

**Supporting Information for:**

**Pd-Catalyzed C-H Fluorination with Nucleophilic Fluoride**

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## General Procedures

NMR spectra were obtained on a Varian vnmrs 700 (699.76 MHz for  $^1\text{H}$ ; 175.95 MHz for  $^{13}\text{C}$ ), Varian vnmrs 500 (500.03 MHz for  $^1\text{H}$ ; 125.72 MHz for  $^{13}\text{C}$ ; 470.56 MHz for  $^{19}\text{F}$ ) or Varian Inova 400 (399.96 MHz for  $^1\text{H}$ ; 100.71 MHz for  $^{13}\text{C}$ ; 375.94 MHz for  $^{19}\text{F}$ ) spectrometer.  $^1\text{H}$  NMR chemical shifts are reported in parts per million (ppm), with the residual solvent peak used as an internal reference. Multiplicities are reported as follows: singlet (s), doublet (d), doublet of doublets (dd), doublet of doublets of doublets (ddd), doublet of triplets (dt), triplet (t), and multiplet (m). IR spectra were obtained on a Perkin-Elmer Spectrum BX FT-IR spectrometer.

## Materials and Methods

The substrates 8-methyl-5-nitroquinoline,<sup>1</sup> 5-fluoro-8-methylquinoline,<sup>1</sup> 5-bromo-8-methylquinoline,<sup>1</sup> 5-methoxy-8-methylquinoline,<sup>1</sup> 5-iodo-8-methylquinoline,<sup>2</sup> 8-methylquinoline-5-carbonitrile,<sup>2</sup> methyl 8-methylquinoline-5-carboxylate,<sup>3</sup> and 5,8-dimethylquinoline<sup>4</sup> were prepared according to literature procedures. The substrate 8-methyl-5-phenylquinoline was prepared by Pd-catalyzed Suzuki coupling between phenylboronic acid and 5-bromo-8-methylquinoline.<sup>5</sup> The substrate 7-bromo-8-methylquinoline was prepared by the reaction of 3-bromo-2-methylaniline with glycerine.<sup>4</sup> 8-Methylquinoline was obtained from Oakwood Products and used as received.  $\text{PhI}(\text{OPiv})_2$  was prepared by the reaction of  $\text{PhI}(\text{OAc})_2$  with trimethylacetic acid according to a literature procedure.<sup>6</sup> Iodosobenzene was prepared by the reaction of  $\text{PhI}(\text{OAc})_2$  with sodium hydroxide.<sup>7</sup>  $\text{PhI}(\text{OTs})(\text{OH})$  and  $\text{CsF}$  were obtained from Matrix Scientific.  $\text{PhI}(\text{O}_2\text{CCF}_3)_2$ ,  $\text{PhI}(\text{OAc})_2$ ,  $\text{MesI}(\text{OAc})_2$  were obtained from Acros, Sigma-Aldrich, and TCI America, respectively. All other oxidants were synthesized from  $\text{PhI}(\text{OAc})_2$  with the corresponding acid.<sup>6</sup>  $\text{Pd}(\text{OAc})_2$ ,  $\text{AgF}$ ,  $\text{AgOTf}$ , and  $\text{MgSO}_4$  were obtained from Pressure Chemical, Oakwood Products, SynQuest Labs, and Fisher Scientific, respectively. All were used as received unless otherwise noted. Dichloromethane and other solvents were obtained from Fisher Scientific and used as received. Flash chromatography was performed on Dynamic Adsorbents silica gel 60A (0.032–0.063 mm particle size, 230–400 mesh) or Biotage silica gel (0.050 mm particle size) and thin layer chromatography was performed on EMD Chemicals Inc. plates pre-coated with silica gel 60 F<sub>254</sub>.

<sup>1</sup> Evans et al. *Tetrahedron* **2005**, 61, 9696.

<sup>2</sup> Tochilkin, A. I. et al. *Chem. Heterocycl. Compd.* (Russ) **1983**, 1373.

<sup>3</sup> Tochilkin, A. I. et al. *Chem. Heterocycl. Compd.* (Russ) **1982**, 785.

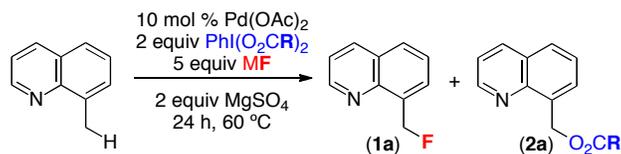
<sup>4</sup> O'Murchu, C. *Synthesis* **1989**, 11, 880.

<sup>5</sup> Leadbeater, N. E.; Griffiths, C. *Tetrahedron Lett.* **2000**, 41 2487.

<sup>6</sup> Stang, P. J.; Boehshar, M.; Wingert, H.; Kitamura, T. *J. Am. Chem. Soc.* **1988**, 110, 3272.

<sup>7</sup> Blackmond, D. G.; Hodnett, N. S.; Lloyd-Jones, G. C. *J. Am. Chem. Soc.* **2006**, 128, 7450.

## Procedures for Reactions in Table 1



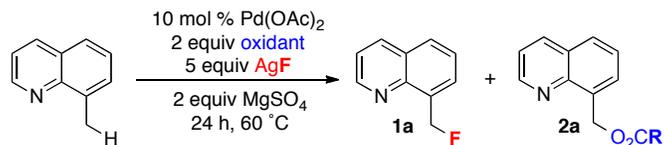
### Non-pre-stirred conditions (entries 1-3):

On the benchtop, PhI(O<sub>2</sub>CR)<sub>2</sub> (0.38 mmol, 2.0 equiv), MgSO<sub>4</sub> (45.9 mg, 0.38 mmol, 2.0 equiv), Pd(OAc)<sub>2</sub> (4.3 mg, 0.02 mmol, 0.10 equiv), and AgF (121 mg, 0.96 mmol, 5.0 equiv) were weighed into a 4 mL vial. 8-Methylquinoline (27.4 mg, 0.19 mmol, 1.0 equiv) and solvent (1.9 mL) were added, and the vial was sealed with a Teflon-lined cap (with Teflon tape covering the threads of the vial). This mixture was heated at 60 °C for 24 h. GC yields were obtained by analysis of the crude reaction mixture using hexadecane as a standard.

### Pre-stirred conditions (entries 4-8):

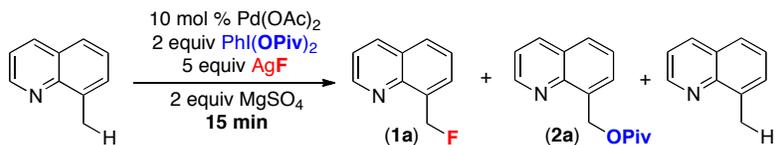
On the benchtop, PhI(OPiv)<sub>2</sub> (154 mg, 0.38 mmol, 2.0 equiv) and MF (0.96 mmol, 5.0 equiv) were weighed into a 4 mL vial, and DCM (1.9 mL) was added. The vial was sealed with a Teflon-lined cap (with Teflon tape covering the threads of the vial), and this mixture was heated at 60 °C for 1 h. The vial was removed from the heat and allowed to cool to rt (~5 min). Then Pd(OAc)<sub>2</sub> (4.3 mg, 0.02 mmol, 0.10 equiv), MgSO<sub>4</sub> (45.9 mg, 0.38 mmol, 2.0 equiv), and 8-methylquinoline (27.4 mg, 0.19 mmol, 1.0 equiv) were added. The vial was sealed with a Teflon-lined cap and heated at 60 °C for an additional 24 h. GC yields were obtained by analysis of the crude reaction mixture using hexadecane as a standard.

### Data for Oxidant Screen



Oxidant	Yield of 1a
PhI(OPiv) <sub>2</sub>	60%
PhI[O <sub>2</sub> C( <i>p</i> -MeOC <sub>6</sub> H <sub>4</sub> )] <sub>2</sub>	33%
PhI[O <sub>2</sub> C( <i>p</i> -PhOC <sub>6</sub> H <sub>4</sub> )] <sub>2</sub>	32%
PhI(O <sub>2</sub> CPh) <sub>2</sub>	29%
PhI[O <sub>2</sub> C( <i>p</i> -AcOC <sub>6</sub> H <sub>4</sub> )] <sub>2</sub>	25%
MesI(O <sub>2</sub> CPh) <sub>2</sub>	25%
PhI[O <sub>2</sub> C( <i>p</i> -ClC <sub>6</sub> H <sub>4</sub> )] <sub>2</sub>	24%
PhI[O <sub>2</sub> C( <i>p</i> -FC <sub>6</sub> H <sub>4</sub> )] <sub>2</sub>	22%
PhI[O <sub>2</sub> C( <i>p</i> -PhC <sub>6</sub> H <sub>4</sub> )] <sub>2</sub>	18%
MesI(OAc) <sub>2</sub>	15%
PhI(OAc) <sub>2</sub>	13%
PhI[O <sub>2</sub> C( <i>p</i> -CF <sub>3</sub> C <sub>6</sub> H <sub>4</sub> )] <sub>2</sub>	12%
PhI[O <sub>2</sub> C( <i>p</i> -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> )] <sub>2</sub>	11%
PhI[O <sub>2</sub> C( <i>p</i> -CNC <sub>6</sub> H <sub>4</sub> )] <sub>2</sub>	9%
PhI[O <sub>2</sub> C( <i>p</i> -BrC <sub>6</sub> H <sub>4</sub> )] <sub>2</sub>	7%
PhI(OTs)(OH)	3%
PhI(O <sub>2</sub> CCF <sub>3</sub> ) <sub>2</sub>	2%
PhI(O <sub>2</sub> CC <sub>9</sub> F <sub>19</sub> ) <sub>2</sub>	2%
PhI=O	1%

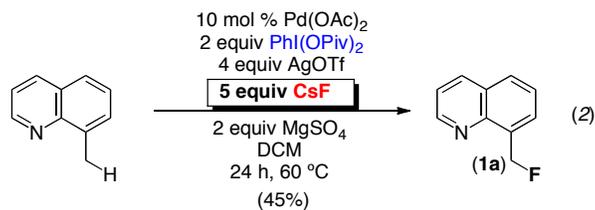
### Data for Accelerated Reaction Times



Temperature (°C)	Yield of 1a	Yield of 2a	Yield of Starting Material
60	29%	8%	45%
70	32%	26%	31%
80	41%	32%	19%
90	42%	43%	6%
100	39%	45%	4%

On the benchtop, PhI(OPiv)<sub>2</sub> (154 mg, 0.38 mmol, 2.0 equiv), MgSO<sub>4</sub> (45.9 mg, 0.38 mmol, 2.0 equiv), Pd(OAc)<sub>2</sub> (4.3 mg, 0.02 mmol, 0.10 equiv), and AgF (121 mg, 0.96 mmol, 5.0 equiv) were weighed into a 4 mL vial. 8-Methylquinoline (27.4 mg, 0.19 mmol, 1.0 equiv) and DCM (1.9 mL) were added and the vial was sealed with a Teflon-lined cap (with Teflon tape covering the threads of the vial). This mixture was heated for 15 min at the appropriate temperature and the cooled to rt. GC yields were obtained by analysis of the crude reaction mixture using hexadecane as a standard.

## Procedure for CsF Fluorination



On the benchtop, PhI(OPiv)<sub>2</sub> (154 mg, 0.38 mmol, 2.0 equiv), CsF (145 mg, 0.96 mmol, 5.0 equiv), and AgOTf (196 mg, 0.76 mmol, 4.0 equiv) were weighed into a 4 mL vial, and DCM (1.9 mL) was added. The vial was sealed with a Teflon-lined cap (with Teflon tape covering the threads of the vial), and this mixture was heated at 60 °C for 1 h. The vial was removed from the heat and allowed to cool to rt (~5 min). Then Pd(OAc)<sub>2</sub> (4.3 mg, 0.02 mmol, 0.10 equiv), MgSO<sub>4</sub> (45.9 mg, 0.38 mmol, 2.0 equiv), and 8-methylquinoline (27.4 mg, 0.19 mmol, 1.0 equiv) were added. The vial was sealed with a Teflon-lined cap and heated at 60 °C for an additional 24 h. GC yields were obtained by analysis of the crude reaction mixture using hexadecane as a standard.

## Mechanistic Experiments

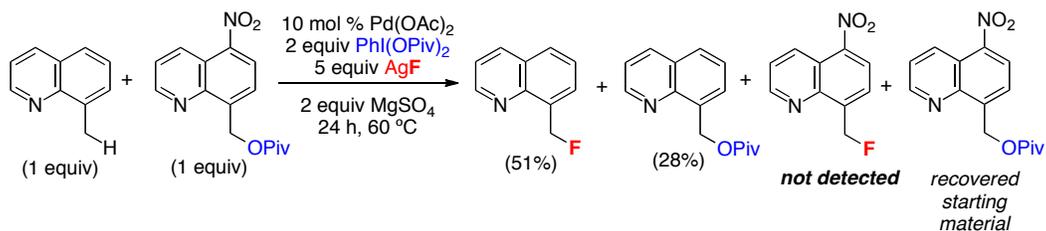
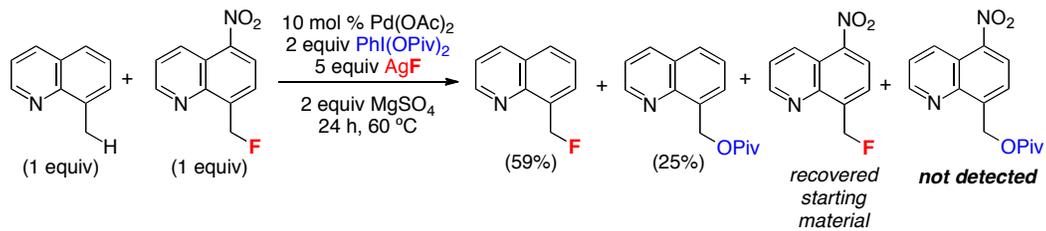
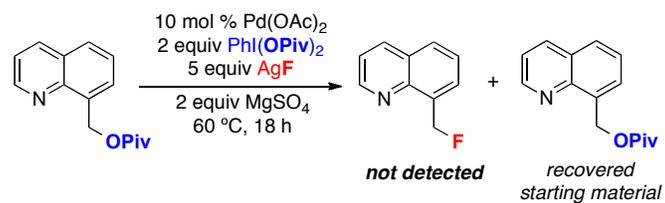
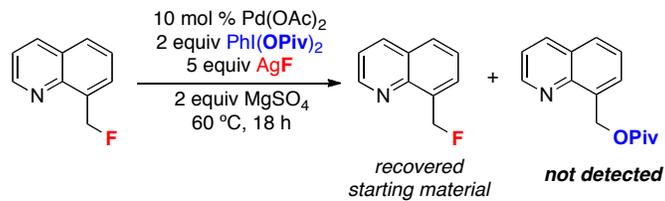
### Observation of *p*-MeC<sub>6</sub>H<sub>4</sub>IF<sub>2</sub> by <sup>19</sup>F NMR Spectroscopy:

On the benchtop, *p*-MeC<sub>6</sub>H<sub>4</sub>I(OPiv)<sub>2</sub> (21.0 mg, 0.05 mmol, 1.0 equiv) and AgF (15.9 mg, 0.13 mmol, 2.5 equiv) were weighed into a 4 mL vial, and CD<sub>2</sub>Cl<sub>2</sub> (0.5 mL) was added. The vial was sealed with a Teflon-lined cap (with Teflon tape covering the threads of the vial), and this mixture was heated at 60 °C for 30 min. The reaction mixture was cooled to rt and was then transferred to an NMR tube for analysis. The <sup>19</sup>F NMR spectrum of this crude mixture is shown on p. S30 and that for an authentic sample of *p*-MeC<sub>6</sub>H<sub>4</sub>IF<sub>2</sub> is shown on p. S31.

### Testing *p*-MeC<sub>6</sub>H<sub>4</sub>IF<sub>2</sub> as fluorination reagent:

*p*-MeC<sub>6</sub>H<sub>4</sub>IF<sub>2</sub> (49.2 mg, 0.19 mmol, 2.0 equiv, stored in glovebox, but reaction was set up on the benchtop), MgSO<sub>4</sub> (23.1 mg, 0.19 mmol, 2.0 equiv), and Pd(OAc)<sub>2</sub> (2.2 mg, 0.01 mmol, 0.10 equiv) were weighed into a 4 mL vial. Dry DCM (0.96 mL) was added and the vial was sealed with a Teflon-lined cap (with Teflon tape covering the threads of the vial) and removed from the glovebox. 8-Methylquinoline (13.8 mg, 0.10 mmol, 1.0 equiv) was added through the septum vial cap, and the mixture was heated at 60 °C for 24 h. GC yields were obtained by analysis of the crude reaction mixture using hexadecane as a standard. Notably, nearly identical results were obtained when this reaction was set up in the glovebox.

## Control Experiments



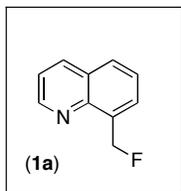
## Procedures/Spectral Characterization of Products in Table 2

### General Procedure

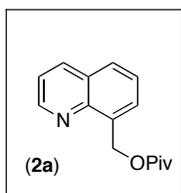
On the benchtop,  $\text{PhI}(\text{OPiv})_2$  (650 mg, 1.60 mmol, 2.0 equiv) and AgF (508 mg, 4.00 mmol, 5.0 equiv) were weighed into a 20 mL vial, and solvent (8.0 mL) was added. The vial was sealed with a Teflon-lined cap (with Teflon tape covering the threads of the vial), and this mixture was heated at 60 °C for 1 h. The vial was removed from the heat and allowed to cool to rt (~10 min). Then  $\text{Pd}(\text{OAc})_2$  (18 mg, 0.08 mmol, 0.10 equiv),  $\text{MgSO}_4$  (193 mg, 1.60 mmol, 2.0 equiv), and substrate (0.80 mmol, 1.0 equiv) were added. The vial was sealed with a Teflon-lined cap and heated at 60 °C for an additional 16-28 h. GC yields were obtained by analysis of the crude reaction mixture using hexadecane as a standard. The reaction mixture was then filtered through a plug of Celite, the plug was washed with  $\text{CH}_2\text{Cl}_2$  (100 mL), and the filtrate was concentrated under vacuum. The resulting crude product was purified by chromatography on silica gel.

## 8-Methylquinoline

8-Methylquinoline was obtained from Oakwood Products and used as received.



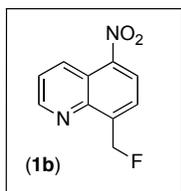
8-Methylquinoline (115 mg, 0.80 mmol, 1.0 equiv) was used as the substrate. The solvent was dichloromethane (8.0 mL), and the reaction was heated for 16 h. GC analysis of the crude reaction mixture showed 67% yield of **1a** and 16% yield of **2a**. The crude brown oil was purified by chromatography on silica gel with a gradient of 0%-8% EtOAc in CH<sub>2</sub>Cl<sub>2</sub>. Product **1a** was obtained as a viscous yellow oil (63.0 mg, 49% isolated yield, R<sub>f</sub> = 0.49 in 96% CH<sub>2</sub>Cl<sub>2</sub>/4% EtOAc). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 8.93 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.18 (dd, *J* = 8.2, 1.8 Hz, 1H), 7.83-7.81 (multiple peaks, 2H), 7.58 (t, *J* = 7.7 Hz, 1H), 7.44 (dd, *J* = 8.3, 4.2 Hz, 1H), 6.15 (d, *J*<sub>HF</sub> = 48 Hz, 2H). <sup>13</sup>C{<sup>1</sup>H} NMR (175 MHz, CDCl<sub>3</sub>): δ 150.1, 145.7 (d, *J*<sub>CF</sub> = 3.3 Hz), 136.4, 134.9 (d, *J*<sub>CF</sub> = 16 Hz), 128.4 (d, *J*<sub>CF</sub> = 2.1 Hz), 128.2, 127.6 (d, *J*<sub>CF</sub> = 10 Hz), 126.5, 121.6, 82.0 (d, *J*<sub>CF</sub> = 165 Hz). <sup>19</sup>F NMR (470 MHz, CDCl<sub>3</sub>): δ -219.2 (t, *J*<sub>FH</sub> = 48 Hz). IR (thin film, CH<sub>2</sub>Cl<sub>2</sub>): 2924, 1596, 1581, 1500, 1353 cm<sup>-1</sup>. HRMS [M+H]<sup>+</sup> Calcd for C<sub>10</sub>H<sub>8</sub>NF: 161.0641; Found: 161.0638.



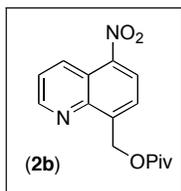
Product **2a** was also obtained from this transformation as a viscous clear oil (21.6 mg, 11% isolated yield, R<sub>f</sub> = 0.29 in 96% CH<sub>2</sub>Cl<sub>2</sub>/4% EtOAc). <sup>1</sup>H NMR (700 MHz, CDCl<sub>3</sub>): δ 8.94 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.17 (dd, *J* = 8.3, 1.7 Hz, 1H), 7.78 (d, *J* = 8.1 Hz, 1H), 7.74 (d, *J* = 6.8 Hz, 1H), 7.54 (app. t, *J* = 7.7 Hz, 1H), 7.43 (dd, *J* = 8.3, 4.2 Hz, 1H), 5.87 (s, 2H), 1.28 (s, 9H). <sup>13</sup>C{<sup>1</sup>H} NMR (175 MHz, CDCl<sub>3</sub>): δ 178.6, 149.9, 146.1, 136.4, 135.1, 128.3, 127.8, 127.6, 126.4, 121.5, 62.9, 39.2, 27.5. IR (thin film, CH<sub>2</sub>Cl<sub>2</sub>): 2972, 1726, 1500, 1282, 1149 cm<sup>-1</sup>. HRMS [M+H]<sup>+</sup> Calcd for C<sub>15</sub>H<sub>17</sub>NO<sub>2</sub>: 244.1332; Found: 244.1332.

## 8-Methyl-5-nitroquinoline

8-Methyl-5-nitroquinoline (**S-b**) was prepared according to a literature procedure.<sup>8</sup> NMR data matched that reported in the literature. <sup>1</sup>H and <sup>13</sup>C NMR spectra for this substrate are provided below.



8-Methyl-5-nitroquinoline (151 mg, 0.80 mmol, 1.0 equiv) was used as the substrate. The solvent was dichloromethane (8.0 mL), and the reaction was heated for 24 h. GC analysis of the crude reaction mixture showed 61% yield of **1b** and 6% yield of **2b**. The orange solid was purified by chromatography on silica gel with a gradient of 0%-2% EtOAc in CH<sub>2</sub>Cl<sub>2</sub>. Product **1b** was obtained as a light yellow solid (67.9 mg, 41% isolated yield, R<sub>f</sub> = 0.55 in 99% CH<sub>2</sub>Cl<sub>2</sub>/1% EtOAc, mp = 143.9-148.6 °C). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 9.06 (dd, J = 8.8, 1.7 Hz, 1H), 8.99 (dd, J = 4.2, 1.7 Hz, 1H), 8.44 (d, J = 8.1 Hz, 1H), 7.93 (d, J = 8.1 Hz, 1H), 7.69 (dd, J = 8.8, 4.2 Hz, 1H), 6.22 (d, J<sub>HF</sub> = 47 Hz, 2H). <sup>13</sup>C{<sup>1</sup>H} NMR (175 MHz, CDCl<sub>3</sub>): δ 150.8, 145.2, 144.8 (d, J<sub>CF</sub> = 3.5 Hz), 143.3 (d, J<sub>CF</sub> = 18 Hz), 132.5, 124.8, 124.4, 123.9 (d, J<sub>CF</sub> = 13 Hz), 120.9, 81.8 (d, J<sub>CF</sub> = 170 Hz). <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>): δ -228.1 (t, J<sub>FH</sub> = 47 Hz). IR (thin film, CH<sub>2</sub>Cl<sub>2</sub>): 3112, 1518, 1335 cm<sup>-1</sup>. HRMS [M+H]<sup>+</sup> Calcd for C<sub>10</sub>H<sub>7</sub>N<sub>2</sub>O<sub>2</sub>F: 206.0491; Found: 206.0491.



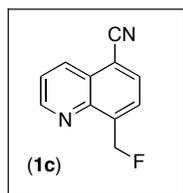
Product **2b** was also obtained from this transformation as a yellow solid (22.2 mg, 10% isolated yield, R<sub>f</sub> = 0.40 in 99% CH<sub>2</sub>Cl<sub>2</sub>/1% EtOAc, mp = 103.4-105.9 °C). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 9.04-9.01 (multiple peaks, 2H), 8.38 (d, J = 8.1 Hz, 1H), 7.79 (d, J = 8.1 Hz, 1H), 7.67 (dd, J = 8.7, 4.3 Hz, 1H), 5.91 (s, 2H), 1.32 (s, 9H). <sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 178.2, 150.8, 145.6, 145.0, 143.3, 132.4, 124.6, 124.6, 124.3, 121.1, 62.8, 39.3, 27.5. IR (thin film, CH<sub>2</sub>Cl<sub>2</sub>):

<sup>8</sup> Evans et al. *Tetrahedron* **2005**, 61, 9696.

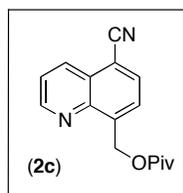
2975, 1730, 1522, 1335, 1145  $\text{cm}^{-1}$ . HRMS  $[\text{M}+\text{H}]^+$  Calcd for  $\text{C}_{15}\text{H}_{16}\text{N}_2\text{O}_4$ : 289.1183; Found: 289.1191.

## 8-Methylquinoline-5-carbonitrile

8-Methylquinoline-5-carbonitrile (**S-c**) was prepared according to a literature procedure.<sup>9</sup> <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 9.05 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.52 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.88 (d, *J* = 7.3 Hz, 1H), 7.63-7.60 (multiple peaks, 2H), 2.88 (s, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 150.9, 146.8, 144.6, 133.8, 133.0, 129.1, 127.9, 123.2, 117.3, 108.3, 18.9. Mp = 108.5-111.3 °C. IR (thin film, CH<sub>2</sub>Cl<sub>2</sub>): 2928, 2222, 1501 cm<sup>-1</sup>. HRMS [M+H]<sup>+</sup> Calcd for C<sub>11</sub>H<sub>8</sub>N<sub>2</sub>: 169.0760; Found: 169.0761.



8-Methylquinoline-5-carbonitrile (135 mg, 0.80 mmol, 1.0 equiv) was used as the substrate. The solvent was dichloromethane (8.0 mL), and the reaction was heated for 16 h. GC analysis of the crude reaction mixture showed 67% yield of **1c** and 12% yield of **2c**. The crude mixture was purified by chromatography on silica gel with a gradient of 0%-4% EtOAc in CH<sub>2</sub>Cl<sub>2</sub>. Product **1c** was obtained as a tan solid (105 mg, 70% isolated yield, *R*<sub>f</sub> = 0.61 in 98% CH<sub>2</sub>Cl<sub>2</sub>/2% EtOAc, mp = 149.8-151.0 °C). <sup>1</sup>H NMR (700 MHz, CDCl<sub>3</sub>): δ 9.01 (dd, *J* = 4, 1.5 Hz, 1H), 8.57 (dd, *J* = 8.4, 1.5 Hz, 1H), 8.03 (d, *J* = 7.4 Hz, 1H), 7.90 (d, *J* = 7.4 Hz, 1H), 7.66 (dd, *J* = 8.4, 4 Hz, 1H), 6.21 (d, *J*<sub>HF</sub> = 47 Hz, 2H). <sup>13</sup>C{<sup>1</sup>H} NMR (100 MHz, CDCl<sub>3</sub>): δ 151.3, 144.3 (d, *J*<sub>CF</sub> = 4.0 Hz), 141.7 (d, *J*<sub>CF</sub> = 18 Hz), 133.8, 133.1, 127.5, 125.1 (d, *J*<sub>CF</sub> = 12 Hz), 123.7, 116.8, 110.1, 81.5 (d, *J*<sub>CF</sub> = 169 Hz). <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>): δ -227.5 (m, *J*<sub>FH</sub> = 47 Hz). IR (thin film, CH<sub>2</sub>Cl<sub>2</sub>): 2227, 1500, 1440, 1353 cm<sup>-1</sup>. HRMS [M+H]<sup>+</sup> Calcd for C<sub>11</sub>H<sub>7</sub>N<sub>2</sub>F: 186.0593; Found: 186.0592.



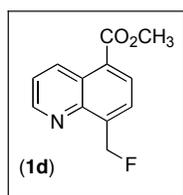
Product **2c** was also obtained from this transformation as a white solid (19.5 mg, 9% isolated yield, *R*<sub>f</sub> = 0.41 in 98% CH<sub>2</sub>Cl<sub>2</sub>/2% EtOAc, mp = 94.4-97.9 °C). <sup>1</sup>H NMR (700 MHz, CDCl<sub>3</sub>): δ 9.04 (m, 1H), 8.54 (dd, *J* = 8.4, 1.5 Hz, 1H), 7.98 (d, *J* = 7.5 Hz, 1H), 7.76 (dd, *J* = 7.5, 0.7 Hz,

<sup>9</sup> Tochilkin, A. I. et al. *Chem. Heterocycl. Compd.* (Russ) **1983**, 1373.

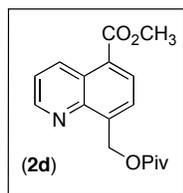
1H), 7.64 (dd,  $J = 8.4, 4.2$  Hz, 1H), 5.89 (s, 2H), 1.31 (s, 9H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (175 MHz,  $\text{CDCl}_3$ ):  $\delta$  178.3, 151.3, 145.2, 141.8, 133.7, 133.0, 127.7, 125.8, 123.6, 116.9, 109.8, 62.4, 39.2, 27.5. IR (thin film,  $\text{CH}_2\text{Cl}_2$ ): 2975, 2227, 1731, 1501, 1158  $\text{cm}^{-1}$ . HRMS  $[\text{M}+\text{H}]^+$  Calcd for  $\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}_2$ : 269.1285; Found: 269.1280.

### Methyl 8-methylquinoline-5-carboxylate

Methyl 8-methylquinoline-5-carboxylate (**S-d**) was prepared according to a literature procedure.<sup>10</sup> <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 9.38 (dd, *J* = 8.8, 1.7 Hz, 1H), 8.97 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.20 (d, *J* = 7.6 Hz, 1H), 7.59 (d, *J* = 7.6 Hz, 1H), 7.52 (dd, *J* = 8.8, 4.2 Hz, 1H), 3.99 (s, 3H), 2.87 (s, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 167.3, 149.5, 147.4, 143.8, 134.8, 131.0, 128.5, 127.2, 124.9, 122.5, 52.4, 19.2. Mp = 55.4-57.8 °C. IR (thin film, CH<sub>2</sub>Cl<sub>2</sub>): 2955, 1712, 1505 cm<sup>-1</sup>. HRMS [M+H]<sup>+</sup> Calcd for C<sub>12</sub>H<sub>11</sub>NO<sub>2</sub>: 202.0863; Found: 202.0865.



Methyl 8-methylquinoline-5-carboxylate (161 mg, 0.80 mmol, 1.0 equiv) was used as the substrate. The solvent was dichloromethane (8.0 mL), and the reaction was heated for 24 h. GC analysis of the crude reaction mixture showed 59% yield of **1d** and 10% yield of **2d**. The crude mixture was purified by chromatography on silica gel with a gradient of 1%-8% EtOAc in CH<sub>2</sub>Cl<sub>2</sub>. Product **1d** was obtained as an off-white solid (104 mg, 59% isolated yield, R<sub>f</sub> = 0.52 in 96% CH<sub>2</sub>Cl<sub>2</sub>/4% EtOAc, mp = 94.0-94.8 °C). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 9.38 (dd, *J* = 8.8, 1.5 Hz, 1H), 8.92 (dd, *J* = 4.2, 1.5 Hz, 1H), 8.33 (d, *J* = 7.6 Hz, 1H), 7.86 (d, *J* = 7.6 Hz, 1H), 7.55 (dd, *J* = 8.8, 4.2 Hz, 1H), 6.20 (d, *J*<sub>HF</sub> = 47 Hz, 2H), 4.01 (s, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (175 MHz, CDCl<sub>3</sub>): δ 167.5, 149.9, 145.1 (d, *J*<sub>CF</sub> = 3.3 Hz), 140.9 (d, *J*<sub>CF</sub> = 18 Hz), 134.8, 130.9, 126.8, 126.5 (d, *J*<sub>CF</sub> = 1.4 Hz), 124.6 (d, *J*<sub>CF</sub> = 12 Hz), 122.9, 81.5 (d, *J*<sub>CF</sub> = 167 Hz), 52.6. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>): δ -226.3 (t, *J*<sub>FH</sub> = 47 Hz). IR (thin film, CH<sub>2</sub>Cl<sub>2</sub>): 2955, 1714, 1506 cm<sup>-1</sup>. HRMS [M+H]<sup>+</sup> Calcd for C<sub>12</sub>H<sub>10</sub>NO<sub>2</sub>F: 220.0768; Found: 220.0772.



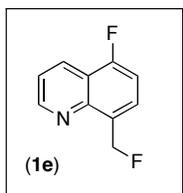
Product **2d** was also obtained from this reaction as an off-white solid (25.4 mg, 11% isolated yield, R<sub>f</sub> = 0.27 in 96% CH<sub>2</sub>Cl<sub>2</sub>/4% EtOAc, mp = 78.8-84.3 °C). <sup>1</sup>H NMR (700 MHz, CDCl<sub>3</sub>): δ

<sup>10</sup> Tochilkin, A. I. et al. *Chem. Heterocycl. Compd.* (Russ) **1982**, 785.

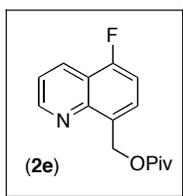
9.36 (dd,  $J = 8.7, 1.7$  Hz, 1H), 8.95 (dd,  $J = 4.0, 1.7$  Hz, 1H), 8.29 (d,  $J = 7.7$  Hz, 1H), 7.73 (dd,  $J = 7.7, 0.9$  Hz, 1H), 7.54 (dd,  $J = 8.7, 4.0$  Hz, 1H), 5.90 (s, 2H), 4.01 (s, 3H), 1.31 (s, 9H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (175 MHz,  $\text{CDCl}_3$ ):  $\delta$  178.4, 167.1, 149.9, 145.9, 141.0, 134.7, 130.7, 127.0, 126.3, 125.3, 122.8, 63.0, 52.5, 39.2, 27.6. IR (thin film,  $\text{CH}_2\text{Cl}_2$ ): 2973, 1717, 1504, 1159  $\text{cm}^{-1}$ . HRMS  $[\text{M}+\text{H}]^+$  Calcd for  $\text{C}_{17}\text{H}_{19}\text{NO}_4$ : 302.1387; Found: 302.1397.

## 5-Fluoro-8-methylquinoline

5-Fluoro-8-methylquinoline (**S-e**) was prepared according to a literature procedure.<sup>11</sup> NMR data matched that reported in the literature. <sup>1</sup>H and <sup>13</sup>C NMR spectra for this substrate are provided below.



5-Fluoro-8-methylquinoline (129 mg, 0.80 mmol, 1.0 equiv) was used as the substrate. The solvent was dichloromethane (8.0 mL), and the reaction was heated for 16 h. GC analysis of the crude reaction mixture showed 44% yield of **1e** and 25% yield of **2e**. The crude mixture was purified by chromatography on silica gel with a gradient of 1%-8% EtOAc in CH<sub>2</sub>Cl<sub>2</sub>. Product **1e** was obtained as a tan solid (43.3 mg, 30% isolated yield, R<sub>f</sub> = 0.49 in 96% CH<sub>2</sub>Cl<sub>2</sub>/4% EtOAc, mp = 52.9-57.0 °C). <sup>1</sup>H NMR (700 MHz, CDCl<sub>3</sub>): δ 9.00 (m, 1H), 8.47 (d, J = 8.5 Hz, 1H), 7.78 (t, J = 6.9 Hz, 1H), 7.53 (dd, J = 8.5, 4.2 Hz, 1H), 7.27 (m, 1H), 6.08 (d, J<sub>HF</sub> = 48 Hz, 2H). <sup>13</sup>C{<sup>1</sup>H} NMR (175 MHz, CDCl<sub>3</sub>): δ 158.2 (dd, J<sub>CF</sub> = 256, 2.6 Hz), 151.0, 146.3 (t, J<sub>CF</sub> = 3.4 Hz), 130.9 (dd, J<sub>CF</sub> = 17, 4.4 Hz), 129.7 (d, J<sub>CF</sub> = 4.8 Hz), 128.0 (t, J<sub>CF</sub> = 9.6 Hz), 121.7 (d, J<sub>CF</sub> = 2.6 Hz), 119.1 (d, J<sub>CF</sub> = 16 Hz), 120.0 (d, J<sub>CF</sub> = 20 Hz), 81.5 (d, J<sub>CF</sub> = 165 Hz). <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>): δ -121.9, -216.0 (t, J<sub>FH</sub> = 48 Hz). IR (thin film, CH<sub>2</sub>Cl<sub>2</sub>): 2917, 1597 cm<sup>-1</sup>. HRMS [M+H]<sup>+</sup> Calcd for C<sub>10</sub>H<sub>7</sub>NF<sub>2</sub>: 180.0619; Found: 180.0616.



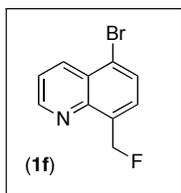
Product **2e** was also obtained from this reaction as a clear oil (50.5 mg, 24% isolated yield, R<sub>f</sub> = 0.27 in 96% CH<sub>2</sub>Cl<sub>2</sub>/4% EtOAc). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 8.97 (dd, J = 4.2, 2 Hz, 1H), 8.43 (dd, J = 8.4, 2 Hz, 1H), 7.68 (m, 1H), 7.49 (dd, J = 8.4, 4.2 Hz, 1H), 7.21 (m, 1H), 5.78 (s, 2H), 1.25 (s, 9H). <sup>13</sup>C{<sup>1</sup>H} NMR (175 MHz, CDCl<sub>3</sub>): δ 178.6, 157.7 (d, J<sub>CF</sub> = 255 Hz), 150.7, 146.7 (d, J<sub>CF</sub> = 3.3 Hz), 131.1 (d, J<sub>CF</sub> = 4.8 Hz), 129.6 (d, J<sub>CF</sub> = 4.8 Hz), 127.8 (d, J<sub>CF</sub> = 8.8 Hz), 121.5 (d,

<sup>11</sup> Evans et al. *Tetrahedron* **2005**, 61, 9696.

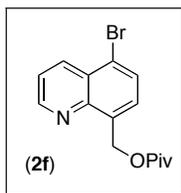
$J_{CF} = 2.8$  Hz), 119.1 (d,  $J_{CF} = 16$  Hz), 109.8 (d,  $J_{CF} = 20$  Hz), 62.5, 39.2, 27.5. IR (thin film,  $\text{CH}_2\text{Cl}_2$ ): 2974, 1728, 1156  $\text{cm}^{-1}$ . HRMS  $[\text{M}+\text{H}]^+$  Calcd for  $\text{C}_{15}\text{H}_{16}\text{NO}_2\text{F}$ : 262.1238; Found: 262.1237.

## 5-Bromo-8-methylquinoline

5-Bromo-8-methylquinoline (**S-f**) was prepared according to a literature procedure.<sup>12</sup> NMR data matched that reported in the literature. <sup>1</sup>H and <sup>13</sup>C NMR spectra for this substrate are provided below.



5-Bromo-8-methylquinoline (178 mg, 0.80 mmol, 1.0 equiv) was used as the substrate. The solvent was dichloromethane (8.0 mL), and the reaction was heated for 16 h. GC analysis of the crude reaction mixture showed 53% yield of **1f** and 19% yield of **2f**. The crude mixture was purified by chromatography on silica gel with a gradient of 0%-4% EtOAc in CH<sub>2</sub>Cl<sub>2</sub>. Product **1f** was obtained as a white solid (83.8 mg, 44% isolated yield, R<sub>f</sub> = 0.65 in 98% CH<sub>2</sub>Cl<sub>2</sub>/2% EtOAc, mp = 67.7-69.0 °C). <sup>1</sup>H NMR (700 MHz, CDCl<sub>3</sub>): δ 8.93 (dd, J = 4.2, 1.6 Hz, 1H), 8.57 (dd, J = 8.6, 1.6 Hz, 1H), 7.87 (d, J = 7.7 Hz, 1H), 7.69 (d, J = 7.7 Hz, 1H), 7.55 (dd, J = 8.6, 4.2 Hz, 1H), 6.10 (d, J<sub>HF</sub> = 47 Hz, 2H). <sup>13</sup>C{<sup>1</sup>H} NMR (175 MHz, CDCl<sub>3</sub>): δ 150.5, 146.0, 136.1, 135.1 (d, J<sub>CF</sub> = 17 Hz), 130.3, 127.6 (d, J<sub>CF</sub> = 11 Hz), 127.5, 122.7, 122.2, 81.7 (d, J<sub>CF</sub> = 167 Hz). <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>): δ -221.4 (t, J<sub>FH</sub> = 47 Hz). IR (thin film, CH<sub>2</sub>Cl<sub>2</sub>): 1569, 1496, 1350 cm<sup>-1</sup>. HRMS [M]<sup>+</sup> Calcd for C<sub>10</sub>H<sub>7</sub>NFBr: 238.9746; Found: 238.9754.



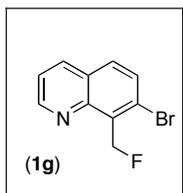
Product **2f** was also obtained from this reaction as an off-white solid (33.4 mg, 13% isolated yield, R<sub>f</sub> = 0.53 in 98% CH<sub>2</sub>Cl<sub>2</sub>/2% EtOAc, mp = 65.0-67.4 °C). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 8.94 (dd, J = 4.2, 1.7 Hz, 1H), 8.55 (dd, J = 8.6, 1.7 Hz, 1H), 7.82 (d, J = 7.8 Hz, 1H), 7.59 (d, J = 7.8 Hz, 1H), 7.54 (dd, J = 8.6, 4.2 Hz, 1H), 5.80 (s, 2H), 1.27 (s, 9H). <sup>13</sup>C{<sup>1</sup>H} NMR (175 MHz, CDCl<sub>3</sub>): δ 178.5, 150.5, 146.7, 135.8, 135.3, 130.2, 127.9, 127.6, 122.6, 121.7, 62.6, 39.2, 27.5.

<sup>12</sup> Evans et al. *Tetrahedron* **2005**, 61, 9696.

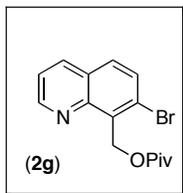
IR (thin film, CH<sub>2</sub>Cl<sub>2</sub>): 2973, 1729, 1156 cm<sup>-1</sup>. HRMS [M+H]<sup>+</sup> Calcd for C<sub>15</sub>H<sub>16</sub>NO<sub>2</sub>Br: 322.0437;  
Found: 322.0432.

## 7-Bromo-8-methylquinoline

7-Bromo-8-methylquinoline (**S-g**) was prepared according to the following procedure. 3-Bromo-2-methylaniline (6.3 g, 33.9 mmol, 1.0 equiv) and NaI (66.0 mg, 0.441 mmol, 0.013 equiv) were dissolved in H<sub>2</sub>SO<sub>4</sub> (8.7 mL) and water (2.2 mL) in a round-bottomed flask. The reaction was heated to 145 °C, and glycerol (3.7 g) was added via addition funnel over 1.5 h. The reaction stirred for 2 h. Once cooled, the crude mixture was extracted twice with toluene. The organic phase was washed with water and brine and dried (MgSO<sub>4</sub>). The filtrate was concentrated under vacuum, and the residue was purified by chromatography on silica gel eluting with hexane/EtOAc (1:1). The product was obtained as a beige solid (5.3 g, 71% yield, R<sub>f</sub> = 0.53, mp = 50.9-52.5 °C). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 8.94 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.11 (dd, *J* = 8, 2 Hz, 1H), 7.69 (d, *J* = 8.8 Hz, 1H), 7.53 (d, *J* = 8.8 Hz, 1H), 7.42 (dd, *J* = 8, 4 Hz, 1H), 2.93 (s, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 150.1, 147.8, 137.3, 136.6, 131.0, 127.4, 126.6, 126.1, 121.2, 17.8. IR (thin film, CH<sub>2</sub>Cl<sub>2</sub>): 2918, 1604, 1490 cm<sup>-1</sup>. HRMS [M+H]<sup>+</sup> Calcd for C<sub>10</sub>H<sub>8</sub>NBr: 221.9913; Found: 221.9914.



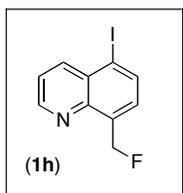
7-Bromo-8-methylquinoline (178 mg, 0.80 mmol, 1.0 equiv) was used as the substrate. The solvent was dichloromethane (8.0 mL), and the reaction was heated for 16 h. GC analysis of the crude reaction mixture showed 49% yield of **1g** and 26% yield of **2g**. The crude mixture was purified by chromatography on silica gel with a gradient of 0%-4% EtOAc in CH<sub>2</sub>Cl<sub>2</sub>. Product **1g** was obtained as a white solid (80.7 mg, 42% isolated yield, R<sub>f</sub> = 0.48 in 98% CH<sub>2</sub>Cl<sub>2</sub>/2% EtOAc, mp = 90.3-94.5 °C). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 9.01 (dd, *J* = 4, 2 Hz, 1H), 8.16 (dd, *J* = 8.3, 2 Hz, 1H), 7.78-7.73 (multiple peaks, 2H), 7.48 (dd, *J* = 8.3, 4 Hz, 1H), 6.30 (d, *J*<sub>HF</sub> = 47 Hz, 2H). <sup>13</sup>C{<sup>1</sup>H} NMR (175 MHz, CDCl<sub>3</sub>): δ 151.5, 147.8 (d, *J*<sub>CF</sub> = 1.4 Hz), 136.4, 133.3 (d, *J*<sub>CF</sub> = 14 Hz), 131.3 (d, *J*<sub>CF</sub> = 3.3 Hz), 130.7 (d, *J*<sub>CF</sub> = 4.8 Hz), 128.4 (d, *J*<sub>CF</sub> = 6.2 Hz), 127.5 (d, *J*<sub>CF</sub> = 1.9 Hz), 121.9, 79.5 (d, *J*<sub>CF</sub> = 163 Hz). <sup>19</sup>F NMR (470 MHz, CDCl<sub>3</sub>): δ -209.8 (t, *J*<sub>FH</sub> = 47 Hz). IR (thin film, CH<sub>2</sub>Cl<sub>2</sub>): 2985, 1570, 1488 cm<sup>-1</sup>. HRMS [M+H]<sup>+</sup> Calcd for C<sub>10</sub>H<sub>7</sub>NFBr: 239.9819; Found: 239.9821.



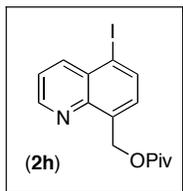
Product **2g** was also obtained from this reaction as a light brown solid (68.2 mg, 26% isolated yield,  $R_f = 0.19$  in 98%  $\text{CH}_2\text{Cl}_2/2\%$  EtOAc, mp = 92.3-102.5 °C).  $^1\text{H}$  NMR (700 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.96 (dd,  $J = 4.1, 1.7$  Hz, 1H), 8.14 (dd,  $J = 8.2, 1.7$  Hz, 1H), 7.74 (d,  $J = 8.9$  Hz, 1H), 7.69 (d,  $J = 8.9$  Hz, 1H), 7.45 (dd,  $J = 8.2, 4.1$  Hz, 1H), 5.94 (s, 2H), 1.19 (s, 9H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (175 MHz,  $\text{CDCl}_3$ ):  $\delta$  178.6, 151.0, 147.8, 136.3, 134.2, 131.2, 129.7, 127.8, 127.4, 121.7, 61.7, 39.2, 27.5. IR (thin film,  $\text{CH}_2\text{Cl}_2$ ): 2971, 1723, 1152  $\text{cm}^{-1}$ . HRMS  $[\text{M}+\text{H}]^+$  Calcd for  $\text{C}_{15}\text{H}_{16}\text{NO}_2\text{Br}$ : 322.0437; Found: 322.0442.

## 5-Iodo-8-methylquinoline

5-Iodo-8-methylquinoline (**S-h**) was prepared according to a literature procedure.<sup>13</sup> <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 8.90 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.37 (dd, *J* = 8.6, 1.7 Hz, 1H), 8.00 (d, *J* = 7.6 Hz, 1H), 7.47 (dd, *J* = 8.6, 4.2 Hz, 1H), 7.30 (m, 1H), 2.78 (s, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 150.1, 148.1, 140.8, 138.7, 137.6, 130.9, 130.1, 122.7, 95.6, 18.3. Mp = 52.1-55.0 °C. IR (thin film, CH<sub>2</sub>Cl<sub>2</sub>): 2919, 1560, 1491 cm<sup>-1</sup>. HRMS [M+H]<sup>+</sup> Calcd for C<sub>10</sub>H<sub>8</sub>NI: 269.9774; Found: 269.9778.



5-Iodo-8-methylquinoline (215 mg, 0.80 mmol, 1.0 equiv) was used as the substrate. The solvent was dichloromethane (8.0 mL), and the reaction was heated for 24 h. GC analysis of the crude reaction mixture showed 67% yield of **1h** and 16% yield of **2h**. The crude mixture was purified by chromatography on silica gel with a gradient of 1%-8% EtOAc in CH<sub>2</sub>Cl<sub>2</sub>. Product **1h** was obtained as a light brown solid (125.5 mg, 55% isolated yield, R<sub>f</sub> = 0.58 in 96% CH<sub>2</sub>Cl<sub>2</sub>/4% EtOAc, mp = 78.5-82.7 °C). <sup>1</sup>H NMR (700 MHz, CDCl<sub>3</sub>): δ 8.88 (m, 1H), 8.40 (d, *J* = 8.6 Hz, 1H), 8.16 (d, *J* = 7.6 Hz, 1H), 7.55 (d, *J* = 7.6 Hz, 1H), 7.52 (dd, *J* = 8, 4.0 Hz, 1H), 6.10 (d, *J*<sub>HF</sub> = 48 Hz, 2H). <sup>13</sup>C{<sup>1</sup>H} NMR (175 MHz, CDCl<sub>3</sub>): δ 150.6 (d, *J*<sub>CF</sub> = 21 Hz), 145.8, 140.6 (d, *J*<sub>CF</sub> = 22 Hz), 137.6 (d, *J*<sub>CF</sub> = 22 Hz), 136.2 (d, *J*<sub>CF</sub> = 19 Hz), 129.9, 128.1 (d, *J*<sub>CF</sub> = 11 Hz), 123.1 (d, *J*<sub>CF</sub> = 22 Hz), 98.6, 81.7 (d, <sup>1</sup>*J*<sub>CF</sub> = 166 Hz). <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>): δ -222.1 (t, *J*<sub>FFH</sub> = 48 Hz). IR (thin film, CH<sub>2</sub>Cl<sub>2</sub>): 1564, 1494, 1344 cm<sup>-1</sup>. HRMS [M+H]<sup>+</sup> Calcd for C<sub>10</sub>H<sub>7</sub>NFI: 286.9607; Found: 286.9608.



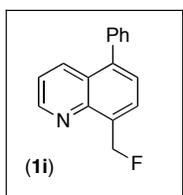
Product **2h** was also obtained from this reaction as a light yellow solid (44.3 mg, 15% isolated yield, R<sub>f</sub> = 0.38 in 96% CH<sub>2</sub>Cl<sub>2</sub>/4% EtOAc, mp = 78.5-82.7 °C). <sup>1</sup>H NMR (700 MHz, CDCl<sub>3</sub>): δ

<sup>13</sup> Tochilkin, A. I. et al. *Chem. Heterocycl. Compd.* (Russ) **1983**, 1373.

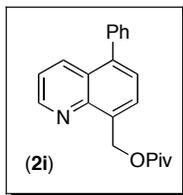
8.89 (dd,  $J = 4.1, 1.4$  Hz, 1H), 8.39 (dd,  $J = 8.5, 1.4$  Hz, 1H), 8.12 (d,  $J = 7.7$  Hz, 1H), 7.51 (dd,  $J = 8.5, 4.1$  Hz, 1H), 7.45 (dd,  $J = 7.7, 0.8$  Hz, 1H), 5.81 (s, 2H), 1.27 (s, 9H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  178.5, 150.6, 146.5, 140.6, 137.6, 136.4, 130.1, 128.6, 123.1, 98.1, 62.6, 39.2, 27.5. IR (thin film,  $\text{CH}_2\text{Cl}_2$ ): 2972, 1729, 1156  $\text{cm}^{-1}$ . HRMS  $[\text{M}+\text{H}]^+$  Calcd for  $\text{C}_{15}\text{H}_{16}\text{NO}_2$ : 370.0298; Found: 370.0300.

## 8-Methyl-5-phenylquinoline

8-Methyl-5-phenylquinoline (**S-i**) was prepared according to the following procedure. 5-Bromo-8-methylquinoline (1.00 g, 4.50 mmol, 1.0 equiv), phenylboronic acid (603 mg, 4.95 mmol, 1.1 equiv), Pd<sub>2</sub>(dba)<sub>3</sub> (206 mg, 0.225 mmol, 0.05 equiv), and K<sub>3</sub>PO<sub>4</sub> (1.91 g, 9.01 mmol, 2.0 equiv) were added to an oven-dried Schlenk flask equipped with a stirbar. Dioxane (20 mL) and P(OMe)<sub>3</sub> (55.8 mg, 0.450 mmol, 0.1 equiv) were added in a glovebox. The reaction was heated to 95 °C for 24 h. The reaction mixture was cooled to rt, diluted with diethyl ether, and filtered through celite. The filtrate was concentrated under vacuum and the residue was purified by chromatography on silica gel eluting with DCM/EtOAc (98:2). The product was obtained as a white solid (0.56 g, 57% yield, R<sub>f</sub> = 0.39, mp = 56.6-58.4 °C). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 8.97 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.24 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.62 (dd, *J* = 7, 1 Hz, 1H), 7.51-7.48 (multiple peaks, 2H), 7.46-7.40 (multiple peaks, 4H), 7.36 (dd, *J* = 8.6, 4 Hz, 1H), 2.87 (s, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 149.2, 147.6, 139.8, 138.7, 136.7, 134.8, 130.3, 129.3, 128.6, 127.6, 127.1, 126.8, 121.0, 18.6. IR (thin film, CH<sub>2</sub>Cl<sub>2</sub>): 2920, 1592, 1504 cm<sup>-1</sup>. HRMS [M+H]<sup>+</sup> Calcd for C<sub>16</sub>H<sub>13</sub>N: 220.1121; Found: 220.1126.



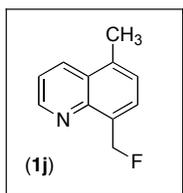
8-Methyl-5-phenylquinoline (175 mg, 0.80 mmol, 1.0 equiv) was used as the substrate. The solvent was fluorobenzene (8.0 mL), and the reaction was heated for 28 h. GC analysis of the crude reaction mixture showed 43% yield of **1i** and 35% yield of **2i**. The crude mixture was purified by chromatography on silica gel with a gradient of 0%-4% EtOAc in CH<sub>2</sub>Cl<sub>2</sub>. Product **1i** was obtained as an off-white solid (74.6 mg, 39% isolated yield, R<sub>f</sub> = 0.45 in 98% CH<sub>2</sub>Cl<sub>2</sub>/2% EtOAc, mp = 63.5-67.0 °C). <sup>1</sup>H NMR (700 MHz, CDCl<sub>3</sub>): δ 8.94 (dd, *J* = 4.1, 1.5 Hz, 1H), 8.26 (dd, *J* = 8.6, 1.5 Hz, 1H), 7.88 (d, *J* = 7.2 Hz, 1H), 7.55 (d, *J* = 7.2 Hz, 1H), 7.52 (m, 2H), 7.48-7.45 (multiple peaks, 3H), 7.39 (dd, *J* = 8.5, 4.1 Hz, 1H), 6.20 (d, *J*<sub>HF</sub> = 48 Hz, 2H). <sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 149.8, 145.8 (d, *J*<sub>CF</sub> = 2.9 Hz), 141.1 (d, *J*<sub>CF</sub> = 2.9 Hz), 139.3, 134.9, 134.2 (d, *J*<sub>CF</sub> = 16 Hz), 130.2, 128.7, 128.0, 127.1 (d, *J*<sub>CF</sub> = 3.8 Hz), 127.1, 126.7, 121.5, 82.1 (d, *J*<sub>CF</sub> = 166 Hz). <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>): δ -219.1 (t, *J*<sub>FH</sub> = 48 Hz). IR (thin film, CH<sub>2</sub>Cl<sub>2</sub>): 3029, 1584, 1504, 1352 cm<sup>-1</sup>. HRMS [M+H]<sup>+</sup> Calcd for C<sub>16</sub>H<sub>12</sub>NF: 238.1027; Found: 238.1033.



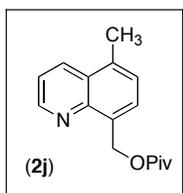
Product **2i** was also obtained from this reaction as a light yellow oil (91.0 mg, 36% isolated yield,  $R_f = 0.21$  in 98%  $\text{CH}_2\text{Cl}_2/2\%$  EtOAc).  $^1\text{H}$  NMR (700 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.94 (dd,  $J = 4.1, 1.7$  Hz, 1H), 8.24 (dd,  $J = 8.5, 1.7$  Hz, 1H), 7.77 (d,  $J = 7.3$  Hz, 1H), 7.51-7.50 (multiple peaks, 3H), 7.47-7.45 (multiple peaks, 3H), 7.38 (dd,  $J = 8.5, 4.1$  Hz, 1H), 5.91 (s, 2H), 1.30 (s, 9H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  178.6, 149.6, 146.2, 140.4, 139.5, 134.8, 134.4, 130.2, 128.7, 127.9, 127.1, 127.1, 126.7, 121.4, 63.0, 39.2, 27.6. IR (thin film,  $\text{CH}_2\text{Cl}_2$ ): 3060, 2972, 1726, 1504, 1480, 1280  $\text{cm}^{-1}$ . HRMS  $[\text{M}+\text{H}]^+$  Calcd for  $\text{C}_{21}\text{H}_{21}\text{NO}_2$ : 320.1645; Found: 320.1652.

## 5,8-Dimethylquinoline

5,8-Dimethylquinoline (**S-j**) was prepared according to a literature procedure.<sup>14</sup> <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 8.96 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.31 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.46-7.41 (multiple peaks, 2H), 7.27 (m, 1H), 2.79 (s, 3H), 2.65 (s, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 148.9, 147.7, 135.1, 132.9, 132.5, 129.5, 127.8, 126.9, 120.6, 18.7, 18.4. IR (thin film, CH<sub>2</sub>Cl<sub>2</sub>): 2921, 1600, 1501 cm<sup>-1</sup>. HRMS [M+H]<sup>+</sup> Calcd for C<sub>11</sub>H<sub>11</sub>N: 158.0964; Found: 158.0964.



5,8-Dimethylquinoline (126 mg, 0.80 mmol, 1.0 equiv) was used as the substrate. The solvent was fluorobenzene (8.0 mL), and the reaction was heated for 16 h. GC analysis of the crude reaction mixture showed 41% yield of **1j** and 40% yield of **2j**. The crude mixture was purified by chromatography on silica gel with a gradient of 1%-8% EtOAc in CH<sub>2</sub>Cl<sub>2</sub>. Product **1j** was obtained as a viscous yellow oil (54.1 mg, 39% isolated yield, *R*<sub>f</sub> = 0.44 in 96% CH<sub>2</sub>Cl<sub>2</sub>/4% EtOAc). <sup>1</sup>H NMR (700 MHz, CDCl<sub>3</sub>): δ 8.94 (m, 1H), 8.34 (dd, *J* = 8.5, 1.5 Hz, 1H), 7.70 (d, *J* = 7.2 Hz, 1H), 7.46 (dd, *J* = 8.5, 4.1 Hz, 1H), 7.40 (d, *J* = 7.2 Hz, 1H), 6.10 (d, *J*<sub>HF</sub> = 48 Hz, 2H), 2.69 (s, 3H). <sup>13</sup>C{<sup>1</sup>H} NMR (125 MHz, CDCl<sub>3</sub>): δ 149.5, 145.8, 135.5 (d, *J*<sub>CF</sub> = 2.4 Hz), 133.0, 132.7 (d, *J*<sub>CF</sub> = 16.3 Hz), 127.9 (d, *J*<sub>CF</sub> = 9.6 Hz), 127.6, 126.9, 121.1, 82.1 (d, <sup>1</sup>*J*<sub>CF</sub> = 165 Hz), 18.9. <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>): δ -216.6 (t, *J*<sub>FH</sub> = 48 Hz). IR (thin film, CH<sub>2</sub>Cl<sub>2</sub>): 2951, 1599, 1504, 1352 cm<sup>-1</sup>. HRMS [M+H]<sup>+</sup> Calcd for C<sub>11</sub>H<sub>10</sub>NF: 175.0797; Found: 175.0794.



Product **2j** was also obtained from this reaction as a viscous yellow oil (77.9 mg, 38% isolated yield, *R*<sub>f</sub> = 0.19 in 96% CH<sub>2</sub>Cl<sub>2</sub>/4% EtOAc). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ 8.95 (dd, *J* = 4.1, 1.5 Hz, 1H), 8.35 (dd, *J* = 8.5, 1.5 Hz, 1H), 7.63 (d, *J* = 7.2 Hz, 1H), 7.47 (dd, *J* = 8.5, 4.1 Hz, 1H),

<sup>14</sup> O'Murchu, C. *Synthesis* **1989**, 11, 880.

7.38 (d,  $J = 7.2$  Hz, 1H), 5.83 (s, 2H), 2.69 (s, 3H), 1.26 (s, 9H).  $^{13}\text{C}\{^1\text{H}\}$  NMR (175 MHz, acetone- $d_6$ ):  $\delta$  179.0, 151.0, 147.8, 136.4, 134.6, 134.3, 129.0, 128.8, 128.2, 122.7, 64.0, 40.2, 28.3, 19.3. IR (thin film,  $\text{CH}_2\text{Cl}_2$ ): 2972, 1726, 1480, 1282, 1159  $\text{cm}^{-1}$ . HRMS  $[\text{M}+\text{H}]^+$  Calcd for  $\text{C}_{16}\text{H}_{19}\text{NO}_2$ : 257.1416; Found: 257.1415.

### 5-Methoxy-8-methylquinoline

5-Methoxy-8-methylquinoline (**S-k**) was prepared according to a literature procedure.<sup>15</sup> NMR data matched that reported in the literature.

On the benchtop,  $\text{PhI}(\text{OPiv})_2$  (154 mg, 0.38 mmol, 2.0 equiv) and AgF (121 mg, 0.96 mmol, 5.0 equiv) were weighed into a 4 mL vial, and DCM (1.9 mL) was added. The vial was sealed with a Teflon-lined cap (with Teflon tape covering the threads of the vial), and this mixture was heated at 60 °C for 1 h. The vial was removed from the heat and allowed to cool to rt (~5 min). Then  $\text{Pd}(\text{OAc})_2$  (4.3 mg, 0.02 mmol, 0.10 equiv),  $\text{MgSO}_4$  (45.9 mg, 0.38 mmol, 2.0 equiv), and 5-methoxy-8-methylquinoline (33.1 mg, 0.19 mmol, 1.0 equiv) were added. The vial was sealed with a Teflon-lined cap and heated at 60 °C for an additional 24 h. GC analysis of the crude reaction mixture showed <1% yield of **1k** and ~55% yield of **2k**, using hexadecane as a standard.

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<sup>15</sup> Evans et al. *Tetrahedron* **2005**, 61, 9696.

Fluorine-19



Sample Name:



Data Collected on:  
Ga.Chem.USA.UMich.edu-vnmrs400  
Archive directory:

Sample directory:

FidFile: kb4-144-02

Pulse Sequence: FLUORINE (s2pul)  
Solvent: cd2cl2  
Data collected on: Jun 7 2012

Operator: kbut1

Relax. delay 1.000 sec  
Pulse 30.0 degrees  
Acq. time 0.603 sec  
Width 108.7 kHz  
16 repetitions

OBSERVE F19, 375.9380190 MHz

DATA PROCESSING

Line broadening 0.5 Hz  
Ft size 131072  
Total time 0 min 29 sec



-129.632

-175.961



Fluorine-19

Sample Name:

Data Collected on:  
Te-vnmrs500  
Archive directory:

Sample directory:

FidFile: kb-pMeC6H4IF2-F

Pulse Sequence: FLUORINE (s2pul)  
Solvent: cd2cl2  
Data collected on: Jun 8 2012

Temp. 25.0 C / 298.1 K  
Operator: kbutl

Relax. delay 1.000 sec  
Pulse 30.0 degrees  
Acq. time 0.498 sec  
Width 131.6 kHz  
16 repetitions

OBSERVE F19, 470.5586258 MHz  
DATA PROCESSING  
Line broadening 1.5 Hz  
FT size 131072  
Total time 0 min 27 sec



Authentic *p*-MeC<sub>6</sub>H<sub>4</sub>IF<sub>2</sub>

175.946

-20 -40 -60 -80 -100 -120 -140 -160 -180 ppm

Sample Name:

Data Collected on:  
 Sn.Chem.I.SA.UMich.edu-inova500  
 Archive directory:

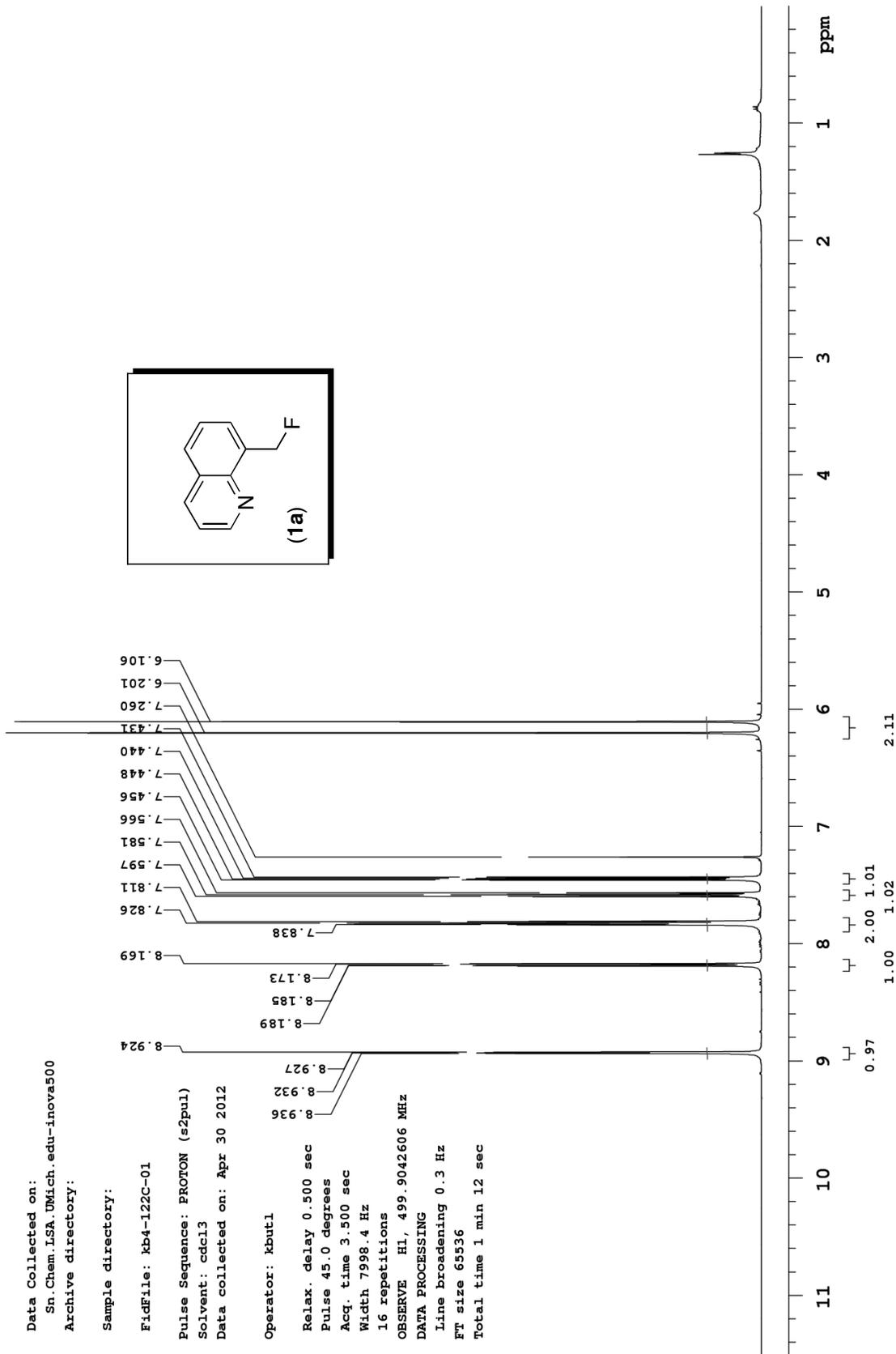
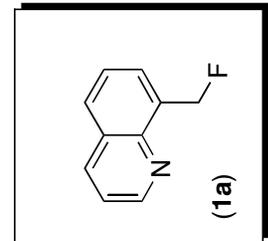
Sample directory:

FidFile: kb4-122C-01

Pulse Sequence: PROTON (s2pul)  
 Solvent: cdcl3  
 Data collected on: Apr 30 2012

Operator: kbut1

Relax. delay 0.500 sec  
 Pulse 45.0 degrees  
 Acq. time 3.500 sec  
 Width 7998.4 Hz  
 16 repetitions  
 OBSERVE H1, 499.9042606 MHz  
 DATA PROCESSING  
 Line broadening 0.3 Hz  
 FT size 65536  
 Total time 1 min 12 sec





Sample Name:

Data Collected on:  
Yb-vmrs700  
Archive directory:

Sample directory:

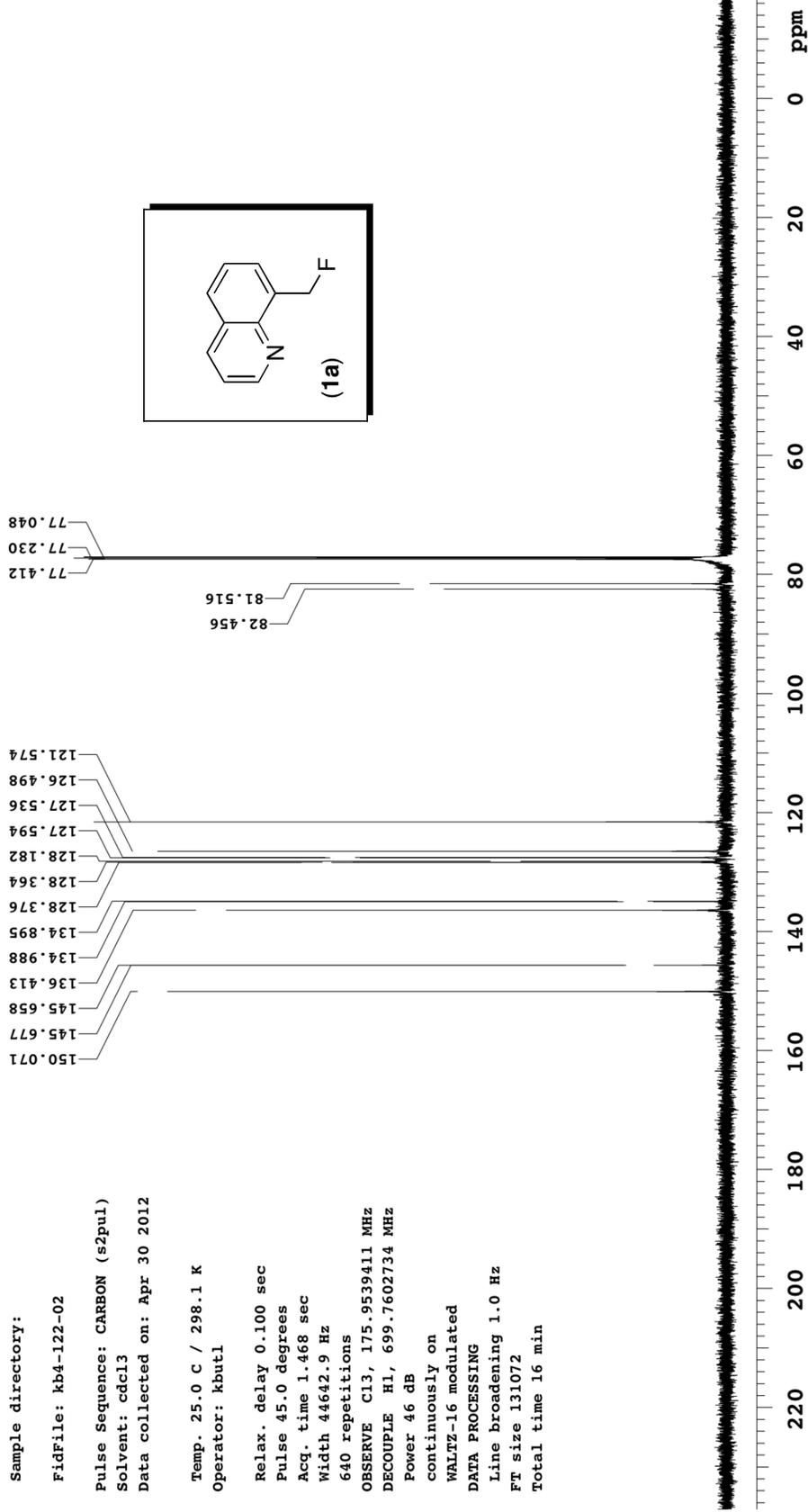
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Pulse Sequence: CARBON (s2pul)  
Solvent: cdcl3  
Data collected on: Apr 30 2012

Temp. 25.0 C / 298.1 K  
Operator: kbutl

Relax. delay 0.100 sec  
Pulse 45.0 degrees  
Acq. time 1.468 sec  
Width 44642.9 Hz  
640 repetitions

OBSERVE C13, 175.9539411 MHZ  
DECOUPLE H1, 699.7602734 MHZ  
Power 46 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
Ft size 131072  
Total time 16 min



pg. 130 fr. 21-27 C-O

Sample Name:

Data Collected on:

Te-vnmrs500

Archive directory:

Sample directory:

FidFile: kb4-130-07

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Jun 11 2012

Temp. 25.0 C / 298.1 K

Operator: kbut1

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 8012.8 Hz

16 repetitions

9.40

5

0.31692 MHz

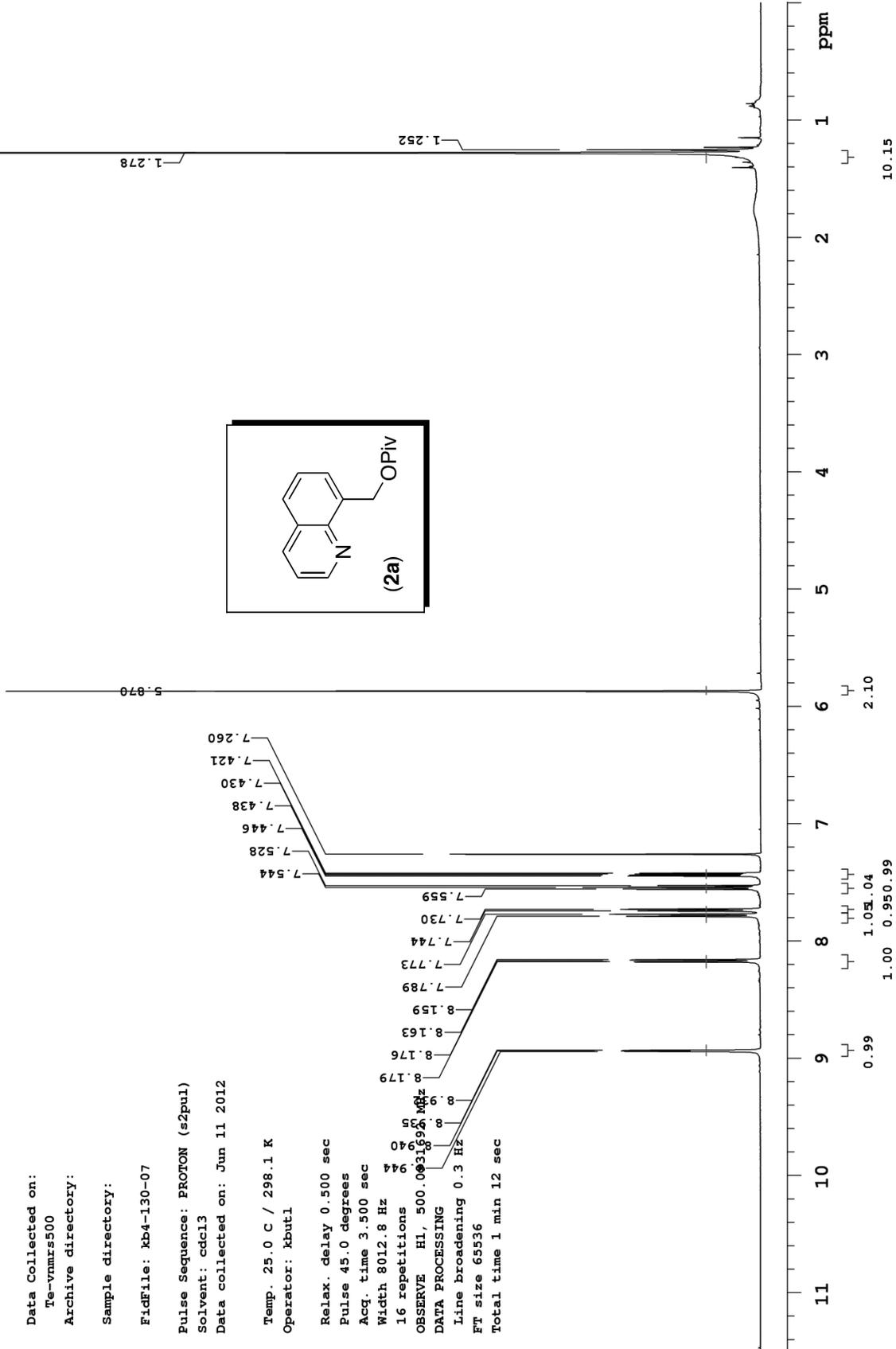
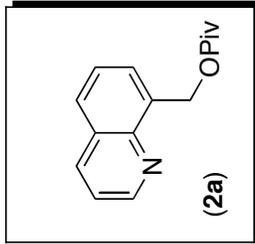
OBSERVE H1, 500.0

DATA PROCESSING

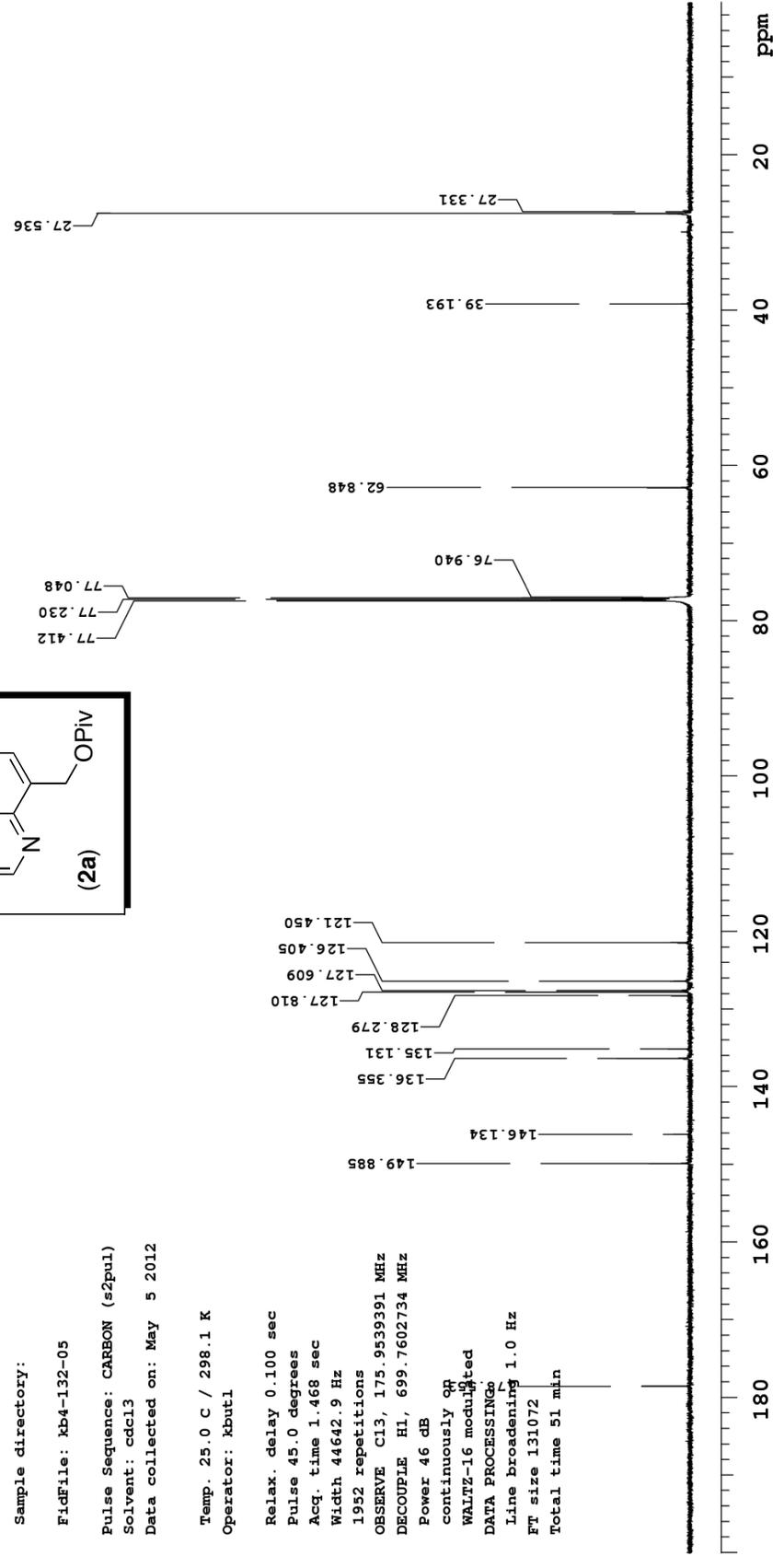
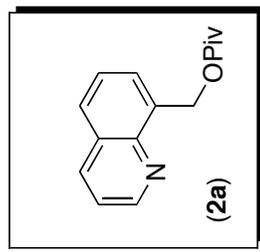
Line broadening 0.3 Hz

FT size 65536

Total time 1 min 12 sec



Sample Name:  
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 Yb-vnmrs700  
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 Pulse Sequence: CARBON (s2pul)  
 Solvent: cdcl3  
 Data collected on: May 5 2012  
 Temp. 25.0 C / 298.1 K  
 Operator: kbut1  
 Relax. delay 0.100 sec  
 Pulse 45.0 degrees  
 Acq. time 1.468 sec  
 Width 44642.9 Hz  
 1952 repetitions  
 OBSERVE C13, 175.9539391 MHz  
 DECOUPLE H1, 699.7602734 MHz  
 Power 46 dB  
 continuously on  
 WALTZ-16 modulated  
 DATA PROCESSING:  
 Line broadening 1.0 Hz  
 FT size 131072  
 Total time 51 min



pg. ? NO2sub

Sample Name:

Data Collected on:

Te-vnmrs500

Archive directory:

Sample directory:

FidFile: kb-NO2sub-H

Pulse Sequence: PROTON (sqpul)

Solvent: cdcl3

Data collected on: Jun 19 2012

Operator: kbut1

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 8012.8 Hz

16 repetitions

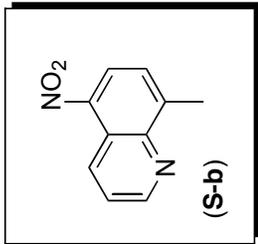
OBSERVE H1, 500.0931690 MHz

DATA PROCESSING

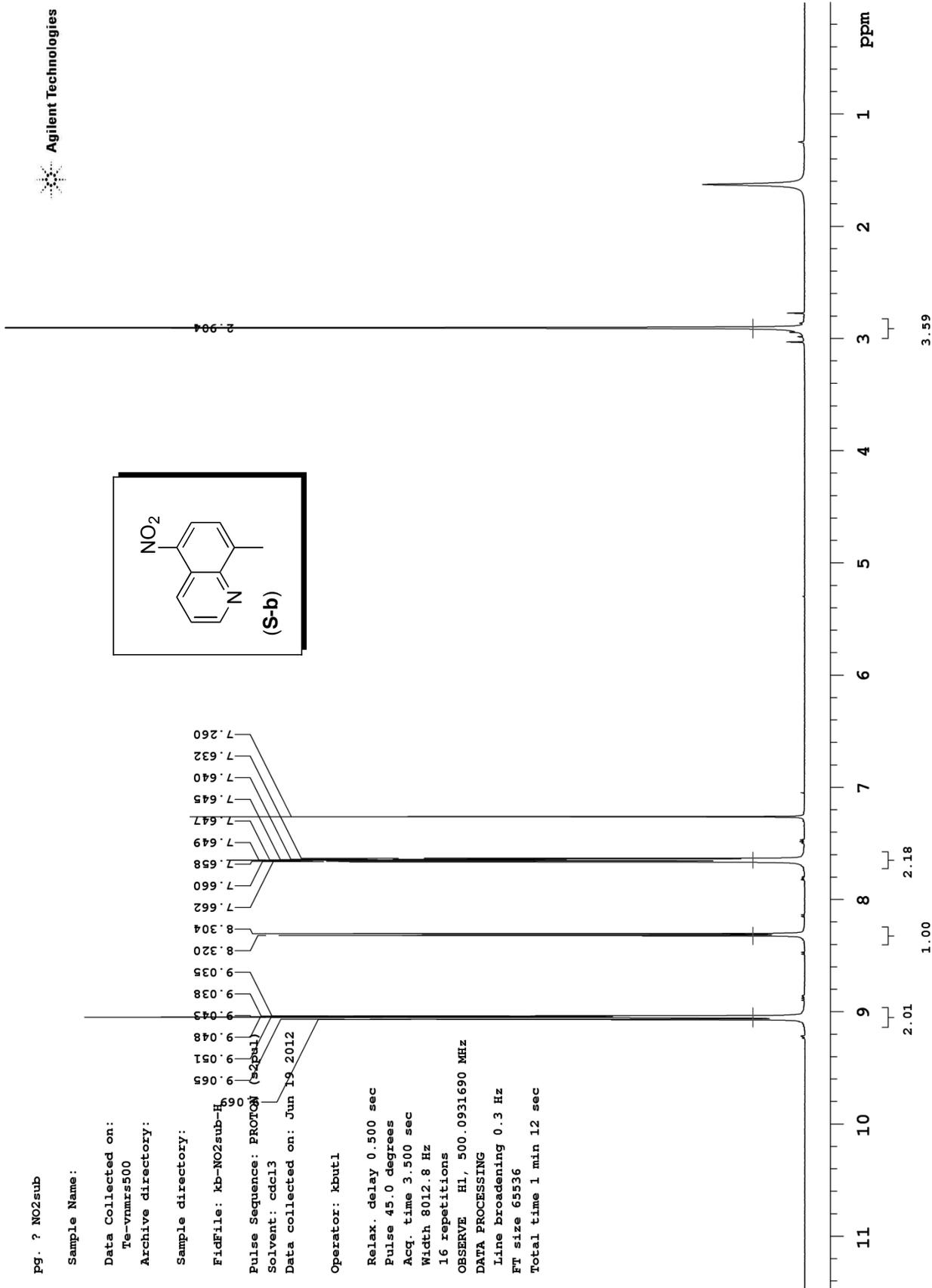
Line broadening 0.3 Hz

FT size 65536

Total time 1 min 12 sec



7.260  
7.632  
7.640  
7.645  
7.647  
7.649  
7.658  
7.660  
7.662  
8.304  
8.320  
9.035  
9.038  
9.043  
9.048  
9.051  
9.055



Sample Name:

Data Collected on:

YB-vnmrs700

Archive directory:

Sample directory:

00

FidFile: kb-NO2sub-C

00

Pulse Sequence: CARBON (s2pul)

Solvent: acetone

Data collected on: Jun 21 2012

Temp. 25.0 C / 298.1 K

Operator: kbutl

Relax. delay 0.100 sec

Pulse 45.0 degrees

Acq. time 1.468 sec

Width 44642.9 Hz

1232 repetitions

OBSERVE CH3, 175.9547606 MHz

DECOUPLE H1, 699.7639052 MHz

Power 46 dB

continuously on

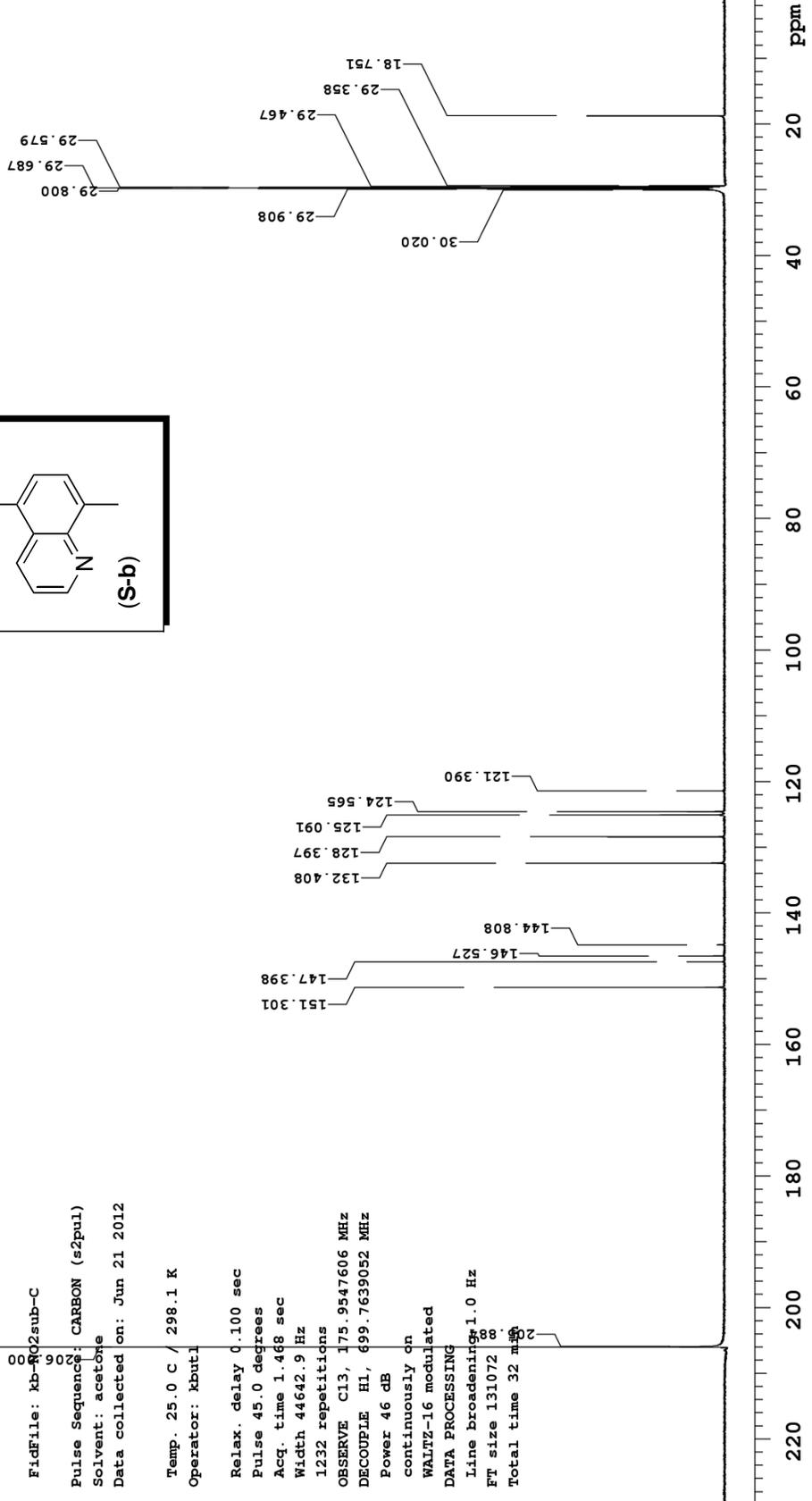
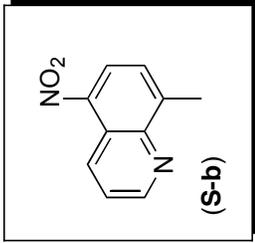
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 32 min



Sample Name:

Data Collected on:

Te-vmms500

Archive directory:

Sample directory:

FidFile: kb4-132-01

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: May 7 2012

Operator: kbutl

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 8012.8 Hz

16 repetitions

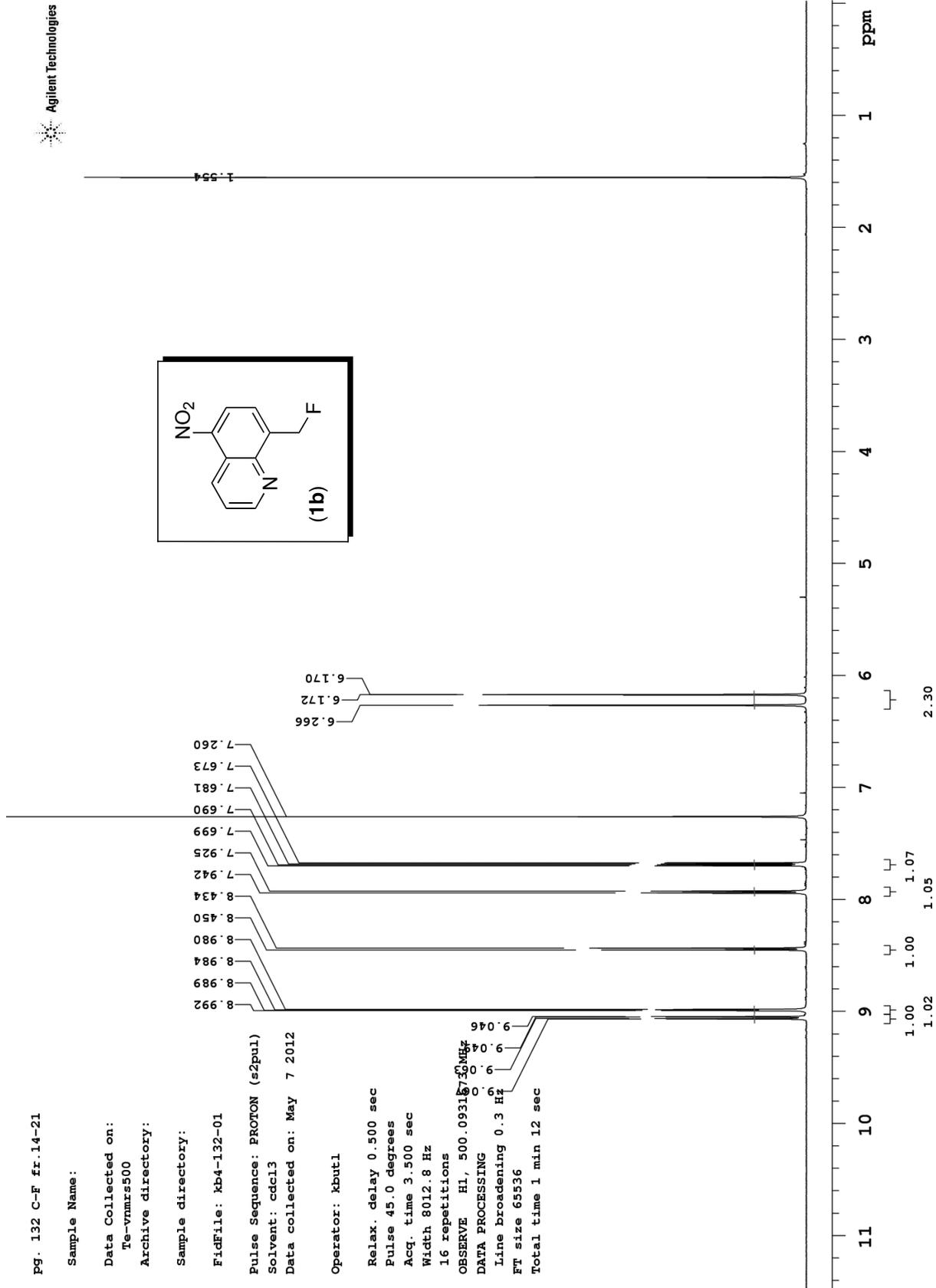
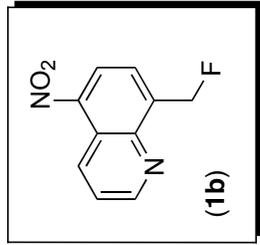
OBSERVE H1, 500.09318733 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 65536

Total time 1 min 12 sec



Sample Name:

Data Collected on:

Yb-vmrs700

Archive directory:

Sample directory:

FidFile: kb4-132-04

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: May 9 2012

Temp. 25.0 C / 298.1 K

Operator: kbut1

Relax. delay 0.100 sec

Pulse 45.0 degrees

Acq. time 1.468 sec

Width 44642.9 Hz

2208 repetitions

OBSERVE C13, 175.9539377 MHz

DECOUPLE H1, 699.7602734 MHz

Power 46 dB

continuously on

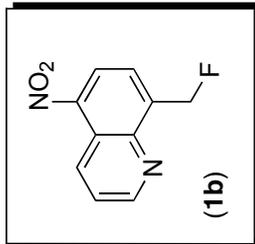
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 57 min



77.052  
77.230  
77.412

81.341  
82.305

123.935  
124.353  
124.798  
132.526

123.862

120.908

144.798  
144.818  
145.174  
150.842

143.227  
143.327

180 160 140 120 100 80 60 40 20 ppm

Sample Name:

Data Collected on:

Te-vnmrs500

Archive directory:

Sample directory:

FidFile: kb3-102-C-O

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: May 16 2012

Temp. 25.0 C / 298.1 K

Operator: kbut1

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 8012.8 Hz

16 repetitions

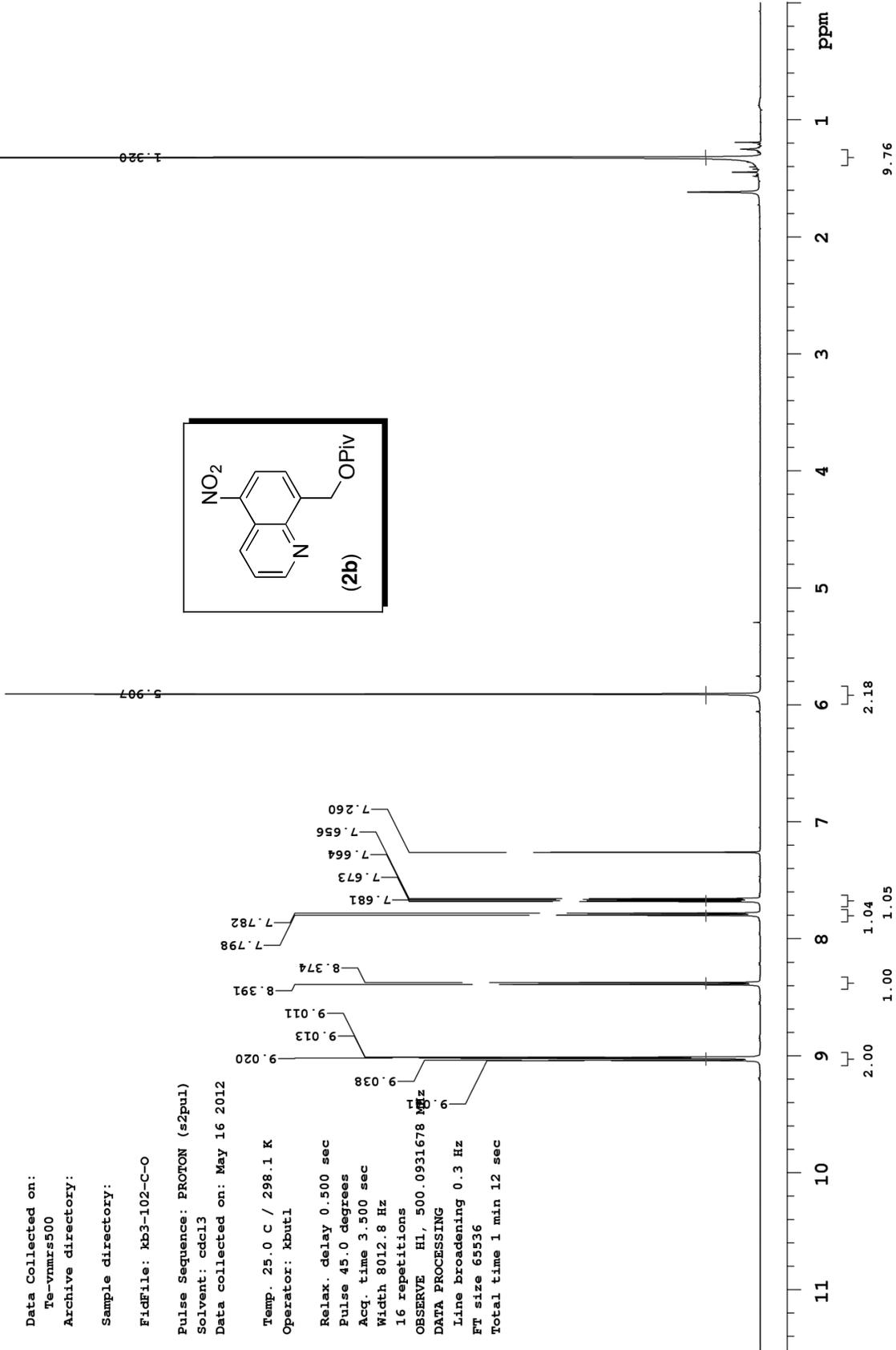
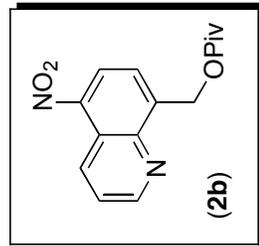
OBSERVE H1, 500.0931678 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 65536

Total time 1 min 12 sec



Sample Name:

Data Collected on:

Te-vmr500

Archive directory:

Sample directory:

FidFile: kb3-102-C-O-carbon

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: May 16 2012

Temp. 25.0 C / 298.1 K

Operator: kbuhl

Relax. delay 0.100 sec

Pulse 45.0 degrees

Acq. time 1.049 sec

Width 31250.0 Hz

4304 repetitions

OBSERVE C13, 125.7484989 MHz

DECOUPLE H1, 500.0956704 MHz

Power 41 dB

continuously on

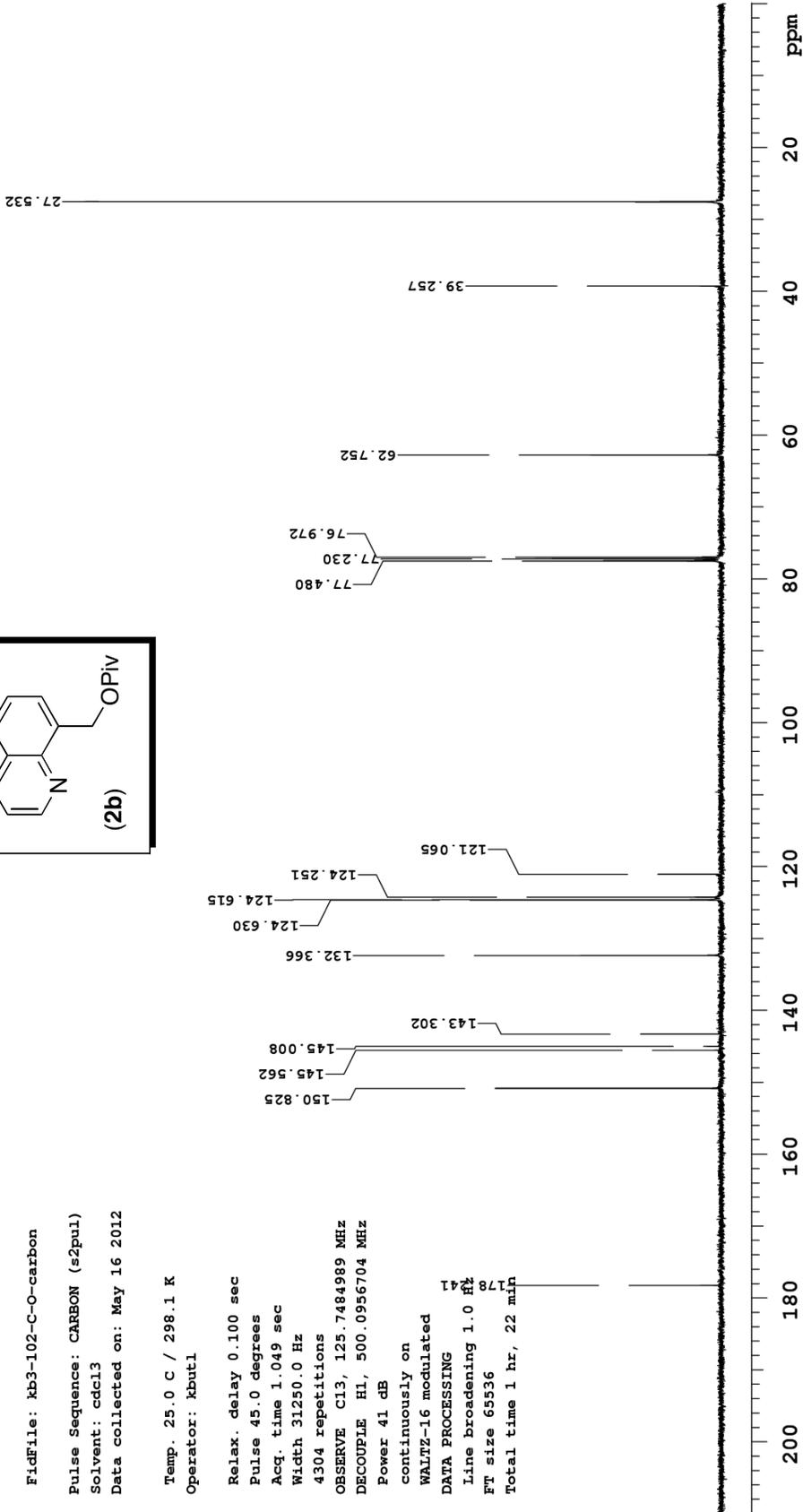
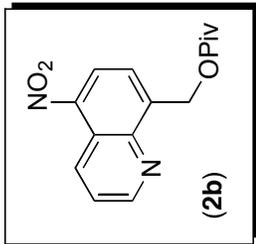
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 1 hr, 22 min



Pg. 196 CN Sub



Sample Name:

Data Collected on:

Te-vmrs500

Archive directory:

Sample directory:

FidFile: kb-CNsub-H

Pulse Sequence: PROTON(s2p12)

Solvent: cdcl3

Data collected on: Jun 18 2012

Operator: kbut1

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 8012.8 Hz

16 repetitions

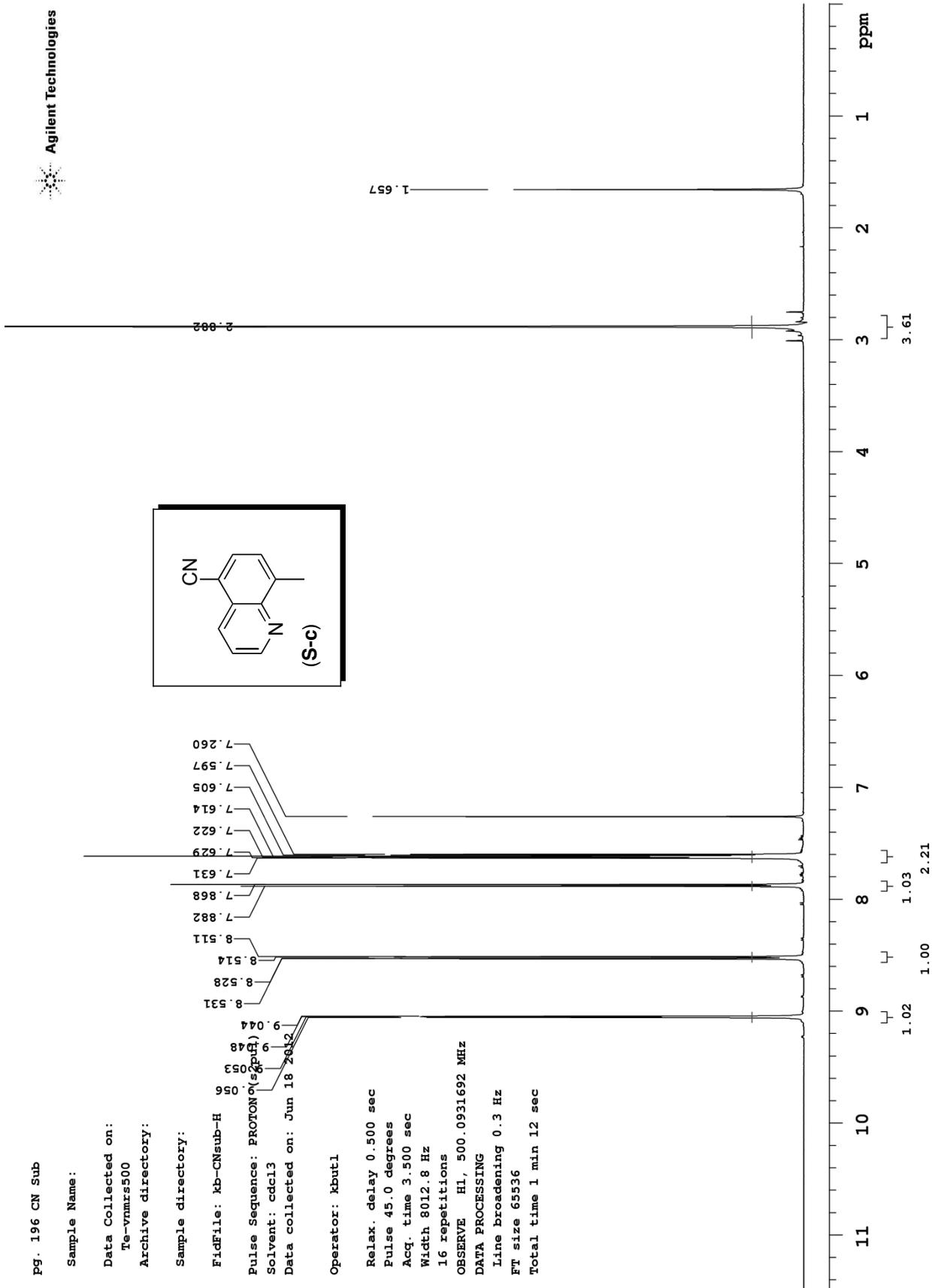
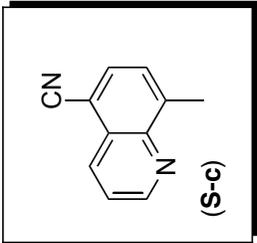
OBSERVE HI, 500.0931692 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 65536

Total time 1 min 12 sec



Pg. 196 CN Sub

Sample Name:

Data Collected on:

Te-vnmrs500

Archive directory:

Sample directory:

FidFile: kb-CNsub-C

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Jun 18 2012

Operator: kbut1

Relax. delay 0.100 sec

Pulse 45.0 degrees

Acq. time 1.049 sec

Width 31250.0 Hz

464 repetitions

OBSERVE C13, 125.7485018 MHz

DECOUPLE H1, 500.0956704 MHz

Power 41 dB

continuously on

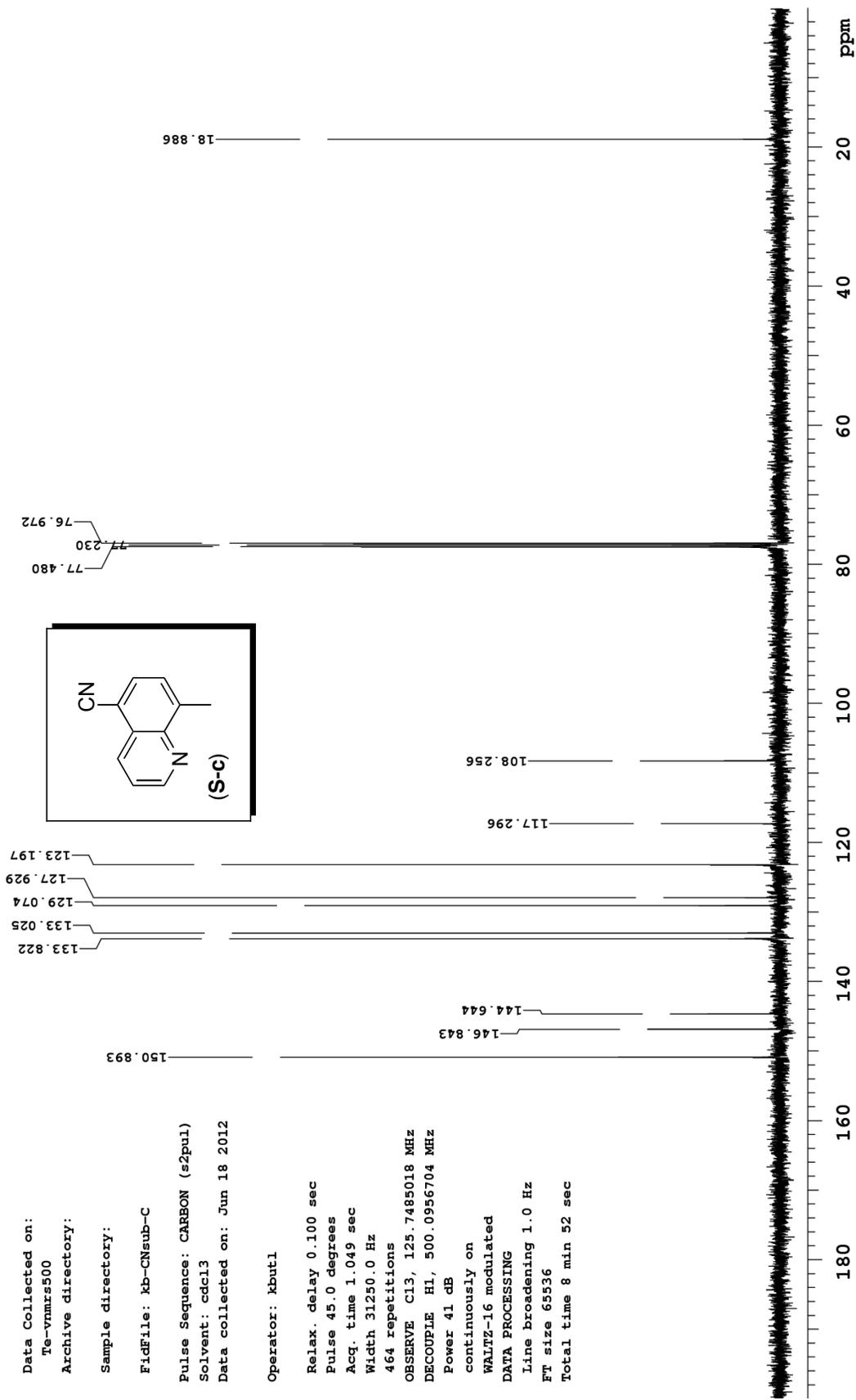
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 8 min 52 sec



Pg. 180 fr. 35-38

Sample Name:

Data Collected on:

Yb-vnmrs700

Archive directory:

Sample directory:

FidFile: kb2-180-01

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Jan 31 2012

Temp. 21.0 C / 294.1 K

Operator: kbutl

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 11261.3 Hz

16 repetitions

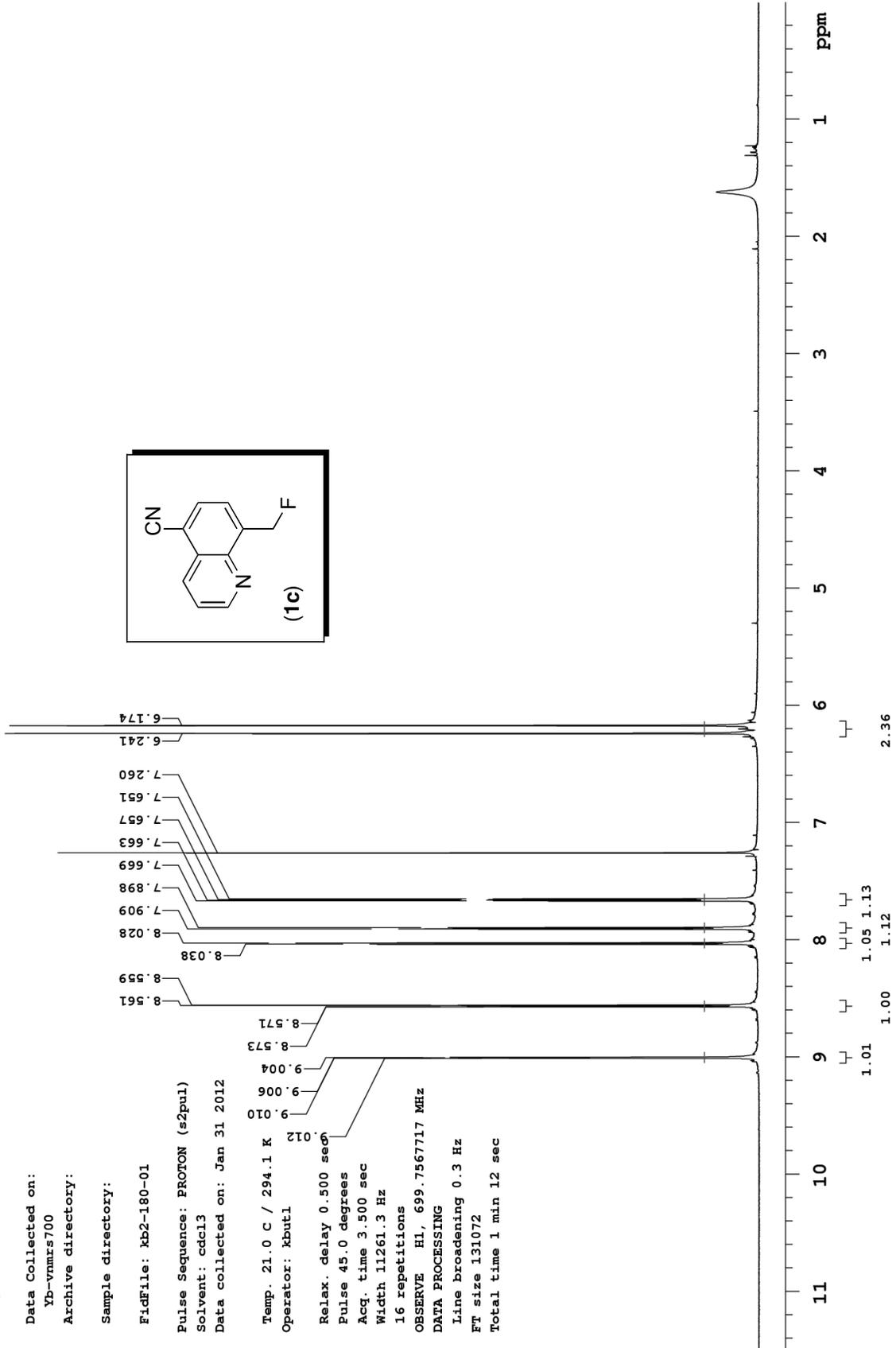
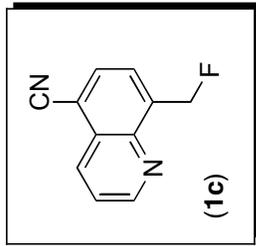
OBSERVE H1, 699.7567717 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 131072

Total time 1 min 12 sec



Sample Name:

Data Collected on:  
Co. Chem. ISA, UMich. edu-vmrs400  
Archive directory:

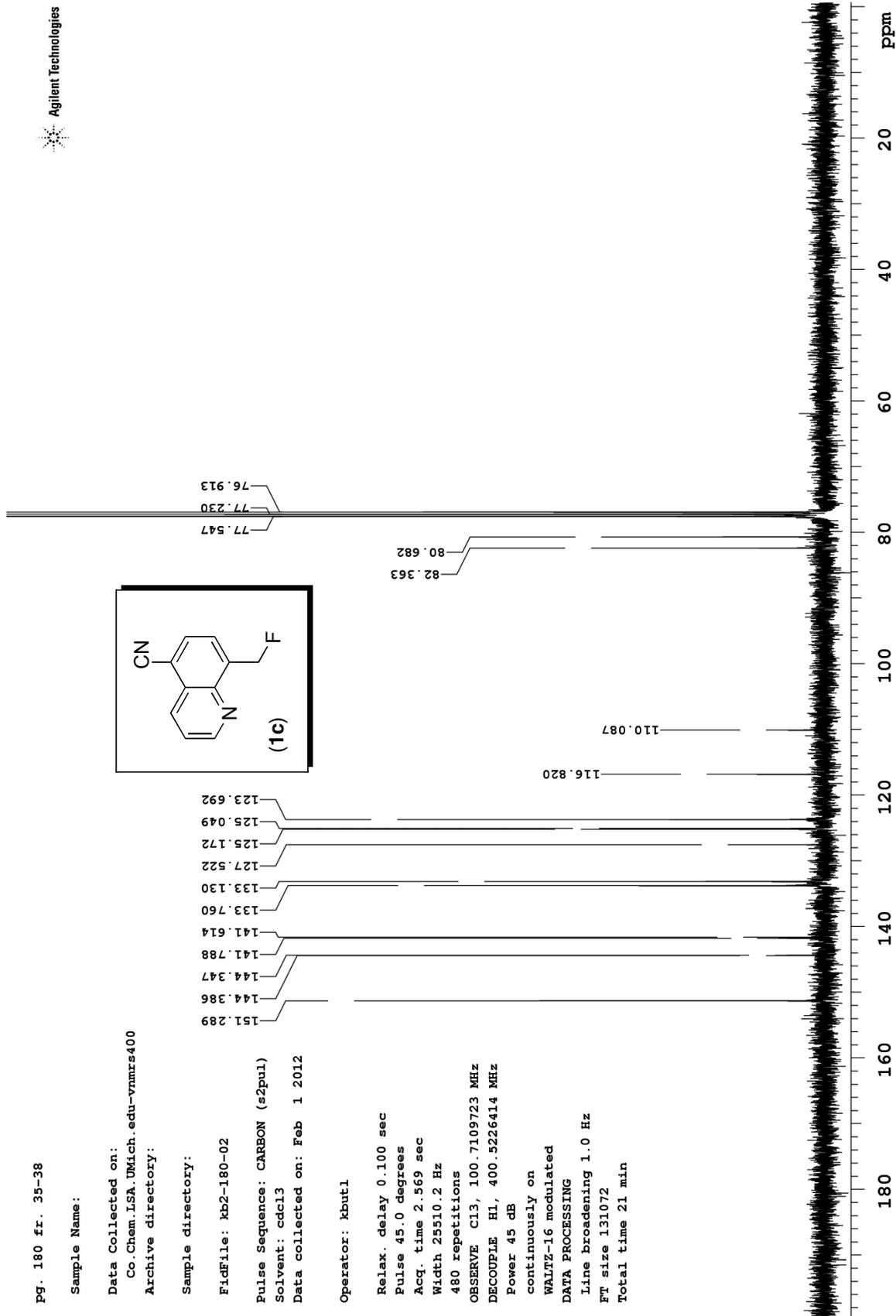
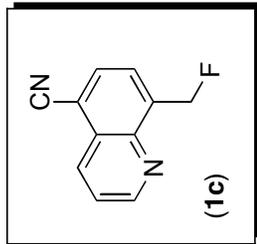
Sample directory:

FidFile: kb2-180-02

Pulse Sequence: CARBON (s2pul)  
Solvent: cdcl3  
Data collected on: Feb 1 2012

Operator: kbutl

Relax. delay 0.100 sec  
Pulse 45.0 degrees  
Acq. time 2.569 sec  
Width 25510.2 Hz  
480 repetitions  
OBSERVE C13, 100.7109723 MHz  
DECOUPLE H1, 400.5226414 MHz  
Power 45 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 131072  
Total time 21 min



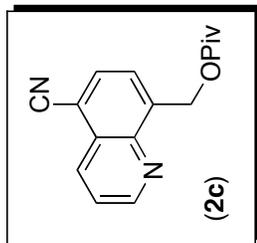


Pg. 99-2 C-O fr. 14-20



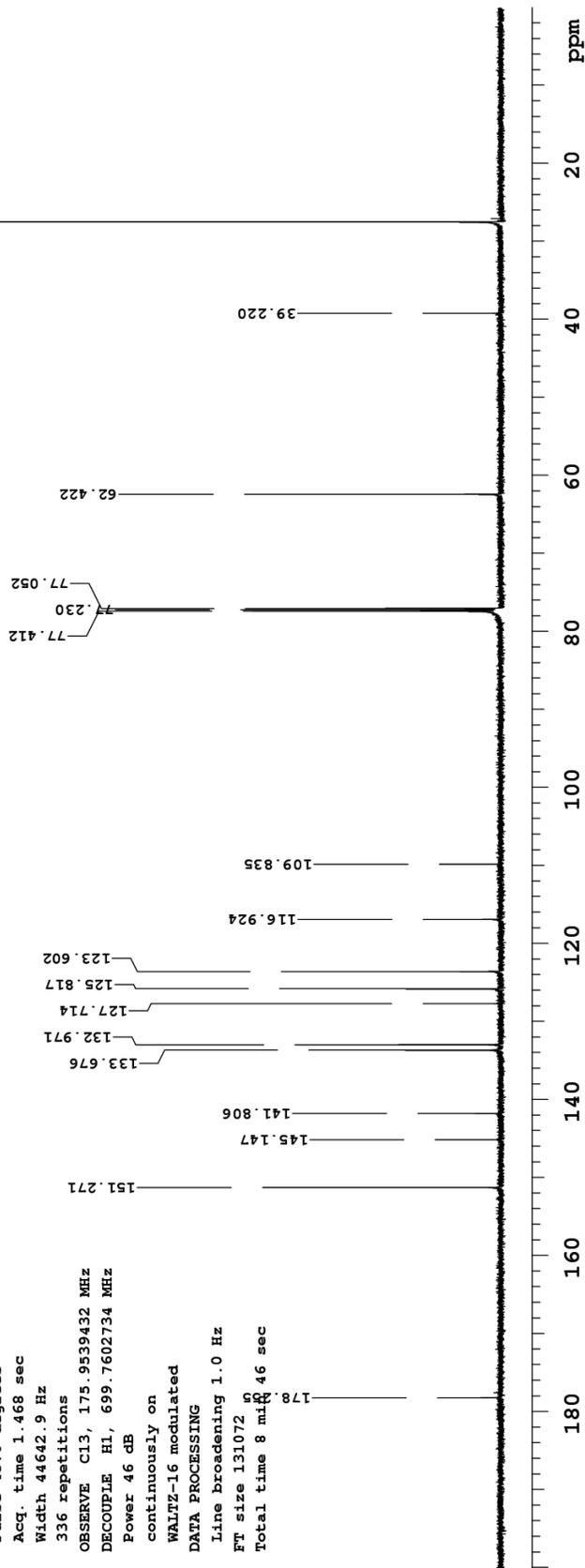
Sample Name:

Data Collected on:  
Yb-vnmrs700  
Archive directory:  
Sample directory:  
FidFile: kb4-99-04  
Pulse Sequence: CARBON (s2pul)  
Solvent: cdcl3  
Data collected on: Apr 10 2012



Operator: kbut1

Relax. delay 0.100 sec  
Pulse 45.0 degrees  
Acq. time 1.468 sec  
Width 44642.9 Hz  
336 repetitions  
OBSERVE C13, 175.9539432 MHz  
DECOUPLE H1, 699.7602734 MHz  
Power 46 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
Ft size 131072  
Total time 8 min 0.46 sec



pg. 200 CO2Mesub

Sample Name:

Data Collected on:

Te-vmmrs500

Archive directory:

Sample directory:

FidFile: kb-CO2Mesub-H

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Jun 19 2012

Operator: kbutl

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 8012.8 Hz

16 repetitions

OBSERVE H1, 500.0931690 MHz

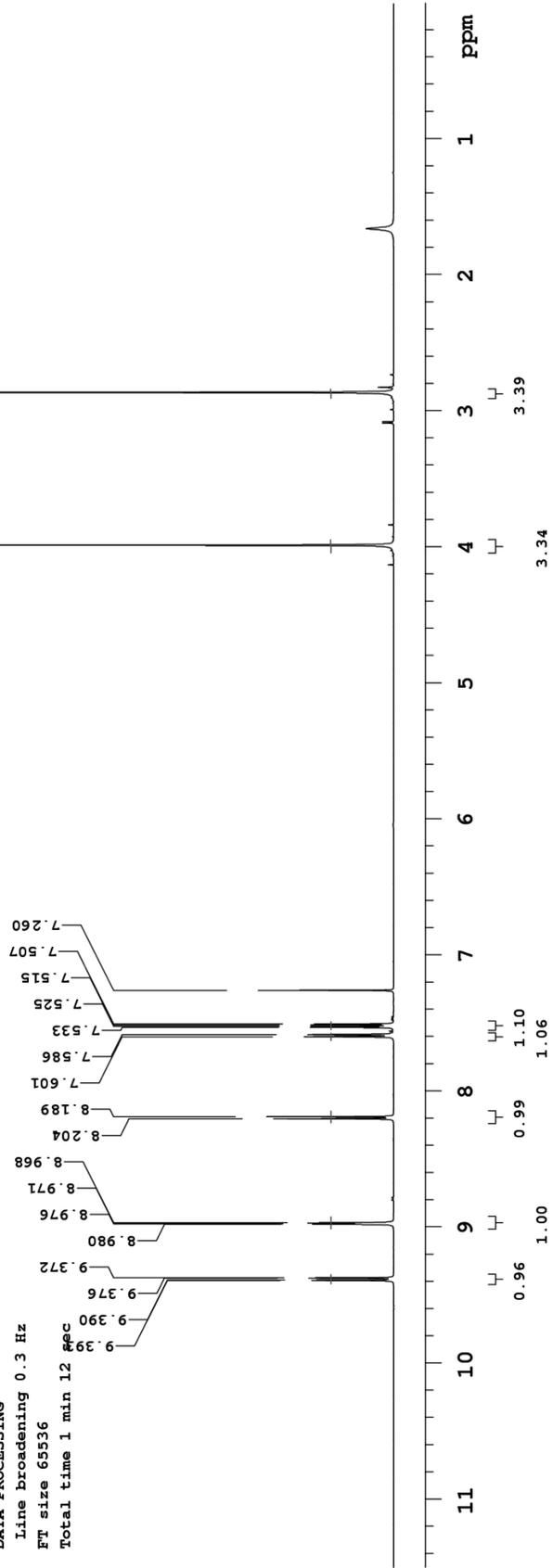
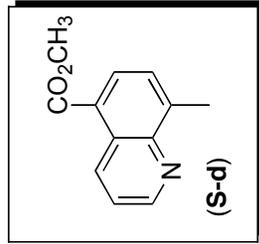
DATA PROCESSING

Line broadening 0.3 Hz

FT size 65536

Total time 1 min 12 sec

Agilent Technologies



Sample Name:

Data Collected on:  
Te-vmmrs500  
Archive directory:

Sample directory:

FidFile: kb-CO2Mesub-C

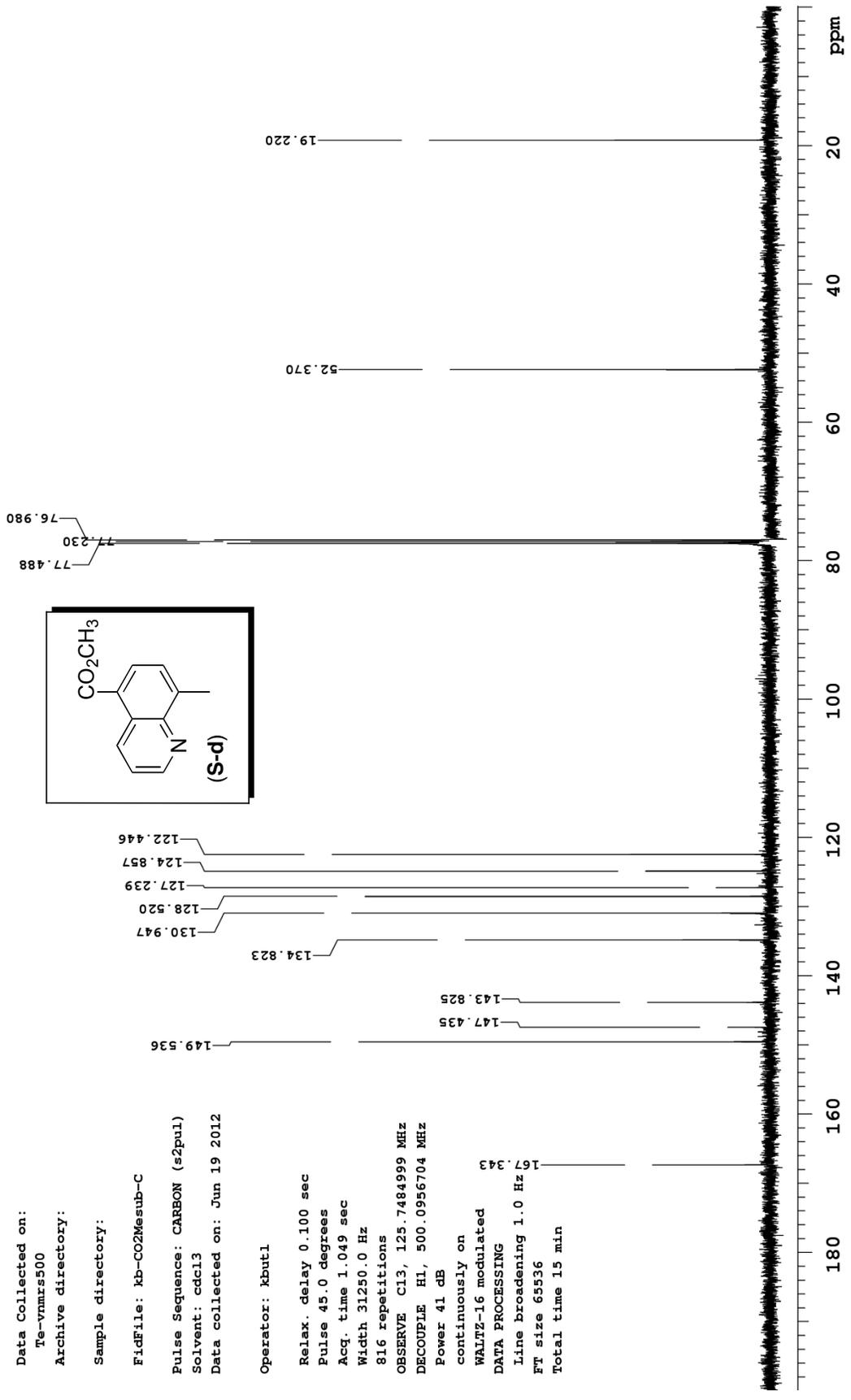
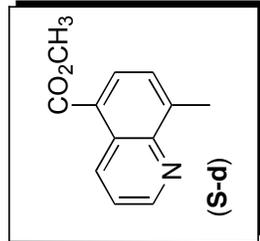
Pulse Sequence: CARBON (s2pul)  
Solvent: cdc13  
Data collected on: Jun 19 2012

Operator: kbut1

Relax. delay 0.100 sec  
Pulse 45.0 degrees  
Acq. time 1.049 sec  
Width 31250.0 Hz  
816 repetitions

OBSERVE C13, 125.7484999 MHZ  
DECOUPLE H1, 500.0956704 MHZ

Power 41 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 15 min



Sample Name:

Data Collected on:

Te-vmrms500

Archive directory:

Sample directory:

FidFile: kb4-100-last

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Jun 21 2012

Temp. 25.0 C / 298.1 K

Operator: kbutl

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 8012.8 Hz

16 repetitions

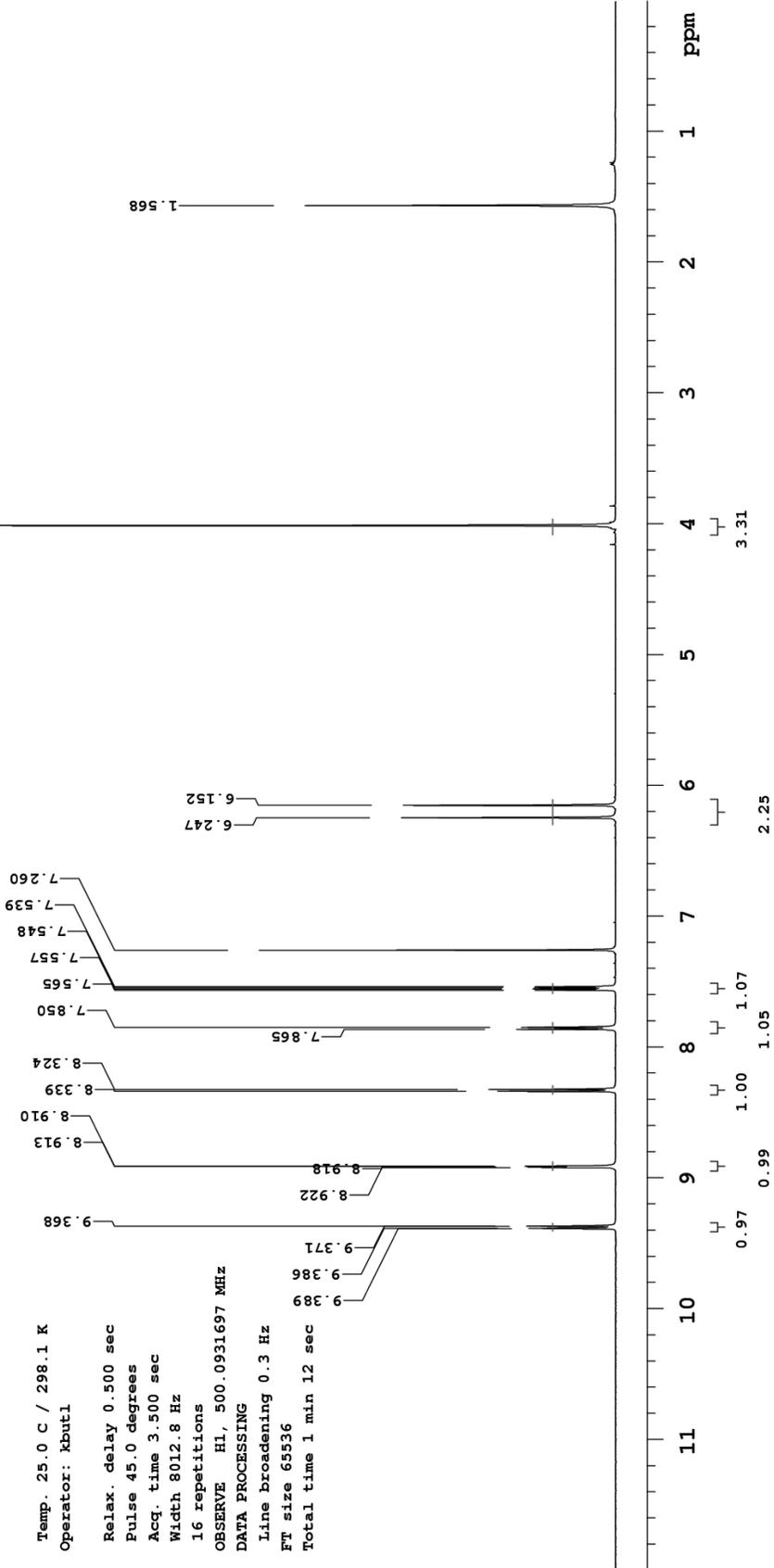
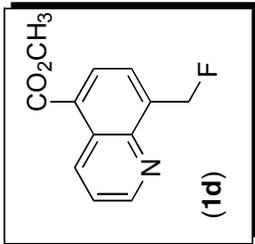
OBSERVE H1, 500.0931697 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 65536

Total time 1 min 12 sec



Sample Name:

Data Collected on:

YB-vnmrs700

Archive directory:

Sample directory:

FidFile: kb4-100-02

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Mar 26 2012

Operator: kbut1

Relax. delay 0.100 sec

Pulse 45.0 degrees

Acq. time 1.468 sec

Width 44642.9 Hz

1888 repetitions

OBSERVE C13, 175.9539398 MHz

DECOUPLE H1, 699.7602734 MHz

Power 46 dB

continuously on

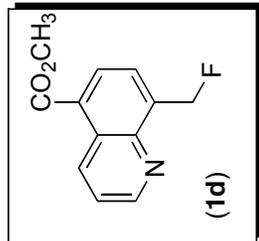
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 49 min



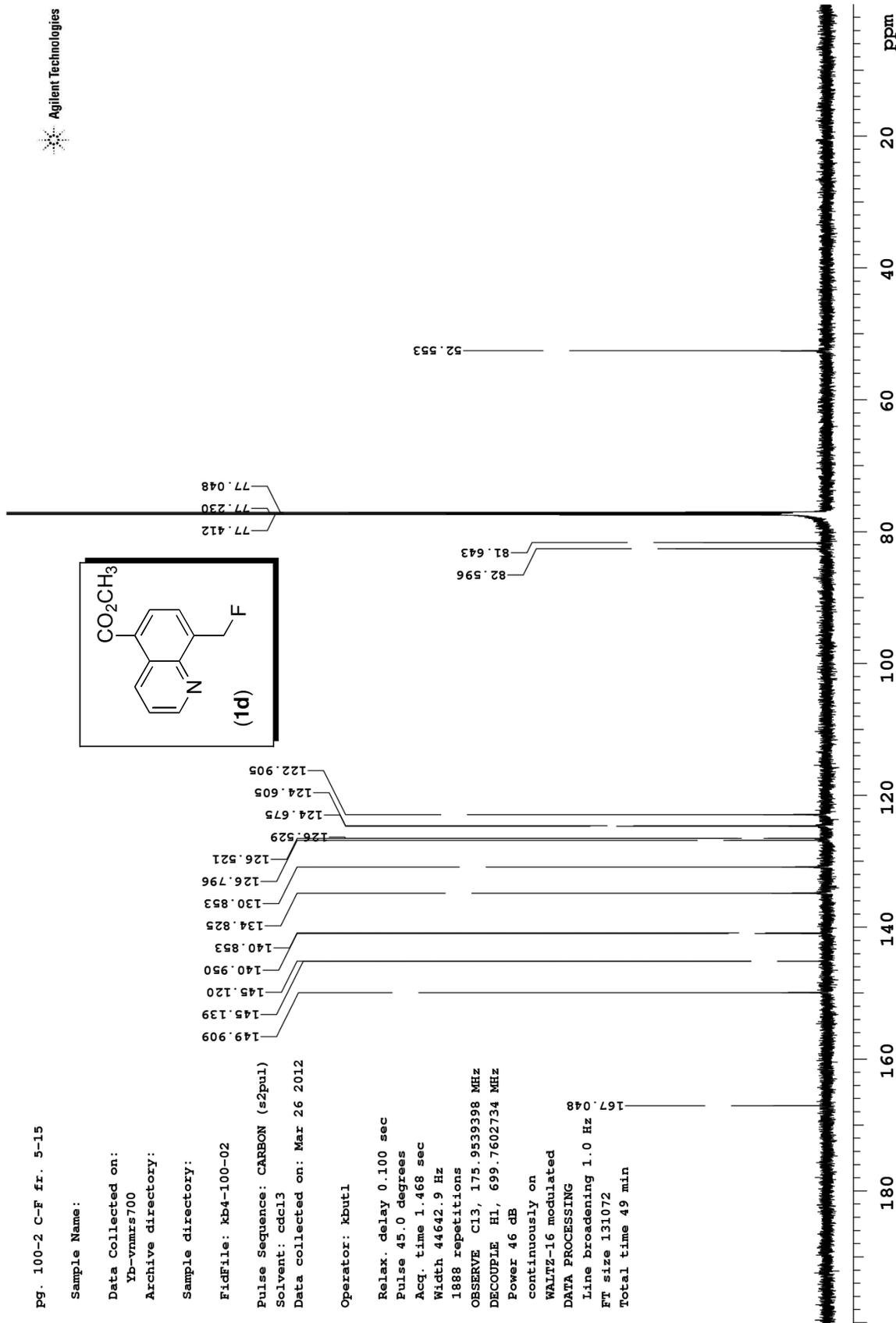
149.909  
145.139  
145.120  
140.950  
140.853  
134.825  
130.853  
126.796  
126.521  
126.529  
124.675  
124.605  
122.905

77.412  
77.230  
77.048

82.596  
81.643

52.553

167.048



Sample Name:

Data Collected on:

Yb-vmrs700

Archive directory:

Sample directory:

FidFile: kb4-100-03

Pulse Sequence: PROTON (s2pul)

Solvent: cdc13

Data collected on: Mar 30 2012

Temp. 25.0 C / 298.1 K

Operator: kbut1

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 11160.7 Hz

16 repetitions

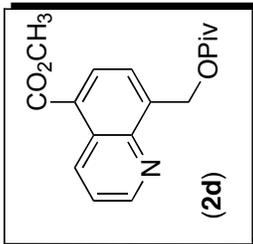
OBSERVE H1, 699.7567683 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 131072

Total time 1 min 17.850



5.902

7.260  
7.533  
7.539  
7.546  
7.551  
7.725

7.727  
7.736  
7.737

8.280  
8.291

8.944  
8.946

8.949  
8.952

9.355  
9.358  
9.360

1.397

4.086

11 10 9 8 7 6 5 4 3 2 1 ppm

9.25

3.30

2.16

1.08

0.98

1.00

0.96

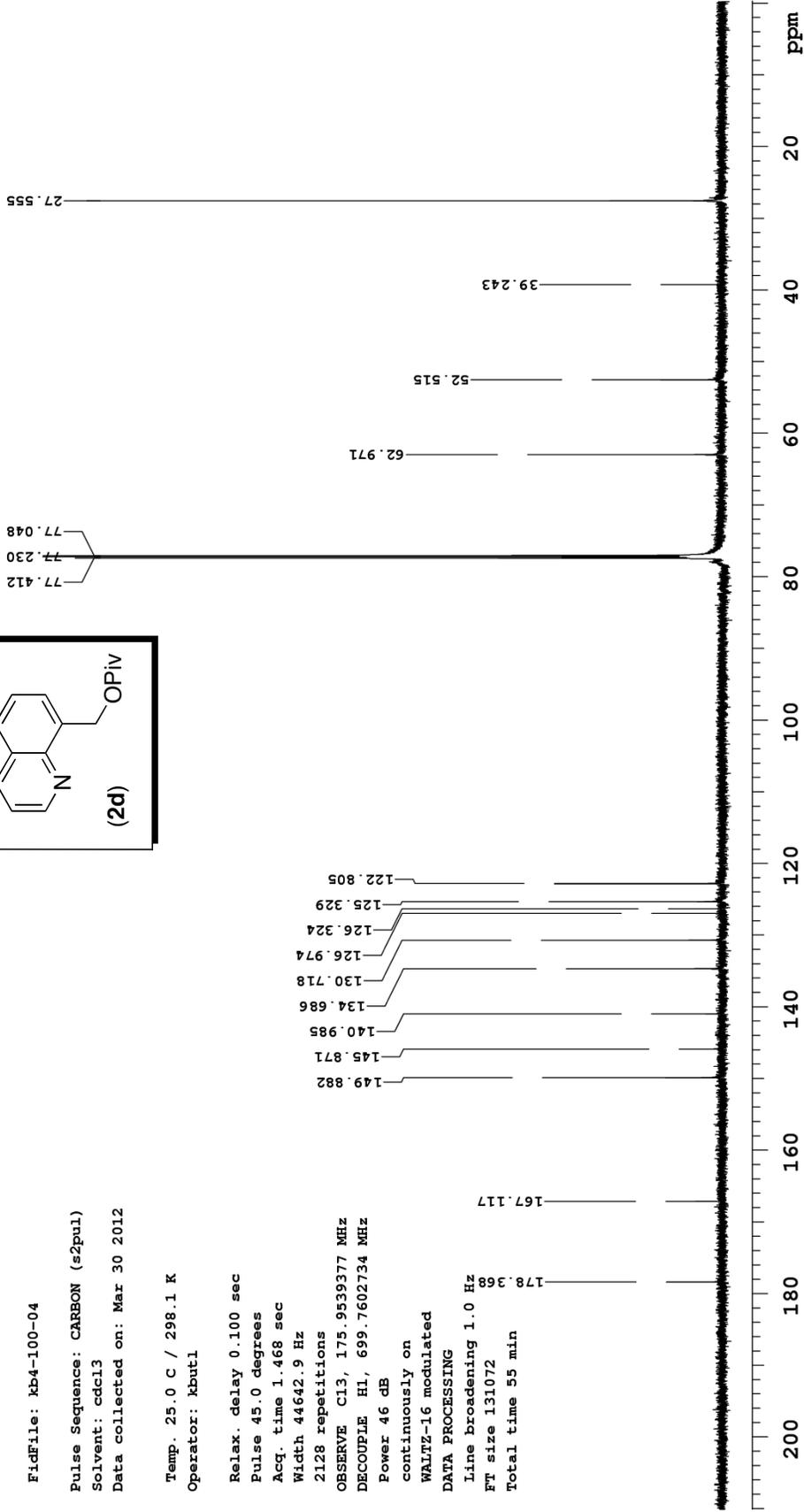
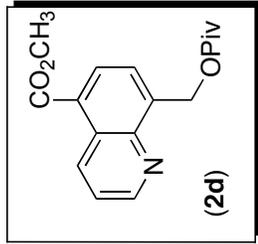
0.90

Sample Name:

Data Collected on: Yb-vnmrs700  
Archive directory:  
Sample directory:  
FidFile: kb4-100-04  
Pulse Sequence: CARBON (s2pul)  
Solvent: cdcl3  
Data collected on: Mar 30 2012

Temp. 25.0 C / 298.1 K  
Operator: kbutl

Relax. delay 0.100 sec  
Pulse 45.0 degrees  
Acq. time 1.468 sec  
Width 44642.9 Hz  
2128 repetitions  
OBSERVE C13, 175.9539377 MHz  
DECOUPLE H1, 699.7602734 MHz  
Power 46 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 131072  
Total time 55 min



Found Fsub

Sample Name:

Data Collected on:

Te-vnmrs500

Archive directory:

Sample directory:

FidFile: kb-Fsub-H

Pulse Sequence: PROTON (s2pu1)

Solvent: cdcl3

Data collected on: Jun 19 2012

Operator: kbut1

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 8012.8 Hz

16 repetitions

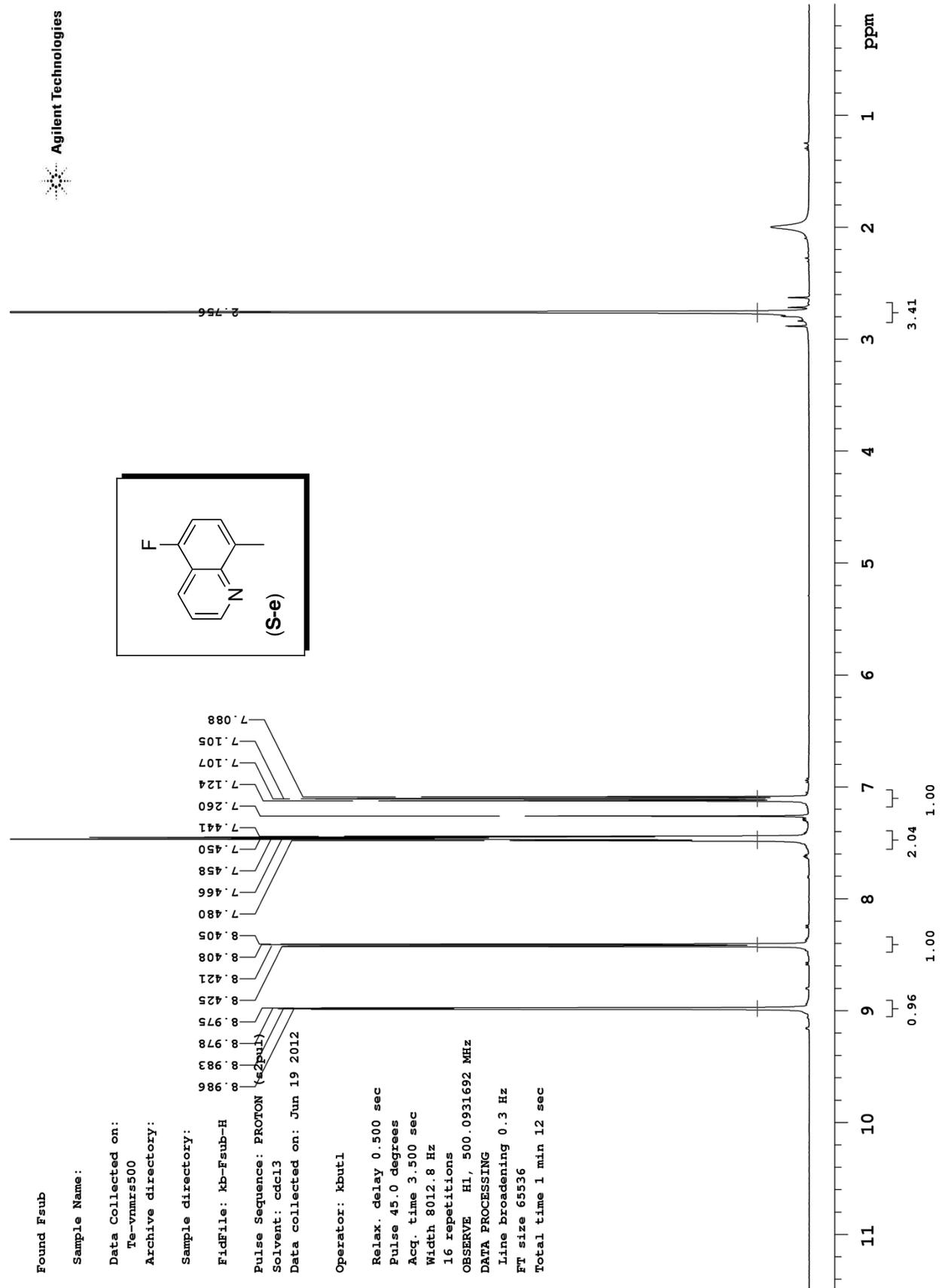
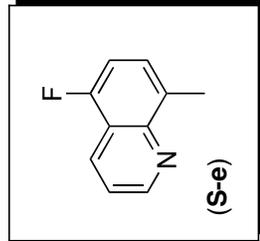
OBSERVE H1, 500.0931692 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 65536

Total time 1 min 12 sec



Found Fsub

Sample Name:

Data Collected on:

Te-vmr500

Archive directory:

Sample directory:

FidFile: kb-Fsub-C

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Jun 19 2012

Operator: kbutl

Relax. delay 0.100 sec

Pulse 45.0 degrees

Acq. time 1.049 sec

Width 31250.0 Hz

592 repetitions

OBSERVE C13, 125.7485008 MHz

DECOUPLE H1, 500.0956704 MHz

Power 41 dB

continuously on

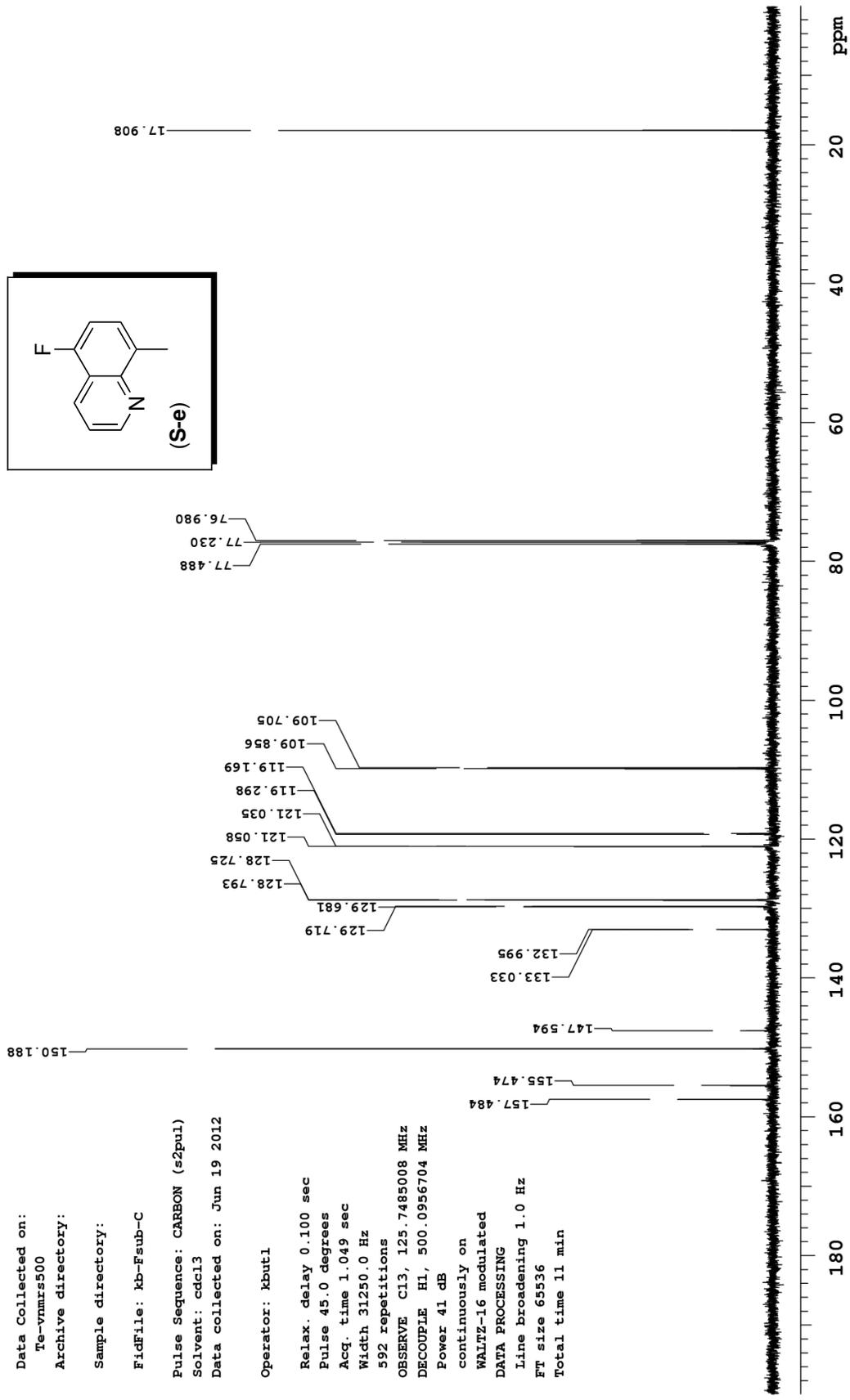
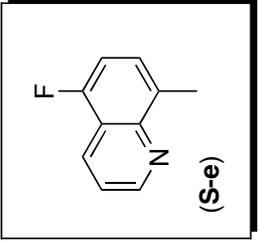
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 11 min



Sample Name:

Data Collected on:

Yb-vmrs700

Archive directory:

Sample directory:

FidFile: kb4-117-01

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Apr 16 2012

Operator: kbut1

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 11160.7 Hz

16 repetitions

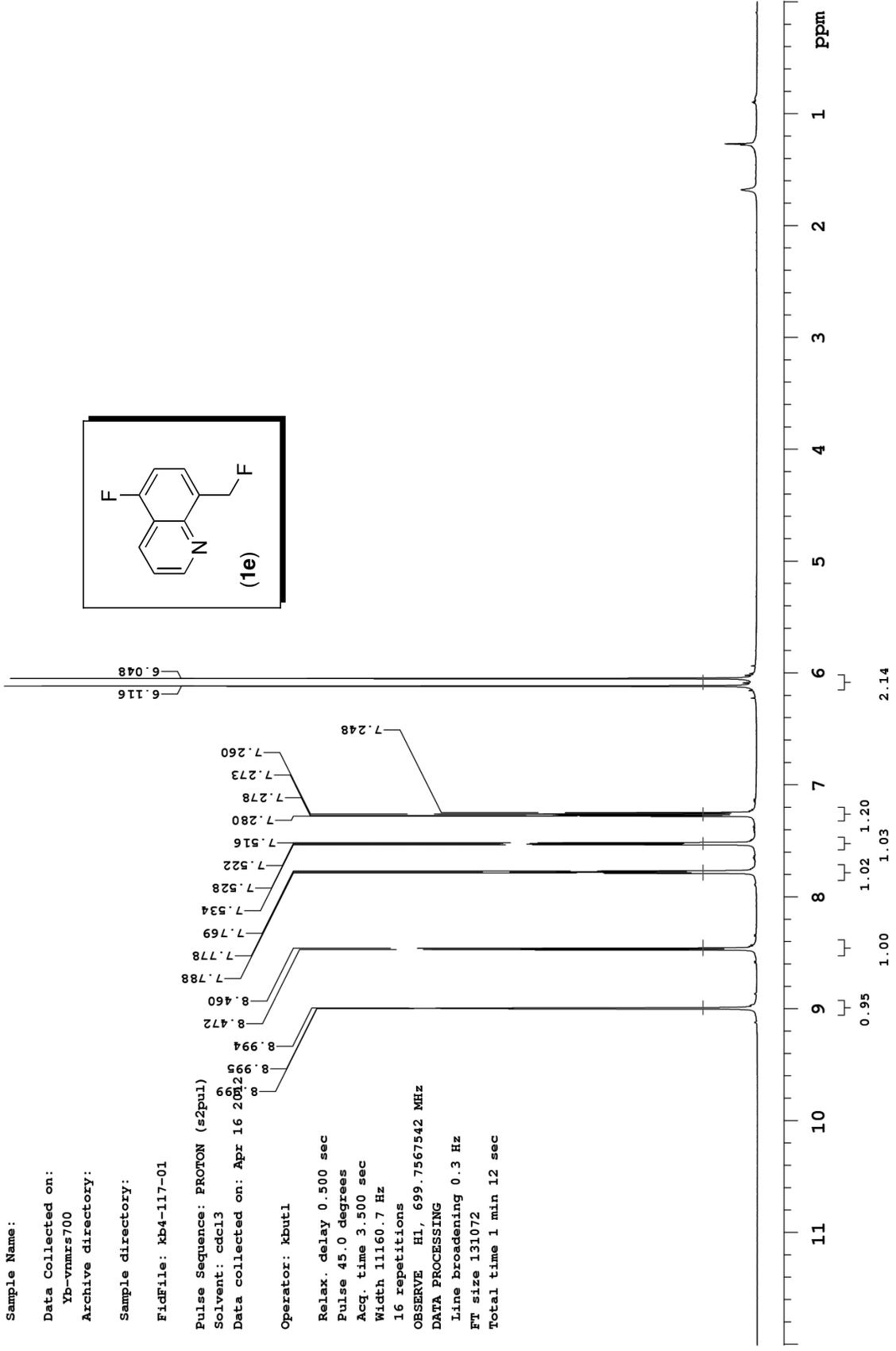
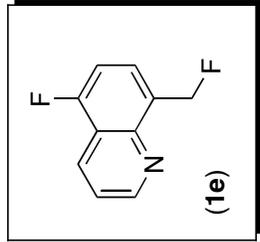
OBSERVE H1, 699.7567542 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 131072

Total time 1 min 12 sec



Sample Name:

Data Collected on:

Yb-vnmrs700

Archive directory:

Sample directory:

FidFile: kb4-117-02

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Apr 16 2012

Operator: kbut1

Relax. delay 0.100 sec

Pulse 45.0 degrees

Acq. time 1.468 sec

Width 44642.9 Hz

896 repetitions

OBSERVE C13, 175.9539418 MHz

DECOUPLE H1, 699.7602734 MHz

Power 46 dB

continuously on

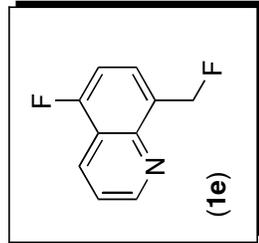
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 23 min



77.048  
77.230  
77.412

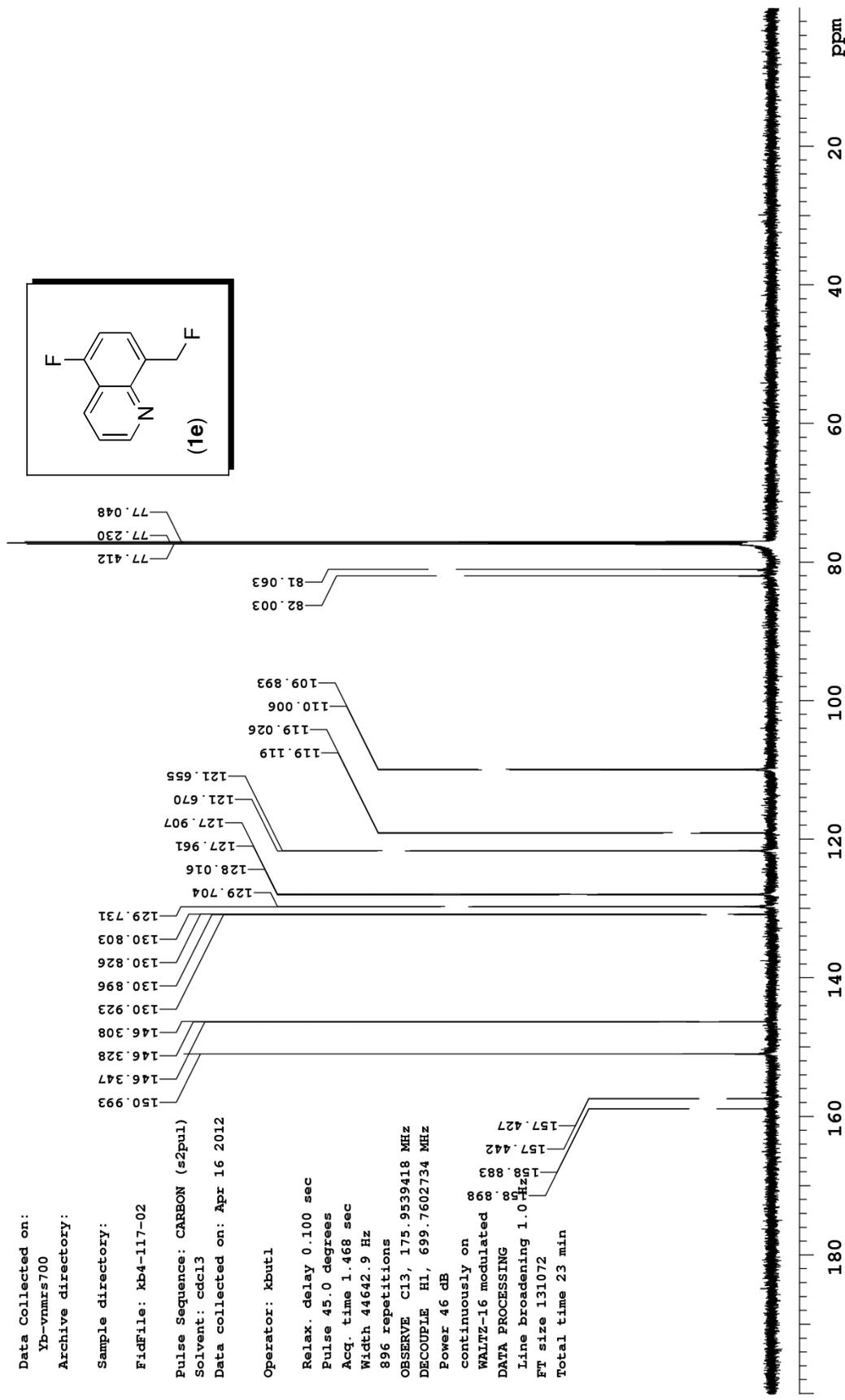
81.063  
82.003

109.893  
110.006  
119.026  
119.119

121.655  
121.670  
127.907  
127.961  
128.016  
129.704

129.731  
130.803  
130.826  
130.896  
130.923  
146.308  
146.328  
146.347  
150.993

157.427  
157.442  
158.883



Sample Name:

Data Collected on:  
Sn.Chem.LSA.UMich.edu-inova500  
Archive directory:

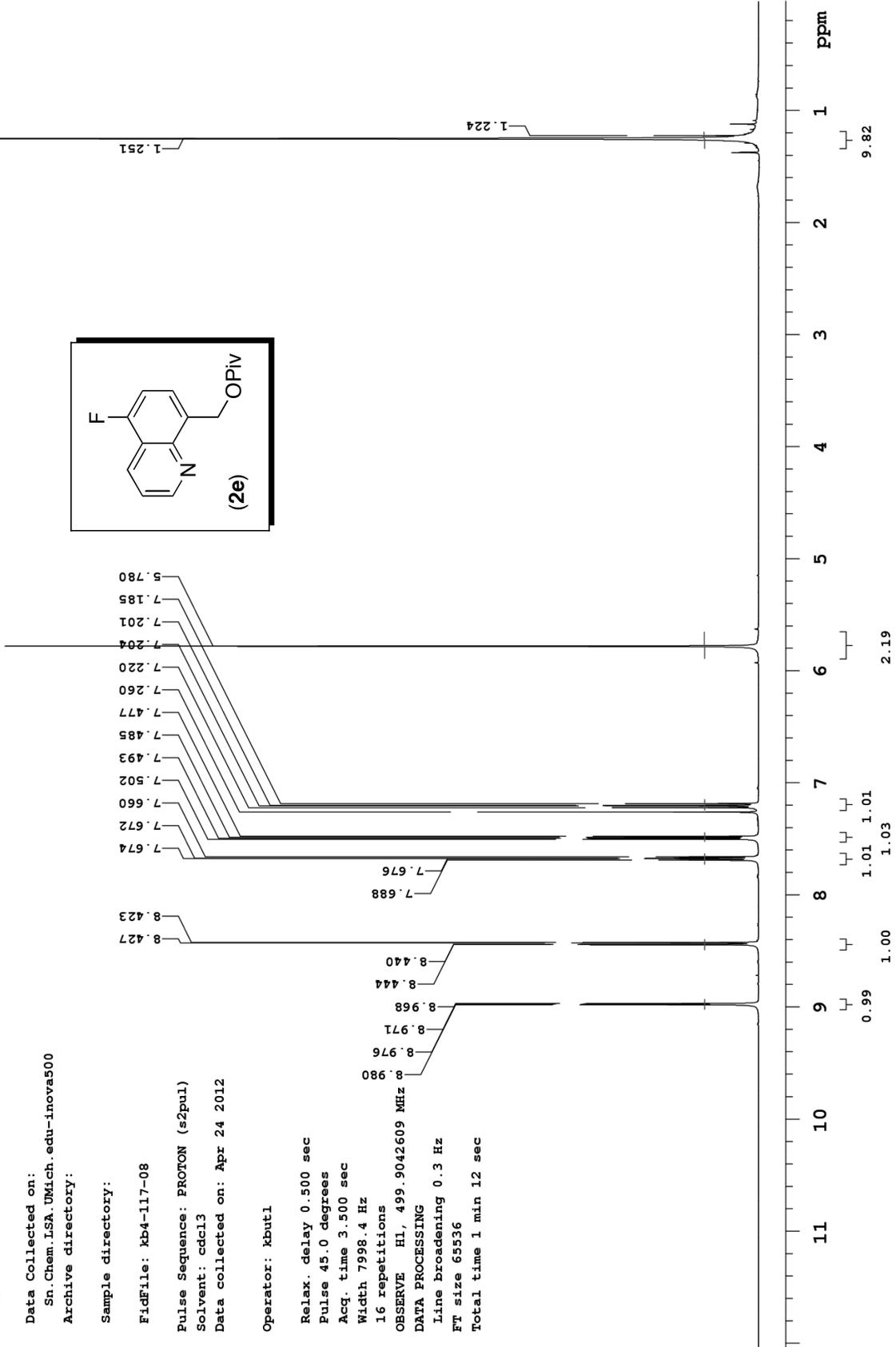
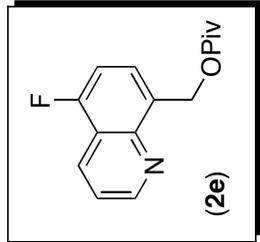
Sample directory:

FidFile: kb4-117-08

Pulse Sequence: PROTON (s2pul)  
Solvent: cdcl3  
Data collected on: Apr 24 2012

Operator: kbut1

Relax. delay 0.500 sec  
Pulse 45.0 degrees  
Acq. time 3.500 sec  
Width 7998.4 Hz  
16 repetitions  
OBSERVE H1, 499.9042609 MHz  
DATA PROCESSING  
Line broadening 0.3 Hz  
FT size 65536  
Total time 1 min 12 sec

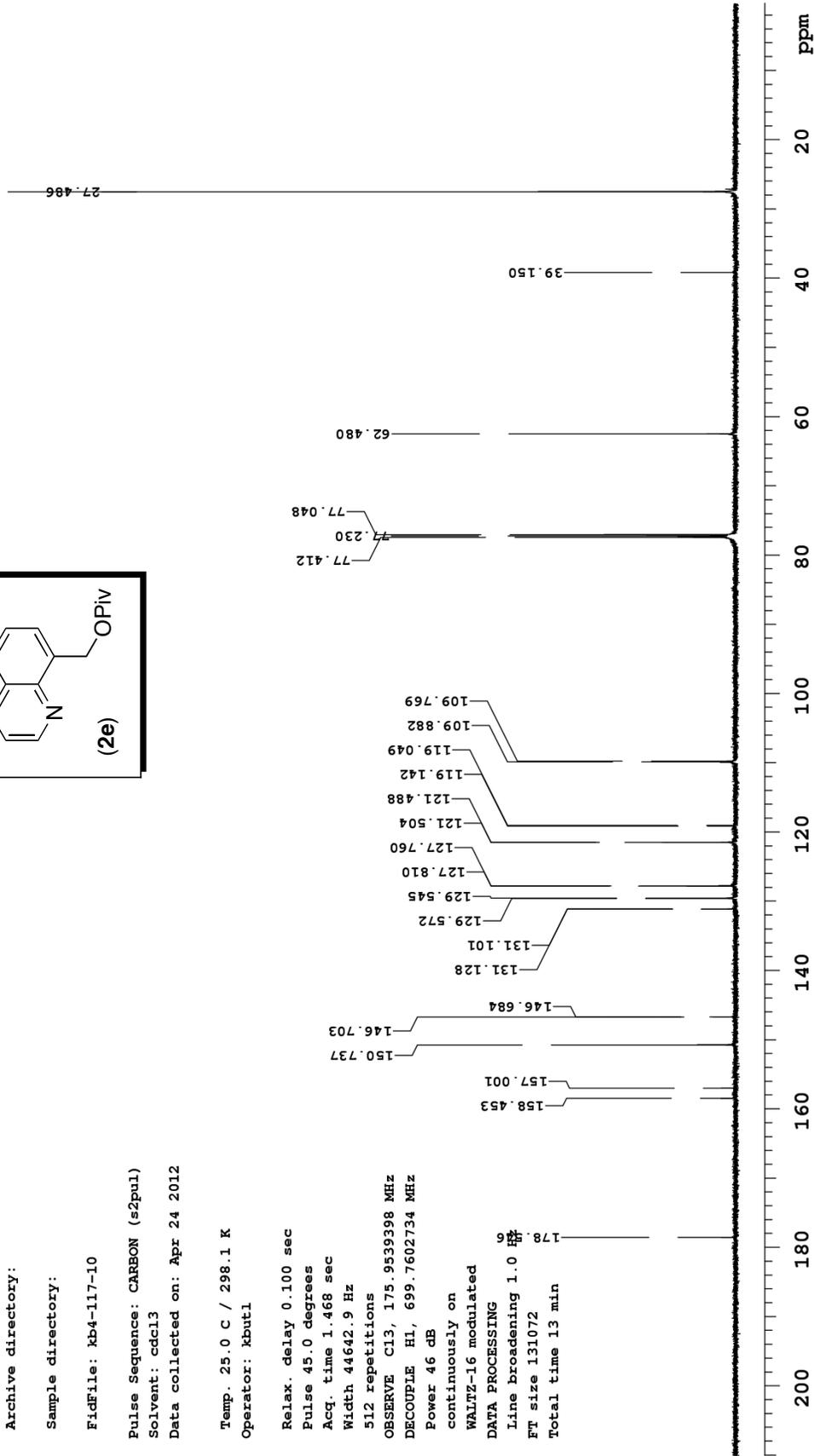
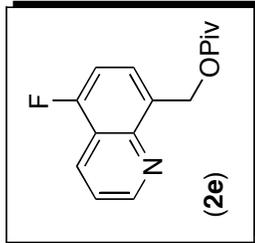


Sample Name:

Data Collected on: Yb-vnmrs700  
Archive directory:  
Sample directory:  
FidFile: kb4-117-10  
Pulse Sequence: CARBON (s2pul)  
Solvent: cdcl3  
Data collected on: Apr 24 2012

Temp. 25.0 C / 298.1 K  
Operator: kbut1

Relax. delay 0.100 sec  
Pulse 45.0 degrees  
Acq. time 1.468 sec  
Width 44642.9 Hz  
512 repetitions  
OBSERVE C13, 175.9539398 MHz  
DECOUPLE H1, 699.7602734 MHz  
Power 46 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 131072  
Total time 13 min



PG\_47 Br Sub

Sample Name:

Data Collected on:

Te-vnmrs500

Archive directory:

Sample directory:

FidFile: kb-Brsub-H

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Jun 18 2012

Operator: kbut1

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 8012.8 Hz

16 repetitions

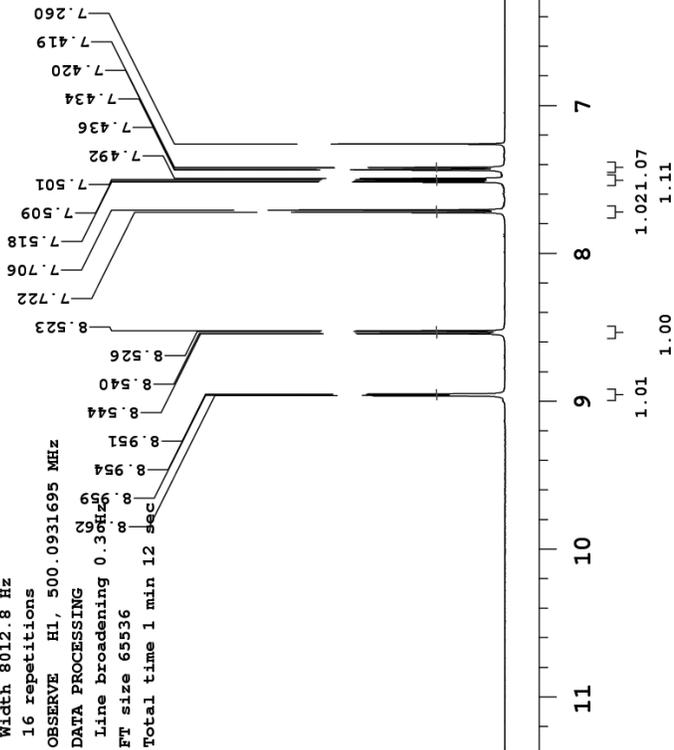
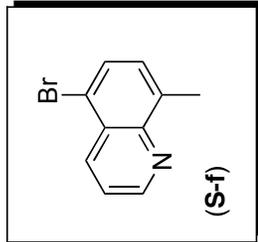
OBSERVE H1, 500.0931695 MHz

DATA PROCESSING

Line broadening 0.30 Hz

FT size 65536

Total time 1 min 12 sec

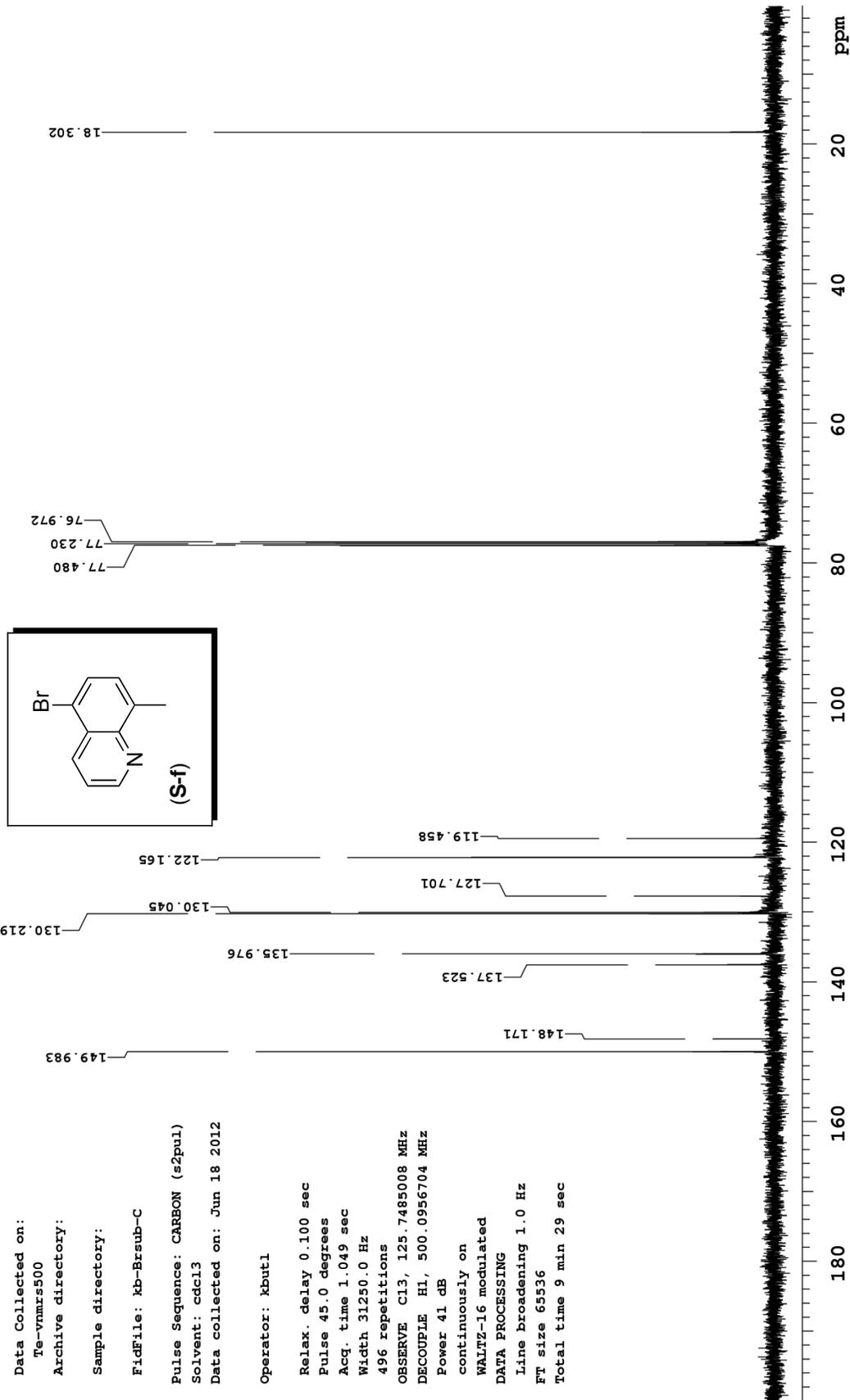


Sample Name :

Data Collected on: Te-vmrms500  
Archive directory:  
Sample directory:  
FidFile: kb-Brsub-C  
Pulse Sequence: CARBON (s2pul)  
Solvent: cdcl3  
Data collected on: Jun 18 2012

Operator: kbutl

Relax. delay 0.100 sec  
Pulse 45.0 degrees  
Acq. time 1.049 sec  
Width 31250.0 Hz  
496 repetitions  
OBSERVE C13, 125.7485008 MHz  
DECOUPLE H1, 500.0956704 MHz  
Power 41 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 9 min 29 sec



PG\_101 C-F fr. 8-10

Sample Name:

Data Collected on:

Yb-vmrs700

Archive directory:

Sample directory:

FidFile: kb4-101-02

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Mar 30 2012

Temp. 25.0 C / 298.1 K

Operator: kbut1

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 11160.7 Hz

16 repetitions

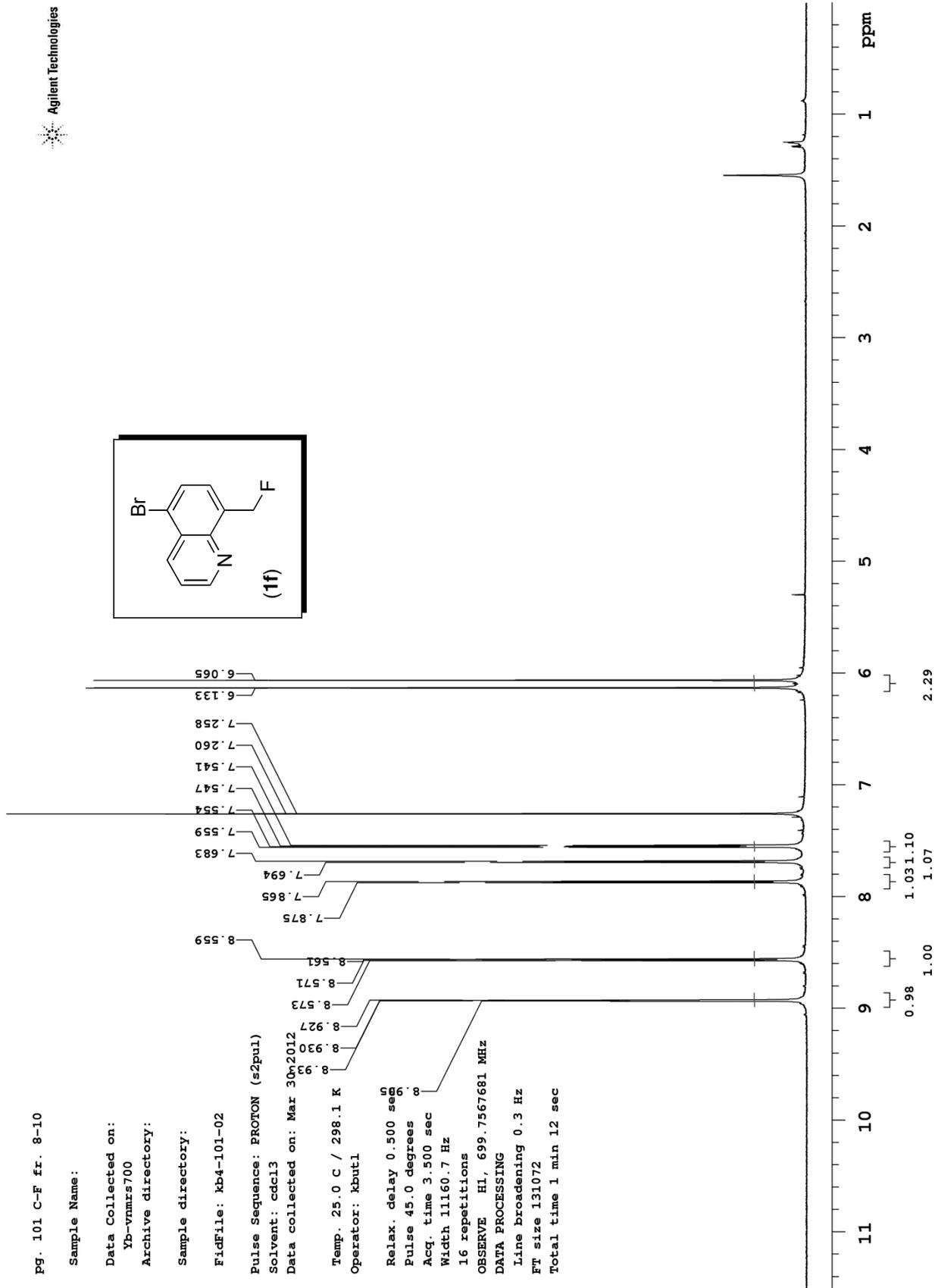
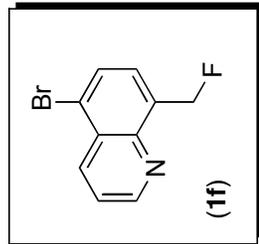
OBSERVE H1, 699.7567681 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 131072

Total time 1 min 12 sec

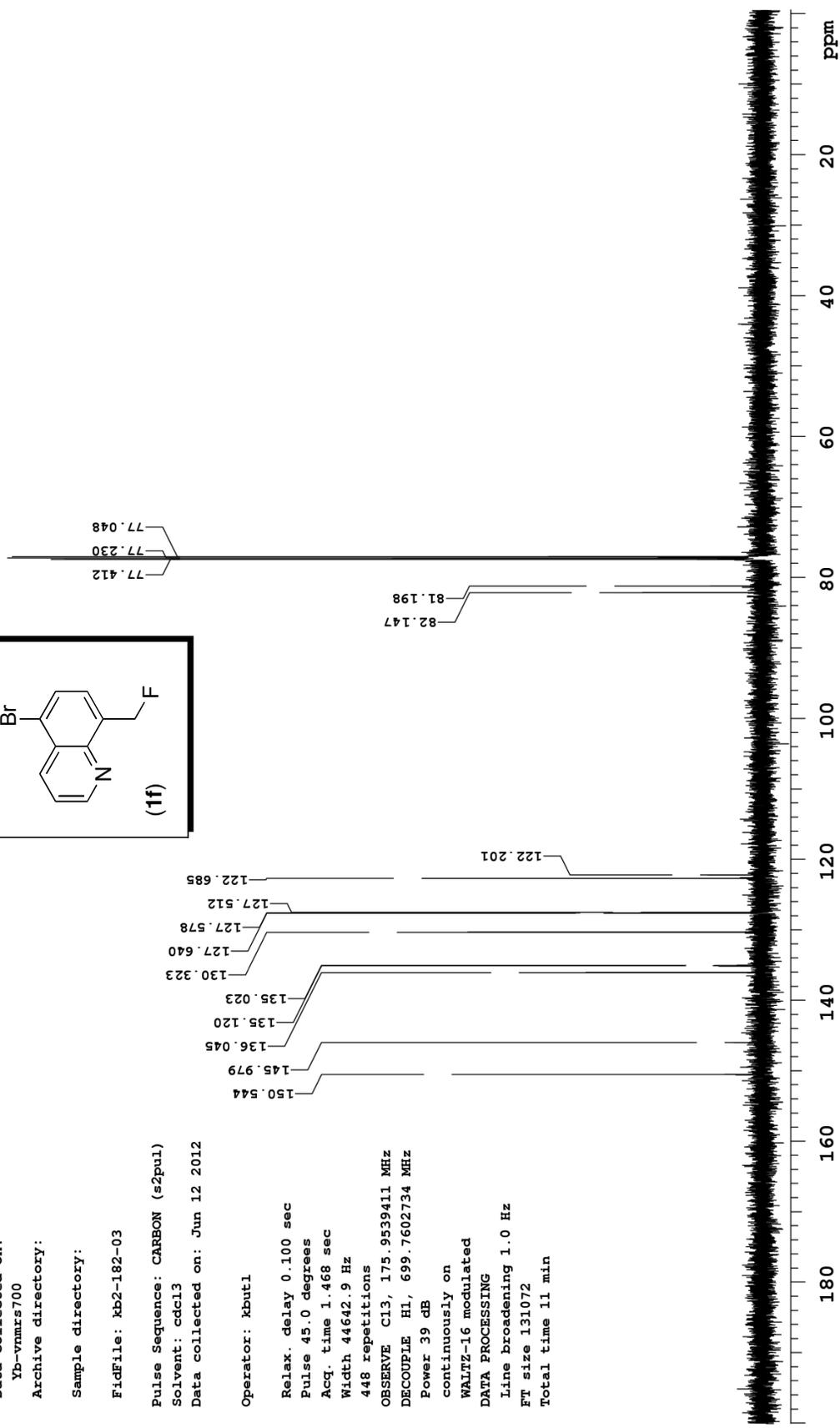
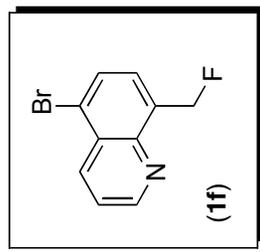


Sample Name:

Data Collected on: Yb-vnmrs700  
Archive directory:  
Sample directory:  
FidFile: kb2-182-03  
Pulse Sequence: CARBON (s2pul)  
Solvent: cdcl3  
Data collected on: Jun 12 2012

Operator: kbutl

Relax. delay 0.100 sec  
Pulse 45.0 degrees  
Acq. time 1.468 sec  
Width 44642.9 Hz  
448 repetitions  
OBSERVE C13, 175.9539411 MHz  
DECOUPLE H1, 699.7602734 MHz  
Power 39 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 131072  
Total time 11 min



Pg. 101-2 fr22-28 C-O

Sample Name:

Data Collected on:

Te-vmr500

Archive directory:

Sample directory:

FidFile: kb4-101-06

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Jun 11 2012

Temp. 25.0 C / 298.1 K

Operator: kbut1

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 8012.8 Hz

16 repetitions

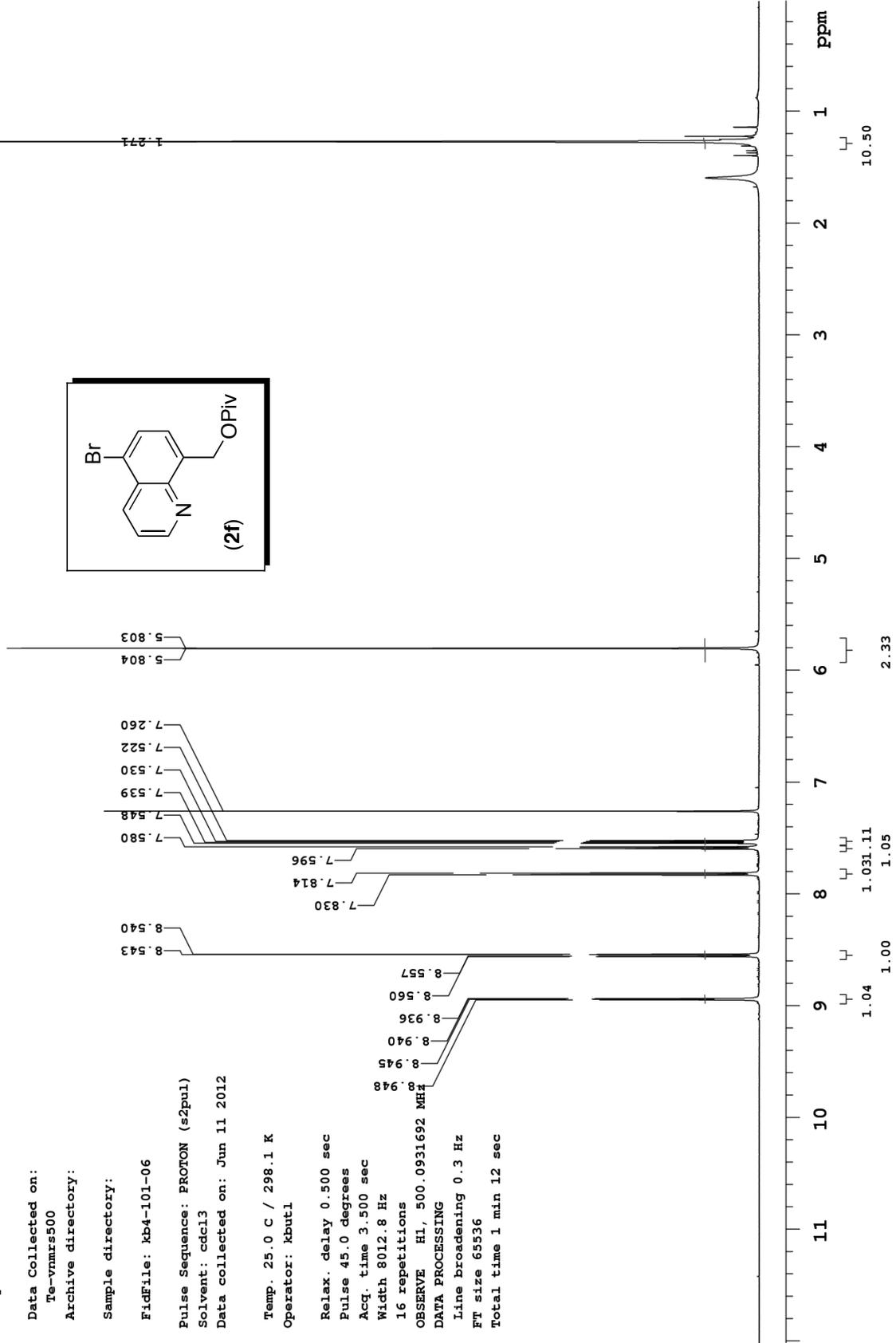
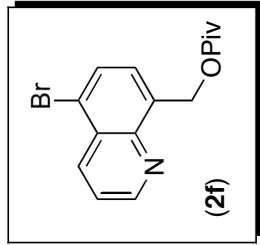
OBSERVE H1, 500.0931692 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 65536

Total time 1 min 12 sec



Sample Name:

Data Collected on:

Yb-vnmrs700

Archive directory:

Sample directory:

File: Kb4-101-05

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Apr 5 2012

Operator: kbut1

Relax. delay 0.100 sec

Pulse 45.0 degrees

Acq. time 1.468 sec

Width 44642.9 Hz

1360 repetitions

OBSERVE C13, 175.9539398 MHz

DECOUPLE H1, 699.7602734 MHz

Power 46 dB

continuously on

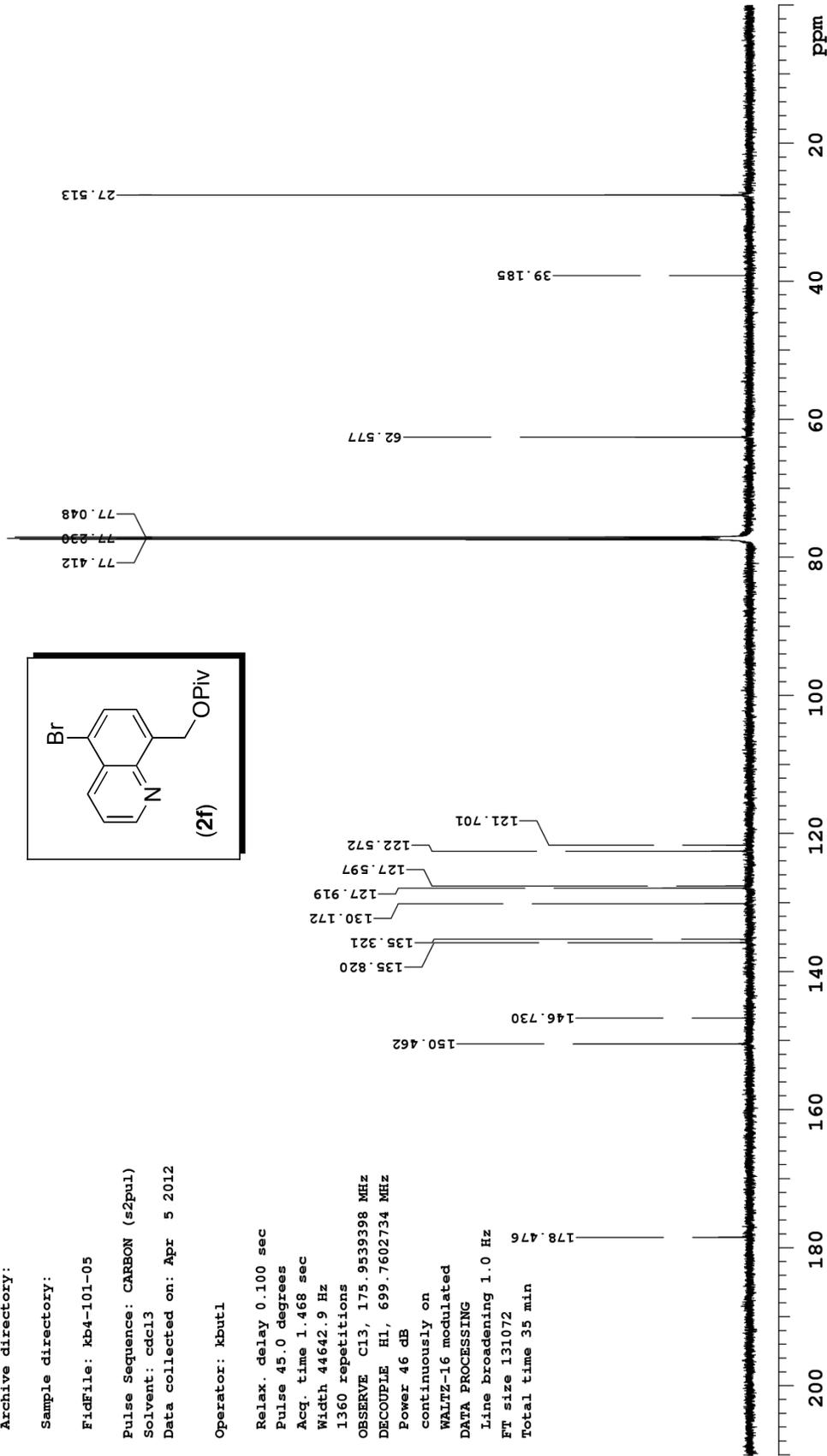
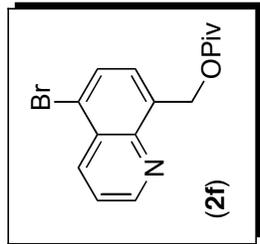
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 35 min



pg. 19 (NDB) 7-Br Sub

Sample Name:

Data Collected on:

Te-vmnrs500

Archive directory:

Sample directory:

FidFile: kb-7Brsub-H

Pulse Sequence: PROTON (2) Full

Solvent: cdcl3

Data collected on: Jun 18 2012

Operator: kbut1

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 8012.8 Hz

16 repetitions

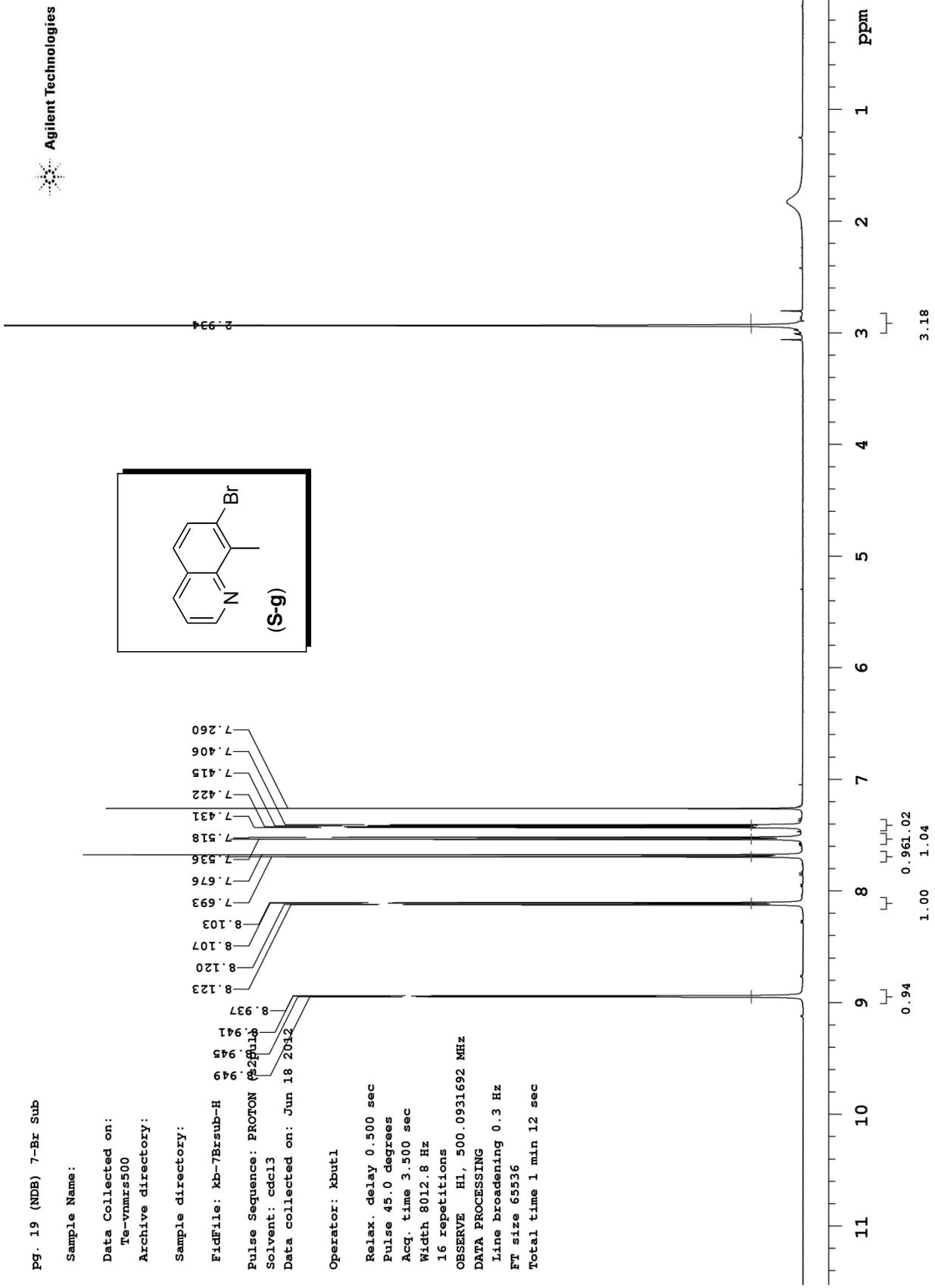
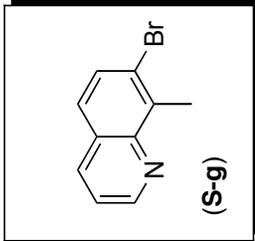
OBSERVE H1, 500.0931692 MHZ

DATA PROCESSING

Line broadening 0.3 Hz

FT size 65536

Total time 1 min 12 sec

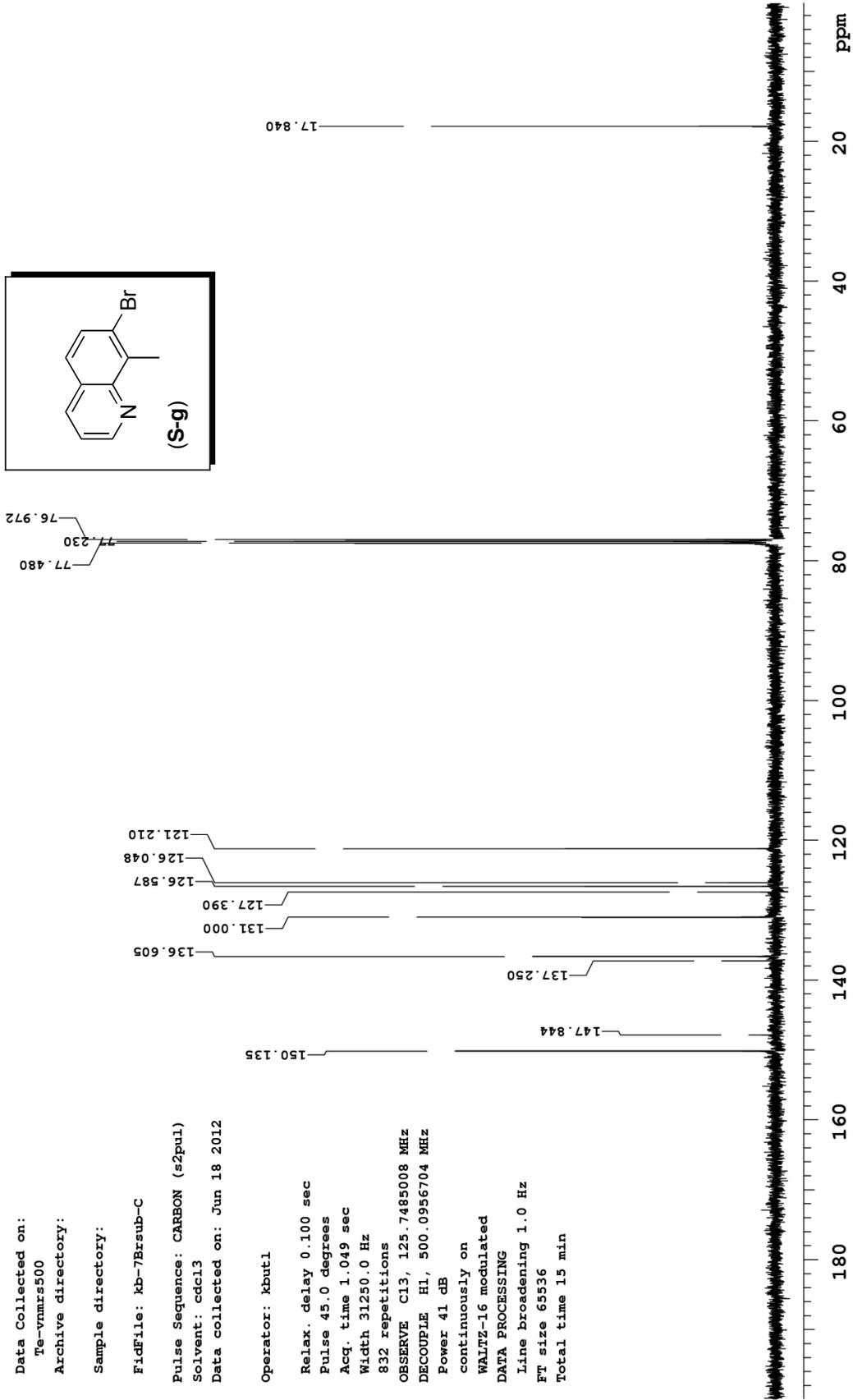


Sample Name:

Data Collected on: Te-vmr500  
Archive directory:  
Sample directory:  
FidFile: kb-7Brsub-C  
Pulse Sequence: CARBON (s2pul)  
Solvent: cdcl3  
Data collected on: Jun 18 2012

Operator: kbutl

Relax. delay 0.100 sec  
Pulse 45.0 degrees  
Acq. time 1.049 sec  
Width 31250.0 Hz  
832 repetitions  
OBSERVE C13, 125.7485008 MHz  
DECOUPLE H1, 500.0956704 MHz  
Power 41 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 15 min



Sample Name:

Data Collected on:

Te-vnmrs500

Archive directory:

Sample directory:

FidFile: kb4-98-09

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Jun 11 2012

Temp. 25.0 C / 298.1 K

Operator: kbut1

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 8012.8 Hz

16 repetitions

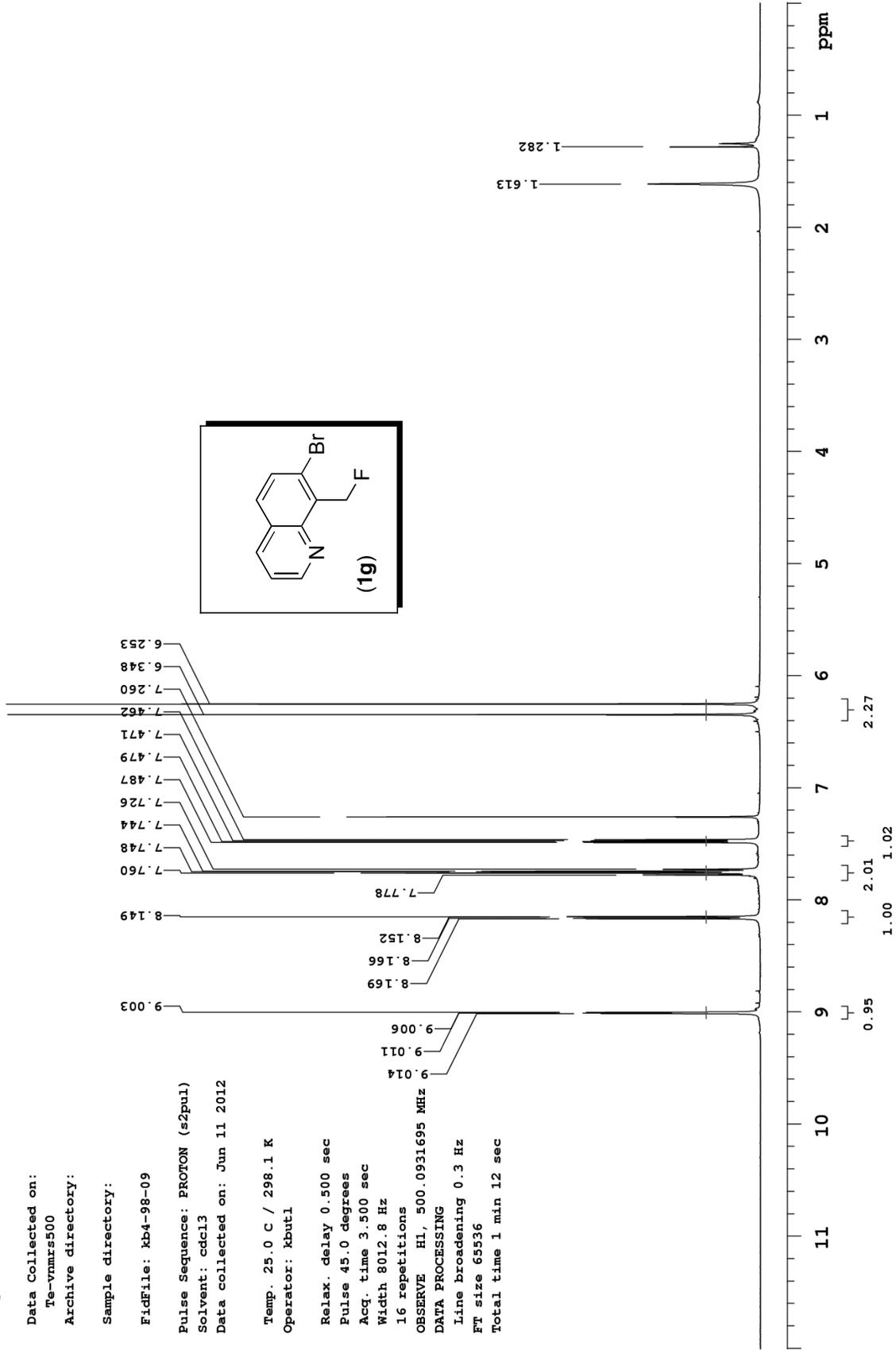
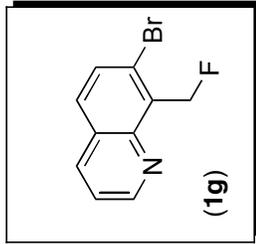
OBSERVE H1, 500.0931695 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 65536

Total time 1 min 12 sec





Sample Name:

Data Collected on:  
Yb-vnmrs700  
Archive directory:

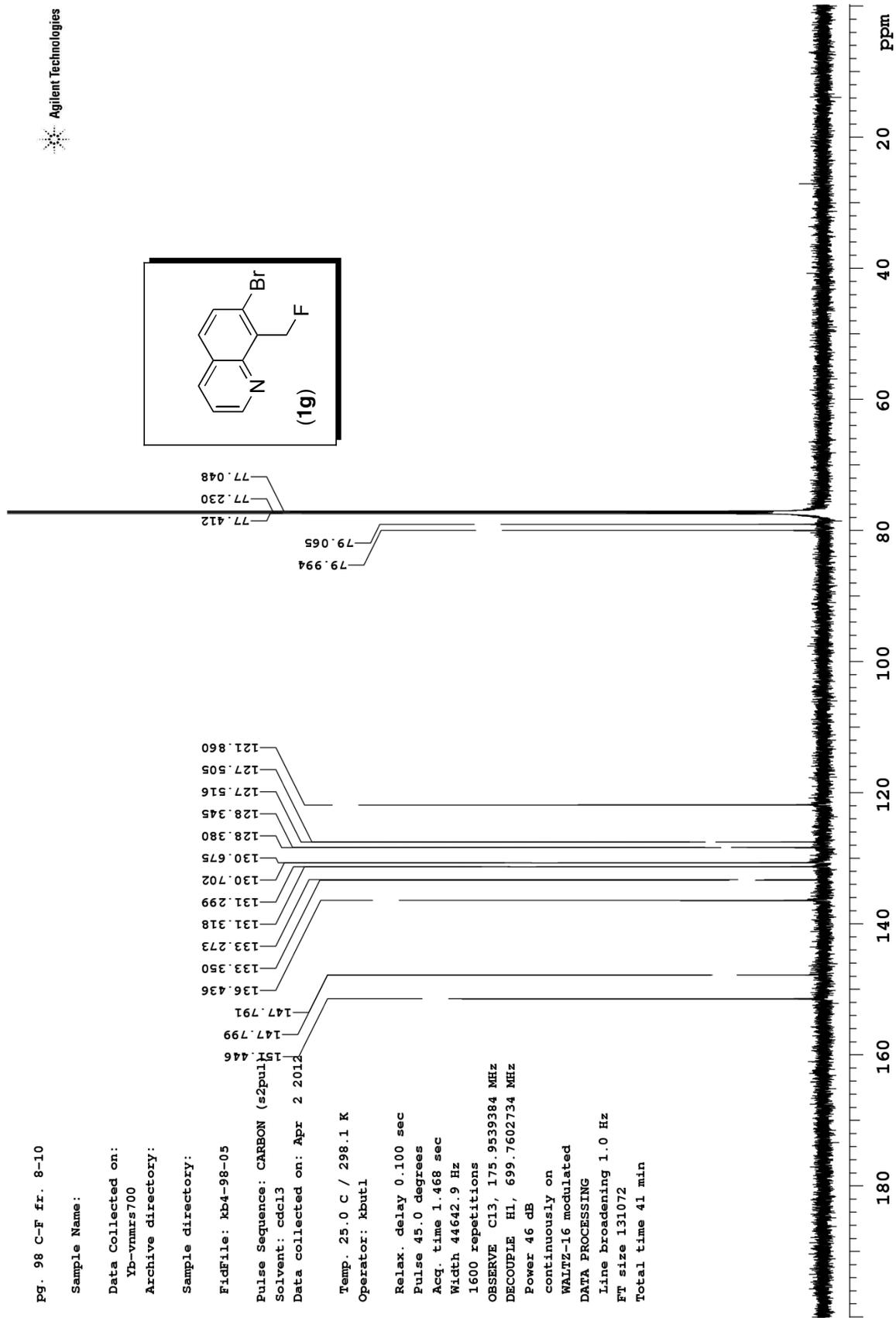
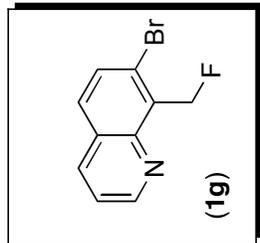
Sample directory:

File: kb4-98-05

Pulse Sequence: CARBON (s2pul)  
Solvent: cdcl3  
Data collected on: Apr 2 2012

Temp. 25.0 C / 298.1 K  
Operator: kbutl

Relax. delay 0.100 sec  
Pulse 45.0 degrees  
Acq. time 1.468 sec  
Width 44642.9 Hz  
1600 repetitions  
OBSERVE C13, 175.9539384 MHz  
DECOUPLE H1, 699.7602734 MHz  
Power 46 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 131072  
Total time 41 min



Sample Name:

Data Collected on:

Yb-vmrs700

Archive directory:

Sample directory:

FidFile: kb4-98-02

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Mar 26 2012

Operator: kbut1

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 11160.7 Hz

16 repetitions

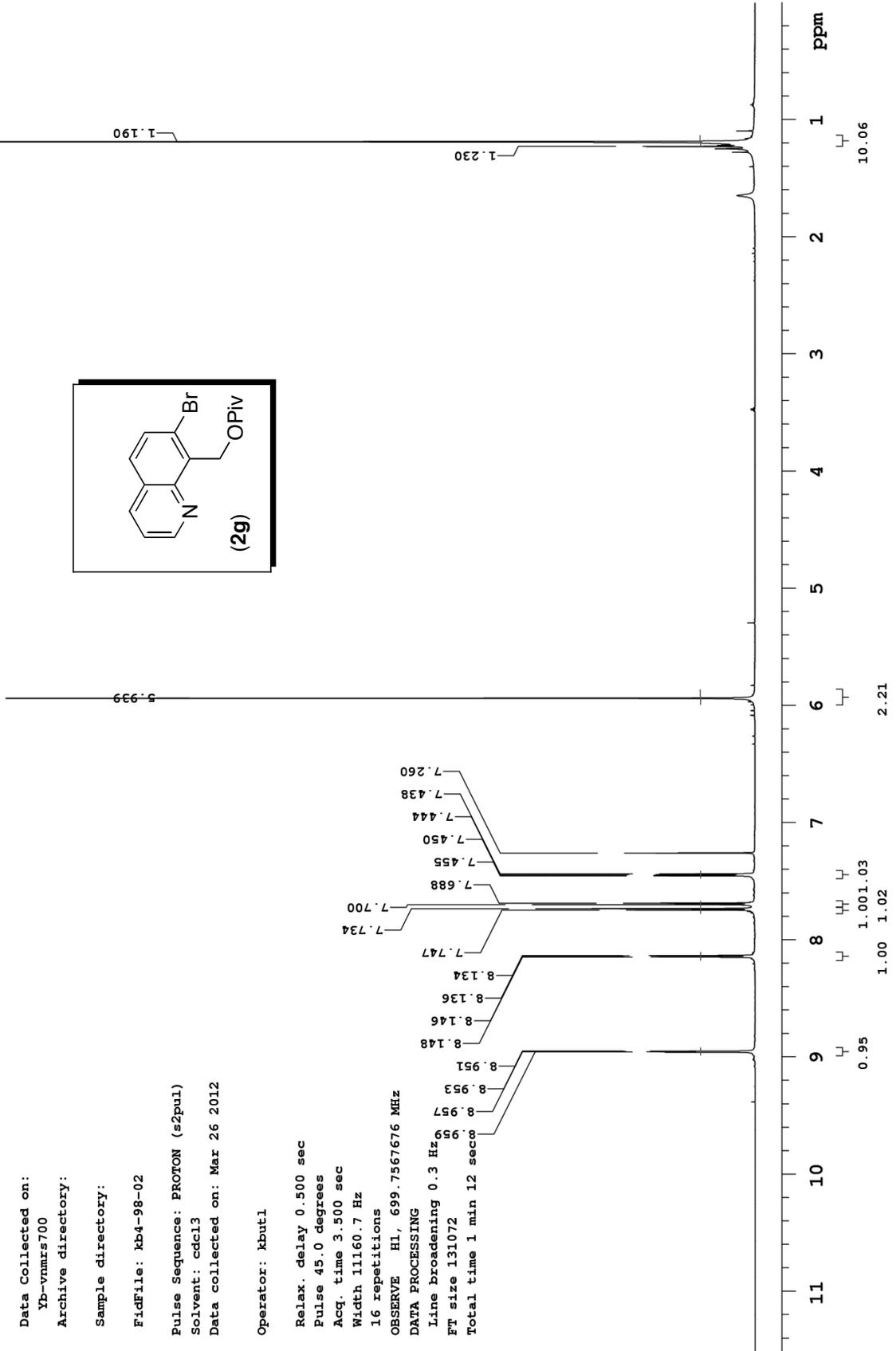
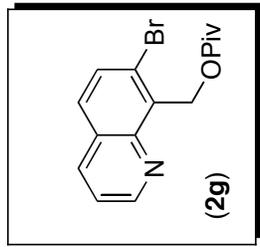
OBSERVE H1, 699.7567676 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 131072

Total time 1 min 12 sec



Sample Name:

Data Collected on:

YB-vmnr700

Archive directory:

Sample directory:

FidFile: kb4-98-03

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Mar 26 2012

Operator: kbutl

Relax. delay 0.100 sec

Pulse 45.0 degrees

Acq. time 1.468 sec

Width 44642.9 Hz

448 repetitions

OBSERVE C13, 175.9539411 MHz

DECOUPLE H1, 699.7602734 MHz

Power 46 dB

continuously on

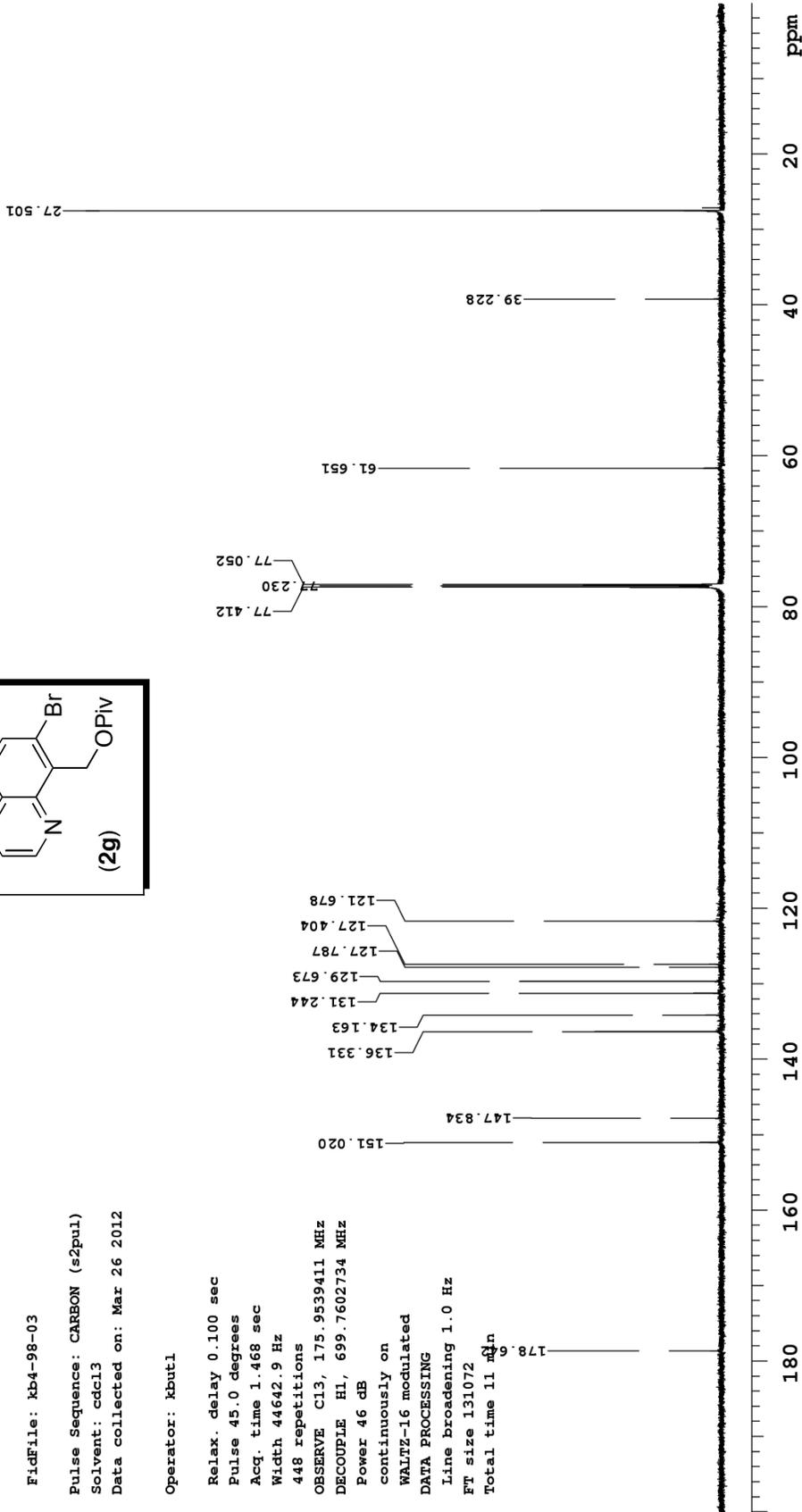
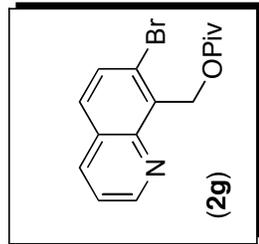
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 11 min



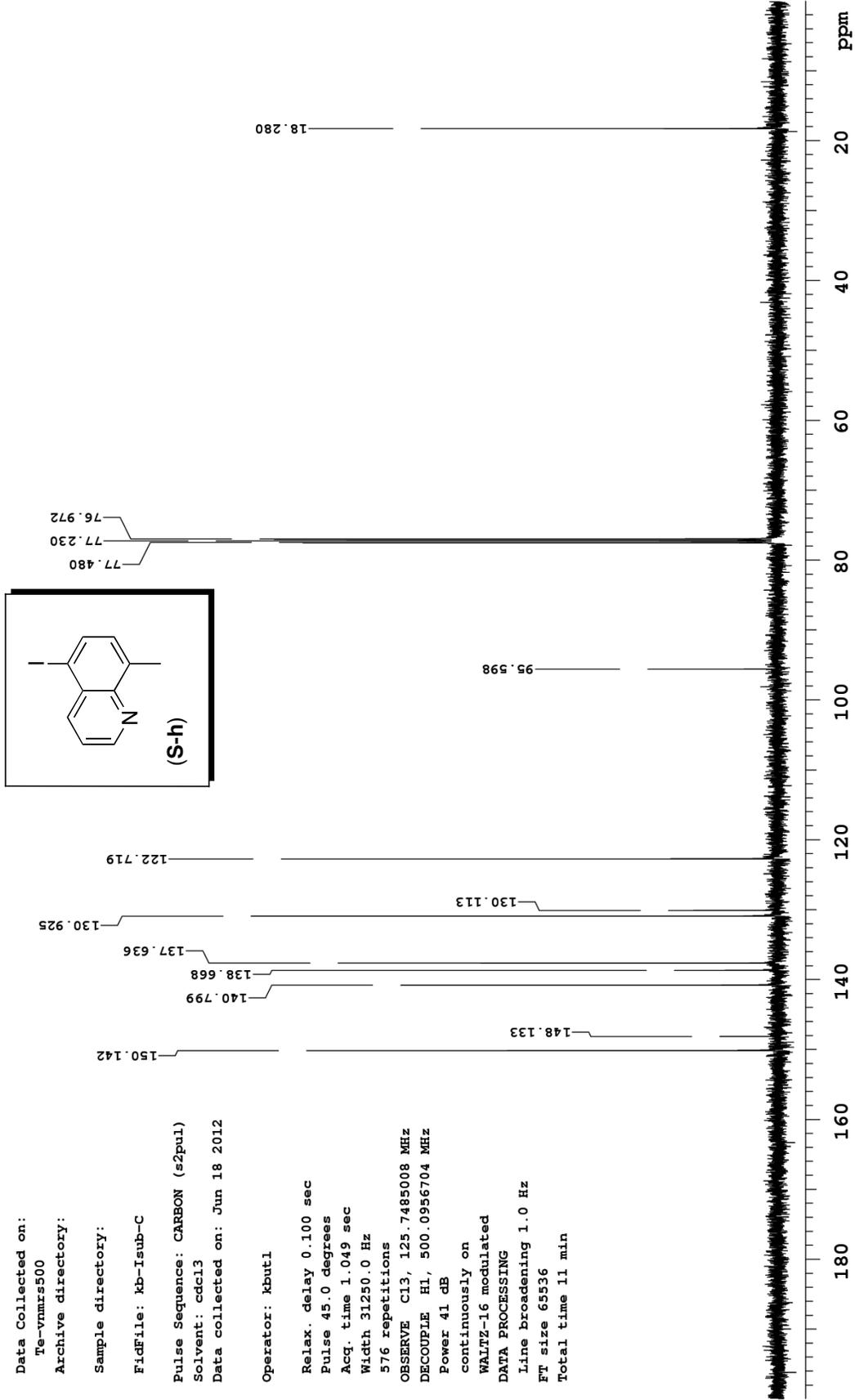


Sample Name:

Data Collected on: Te-vmr500  
Archive directory:  
Sample directory:  
FidFile: kb-Isup-C  
Pulse Sequence: CARBON (s2pul)  
Solvent: cdcl3  
Data collected on: Jun 18 2012

Operator: kbut1

Relax. delay 0.100 sec  
Pulse 45.0 degrees  
Acq. time 1.049 sec  
Width 31250.0 Hz  
576 repetitions  
OBSERVE C13, 125.7485008 MHz  
DECOUPLE H1, 500.0956704 MHz  
Power 41 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 11 min



Sample Name:

Data Collected on:  
Yb-vnmrs700  
Archive directory:

Sample directory:

FidFile: kb2-179-01

Pulse Sequence: PROTON (s2pul)

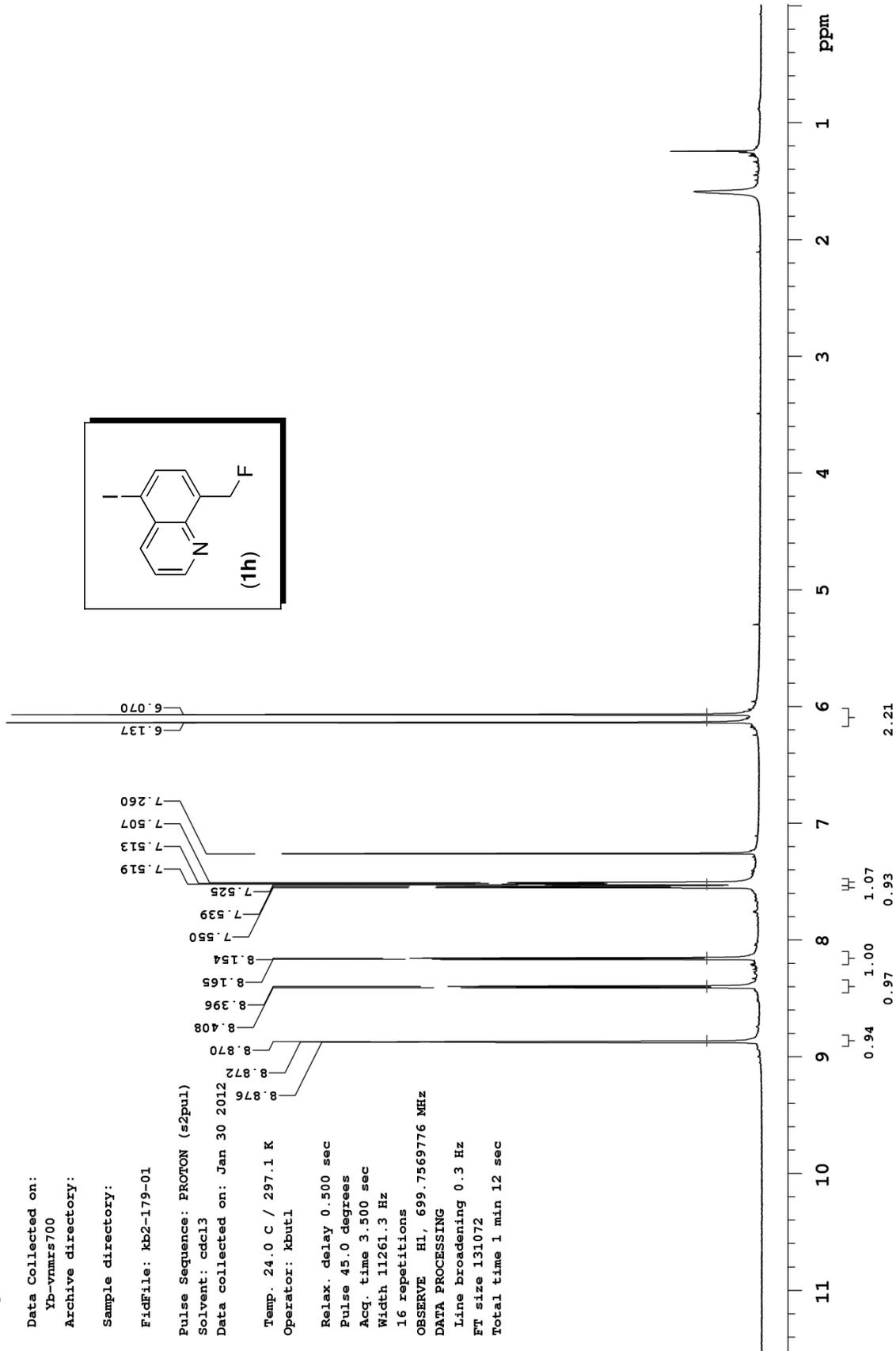
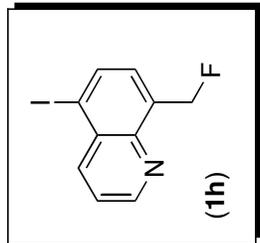
Solvent: cdcl3

Data collected on: Jan 30 2012

Temp. 24.0 C / 297.1 K  
Operator: kbut1

Relax. delay 0.500 sec  
Pulse 45.0 degrees  
Acq. time 3.500 sec  
Width 11261.3 Hz  
16 repetitions

OBSERVE H1, 699.7569776 MHz  
DATA PROCESSING  
Line broadening 0.3 Hz  
FT size 131072  
Total time 1 min 12 sec





Sample Name:

Data Collected on:

Yb-vmrs700

Archive directory:

Sample directory:

FidFile: kb2-179-02

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Feb 1 2012

Operator: kbutl

Relax. delay 0.100 sec

Pulse 45.0 degrees

Acq. time 1.468 sec

Width 44642.9 Hz

3056 repetitions

OBSERVE C13, 175.9539902 MHz

DECOUPLE H1, 699.7602734 MHz

Power 47 dB

continuously on

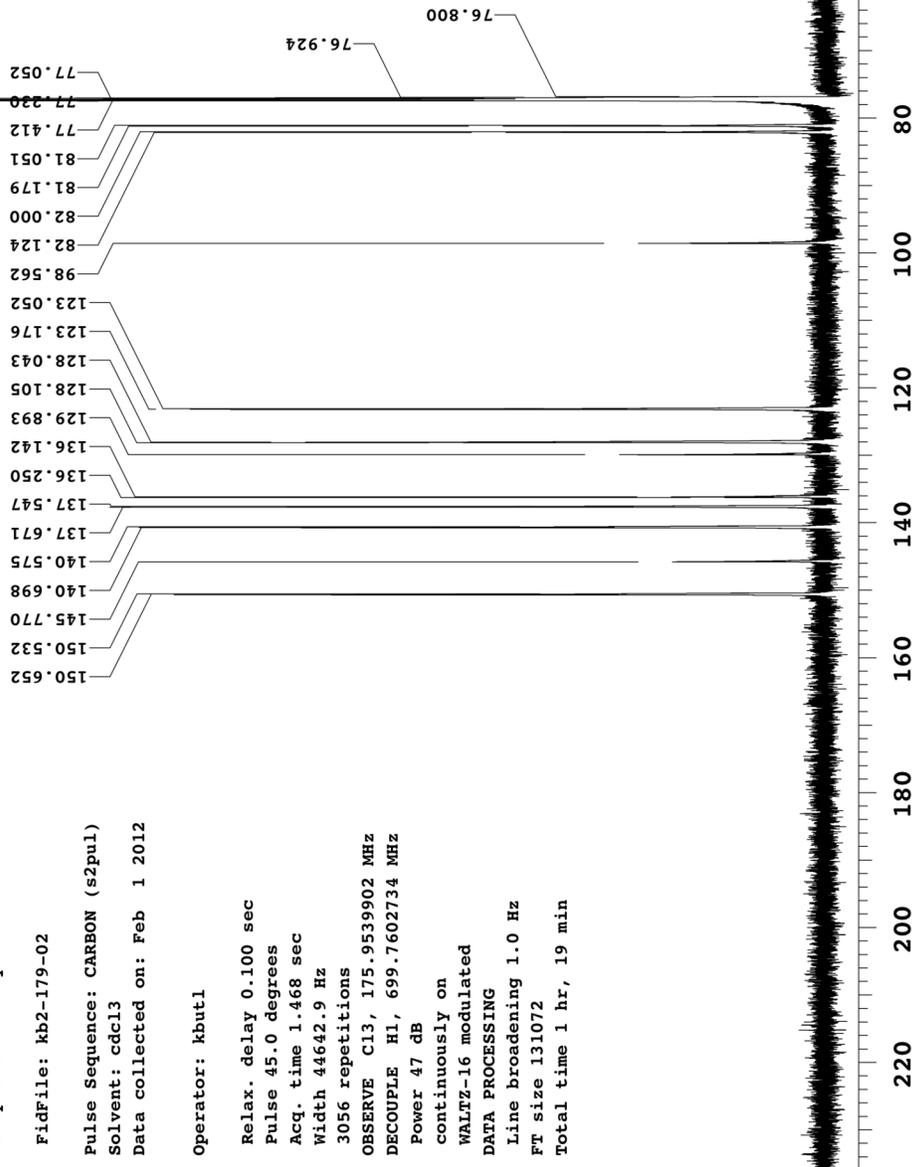
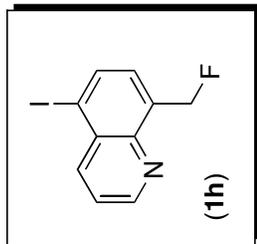
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 1 hr, 19 min





Sample Name:

Data Collected on:

Te-vmr500

Archive directory:

Sample directory:

FidFile: kb4-115-04

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Apr 18 2012

Operator: kbut1

Relax. delay 0.100 sec

Pulse 45.0 degrees

Acq. time 1.049 sec

Width 31250.0 Hz

2704 repetitions

OBSERVE C13, 125.7484999 MHZ

DECOUPLE H1, 500.0956704 MHZ

Power 42 dB

continuously on

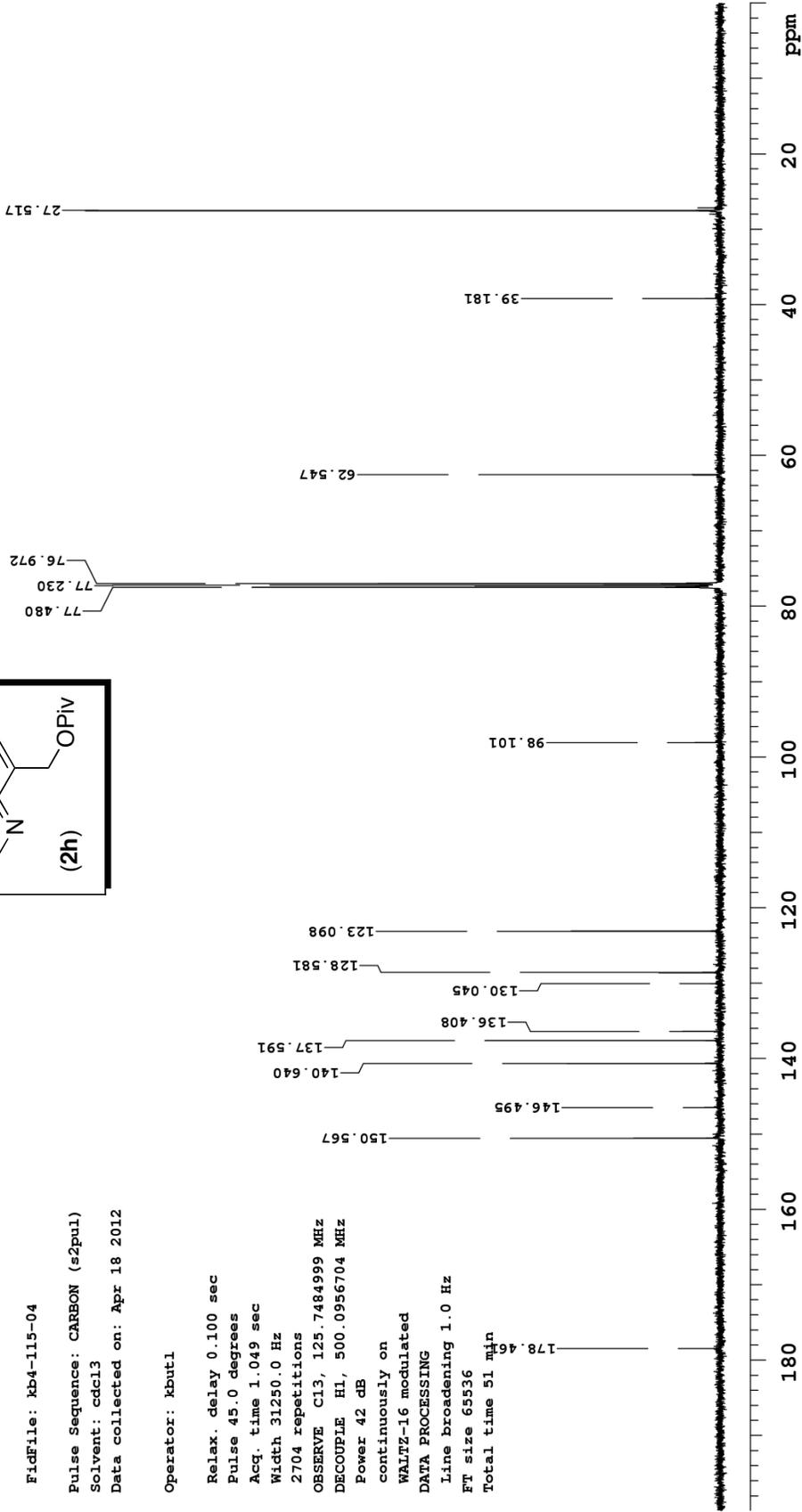
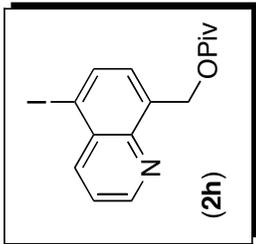
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65936

Total time 51 min



Sample Name:

Data Collected on:

Te-vmr500

Archive directory:

Sample directory:

FidFile: kb-Phsub-H

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Jun 19 2012

Operator: kbut1

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 8012.8 Hz

16 repetitions

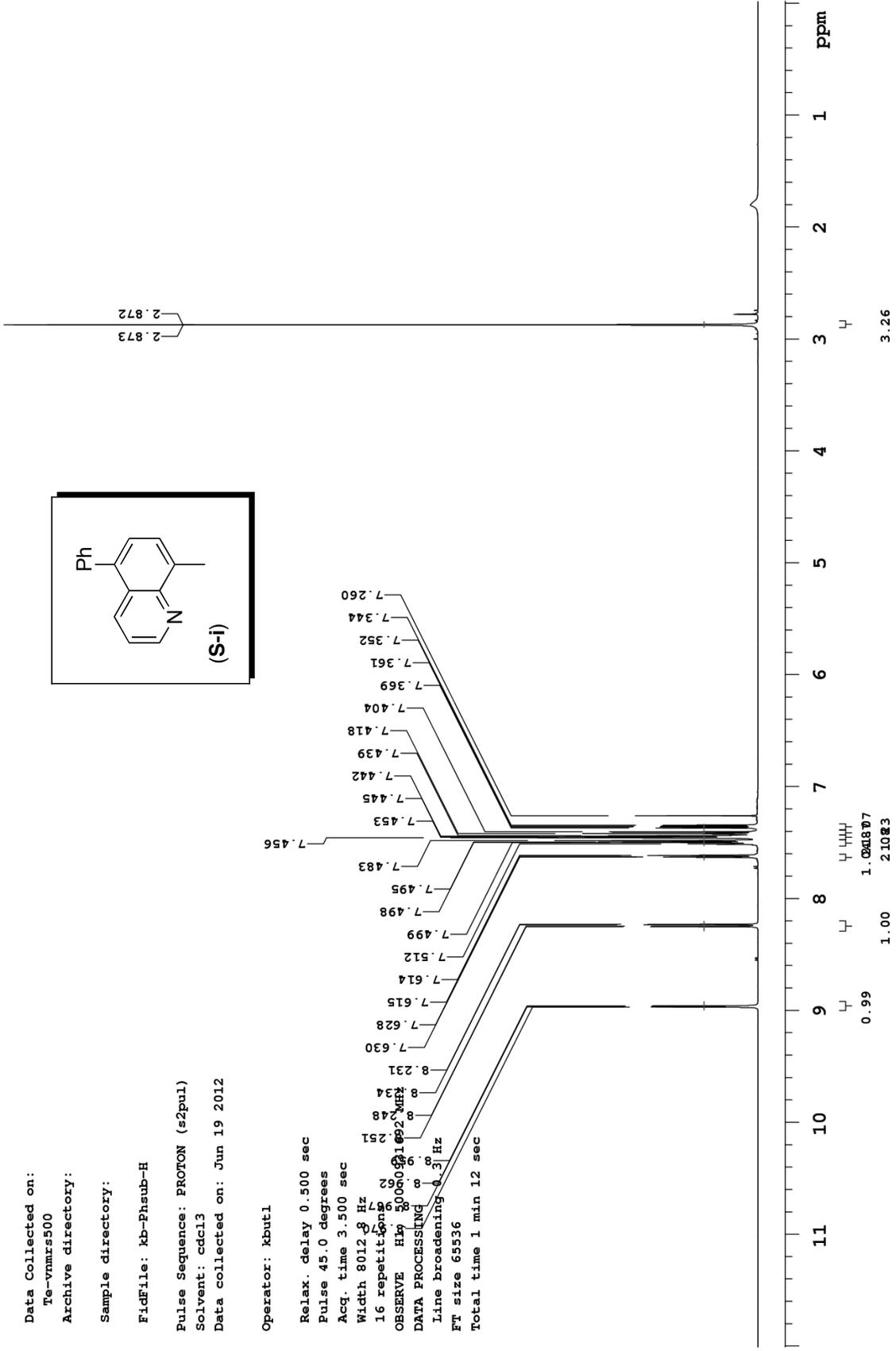
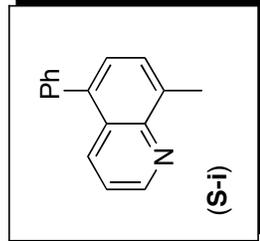
OBSERVE HM 500.1362 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 65536

Total time 1 min 12 sec



Sample Name:

Data Collected on:  
Te-vnmrs500  
Archive directory:

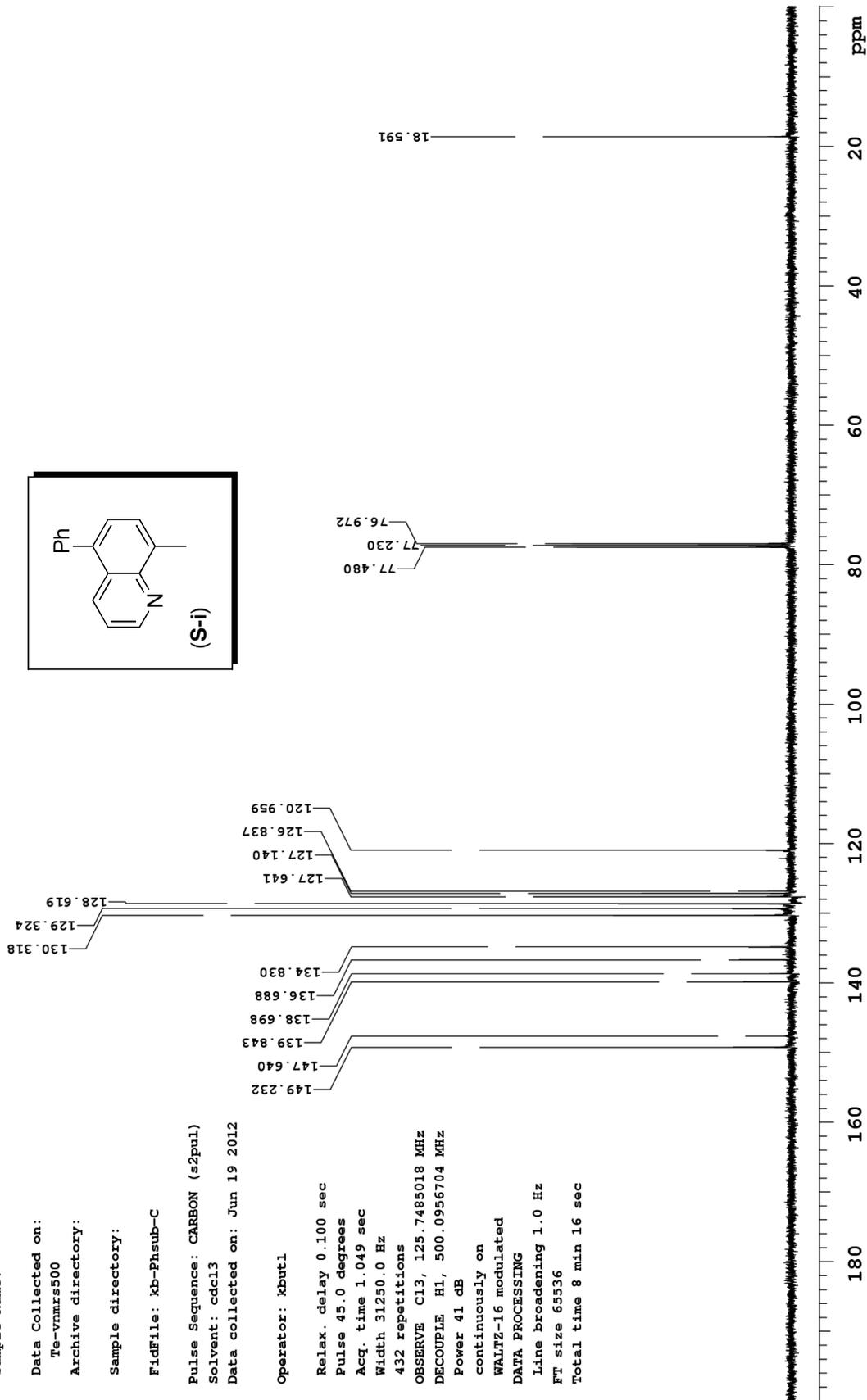
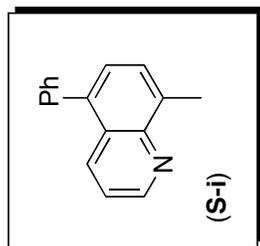
Sample directory:

FidFile: kb-Phsub-C

Pulse Sequence: CARBON (s2pul)  
Solvent: cdcl3  
Data collected on: Jun 19 2012

Operator: kbut1

Relax. delay 0.100 sec  
Pulse 45.0 degrees  
Acq. time 1.049 sec  
Width 31250.0 Hz  
432 repetitions  
OBSERVE C13, 125.7485018 MHz  
DECOUPLE H1, 500.0956704 MHz  
Power 41 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 8 min 16 sec





Pg. 134 C-F fr.14-28

Sample Name:

Data Collected on:

Te-vnmrs500

Archive directory:

Sample directory:

FidFile: kb4-134-12

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Jun 12 2012

Operator: kbut1

Relax. delay 0.100 sec

Pulse 45.0 degrees

Acq. time 1.049 sec

Width 31250.0 Hz

3888 repetitions

OBSERVE C13, 125.7485018 MHz

DECOUPLE H1, 500.0956704 MHz

Power 41 dB

continuously on

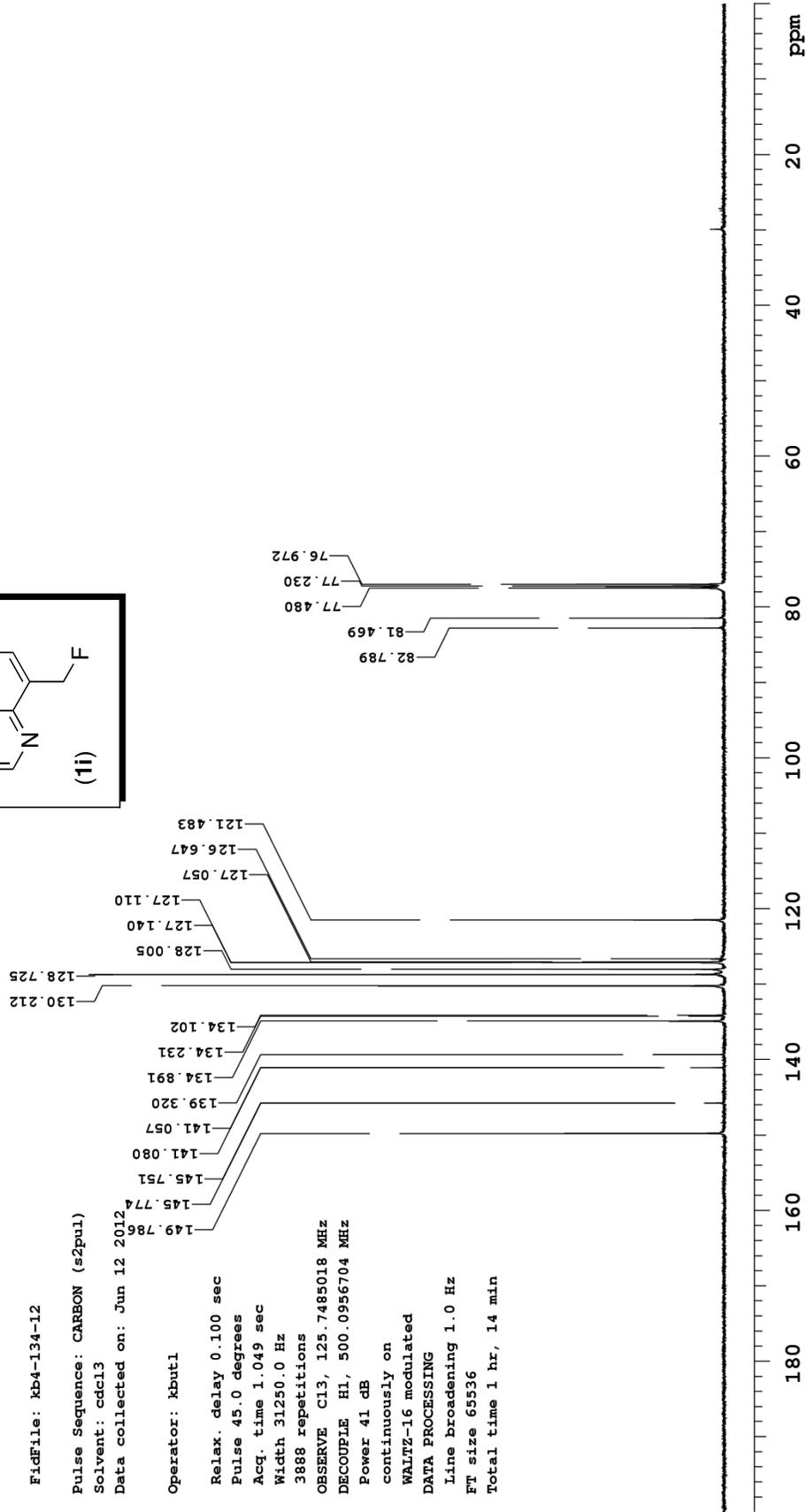
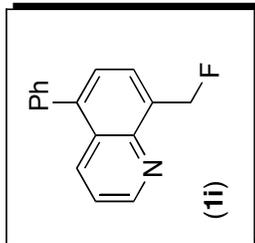
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 1 hr, 14 min





pg. 134 C-O fr.33-41

Sample Name:

Data Collected on:

Te-vnmrs500

Archive directory:

Sample directory:

FidFile: kb4-134-C2

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Jun 12 2012

Temp. 25.0 C / 298.1 K

Operator: kbut1

Relax. delay 0.100 sec

Pulse 45.0 degrees

Acq. time 1.049 sec

Width 31250.0 Hz

1000 repetitions

OBSERVE C13, 125.7484999 MHz

DECOUPLE H1, 500.0956704 MHz

Power 41 dB

continuously on

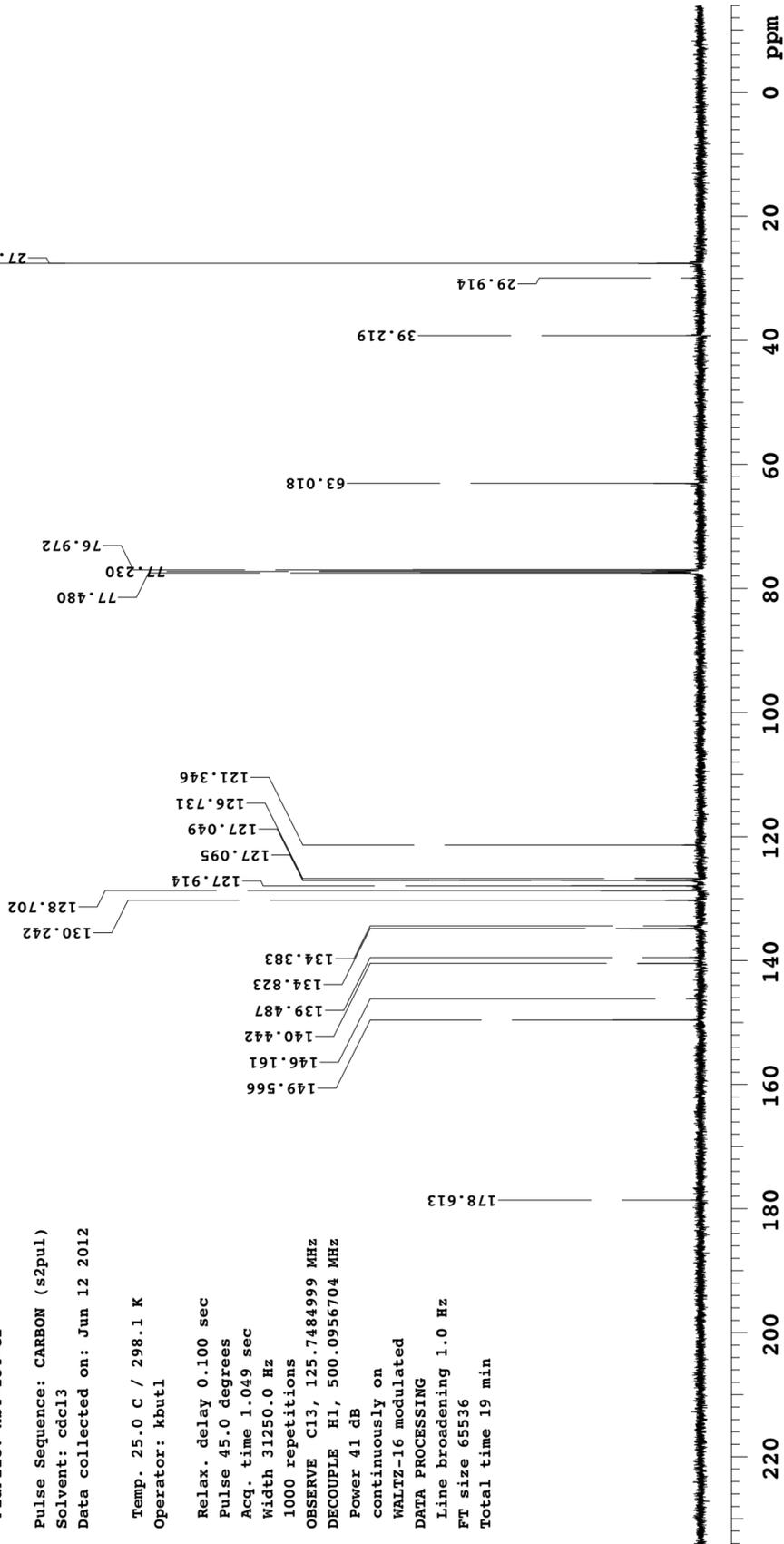
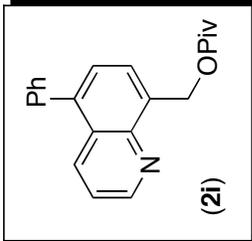
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 19 min



Pg. 123 Me Sub

Sample Name:

Data Collected on:

Te-vmr500

Archive directory:

Sample directory:

FidFile: kb-Mesub-H

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Jun 18 2012

Operator: kbut1

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 8012.8 Hz

16 repetitions

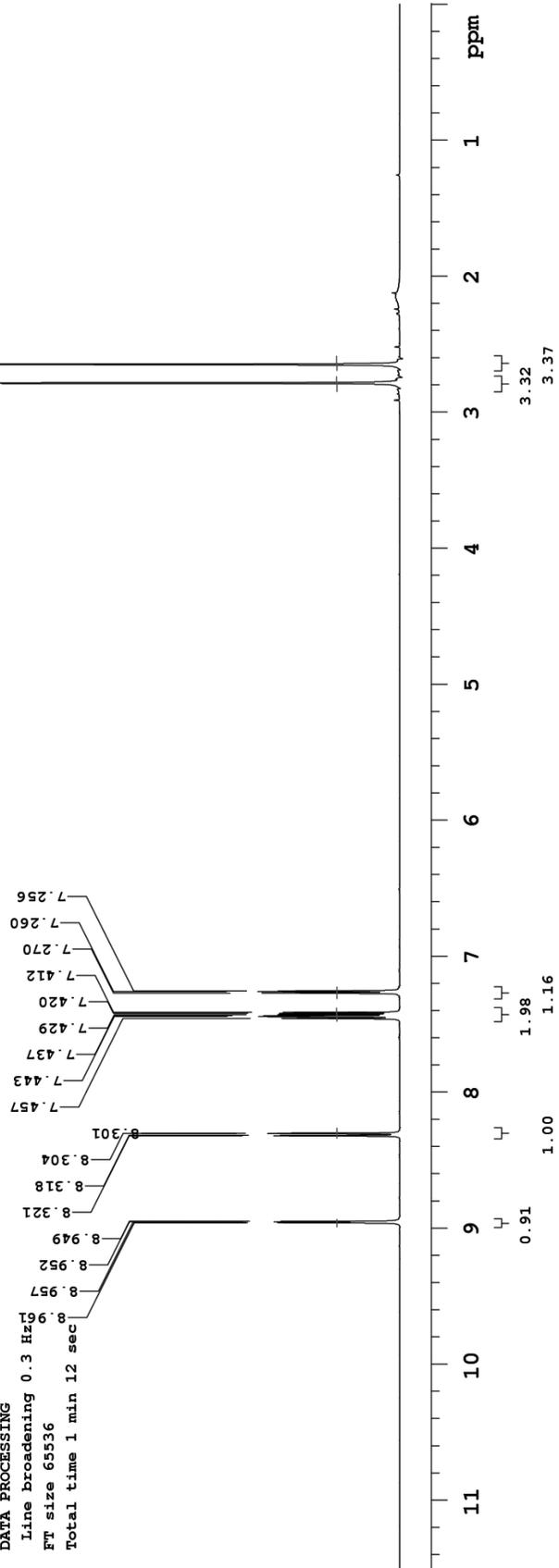
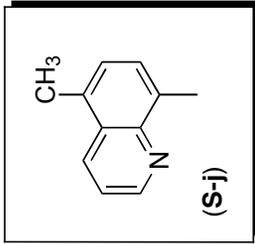
OBSERVE H1, 500.0931692 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 65536

Total time 1 min 12 sec

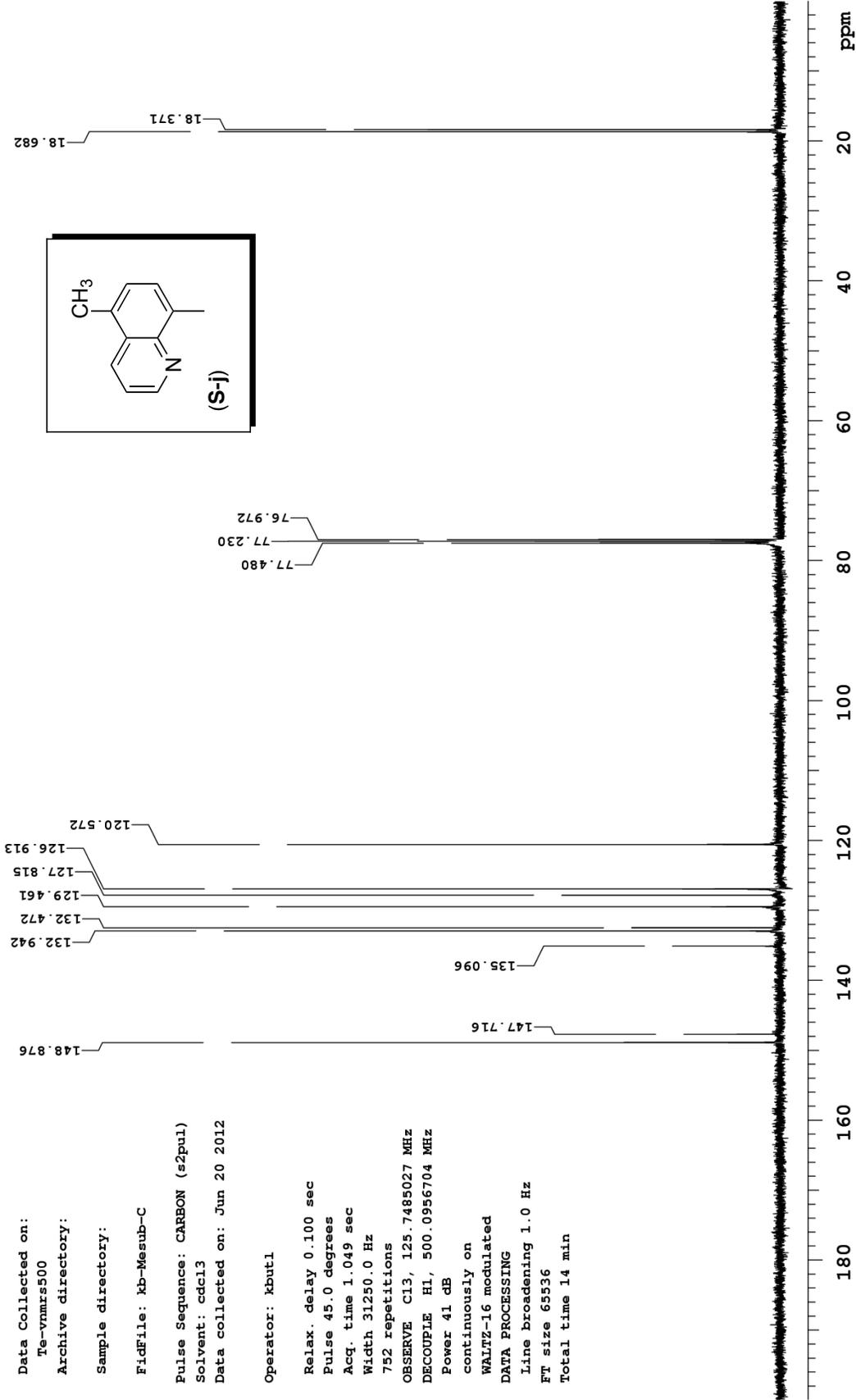
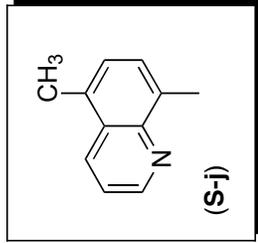


Sample Name:

Data Collected on:  
Te-vmrms500  
Archive directory:  
Sample directory:  
FidFile: kb-Mesub-C  
Pulse Sequence: CARBON (s2pul)  
Solvent: cdcl3  
Data collected on: Jun 20 2012

Operator: kbut1

Relax. delay 0.100 sec  
Pulse 45.0 degrees  
Acq. time 1.049 sec  
Width 31250.0 Hz  
752 repetitions  
OBSERVE C13, 125.7485027 MHz  
DECOUPLE H1, 500.0956704 MHz  
Power 41 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 14 min



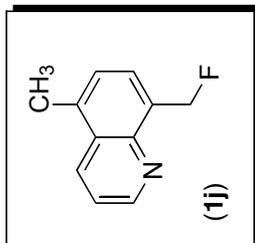
Sample Name:

Data Collected on:  
Yb-vnmrs700  
Archive directory:

Sample directory:

FidFile: kb4-96-01

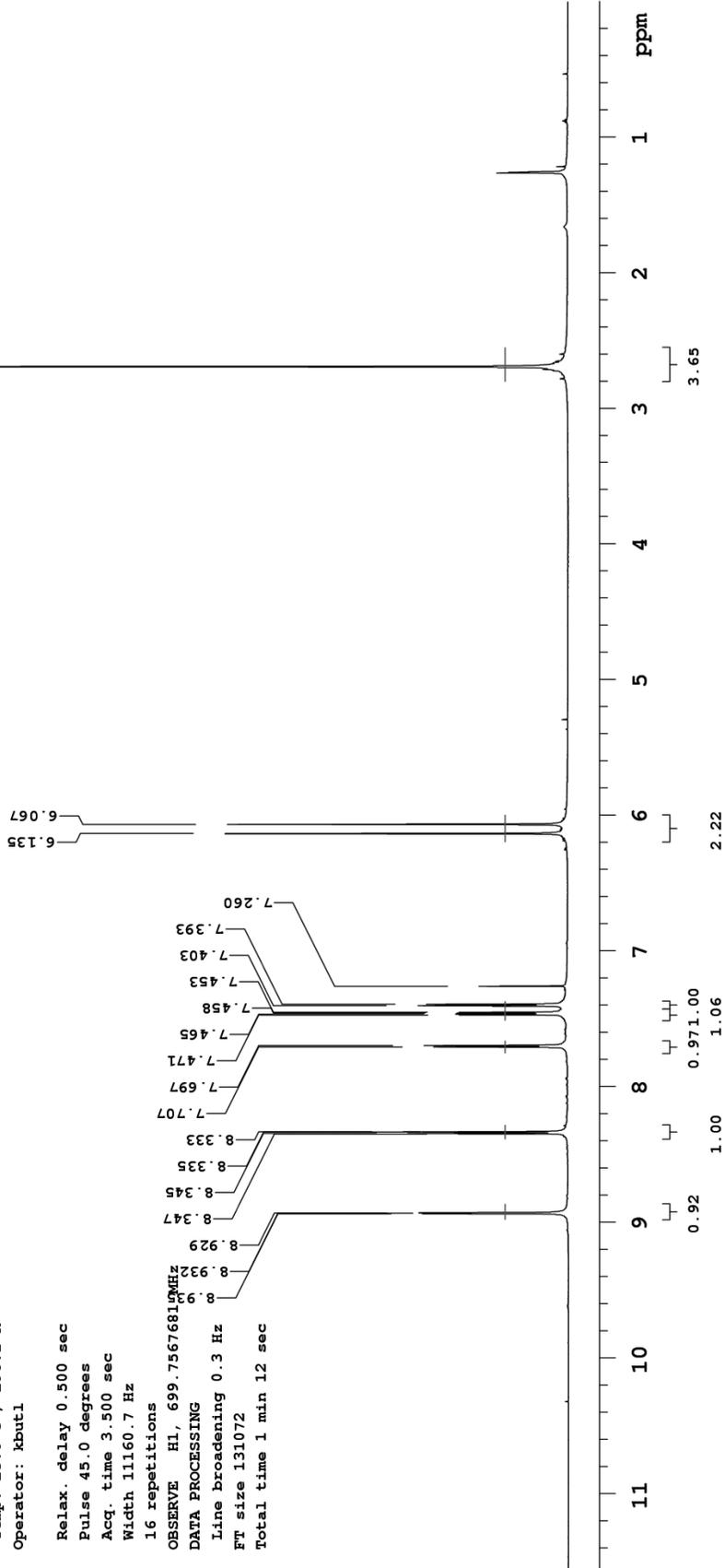
Pulse Sequence: PROTON (s2pul)  
Solvent: cdcl3  
Data collected on: Mar 30 2012



Temp. 25.0 C / 298.1 K  
Operator: kbut1

Relax. delay 0.500 sec  
Pulse 45.0 degrees  
Acq. time 3.500 sec  
Width 11160.7 Hz

16 repetitions  
OBSERVE H1, 699.756768100MHz  
DATA PROCESSING  
Line broadening 0.3 Hz  
FT size 131072  
Total time 1 min 12 sec



Sample Name:

Data Collected on:  
Yb-vnmrs700  
Archive directory:

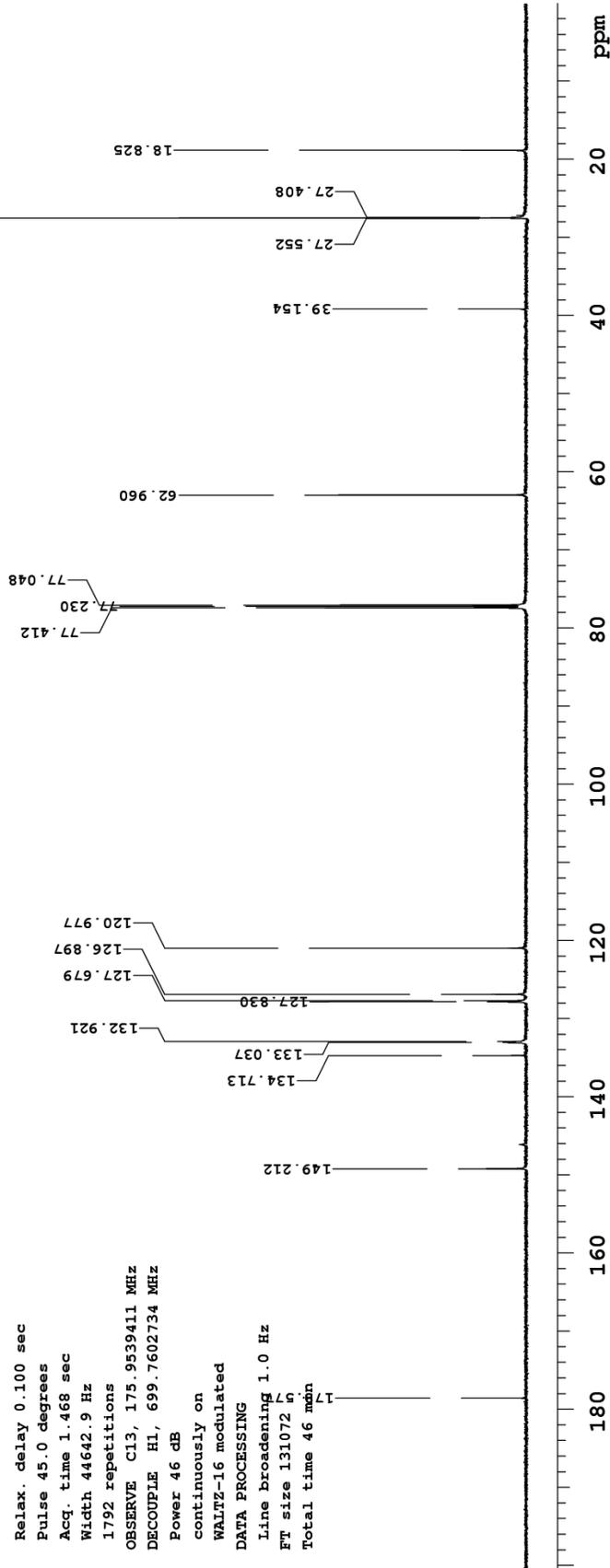
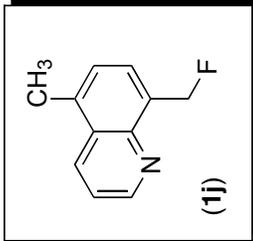
Sample directory:

FidFile: kb2-181-02

Pulse Sequence: CARBON (s2pul)  
Solvent: cdc13  
Data collected on: Feb 9 2012

Temp. 25.0 C / 298.1 K  
Operator: kbut1

Relax. delay 0.100 sec  
Pulse 45.0 degrees  
Acq. time 1.468 sec  
Width 44642.9 Hz  
1792 repetitions  
OBSERVE C13, 175.9539411 MHz  
DECOUPLE H1, 699.7602734 MHz  
Power 46 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 131072 b  
Total time 46 min



Pg. 181 C-O fr. 28-30

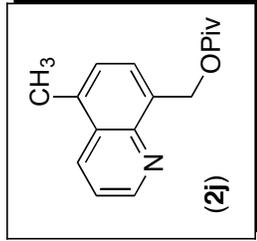
Sample Name:

Data Collected on:  
Co.Chem.USA.UMich.edu-vmrs400  
Archive directory:

Sample directory:  
FidFile: kb2-181-07  
Pulse Sequence: PROTON (s2pul)  
Solvent: cdcl3  
Data collected on: Feb 3 2012

Operator: kbut1

Relax. delay 0.500 sec  
Pulse 45.0 degrees  
Acq. time 3.500 sec  
Width 6410.3 Hz  
16 repetitions  
OBSERVE F1, 400.5206314 MHz  
DATA PROCESSING  
Line broadening 0.3 Hz  
FT size 65536  
Total time 1 min 12 sec



1.259

1.266

2.686

5.825

8.951  
8.948  
8.941  
8.937  
8.363  
8.359  
8.342  
8.338  
7.634  
7.616  
7.482  
7.472  
7.461  
7.450  
7.380  
7.362  
7.260

ppm

1

2

3

4

5

6

7

8

9

9.86

3.33

2.13

1.001.04  
1.01

1.00

0.94

Sample Name:

Data Collected on:

Yb-vnmrs700

Archive directory:

Sample directory:

FidFile: kb2-181-13C-acetone

Pulse Sequence: CARBON (s2pul)

Solvent: acetone

Data collected on: Feb 14 2012

Temp. 25.0 C / 298.1 K

Operator: kbut1

Relax. delay 0.100 sec

Pulse 45.0 degrees

Acq. time 1.468 sec

Width 4642.9 Hz

1312 repetitions

OBSERVE C13, 175.9545980 MHZ

DECOUPLE H1, 699.7639052 MHZ

Power 46 dB

continuously on

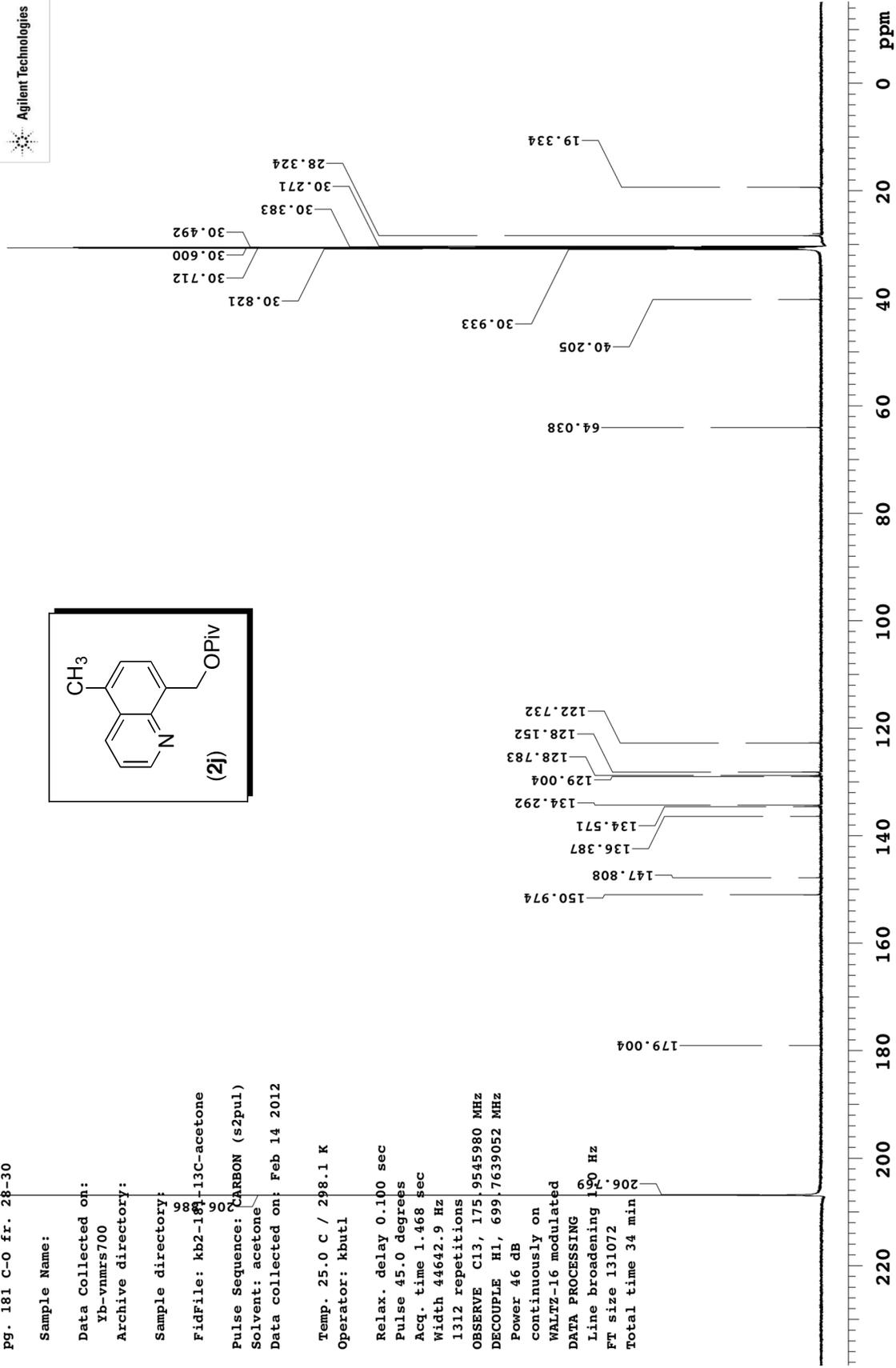
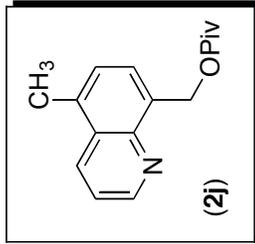
WALTZ-16 modulated

DATA PROCESSING

Line broadening 150 Hz

FT size 131072

Total time 34 min



pg. 199 MeOsub



Sample Name:

Data Collected on:

Te-vmr500

Archive directory:

Sample directory:

FidFile: kb-MeOsub-H

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Jun 19 2012

Operator: kbut1

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 3.500 sec

Width 8012.8 Hz

16 repetitions

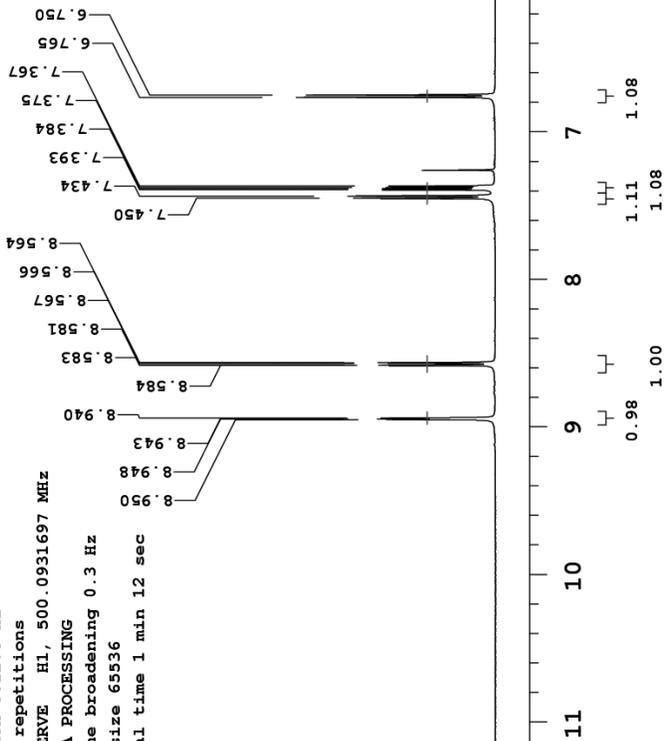
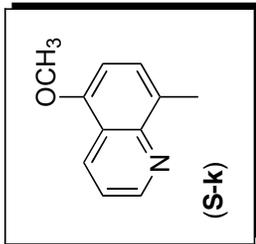
OBSERVE H1, 500.0931697 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 65936

Total time 1 min 12 sec



Sample Name:

Data Collected on:  
Te-vnmrs500  
Archive directory:

Sample directory:

FidFile: kb-MeOsub-C

Pulse Sequence: CARBON (s2pul)  
Solvent: cdcl3  
Data collected on: Jun 19 2012

Operator: kbutl

Relax. delay 0.100 sec  
Pulse 45.0 degrees  
Acq. time 1.049 sec  
Width 31250.0 Hz  
400 repetitions

OBSERVE C13, 125.7485018 MHz  
DECOUPLE H1, 500.0956704 MHz  
Power 41 dB

continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 1.0 Hz  
FT size 65536  
Total time 7 min 39 sec

