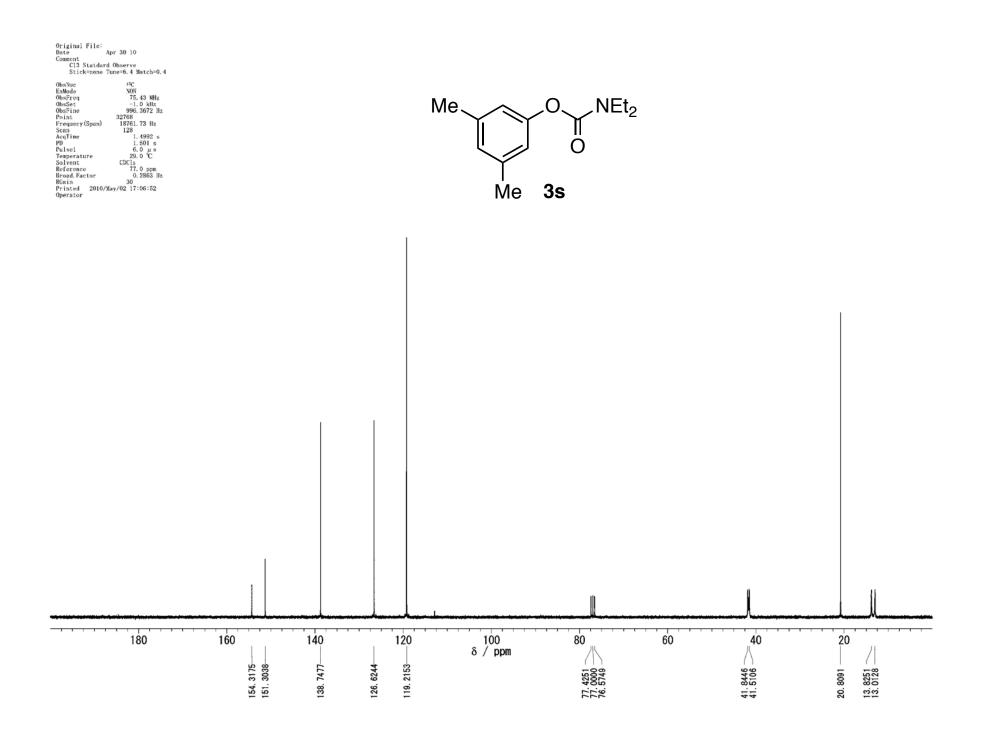


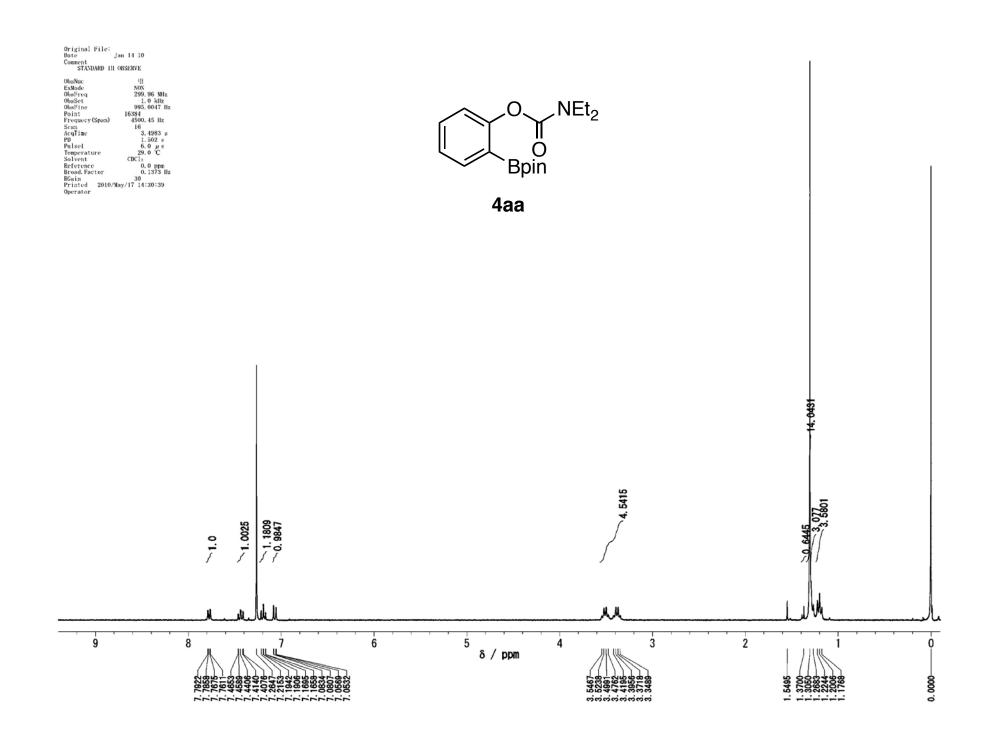


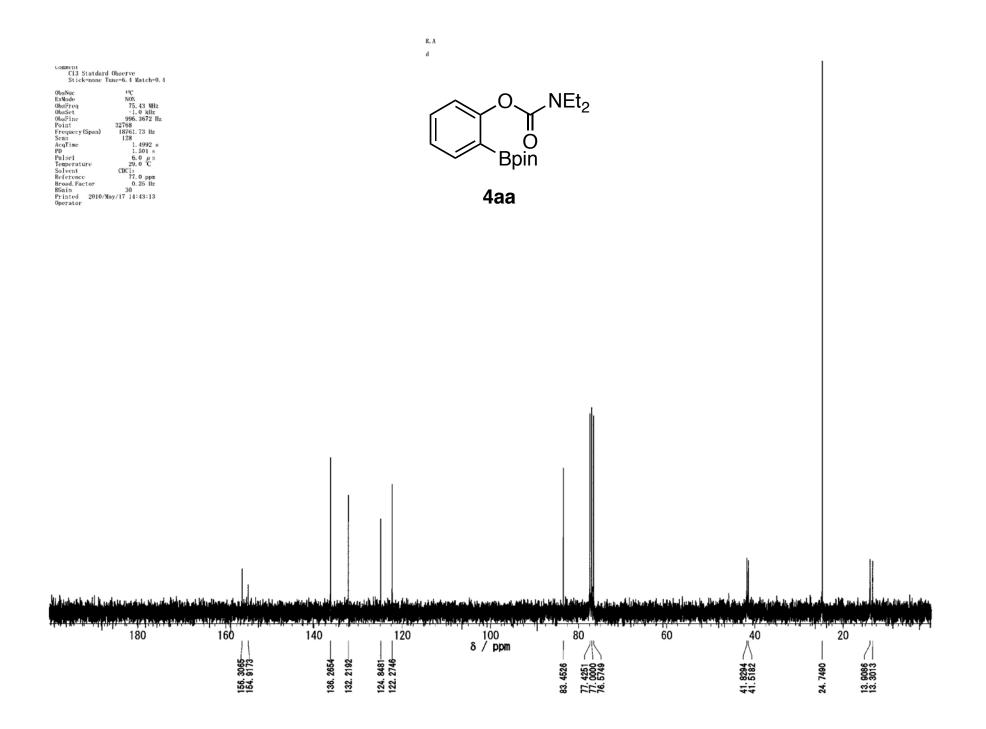


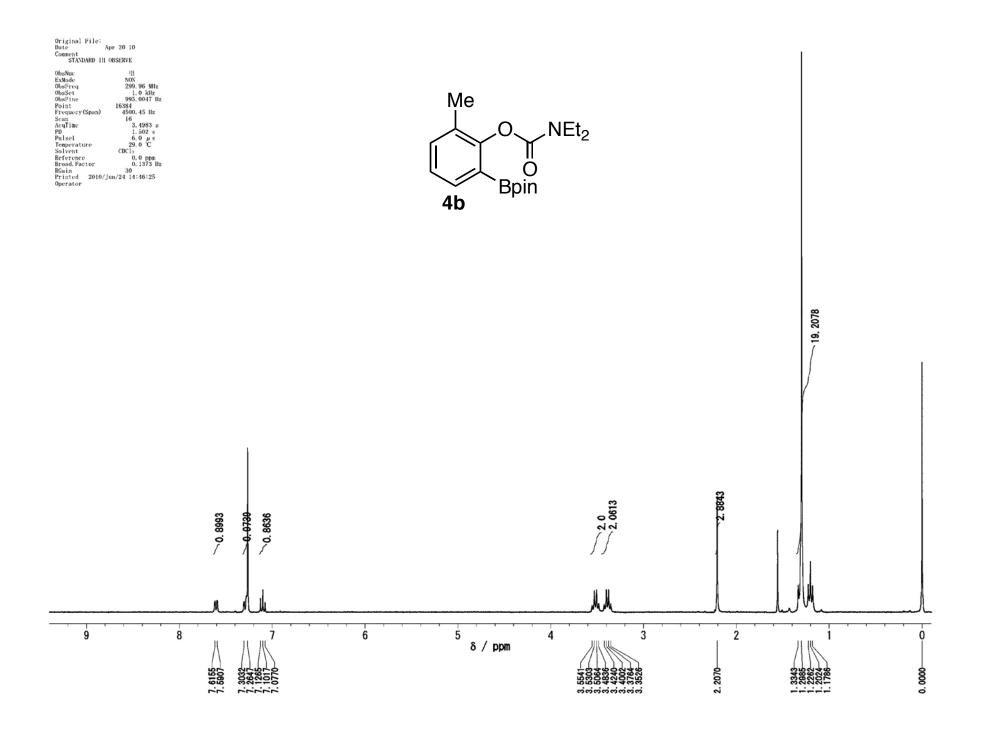


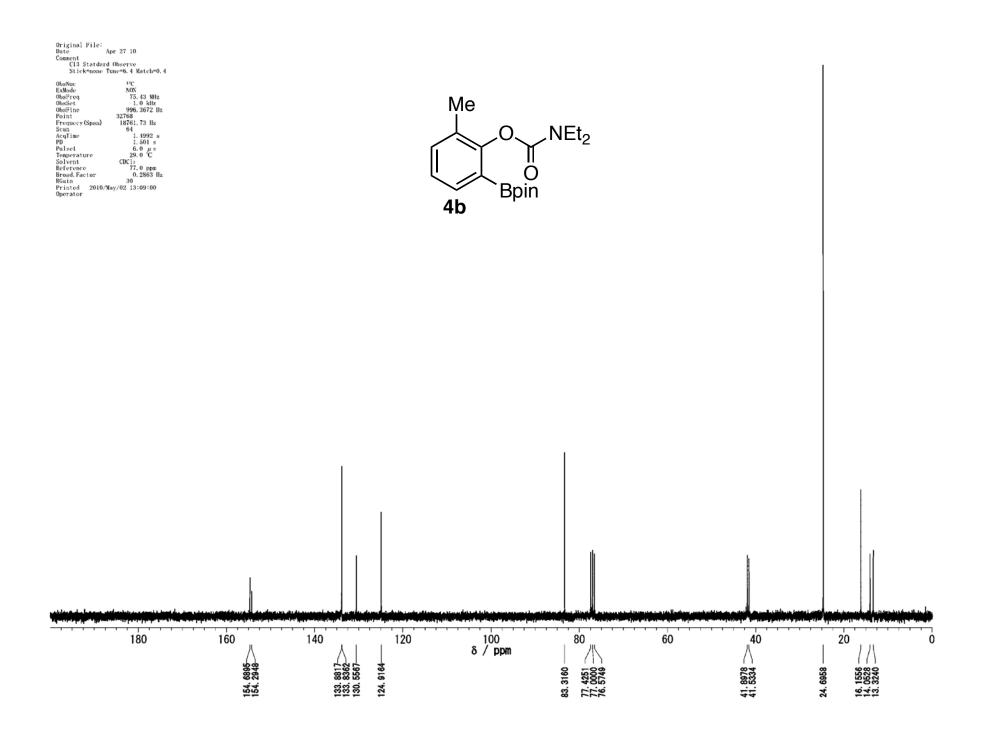


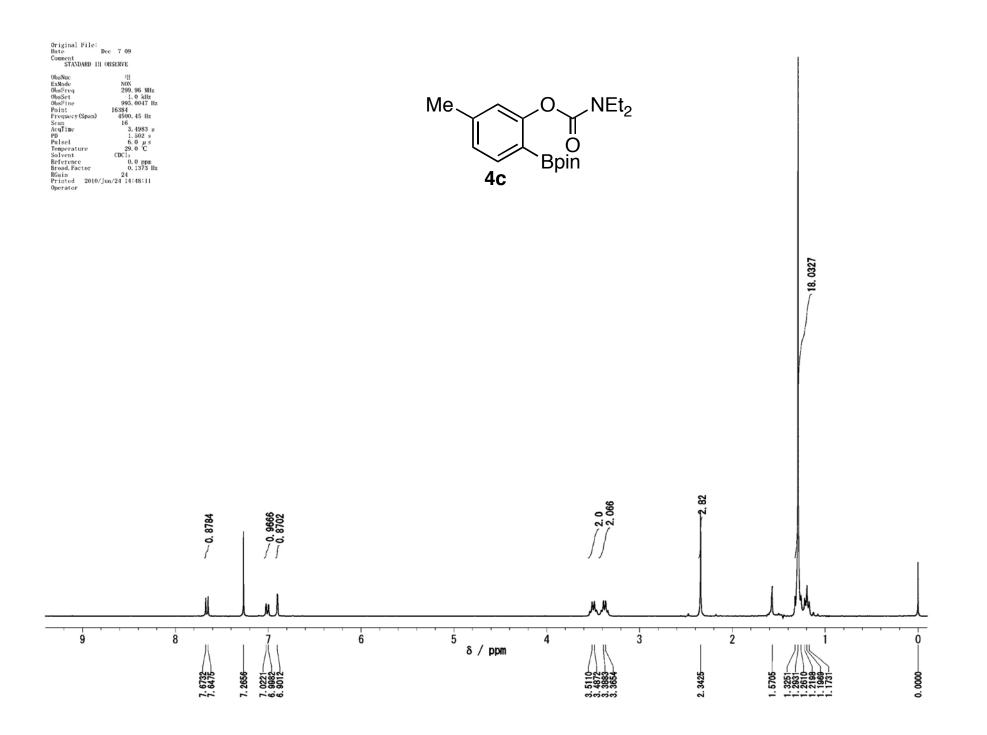


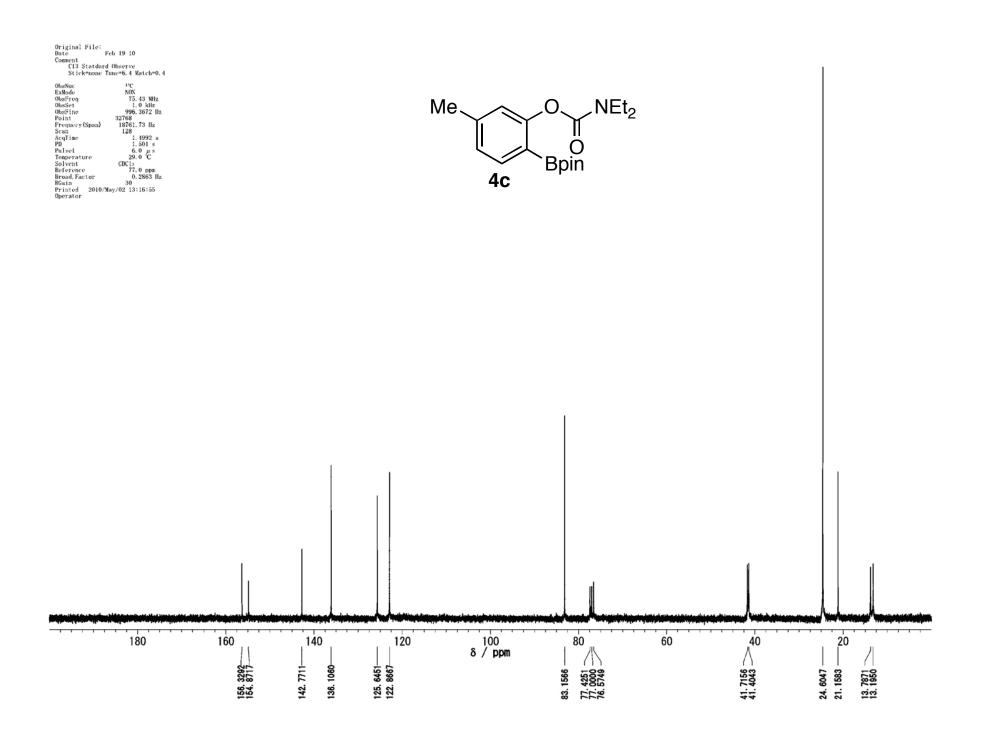




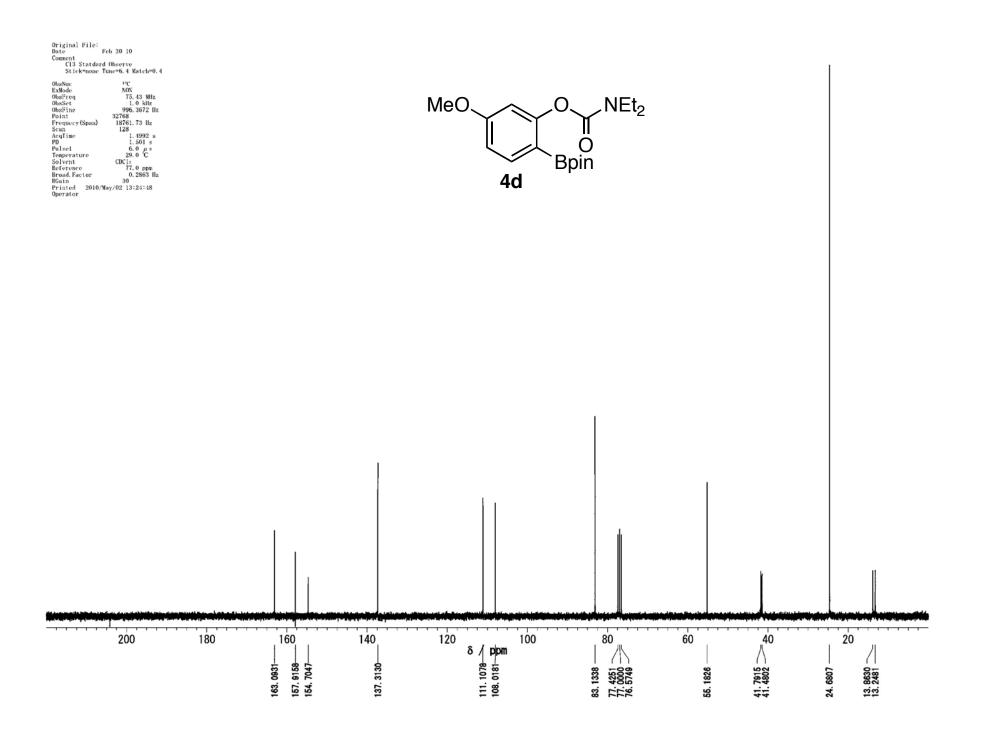


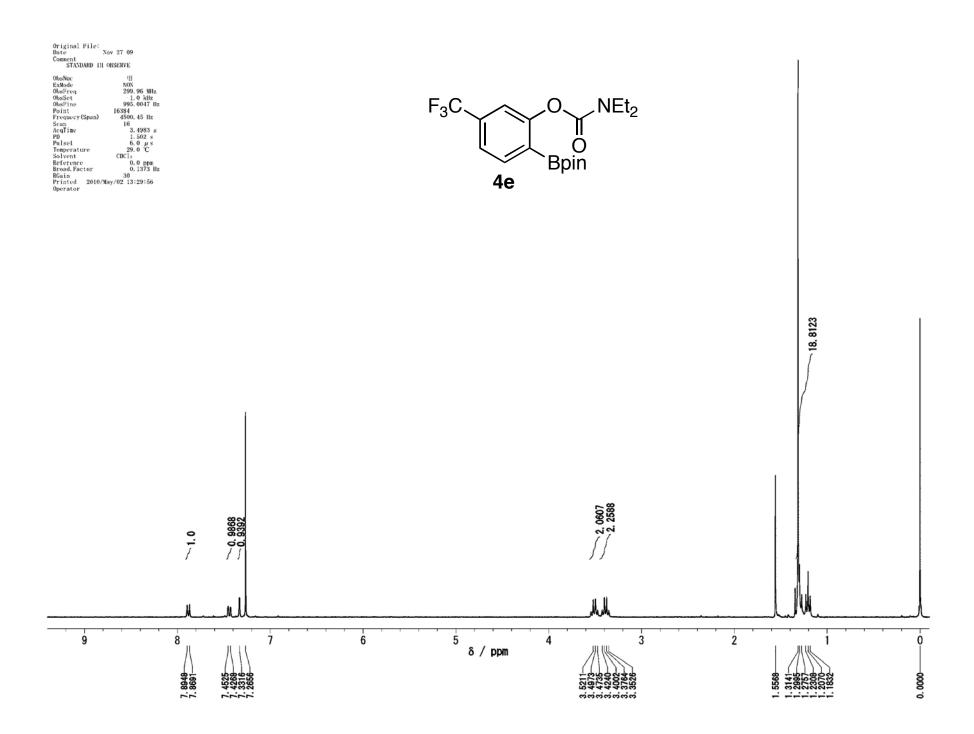




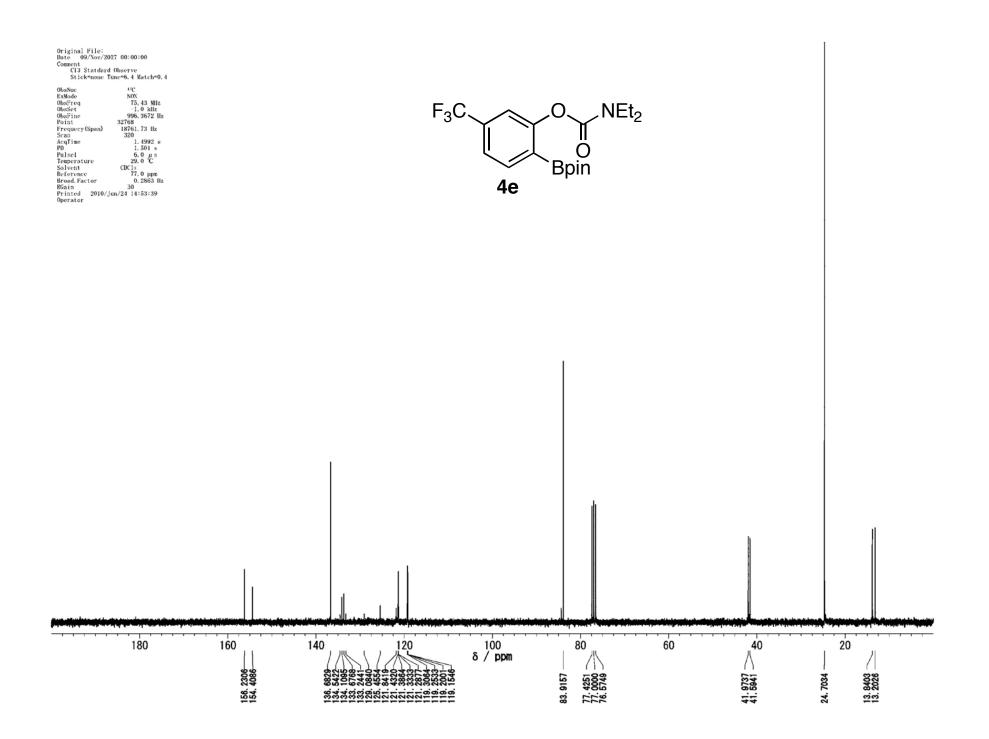


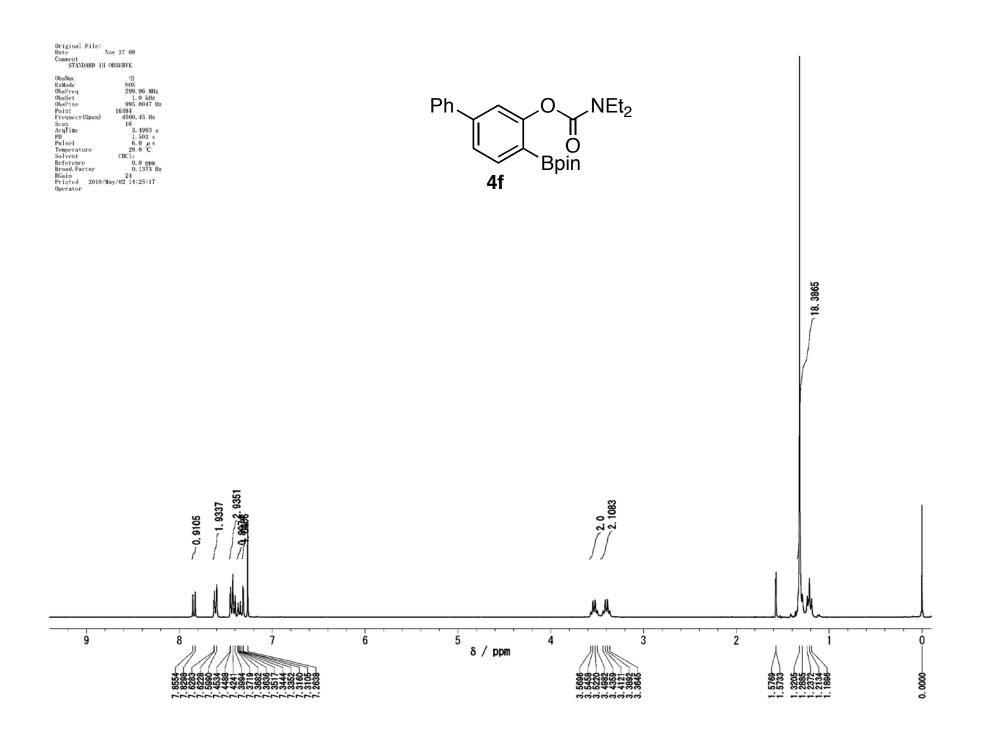


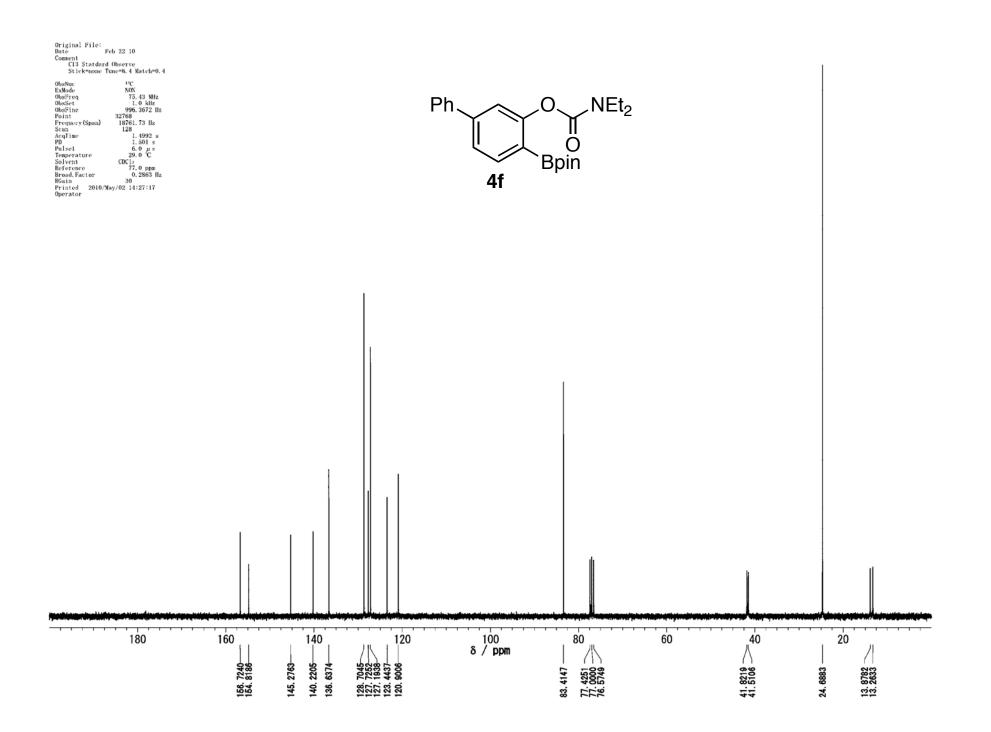


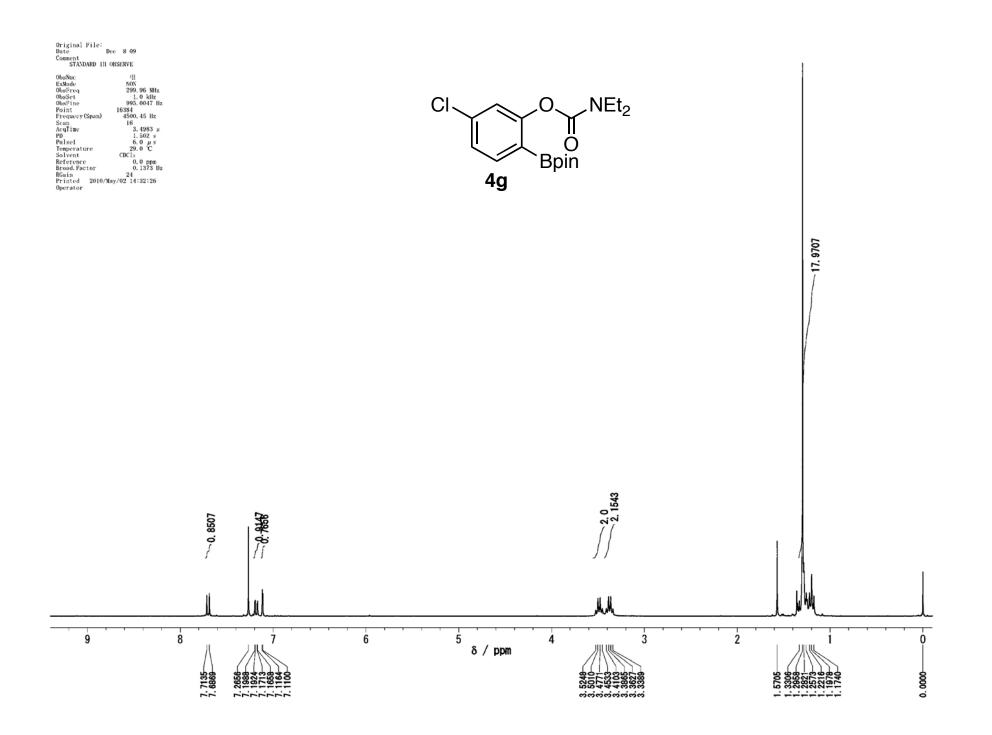


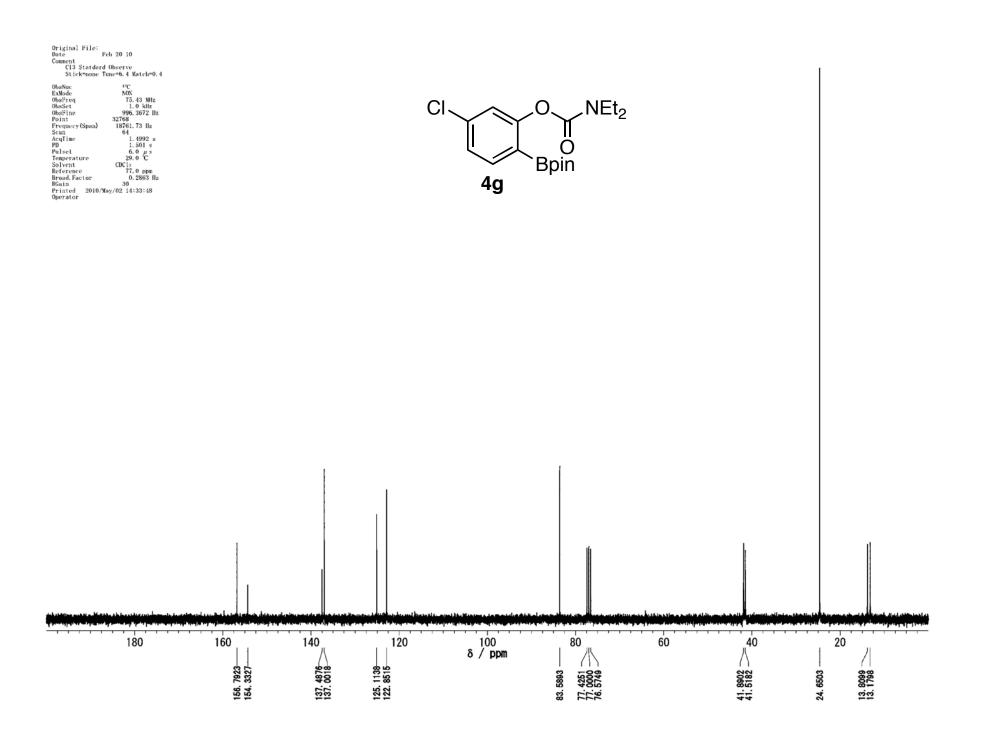




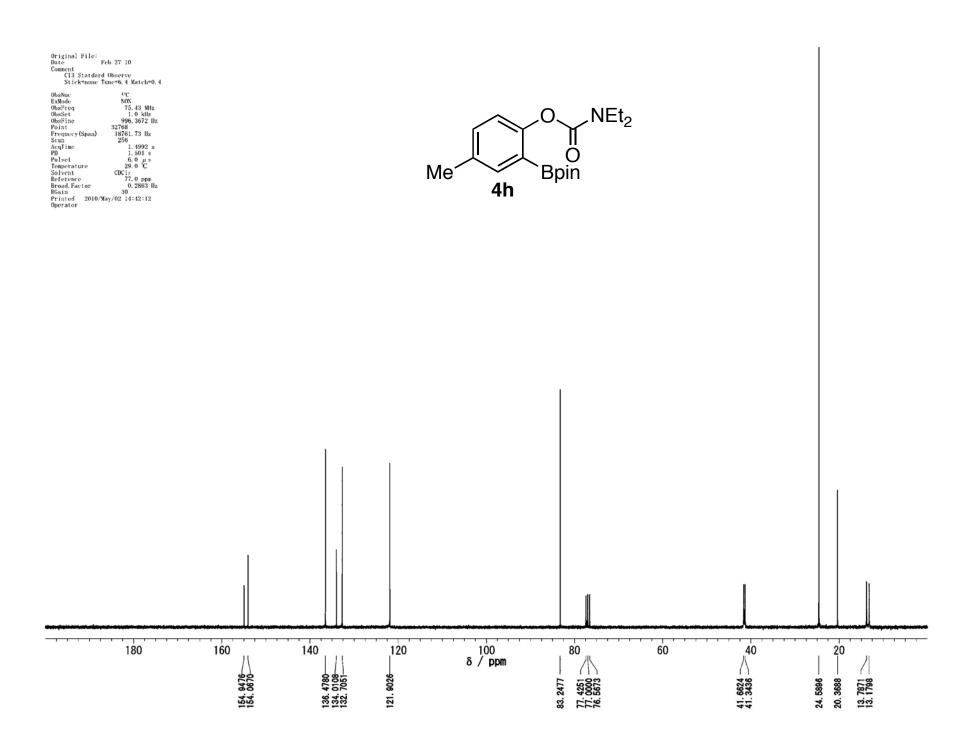


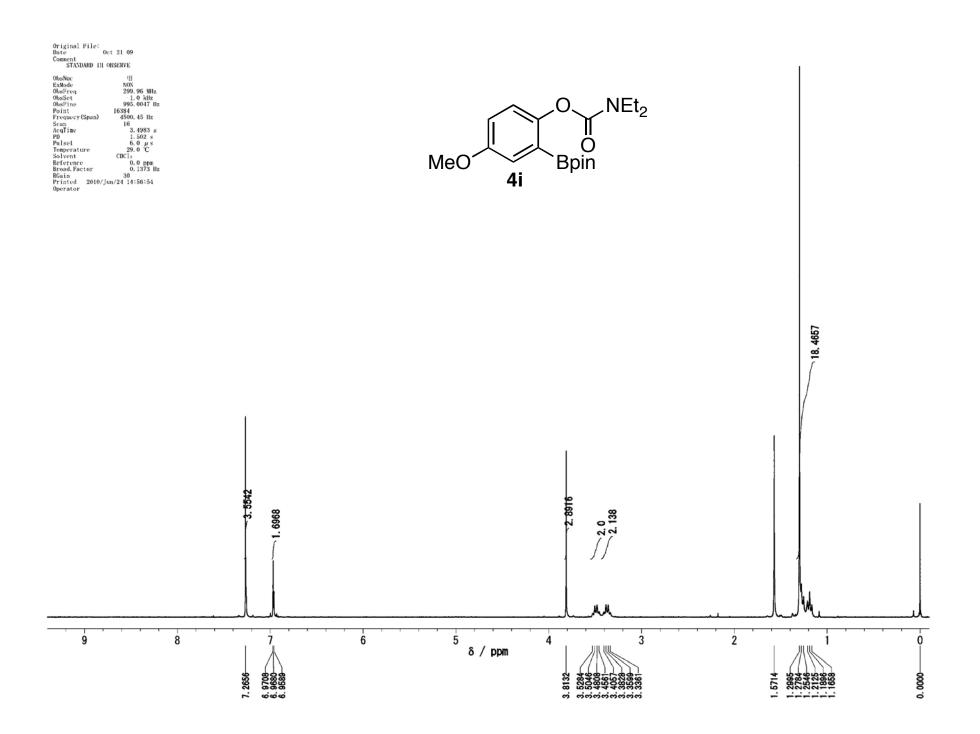


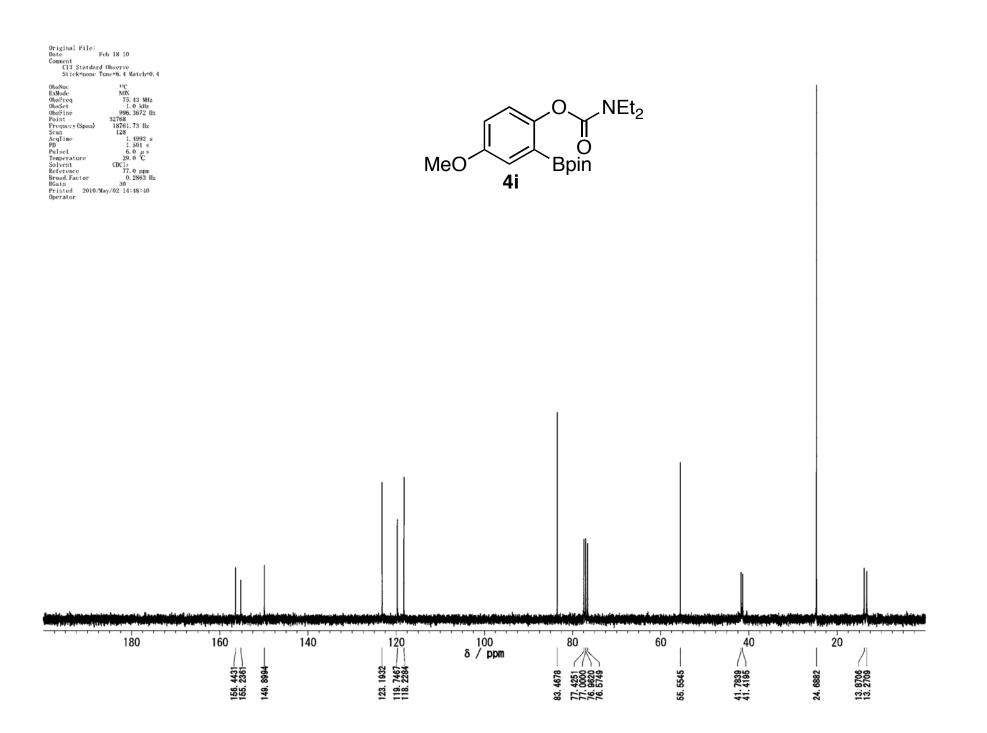


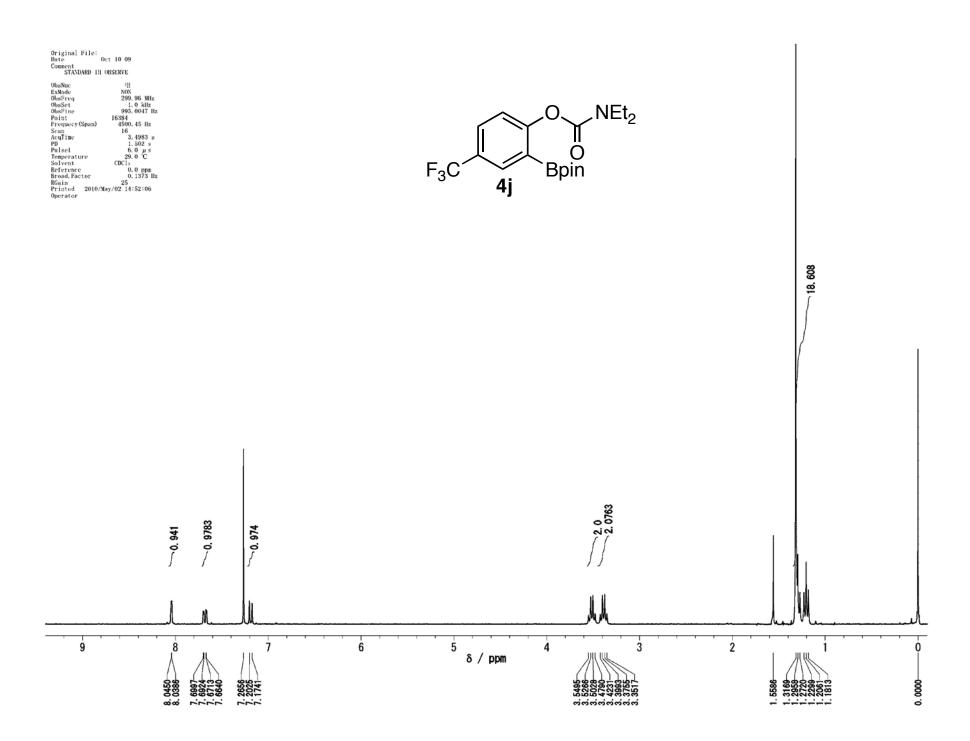






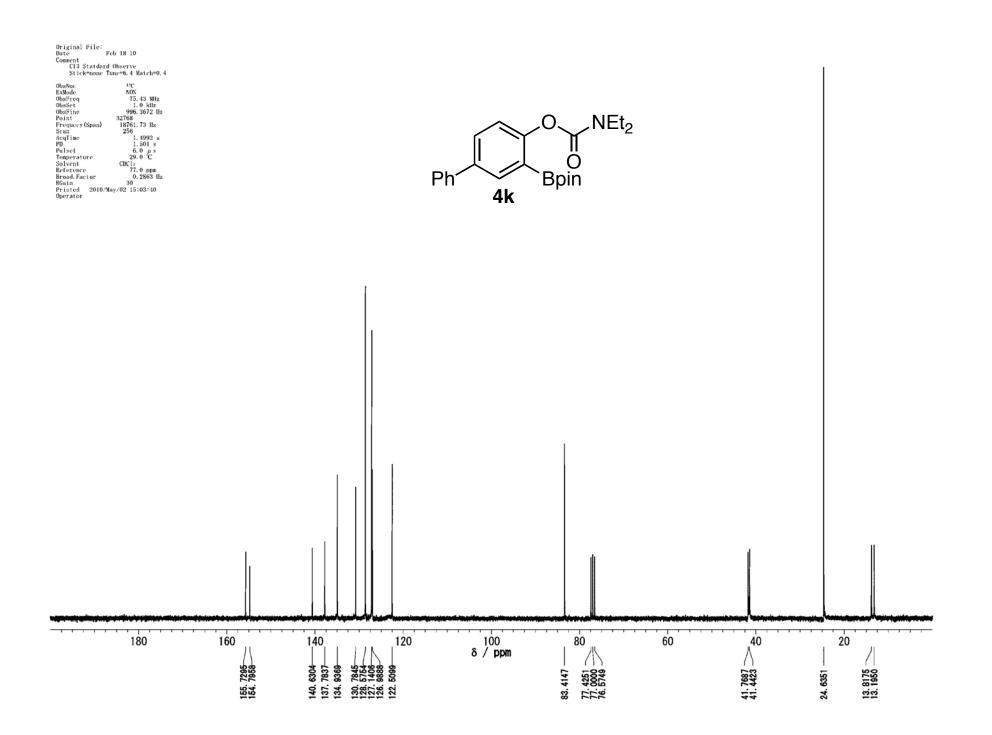


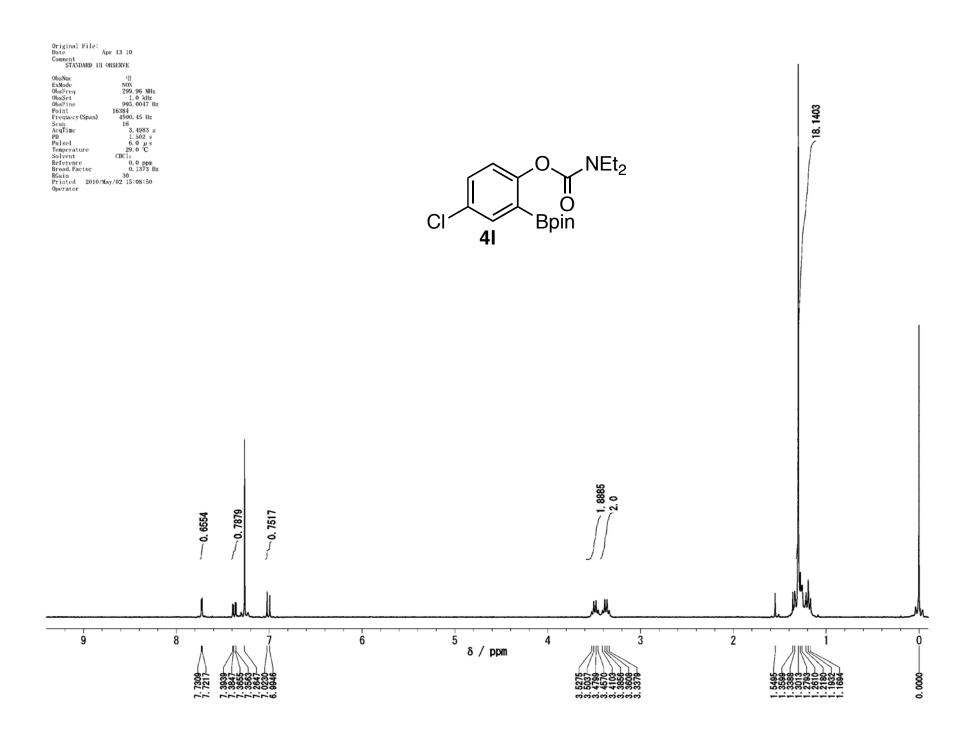


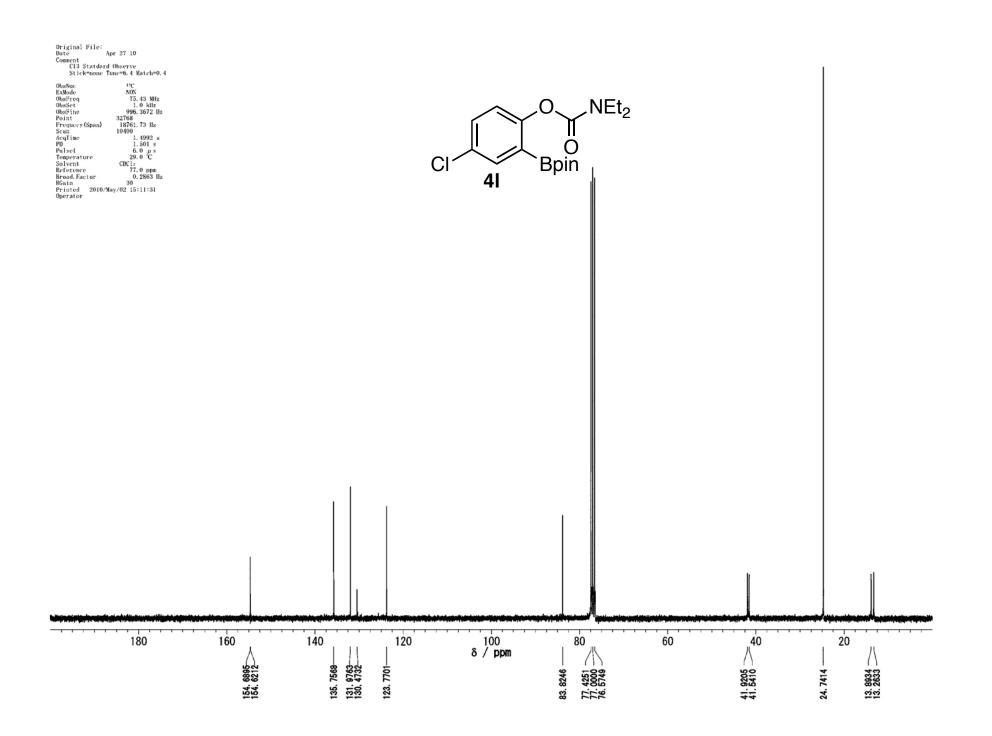


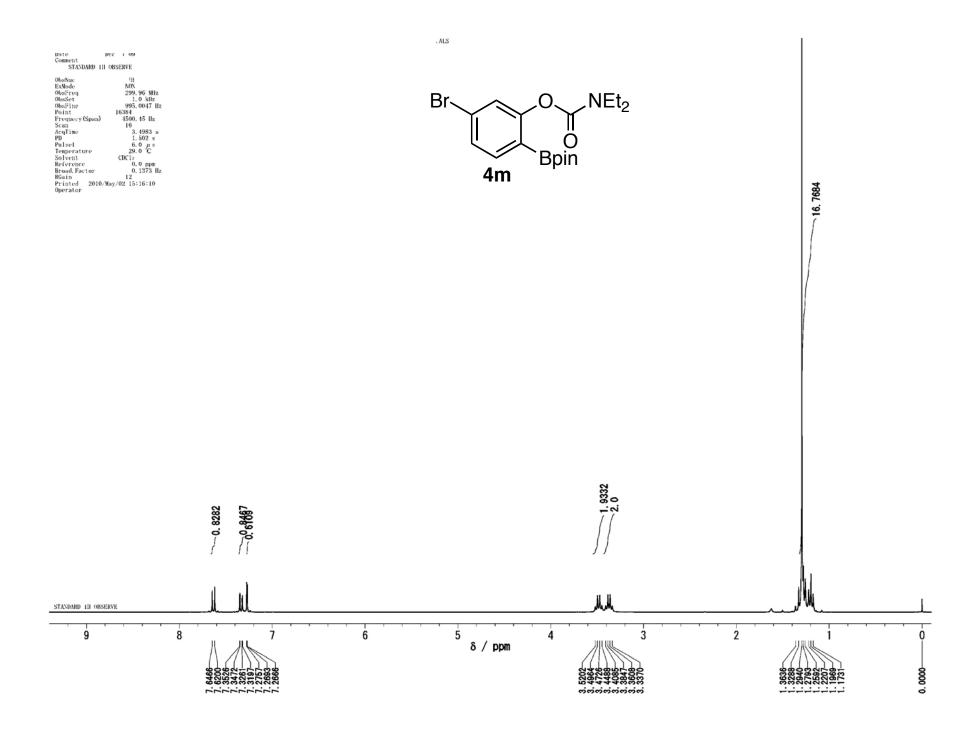


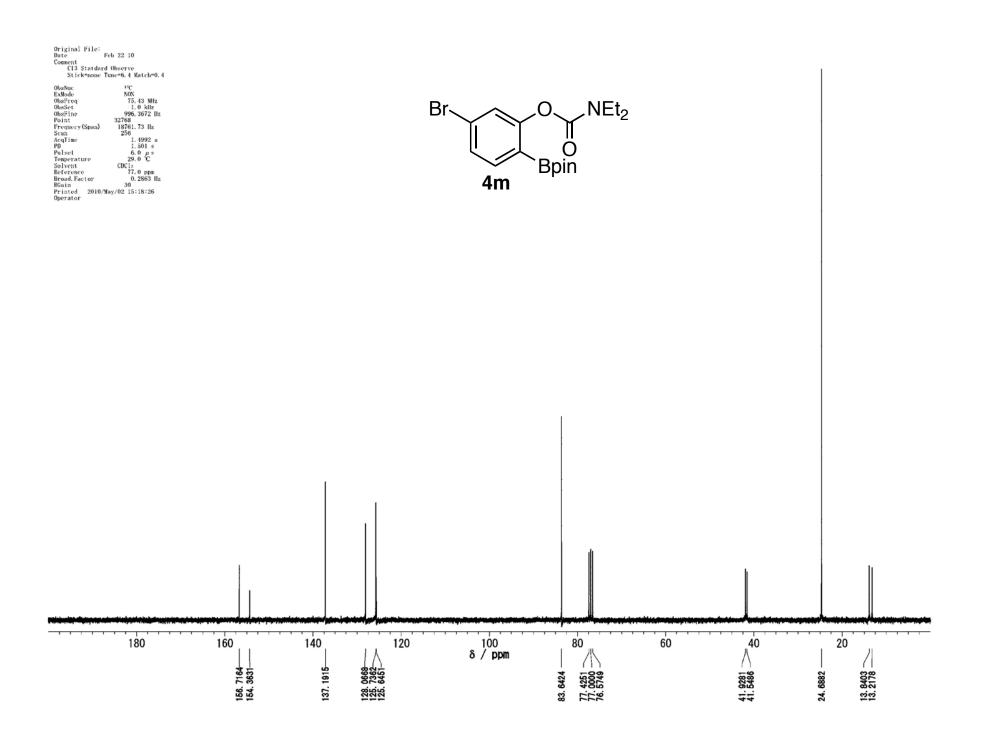


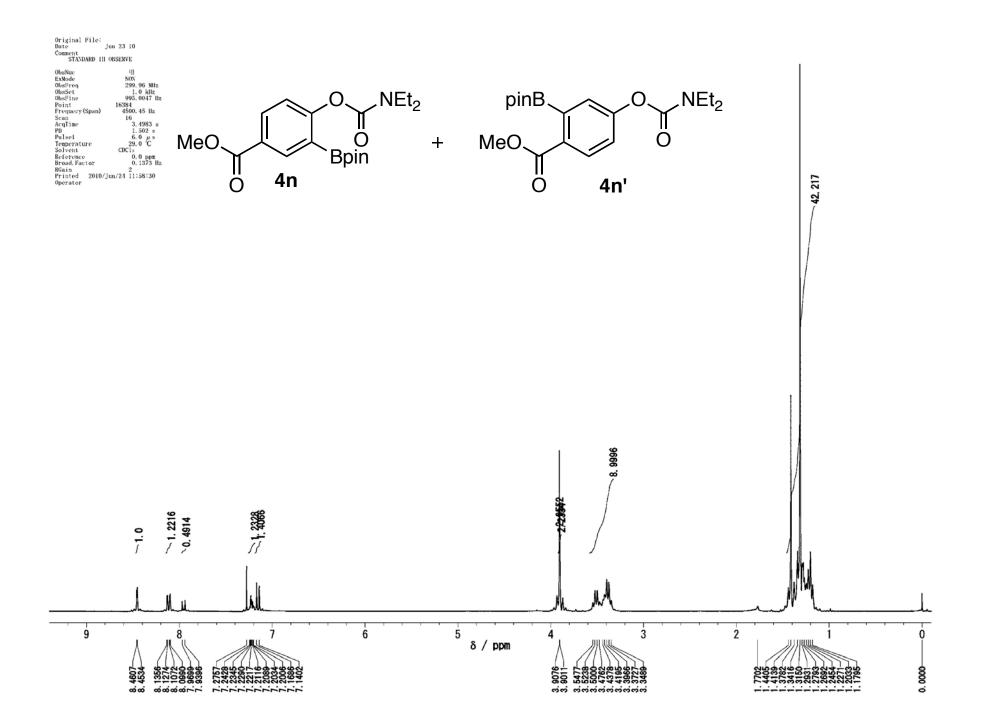


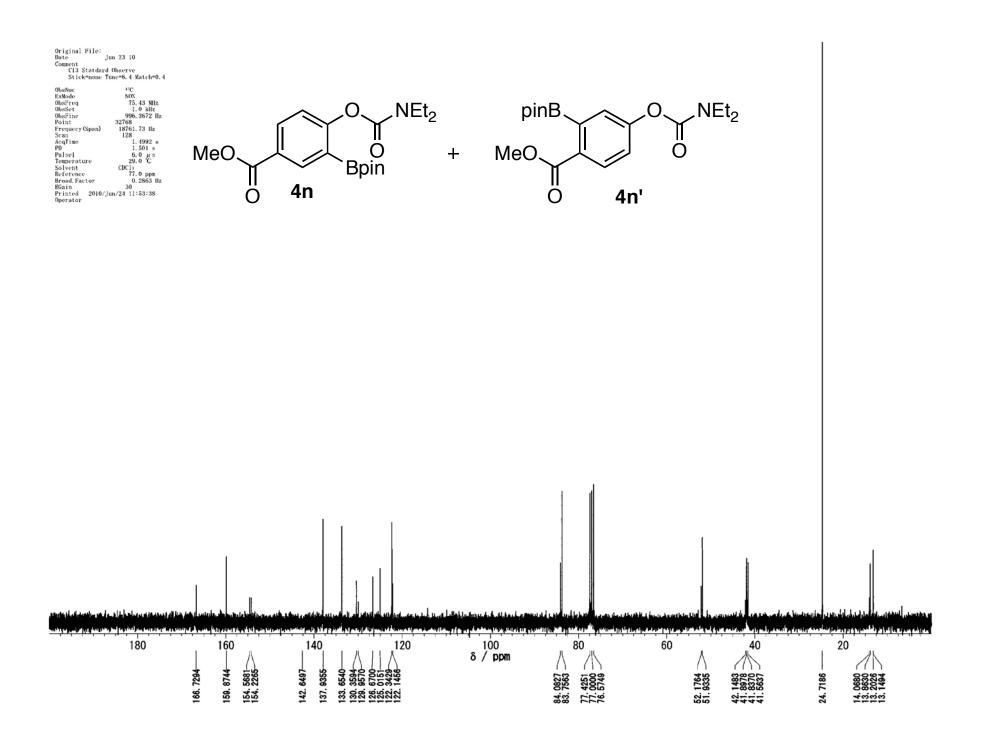


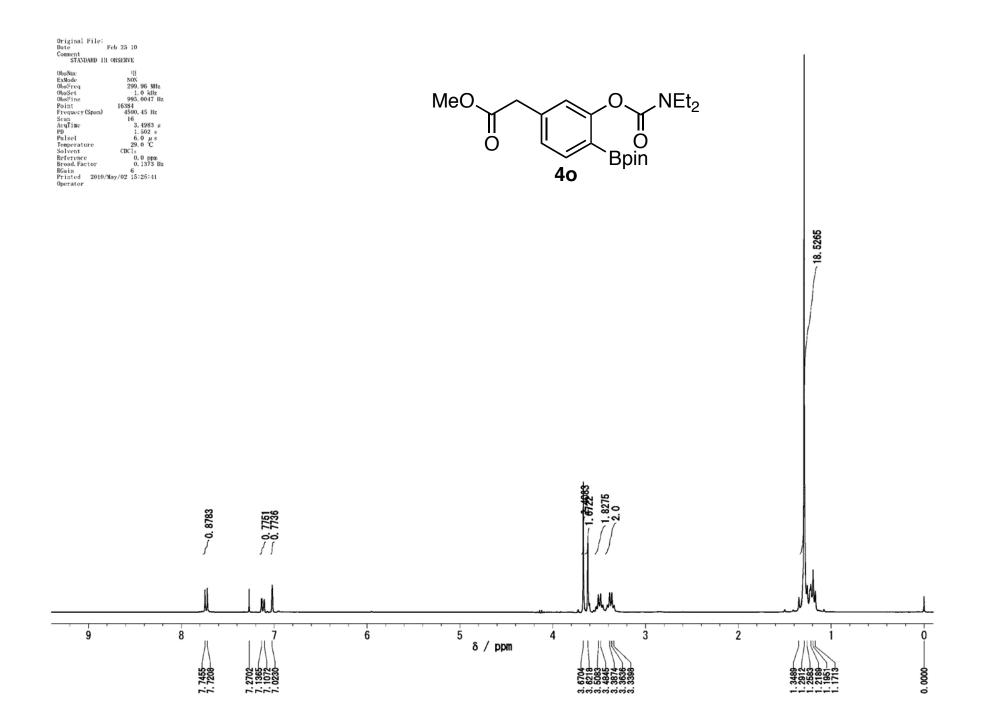




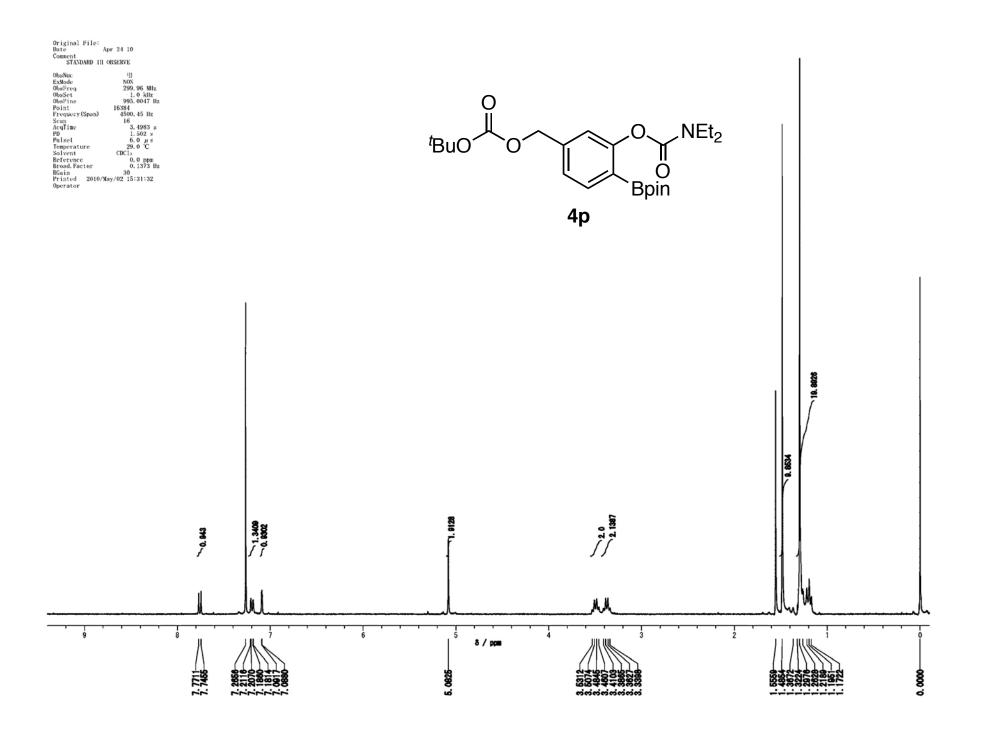


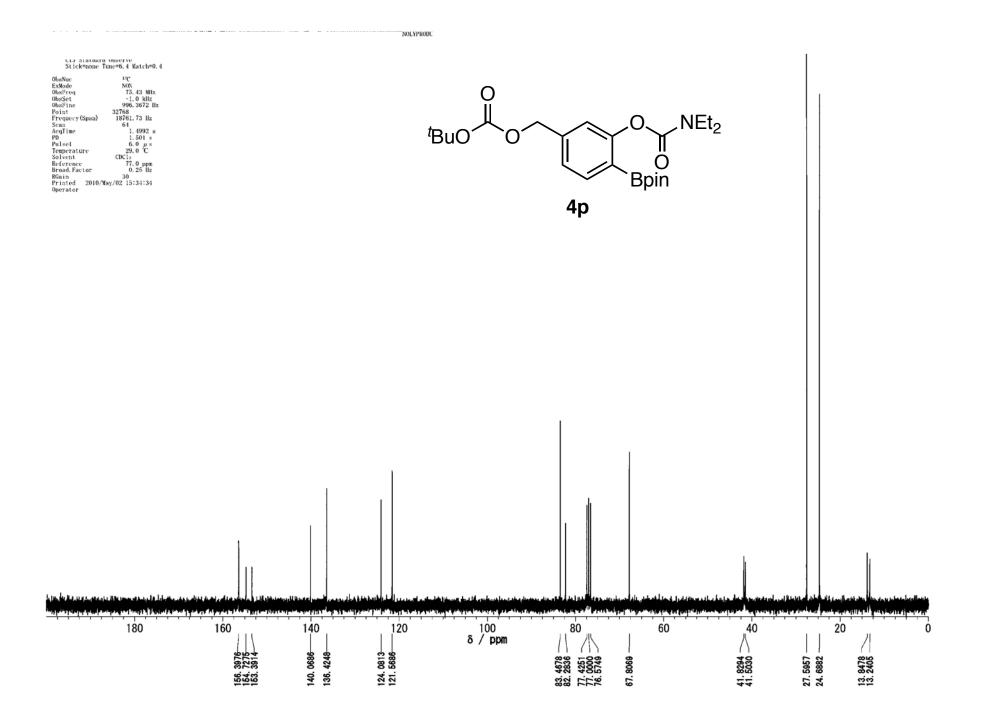






Original File:      Date    Feb 25 10      Comment    Cl3 Statdard Observe      Stick=none Tun=6.4 Match=0.4      ObsNuc    NC      ObsPreq    75.43 WHz      ObsPreq    1.0 kHz      Point    32768      Preptucey(Span)    13761.73 Hz      Scan    1.4 992 s      PD    1.5 Poil s      Pulsel    6.0 $\mu$ s      Pulsel    6.0 $\mu$ s      Broad Factor    0.2 863 Hz      Road Factor    0.2 863 Hz      Roatin    30      Printed    2010/May/02 15:27:38      Operator    15.27:38	MeO	O NEt <sub>2</sub> O Bpin	
		83. 4223 777. 005 76. 5749 76. 5749 7749 7757 7757 7757 7757 7757 7757	441:8294 441:8294 141:8294 141:8294 141:8294 141:8294 132:8906 132:89





S97

