

# A Versatile and Practical Method for Regioselective Synthesis of Polysubstituted Furanonaphthoquinones

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## Supplementary Data

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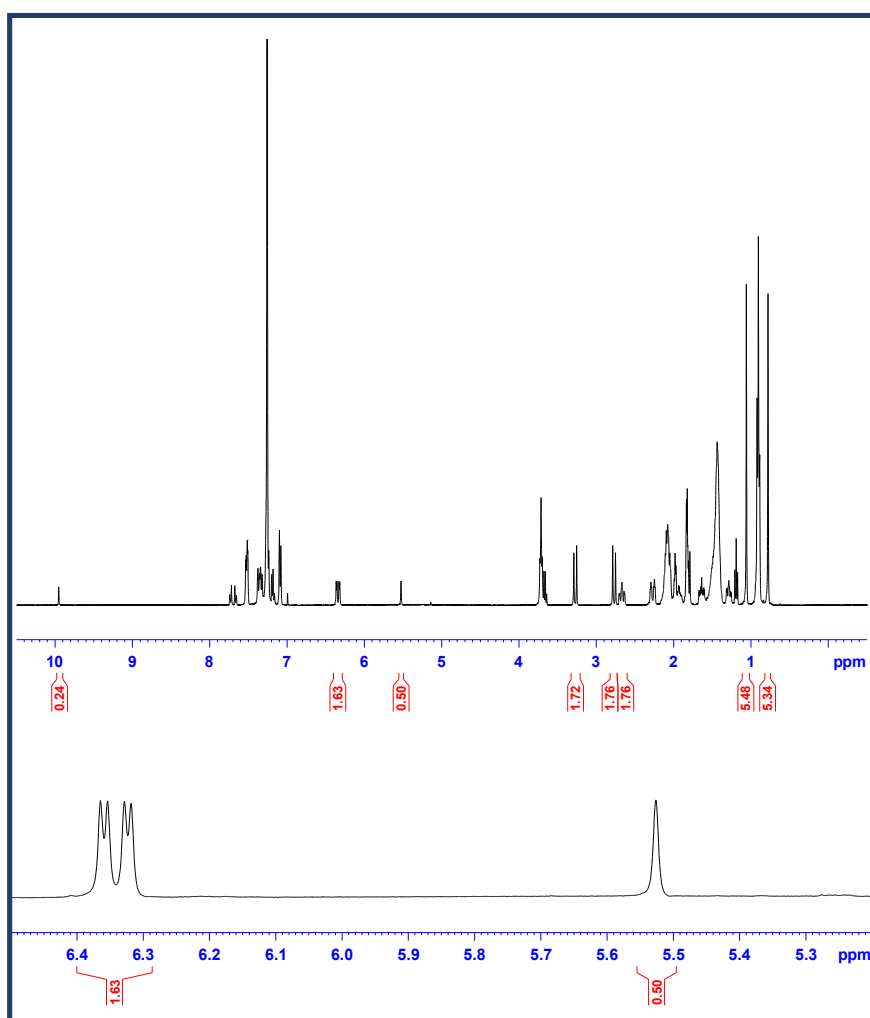
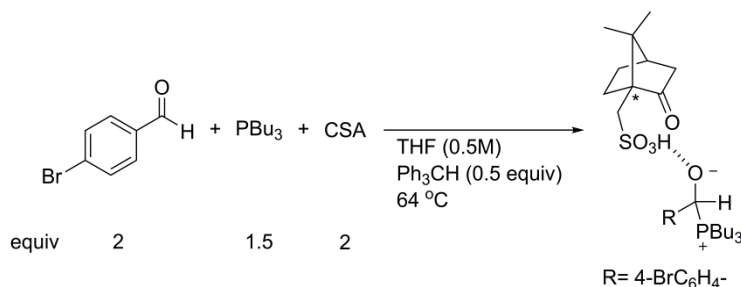
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## I . Extra Schemes and Figures:

### (1) Controlled experiments for mechanism studies:

Three controlled experiments were performed with 4-bromobenzaldehyde **2a**, CSA,  $\text{Bu}_3\text{P}$ , and a known amount of  $\text{Ph}_3\text{CH}$  (as internal reference for calculating the ratio of NMR integrals) in dry THF (0.5 mL) at 64 °C under nitrogen, and the  $^1\text{H}$  NMR spectra of their crude products were measured and discussed as below.

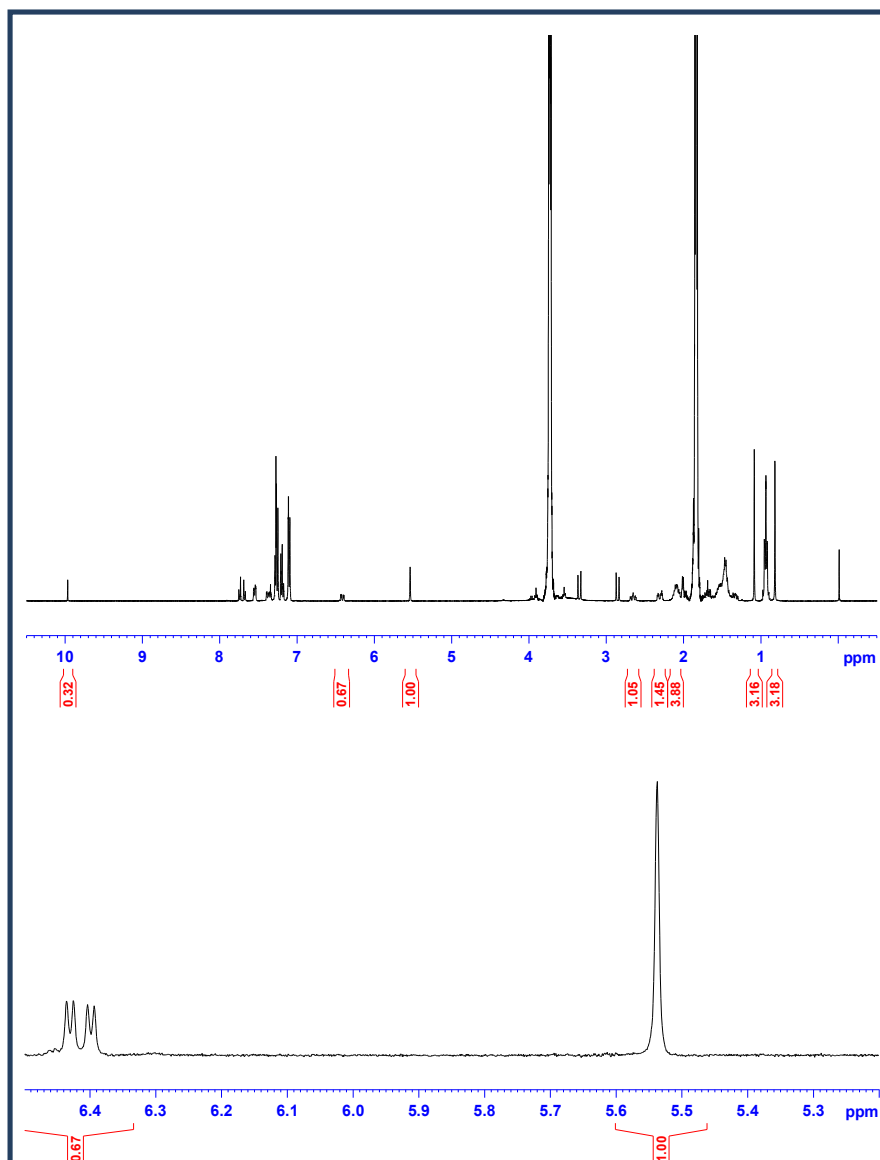
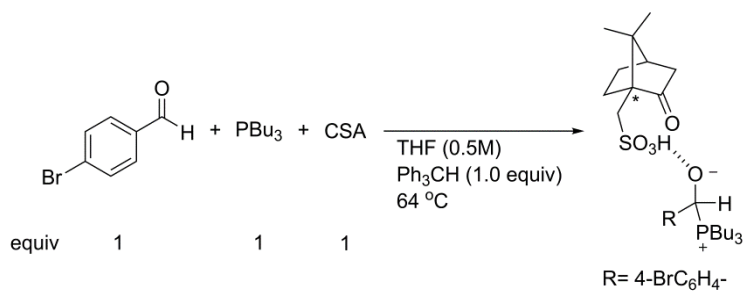
(a)



The  $^1\text{H}$  NMR spectra of the crude product indicated three kinds of protons as singlet, doublet of doublets, and singlet signals near  $\delta$  10, 6.3, and 5.5 ppm assignable to  $\text{RCHO}$  (remaining aldehyde **2a**),  $\text{CSA-RP}^+\text{Ph}_3\text{CHO}^-$ , and  $\text{Ph}_3\text{CH}$  protons respectively. For the signal ratio, the integral value of  $\text{CSA-RP}^+\text{Ph}_3\text{CHO}^-$  was similar to the equivalent of  $\text{PPh}_3$ , and the sum of signals near  $\delta$  10 and 6.3 ppm was close to the equivalent of  $\text{RCHO}$  **2a** initially used. These results showed that aldehyde **2a** reacted with

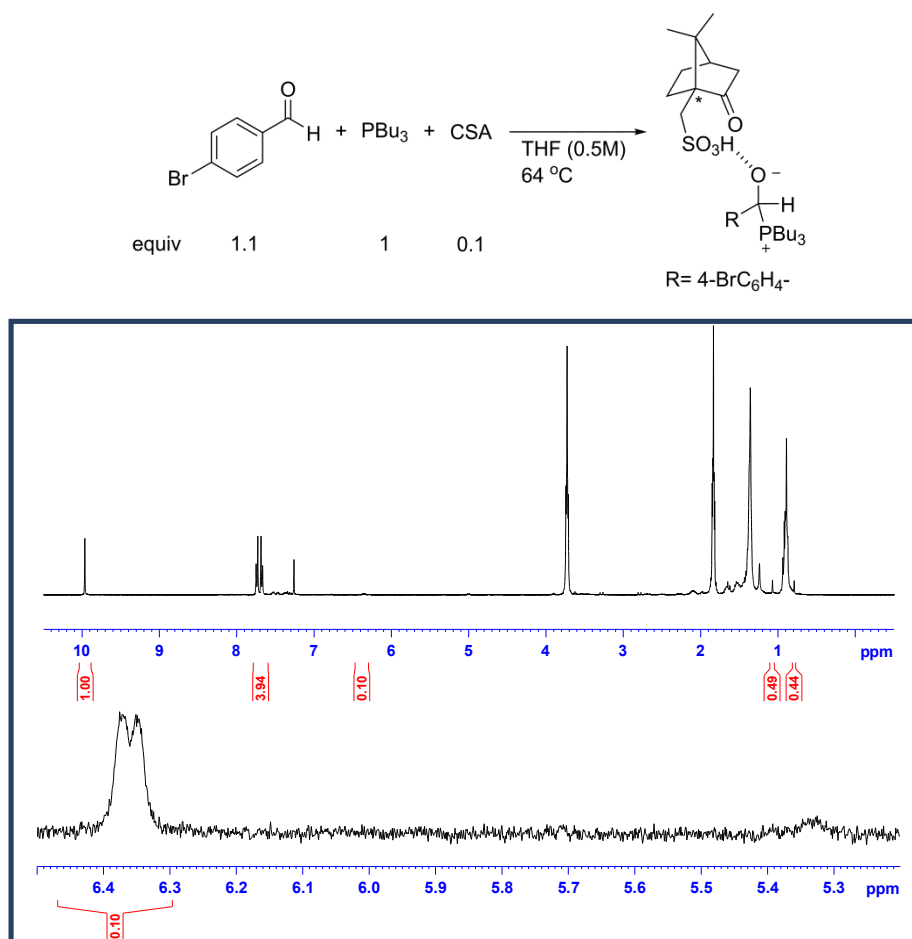
PPh<sub>3</sub> almost quantitatively catalyzed by CSA.

(b)



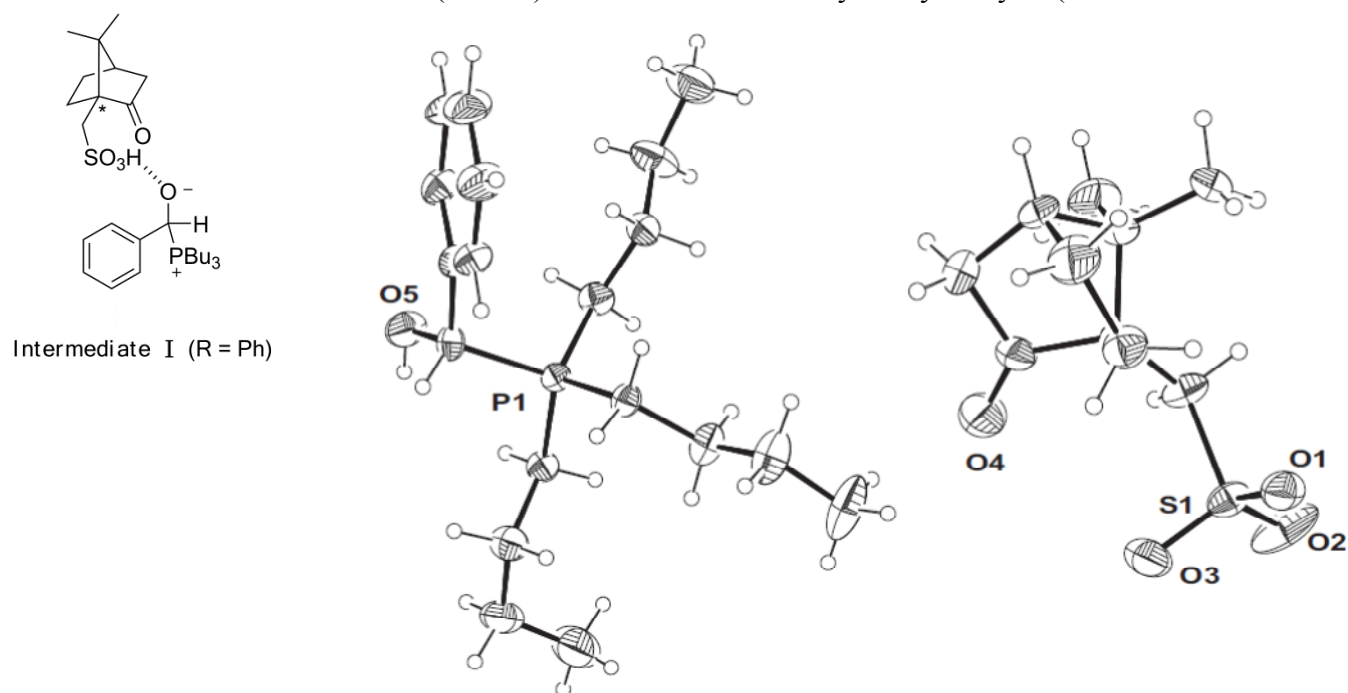
Similarly, when the reaction was performed with equal amounts of **2a**, CSA, Bu<sub>3</sub>P, and Ph<sub>3</sub>CH, the sum of signals near  $\delta$  10 and 6.4 ppm was also very close to the equivalent of **2a** initially used.

(c)

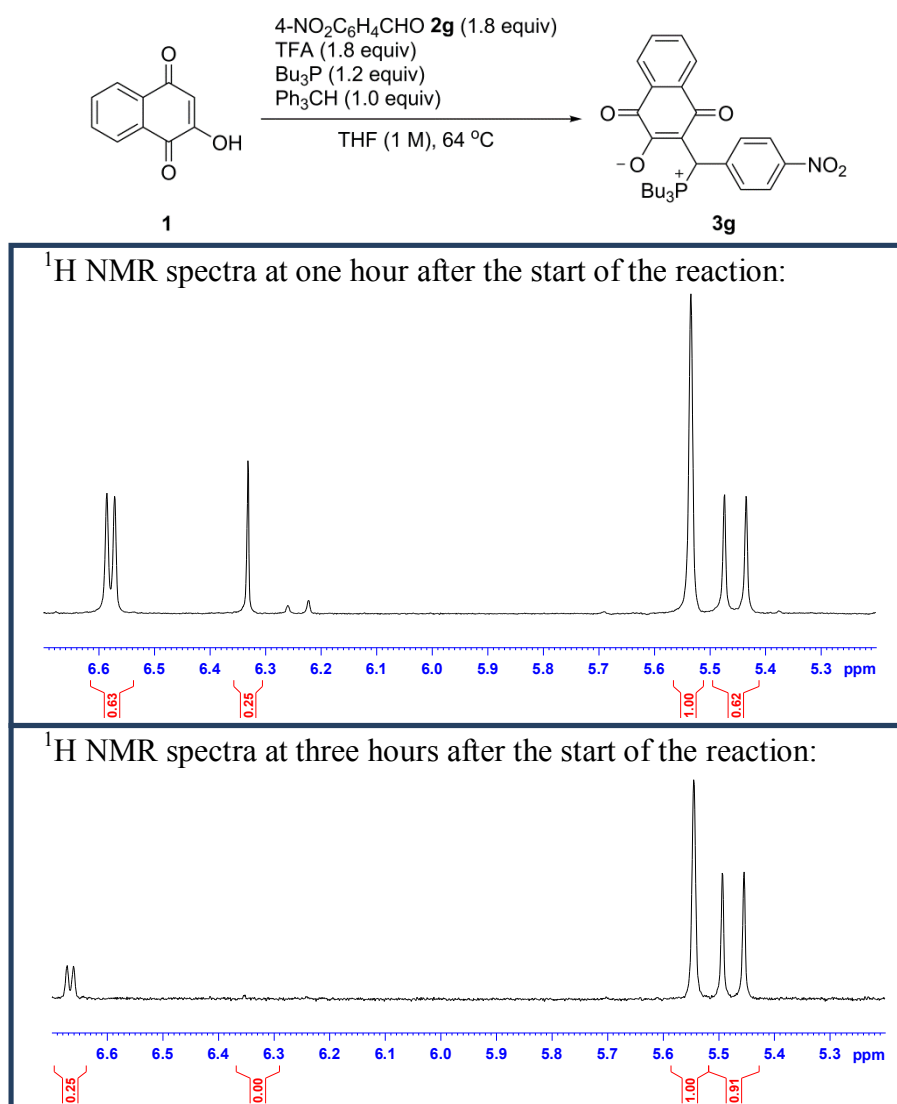


Finally, even when the reaction was performed with catalytic amounts of CSA, the amount of intermediate **I** ( $\text{CSA-RP}^+\text{Ph}_3\text{CHO}^-$ ) formed from the reaction of **2a** and  $\text{Bu}_3\text{P}$  was equal to that of CSA, and the sum of signals near  $\delta$  10 and 6.3 ppm was exactly the same as the equivalent of **2a** initially used. Based on the above results, it was apparent that  $\text{Bu}_3\text{P}$  can react with **2a** to form the intermediate **I** as mentioned in our proposed mechanism and the extent of this reaction depended on the amount of CSA.

(d) Furthermore, the crude products from controlled experiments were purified by recrystallization, and the structure of the intermediate **I** ( $\text{R} = \text{Ph}$ ) was also determined by X-ray analysis (CCDC Number: 887788).



(e) The intermediate **I** could be observed not only in the controlled experiments without 2-hydroxynaphthoquinone **1** but in the reactions with **1**.

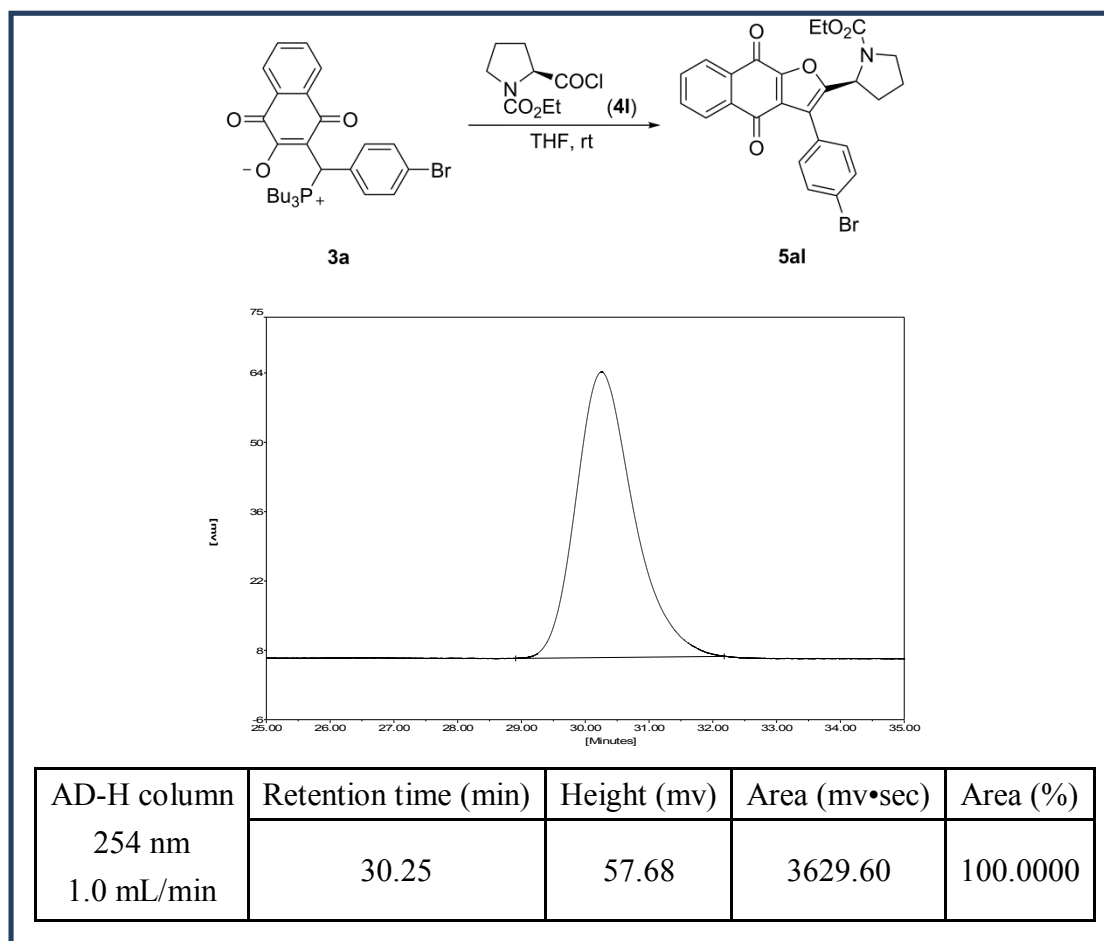
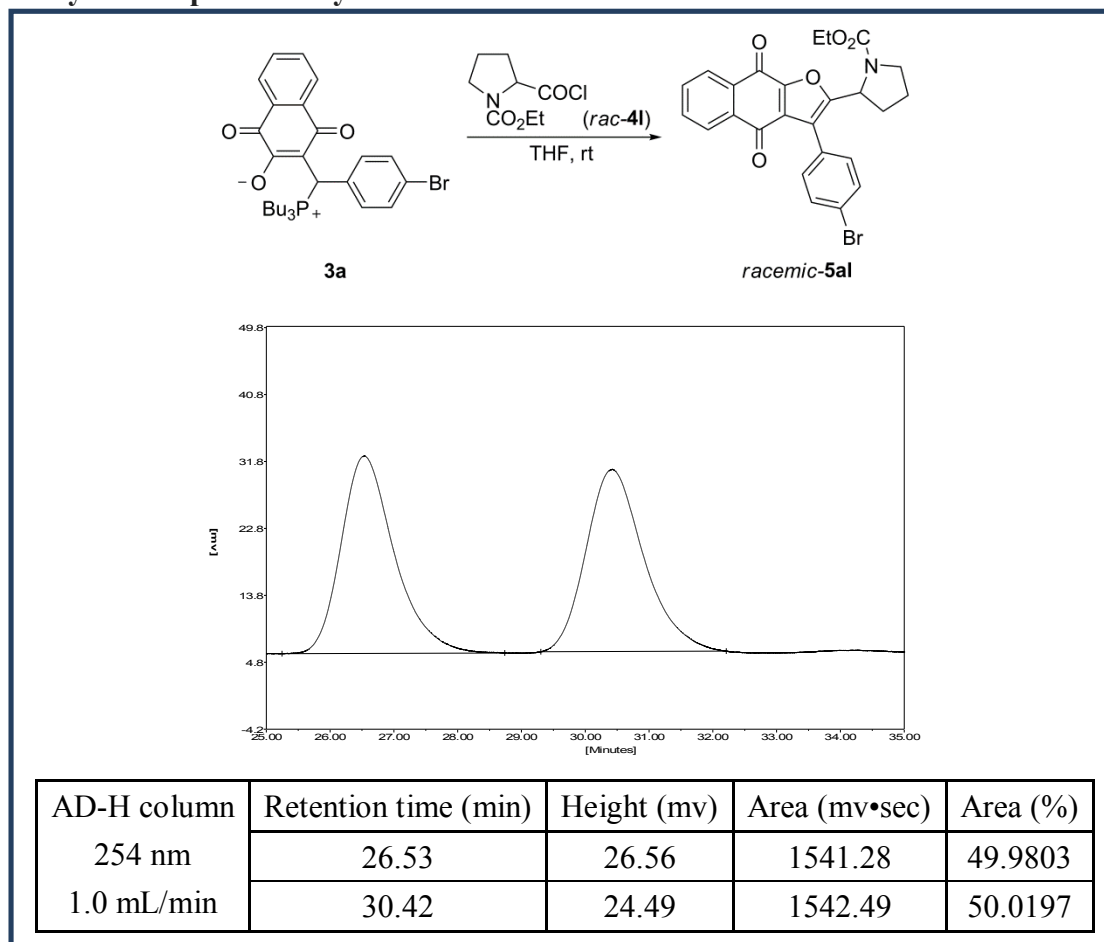


The <sup>1</sup>H NMR spectra during the course of the reaction indicated four kinds of protons as doublet, singlet, singlet, and doublet signals near  $\delta$  6.6, 6.3, 5.5 and 5.4 ppm assignable to intermediate **I**, 2-hydroxynaphthoquinone **1**, Ph<sub>3</sub>CH, and product **3g** protons respectively. At one hour after the start of the reaction, the sum of signals near  $\delta$  6.3 (remaining **1**) and 5.4 (producing **3g**, consuming **1**) ppm was close to the equivalent of **1** initially used, and it indicated that 2-hydroxynaphthoquinone **1** was effectively transformed to product without wasting on forming [2+1] side product **III**. In addition, the sum of signals near  $\delta$  6.6 (intermediate **I**, remaining Bu<sub>3</sub>P) and 5.4 (producing **3g**, consuming Bu<sub>3</sub>P) ppm was close to the equivalent of Bu<sub>3</sub>P initially used, and it showed that Bu<sub>3</sub>P was completely transformed to intermediate **I** and product **3g** without being oxidized by air under such conditions.

On the other hand, after three hours of the reaction, most of the remaining **1** was converted to the product **3g** ( $\delta$  5.4 ppm) to achieve a 91% NMR yield and the peak near  $\delta$  6.3 ppm disappeared. Moreover, the intermediate **I** ( $\delta$  6.6 ppm) also decomposed to provide the necessary aldehyde **2g** and Bu<sub>3</sub>P for reaction with **1** and intermediate **II** respectively, along with a corresponding increase in product **3g**. Therefore, these results proved that the intermediate **I** could be served as a well-protected species to reversibly store and prevent Bu<sub>3</sub>P from being oxidized and consumed by air before reacting with intermediate **II** and gradually release Bu<sub>3</sub>P and aldehyde **2** for further steps during the course of the

reaction.

(2) HPLC analysis for optical study:



## II. General Considerations:

All reactions were carried out under a nitrogen atmosphere in dried glassware. The starting materials purchased from commercial sources were used without further purification. THF was continuously refluxed and freshly distilled from sodium benzophenone ketyl under nitrogen. Yields refer to isolated yields of compounds estimated to be > 95 % pure as determined by <sup>1</sup>H-NMR. <sup>1</sup>H and <sup>13</sup>C spectra were generally recorded in a AV-400 or AV-500 Bruker using CDCl<sub>3</sub> as solvent at 400 or 500 and 100 or 125 MHz, respectively. Chemical shifts are reported in ppm relative to CDCl<sub>3</sub> (δ 7.26 ppm) in indicated cases. Analytical thin layer chromatography (TLC) was performed using Merck 60 F254 precoated silica gel plate (0.2 mm thickness). Flash chromatography was performed using Merck silica gel 60.

## III. Representative Experimental procedures:

### (A) Typical procedure- I (TP- I )

Preparation of **3a** (Table 5, entry 1): A dry and nitrogen-flushed 10-mL Schlenk flask, equipped with a magnetic stirring bar and a septum, was sequentially charged with a solution of **1** (87.1 mg, 0.5 mmol), **2a** (166.5 mg, 1.8 equiv), trifluoroacetic acid (68.9 μL, 1.8 equiv) and Bu<sub>3</sub>P (150 μL, 1.2 equiv) in dry THF (0.5 mL). The reaction mixture was stirred for 8 h at 60-64 °C. Thereafter, the solvent was removed by evaporation *in vacuo*. Purification by flash chromatography (ethyl acetate/hexanes: 1/3; then MeOH/CH<sub>2</sub>Cl<sub>2</sub>: 1/125) furnished **3a** as red solids (225.0 mg, 83% yield).

### (B) Typical procedure- II (TP- II )

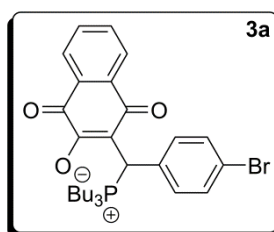
Preparation of **5aa** (Table 7, entry 1): A dry and nitrogen-flushed 25-mL Schlenk tube, equipped with a magnetic stirring bar and a septum, was charged with a solution of **3a** (271.7 mg, 0.5 mmol), **4a** (75.6 μL, 1.3 equiv), and Et<sub>3</sub>N (105 μL, 1.5 equiv) in dry THF (2.5 mL). The reaction mixture was stirred for 1.5 h at room temperature (25-28 °C). Thereafter, the solvent was removed by evaporation *in vacuo*. Purification by flash chromatography (CH<sub>2</sub>Cl<sub>2</sub>/hexanes: 1/2.8) furnished **5aa** as pale yellow solids (197.7 mg, 92% yield).

### (C) Typical procedure- III (TP- III )

Preparation of **7a** (Scheme 5): A dry and nitrogen-flushed 10-mL Schlenk flask, equipped with a magnetic stirring bar and a septum, was sequentially charged with a solution of 4-hydroxycoumarin (81.1 mg, 0.5 mmol), **5qa** (189.2 mg, 1 equiv), Bu<sub>3</sub>P (150 μL, 1.2 equiv), PhCO<sub>2</sub>H (6.1 mg, 0.1 equiv), and pyrrolidine (4 μL, 0.1 equiv) in dry THF (2.5 mL). The reaction mixture was stirred for 1 h at room temperature (25-28 °C). Thereafter, the solvent was removed by evaporation *in vacuo*. Purification by flash chromatography (ethyl acetate/hexanes: 1/3; then MeOH/CH<sub>2</sub>Cl<sub>2</sub>: 1/100) furnished **7a** as orange solids (263.4 mg, 73 %).

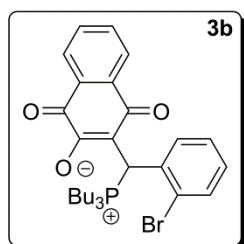
#### IV. Spectra data of the substrates:

##### Synthesis of 3a:



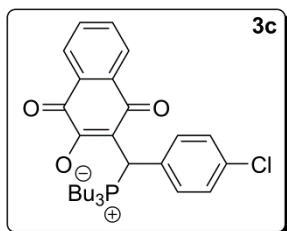
Prepared according to **TP- I** from **1** (87.1 mg, 0.5 mmol), **2a** (166.5 mg, 1.8 equiv), trifluoroacetic acid (68.9  $\mu\text{L}$ , 1.8 equiv), and tributylphosphine (150.0  $\mu\text{L}$ , 1.2 equiv) in dry THF (0.5 mL) [reaction condition: 60-64  $^{\circ}\text{C}$  for 8h]. Purification by *flash*-chromatography (ethyl acetate/hexanes: 1/3; then MeOH/ $\text{CH}_2\text{Cl}_2$ : 1/125) yielded **3** as red solid (225.0 mg, 83%). mp.: 148.6-149.6  $^{\circ}\text{C}$ ;  $R_f$  0.33 (dichloromethane/methane: 30/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 8.07-7.98 (m, 2H), 7.53 (*pseudo t*, 1H,  $J = 7.2$  Hz), 7.43 (*pseudo t*, 1H,  $J = 7.5$  Hz), 7.39-7.29 (m, 4H), 5.24 (d, 1H,  $J = 14.9$  Hz), 2.35-2.20 (m, 3H), 2.16-1.98 (m, 3H), 1.43-1.24 (m, 12H), 0.82 (t, 3H,  $J = 6.5$  Hz).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 185.3, 180.0 (d,  $J = 7.7$  Hz), 171.4, 135.0, 134.7, 133.4, 132.1 (d,  $J = 2.0$  Hz), 131.7, 131.5 (d,  $J = 4.9$  Hz), 126.1, 125.7, 122.2 ( $J = 3.9$  Hz), 113.3, 36.3 ( $J = 49.0$  Hz), 24.2 ( $J = 4.9$  Hz), 24.0 ( $J = 15.0$  Hz), 20.8 ( $J = 47.0$  Hz), 13.2.  $^{31}\text{P}$  (200 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 32.8. **MS** (ESI)  $m/z$  (%): 567 [ $\text{M}+2+\text{Na}$ ] $^+$  (100), 565 (100), 545 (80), 543 (80). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3062 (w), 2947 (m), 1676 (m), 1586 (m), 1533 (s), 1367 (m), 1271 (m), 557 (m). **HRMS** (ESI) for  $\text{C}_{29}\text{H}_{37}\text{BrO}_3\text{P}^+$ , [ $\text{M}+\text{H}$ ] $^+$  (543.1658) found: 543.1683.

##### Synthesis of 3b:



Prepared according to **TP- I** from **1** (87.1 mg, 0.5 mmol), **2b** (105.0  $\mu\text{L}$ , 1.8 equiv), trifluoroacetic acid (68.9  $\mu\text{L}$ , 1.8 equiv), and tributylphosphine (150.0  $\mu\text{L}$ , 1.2 equiv) in dry THF (0.5 mL) [reaction condition: 60-64  $^{\circ}\text{C}$  for 5h]. Purification by *flash*-chromatography (ethyl acetate/hexanes: 1/3; then MeOH/ $\text{CH}_2\text{Cl}_2$ : 1/125) yielded **3b** as red solid (206.6 mg, 76%). mp.: 149.3-150.0  $^{\circ}\text{C}$ ;  $R_f$  0.28 (dichloromethane/methane: 50/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 8.13-7.96 (m, 3H), 7.63-7.53 (m, 3H), 7.23 (*pseudo t*, 1H,  $J = 7.5$  Hz), 7.10 (*pseudo t*, 1H,  $J = 7.5$  Hz), 6.02 (d, 1H,  $J = 14.8$  Hz), 2.51-2.19 (m, 6H), 1.51-1.29 (m, 12H), 0.87 (t, 3H,  $J = 6.6$  Hz).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 185.6, 180.2 (d,  $J = 7.9$  Hz), 171.4 (d,  $J = 2.1$  Hz), 136.4 (d,  $J = 2.0$  Hz), 135.0, 133.5, 133.2 (d,  $J = 2.0$  Hz), 133.1 (d,  $J = 3.5$  Hz), 131.7, 130.7, 129.5 (d,  $J = 2.9$  Hz), 128.4 (d,  $J = 2.7$  Hz), 125.9 (d,  $J = 8.9$  Hz), 124.0 (d,  $J = 7.2$  Hz), 114.9 (d,  $J = 1.9$  Hz), 35.3 (d,  $J = 49.9$  Hz), 24.3 (d,  $J = 4.9$  Hz), 24.0 (d,  $J = 15.4$  Hz), 21.1 (d,  $J = 46.7$  Hz), 13.3.  $^{31}\text{P}$  (200 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 36.1. **MS** (ESI)  $m/z$  (%): 567 (90), 565 [ $\text{M}+\text{Na}$ ] $^+$  (100), 261 (33). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3062 (w), 2957 (m), 1681 (m), 1586 (m), 1524 (s), 1362 (m), 1238 (m), 548 (w). **HRMS** (ESI) for  $\text{C}_{29}\text{H}_{37}\text{BrO}_3\text{P}^+$ , [ $\text{M}+\text{H}$ ] $^+$  (543.1658) found: 543.1673.

##### Synthesis of 3c:

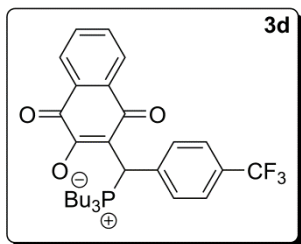


Prepared according to **TP- I** from **1** (87.1 mg, 0.5 mmol), **2c** (126.5 mg, 1.8 equiv), trifluoroacetic acid (68.9  $\mu\text{L}$ , 1.8 equiv), and tributylphosphine (150.0  $\mu\text{L}$ , 1.2 equiv) in dry THF (0.5 mL) [reaction condition: 60-64  $^{\circ}\text{C}$  for 8h]. Purification by *flash*-chromatography (ethyl acetate/hexanes: 1/3; then MeOH/ $\text{CH}_2\text{Cl}_2$ : 1/125) yielded **3c** as red solid (227.1 mg, 91%). mp.: 149.1-150.1  $^{\circ}\text{C}$ ;  $R_f$  0.34 (dichloromethane/methane: 30/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 8.09-8.00 (m, 2H), 7.60 (t, 1H,  $J = 7.4$  Hz), 7.51 (m, 1H,  $J = 7.4$  Hz), 7.48-7.43 (m, 2H), 7.30-7.24 (m, 2H),



5.33 (d, 1H,  $J = 15.0$  Hz), 2.42-2.26 (m, 3H), 2.23-2.08 (m, 3H), 1.50-1.28 (m, 12H), 0.89 (t, 3H,  $J = 7.0$  Hz).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 185.2, 179.8 (d,  $J = 7.4$  Hz), 171.3, 134.8, 134.0 (d,  $J = 2.9$  Hz), 133.9 (d,  $J = 3.7$  Hz), 133.3, 131.5, 131.1 (d,  $J = 4.8$  Hz), 130.6, 128.9 (d,  $J = 2.1$  Hz), 125.9, 125.6, 113.3, 36.0 (d,  $J = 49.0$  Hz), 24.1 (d,  $J = 5.0$  Hz), 23.8 (d,  $J = 15.0$  Hz), 20.5 (d,  $J = 47.1$  Hz), 13.1.  $^{31}\text{P}$  (200 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 33.0. **MS** (ESI)  $m/z$  (%): 499  $[\text{M}+\text{H}]^+$  (100). **IR** (KBr)  $\tilde{\nu}(\text{cm}^{-1})$ : 3062 (w), 2947 (m), 1681 (m), 1581(m), 1533 (s), 1367 (m), 1276 (M), 729 (w). **HRMS** (ESI) for  $\text{C}_{29}\text{H}_{37}\text{ClO}_3\text{P}^+$ ,  $[\text{M}+\text{H}]^+$  (499.2163) found: 499.2134.

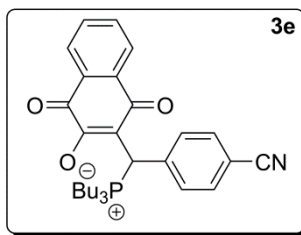
### Synthesis of 3d:



Prepared according to **TP- I** from **1** (87.1 mg, 0.5 mmol), **2d** (122.9  $\mu\text{L}$ , 1.8 equiv), trifluoroacetic acid (68.9  $\mu\text{L}$ , 1.8 equiv), and tributylphosphine (150.0  $\mu\text{L}$ , 1.2 equiv) in dry THF (0.5 mL) [reaction condition: 60-64 °C for 4h]. Purification by *flash*-chromatography (ethyl acetate/hexanes: 1/3; then MeOH/ $\text{CH}_2\text{Cl}_2$ : 1/125) yielded **3d** as red solid (250.3 mg, 94%). mp.: 160.5-161.5 °C;  $R_f$  0.34 (dichloromethane/methane: 30/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ :

8.10-7.98 (m, 2H), 7.70-7.47 (m, 6H), 5.41 (d, 1H,  $J = 15.2$  Hz), 2.45-2.28 (m, 3H), 2.27-2.10 (m, 3H), 1.51-1.26 (m, 12H), 0.88 (t, 9H,  $J = 7.0$  Hz).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 185.0, 179.8 (d,  $J = 7.5$  Hz), 171.3 (d,  $J = 2.8$  Hz), 139.8, 134.7, 133.3, 131.4, 130.6, 130.1 (d,  $J = 4.8$  Hz), 130.0 (d,  $J = 32.5$  Hz), 125.8, 125.6 (m), 125.5, 123.5 (d,  $J = 163.3$  Hz), 112.9 (d,  $J = 2.5$  Hz), 36.4 (d,  $J = 48.8$  Hz), 24.0 (d,  $J = 4.9$  Hz), 23.7 (d,  $J = 15.1$  Hz), 20.5 (d,  $J = 46.9$  Hz), 12.9.  $^{31}\text{P}$  (200 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 33.4. **MS** (ESI)  $m/z$  (%): 533  $[\text{M}+\text{H}]^+$  (100). **IR** (KBr)  $\tilde{\nu}(\text{cm}^{-1})$ : 3062 (w), 2938 (m), 1676 (m), 1581 (m), 1533 (s), 1319 (s), 1124 (m), 1067 (m), 733 (m). **HRMS** (ESI) for  $\text{C}_{30}\text{H}_{37}\text{F}_3\text{O}_3\text{P}^+$ ,  $[\text{M}+\text{H}]^+$  (533.2427) found: 533.2402.

### Synthesis of 3e:

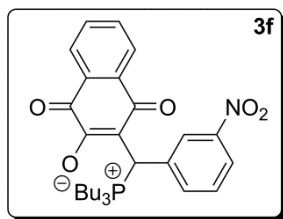


Prepared according to **TP- I** from **1** (87.1 mg, 0.5 mmol), **2e** (118.0 mg, 1.8 equiv), trifluoroacetic acid (68.9  $\mu\text{L}$ , 1.8 equiv), and tributylphosphine (150.0  $\mu\text{L}$ , 1.2 equiv) in dry THF (0.5 mL) [reaction condition: 60-64 °C for 2h]. Purification by *flash*-chromatography (ethyl acetate/hexanes: 1/3; then MeOH/ $\text{CH}_2\text{Cl}_2$ : 1/125) yielded **3e** as red solid (191.3 mg, 78%). mp.: 185.4-186.0 °C;  $R_f$  0.29 (dichloromethane/methane: 30/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ :

8.10-7.98 (m, 2H), 7.69-7.57 (m, 5H), 7.53 (*pseudo* t, 1H,  $J = 7.5$  Hz), 5.41 (d, 1H,  $J = 15.2$  Hz), 2.45-2.28 (m, 3H), 2.24-2.08 (m, 3H), 1.50-1.30 (m, 12H), 0.89 (t, 9H,  $J = 6.9$  Hz).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 184.8, 179.7 (d,  $J = 6.9$  Hz), 171.3, 141.2, 134.5, 133.3, 132.3, 131.4, 130.6, 130.4 (d,  $J = 4.5$  Hz), 125.8, 125.4, 117.9, 112.5, 111.6 (d,  $J = 2.9$  Hz), 36.7 (d,  $J = 48.7$  Hz), 23.9 (d,  $J = 4.9$  Hz), 23.6 (d,  $J = 15.2$  Hz), 20.5 (d,  $J = 46.8$  Hz), 13.0.  $^{31}\text{P}$  (200 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 33.6. **MS** (ESI)  $m/z$  (%): 490  $[\text{M}+\text{H}]^+$  (100). **IR** (KBr)  $\tilde{\nu}(\text{cm}^{-1})$ : 3052 (w), 2947 (m), 2354 (w), 2220 (m), 1680 (s), 1581 (s), 1538 (s), 1366 (s), 1271 (m), 738 (m). **HRMS** (ESI) for  $\text{C}_{30}\text{H}_{37}\text{NO}_3\text{P}^+$ ,  $[\text{M}+\text{H}]^+$  (490.2506) found: 490.2487.

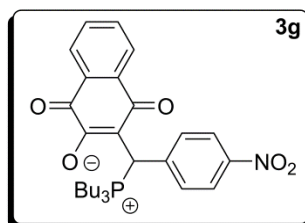
### Synthesis of 3f:

Prepared according to **TP- I** from **1** (87.1 mg, 0.5 mmol), **2f** (136.0 mg, 1.8 equiv), trifluoroacetic acid (68.9  $\mu\text{L}$ , 1.8 equiv), and tributylphosphine (150.0  $\mu\text{L}$ , 1.2 equiv) in dry THF (0.5 mL) [reaction condition:



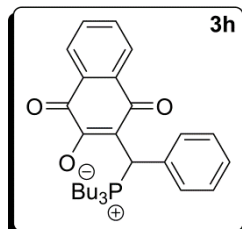
60-64 °C for 4h]. Purification by *flash*-chromatography (ethyl acetate/hexanes: 1/3; then MeOH/CH<sub>2</sub>Cl<sub>2</sub>: 1/125) yielded **3f** as red solid (234.1 mg, 92%). mp.: 153.4-154.5 °C; *R<sub>f</sub>* 0.29 (dichloromethane/methane: 30/1) <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>, 25 °C) δ/ppm: 8.27 (*pseudo s*, 1H), 8.14 (d, 1H, *J* = 7.8 Hz), 8.09-8.00 (m, 2H), 7.98 (d, 1H, *J* = 7.4 Hz), 7.62 (t, 1H, *J* = 7.6 Hz), 7.56-7.45 (m, 2H), 5.47 (d, 1H, *J* = 15.2 Hz), 2.45-2.29 (m, 3H), 2.26-2.10 (m, 3H), 1.51-1.31 (m, 12H), 0.89 (t, 9H, *J* = 6.9 Hz). <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>, 25 °C) δ/ppm: 185.1, 180.0 (d, *J* = 7.3 Hz), 171.6, 148.1, 138.0, 136.4 (d, *J* = 4.1 Hz), 134.7, 133.6, 131.7, 130.9, 130.2, 126.2, 125.7, 124.1 (d, *J* = 5.3 Hz), 123.0, 112.8, 36.5 (d, *J* = 49.4 Hz), 24.2 (d, *J* = 4.9 Hz), 23.9 (d, *J* = 15.1 Hz), 20.8 (d, *J* = 46.9 Hz), 13.2. <sup>31</sup>P (200 MHz, CDCl<sub>3</sub>, 25 °C) δ/ppm: 33.7. MS (ESI) *m/z* (%): 532 [M+Na]<sup>+</sup> (100). IR (KBr)  $\tilde{\nu}$  (cm<sup>-1</sup>): 3072 (w), 2957 (m), 1676 (m), 1585 (m), 1529 (s), 1367 (m), 1348 (m), 1271 (m), 710 (w). HRMS (ESI) for C<sub>29</sub>H<sub>37</sub>NO<sub>5</sub>P<sup>+</sup>, [M+H]<sup>+</sup> (510.2404) found: 510.2390.

### Synthesis of **3g**:



Prepared according to TP- I from **1** (87.1 mg, 0.5 mmol), **2g** (136.1 mg, 1.8 equiv), trifluoroacetic acid (68.9 μL, 1.8 equiv), and tributylphosphine (150.0 μL, 1.2 equiv) in dry THF (0.5 mL) [reaction condition: 60-64 °C for 3h]. Purification by *flash*-chromatography (ethyl acetate/hexanes: 1/3; then MeOH/CH<sub>2</sub>Cl<sub>2</sub>: 1/125) yielded **3g** as red solid (227.1 mg, 90%). mp.: 168.8-169.4 °C; *R<sub>f</sub>* 0.32 (dichloromethane/methane: 30/1). <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>, 25 °C) δ/ppm: 8.15 (d, 2H, *J* = 8.5 Hz), 8.06 (d, 1H, *J* = 7.5 Hz), 8.01 (d, 1H, *J* = 7.5 Hz), 7.71 (d, 2H, *J* = 7.0 Hz), 7.61 (t, 1H, *J* = 7.4 Hz), 7.52 (t, 1H, *J* = 7.3 Hz), 5.46 (d, 1H, *J* = 15.4 Hz), 2.47-2.32 (m, 3H), 2.23-2.08 (m, 3H), 1.50-1.33 (m, 12H), 0.88 (t, 9H, *J* = 6.8 Hz). <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>, 25 °C) δ/ppm: 184.9, 179.8 (d, *J* = 7.3 Hz), 171.4, 147.3 (d, *J* = 3.2 Hz), 143.4 (d, *J* = 2.3 Hz), 134.6, 133.5, 131.5, 130.8, 130.7, 126.0, 125.6, 123.8 (d, *J* = 1.7 Hz), 112.5 (d, *J* = 2.5 Hz), 36.6 (d, *J* = 48.6 Hz), 24.1 (d, *J* = 5.0 Hz), 23.8 (d, *J* = 15.1 Hz), 20.7 (d, *J* = 46.8 Hz), 13.1. <sup>31</sup>P-NMR (200 MHz, CDCl<sub>3</sub>, 25 °C) δ/ppm: 33.6. MS (ESI) *m/z* (%): 510 [M+H]<sup>+</sup> (100). IR (KBr)  $\tilde{\nu}$  (cm<sup>-1</sup>): 3062 (w), 2956 (m), 1680 (s), 1590 (s), 1523 (s), 1371 (s), 1271 (s), 738 (s). HRMS (ESI) for C<sub>29</sub>H<sub>37</sub>NO<sub>5</sub>P, [M+H]<sup>+</sup> (510.2409), found: 510.2414.

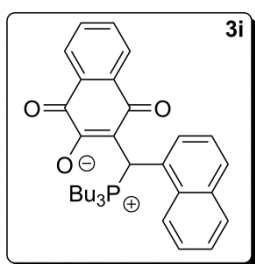
### Synthesis of **3h**:



Prepared according to TP- I from **1** (87.1 mg, 0.5 mmol), **2h** (91.5 μL, 1.8 equiv), trifluoroacetic acid (68.9 μL, 1.8 equiv), and tributylphosphine (150.0 μL, 1.2 equiv) in dry THF (0.5 mL) [reaction condition: 60-64 °C for 12h]. Purification by *flash*-chromatography (ethyl acetate/hexanes: 1/3; then MeOH/CH<sub>2</sub>Cl<sub>2</sub>: 1/125) yielded **3h** as red solid (184.0 mg, 79 %). mp.: 175.0-176.0 °C; *R<sub>f</sub>* 0.31 (dichloromethane/methane: 30/1). <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>, 25 °C) δ/ppm: 8.04 (*pseudo t*, 2H, *J* = 7.8 Hz), 7.60 (*pseudo t*, 1H, *J* = 7.2 Hz), 7.54-7.45 (m, 3H), 7.33-7.22 (m, 3H), 5.36 (d, 1H, *J* = 15.0 Hz), 2.42-2.11 (m, 6H), 1.48-1.23 (m, 12H), 0.87 (t, 9H, *J* = 6.6 Hz). <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>, 25 °C) δ/ppm: 185.6, 180.1 (d, *J* = 7.9 Hz), 171.5, 135.4 (d, *J* = 3.4 Hz), 135.2, 133.4, 131.8, 130.7, 129.9 (d, *J* = 4.9 Hz), 129.0 (d, *J* = 2.4 Hz), 128.1 (d, *J* = 3.0 Hz), 126.1, 125.7, 113.7, 36.6 (d, *J* = 48.8 Hz), 24.3 (d, *J* = 5.0 Hz), 24.0 (d, *J* = 15.1 Hz), 20.7 (d, *J* = 47.2 Hz), 13.3. <sup>31</sup>P (200 MHz, CDCl<sub>3</sub>, 25 °C) δ/ppm: 32.9. MS (ESI) *m/z* (%): 465 [M+H]<sup>+</sup> (100). IR (KBr)  $\tilde{\nu}$  (cm<sup>-1</sup>): 3072 (w), 2957 (m), 1671 (m), 1586 (s),

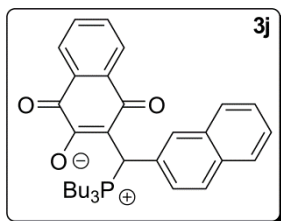
1529 (s), 1362 (m), 957 (m). **HRMS** (ESI) for  $C_{29}H_{38}O_3P^+$ ,  $[M+H]^+$  (465.2553) found: 465.2522.

### Synthesis of 3i:



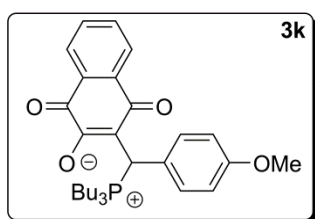
Prepared according to **TP- I** from **1** (87.1 mg, 0.5 mmol), **2i** (122.3  $\mu$ L, 1.8 equiv), trifluoroacetic acid (68.9  $\mu$ L, 1.8 equiv), and tributylphosphine (150.0  $\mu$ L, 1.2 equiv) in dry THF (0.5 mL) [reaction condition: 60-64  $^{\circ}$ C for 8h]. Purification by *flash*-chromatography (ethyl acetate/hexanes: 1/3; then MeOH/CH<sub>2</sub>Cl<sub>2</sub>: 1/125) yielded **3i** as red solid (149.2 mg, 58%). mp.: 193.1-194.0  $^{\circ}$ C;  $R_f$  0.36 (dichloromethane/methane: 30/1).  **$^1H$ -NMR** (400 MHz, CDCl<sub>3</sub>, 25  $^{\circ}$ C)  $\delta$ /ppm: 8.53 (d, 1H,  $J$  = 8.5 Hz), 8.17-8.12 (m, 1H), 8.08-7.99 (m, 2H), 7.85 (d, 1H,  $J$  = 8.1 Hz), 7.77 (d, 1H,  $J$  = 8.0 Hz), 7.64 (t, 1H,  $J$  = 7.6 Hz), 7.57 (d, 1H,  $J$  = 7.3 Hz), 7.53-7.45 (m, 2H), 7.41 (t, 1H,  $J$  = 7.7 Hz), 6.39 (d, 1H,  $J$  = 15.4 Hz), 2.41-2.12 (m, 6H), 1.40-1.22 (m, 12H), 0.82 (t, 9H,  $J$  = 6.7 Hz).  **$^{13}C$ -NMR** (100 MHz, CDCl<sub>3</sub>, 25  $^{\circ}$ C)  $\delta$ /ppm: 185.6, 180.3 (d,  $J$  = 8.0 Hz), 171.5, 140.0, 133.8, 133.3, 132.2 (d,  $J$  = 2.7 Hz), 131.7, 131.4 (d,  $J$  = 5.4 Hz), 130.5, 129.7 (d,  $J$  = 5.2 Hz), 129.0, 128.5 (d,  $J$  = 3.1 Hz), 126.7, 125.9, 125.7, 122.8, 115.2, 30.8 (d,  $J$  = 52.8 Hz), 24.1 (d,  $J$  = 4.9 Hz), 23.9 (d,  $J$  = 15.1 Hz), 21.0 (d,  $J$  = 46.7 Hz), 13.1.  **$^{31}P$ -NMR** (200 MHz, CDCl<sub>3</sub>, 25  $^{\circ}$ C)  $\delta$ /ppm: 35.3. **MS** (ESI)  $m/z$  (%): 515  $[M+H]^+$  (100). **IR** (KBr)  $\tilde{\nu}$  (cm<sup>-1</sup>): 3052 (w), 2956 (m), 1671 (m), 1580 (m), 1519 (s), 1361 (m), 1271 (m), 776 (w). **HRMS** (ESI) for  $C_{33}H_{40}O_3P$ ,  $[M+H]^+$  (515.2715), found: 515.2689.

### Synthesis of 3j:



Prepared according to **TP- I** from **1** (87.1 mg, 0.5 mmol), **2j** (85.6  $\mu$ L, 1.8 equiv), trifluoroacetic acid (68.9  $\mu$ L, 1.8 equiv), and tributylphosphine (150.0  $\mu$ L, 1.2 equiv) in dry THF (0.5 mL) [reaction condition: 60-64  $^{\circ}$ C for 8h]. Purification by *flash*-chromatography (ethyl acetate/hexanes: 1/3; then MeOH/CH<sub>2</sub>Cl<sub>2</sub>: 1/125) yielded **3j** as red solid (180.3 mg, 70%). mp.: 203.9-204.5  $^{\circ}$ C;  $R_f$  0.31 (dichloromethane/methane: 30/1).  **$^1H$ -NMR** (400 MHz, CDCl<sub>3</sub>, 25  $^{\circ}$ C)  $\delta$ /ppm: 8.06 (d, 1H,  $J$  = 7.5 Hz), 8.02 (d, 1H,  $J$  = 7.6 Hz), 7.94-7.90 (m, 1H), 7.84-7.74 (m, 3H), 7.66 (d, 1H,  $J$  = 8.6 Hz), 7.59 (t, 1H,  $J$  = 7.9 Hz), 7.52-7.43 (m, 3H), 5.52 (d, 1H,  $J$  = 15.1 Hz), 2.48-2.33 (m, 3H), 2.27-2.13 (m, 3H), 1.49-1.31 (m, 12H), 0.86 (t, 9H,  $J$  = 6.9 Hz).  **$^{13}C$ -NMR** (100 MHz, CDCl<sub>3</sub>, 25  $^{\circ}$ C)  $\delta$ /ppm: 185.4, 180.1 (d,  $J$  = 8.1 Hz), 171.3, 135.0, 133.3, 133.1 (d,  $J$  = 2.5 Hz), 132.8 (d,  $J$  = 7.5 Hz), 132.7, 131.7, 130.6, 128.7, 128.6 (d,  $J$  = 6.9 Hz), 127.7, 127.5 (d,  $J$  = 3.4 Hz), 127.4, 126.2, 126.1, 125.9, 125.6, 113.5, 36.8 (d,  $J$  = 49.1 Hz), 24.2 (d,  $J$  = 4.9 Hz), 23.9 (d,  $J$  = 15.1 Hz), 20.7 (d,  $J$  = 47.2 Hz), 13.1.  **$^{31}P$ -NMR** (200 MHz, CDCl<sub>3</sub>, 25  $^{\circ}$ C)  $\delta$ /ppm: 33.1. **MS** (ESI)  $m/z$  (%): 515  $[M+H]^+$  (100). **IR** (KBr)  $\tilde{\nu}$  (cm<sup>-1</sup>): 3043 (w), 2956 (m), 1671 (s), 1585 (s), 1533 (s), 1452 (m), 1352 (m), 1276 (s), 733 (m). **HRMS** (ESI) for  $C_{33}H_{40}O_3P$ ,  $[M+H]^+$  (515.2715), found: 515.2692.

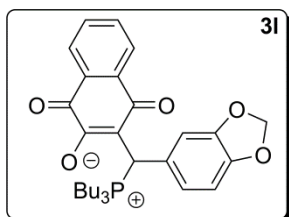
### Synthesis of 3k:



Prepared according to **TP- I** from **1** (87.1 mg, 0.5 mmol), **2k** (109.5  $\mu$ L, 1.8 equiv), trifluoroacetic acid (68.9  $\mu$ L, 1.8 equiv), and tributylphosphine (150.0  $\mu$ L, 1.2 equiv) in dry THF (0.5 mL) [reaction condition: 60-64  $^{\circ}$ C for 15h]. Purification by *flash*-chromatography (ethyl acetate/hexanes: 1/3; then MeOH/CH<sub>2</sub>Cl<sub>2</sub>: 1/125) yielded **3k** as red solid (190.4 mg, 77%). mp.: 164.6-165.6  $^{\circ}$ C;  $R_f$  0.31 (dichloromethane/methane: 30/1).  **$^1H$ -NMR** (400 MHz, CDCl<sub>3</sub>, 25  $^{\circ}$ C)  $\delta$ /ppm: 8.03

(d, 2H,  $J = 7.8$  Hz), 7.59 (t, 1H,  $J = 7.3$  Hz), 7.49 (t, 1H,  $J = 7.5$  Hz), 7.42 (d, 2H,  $J = 8.2$  Hz), 6.81 (d, 2H,  $J = 8.2$  Hz), 5.31 (d, 1H,  $J = 14.7$  Hz), 3.76 (s, 3H), 2.38-2.25 (m, 3H), 2.23-2.09 (m, 3H), 1.47-1.30 (m, 12H), 0.88 (t, 9H,  $J = 6.6$  Hz).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 185.1, 179.5 (d,  $J = 8.3$  Hz), 170.9, 159.0 (d,  $J = 2.5$  Hz), 134.7, 133.0, 131.3, 130.5 (d,  $J = 4.7$  Hz), 130.1, 126.8 (d,  $J = 3.0$  Hz), 125.4, 125.3, 126.8 (d,  $J = 3.0$  Hz), 125.4, 125.3, 113.9, 113.6, 54.8, 35.3 (d,  $J = 48.9$  Hz), 23.8 (d,  $J = 4.9$  Hz), 23.6 (d,  $J = 15.0$  Hz), 20.2 (d,  $J = 47.1$  Hz), 12.9.  $^{31}\text{P-NMR}$  (200 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 32.7. **MS** (ESI)  $m/z$  (%): 495  $[\text{M}+\text{H}]^+$  (100). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3062 (w), 2956 (m), 1676 (s), 1585 (s), 1533 (s), 1271 (s), 1252 (s), 1176 (s), 733 (s). **HRMS** (ESI) for  $\text{C}_{30}\text{H}_{40}\text{O}_4\text{P}$ ,  $[\text{M}+\text{H}]^+$  (495.2664), found: 495.2642.

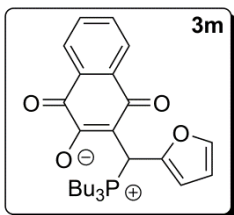
### Synthesis of 3l:



Prepared according to **TP- I** from **1** (87.1 mg, 0.5 mmol), **2l** (135.1  $\mu\text{L}$ , 1.8 equiv), trifluoroacetic acid (68.9  $\mu\text{L}$ , 1.8 equiv), and tributylphosphine (150.0  $\mu\text{L}$ , 1.2 equiv) in dry THF (0.5 mL) [reaction condition: 60-64 °C for 5h]. Purification by *flash*-chromatography (ethyl acetate/hexanes: 1/3; then MeOH/ $\text{CH}_2\text{Cl}_2$ : 1/125) yielded **3l** as red solid (124.6 mg, 49%). mp.: 203.7-204.5 °C;  $R_f$  0.28 (dichloromethane/methane: 30/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 8.04

(t, 1H  $J = 7.2$  Hz), 7.60 (t, 1H  $J = 7.5$  Hz), 7.50 (t, 1H  $J = 7.5$  Hz), 7.06-7.03 (m, 1H), 6.97-6.91 (m, 3H), 6.72 (d, 1H,  $J = 8.0$  Hz), 5.91 (d, 1H,  $J = 4.3$  Hz), 5.27 (d, 1H,  $J = 14.8$  Hz), 2.39-2.12 (m, 6H), 1.50-1.32 (m, 12H), 0.89 (t, 9H,  $J = 6.7$  Hz).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 185.2, 179.7 (d,  $J = 7.9$  Hz), 171.2, 148.0, 147.3 (d,  $J = 3.0$  Hz), 135.0, 133.2, 131.6, 130.4, 128.8, 125.8, 125.5, 123.0 (d,  $J = 5.9$  Hz), 113.6, 110.1, 108.2, 101.0, 36.3 (d,  $J = 49.1$  Hz), 24.1 (d,  $J = 4.7$  Hz), 23.8 (d,  $J = 14.9$  Hz), 20.5 (d,  $J = 47.1$  Hz), 13.1.  $^{31}\text{P-NMR}$  (200 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 32.8. **MS** (ESI)  $m/z$  (%): 509  $[\text{M}+\text{H}]^+$  (100). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3071 (w), 2947 (m), 1676 (s), 1590 (s), 1533 (s), 1366 (m), 1266 (m), 1242 (m), 723 (m). **HRMS** (ESI) for  $\text{C}_{30}\text{H}_{38}\text{O}_5\text{P}$ ,  $[\text{M}+\text{H}]^+$  (509.2457), found: 509.2413.

### Synthesis of 3m:



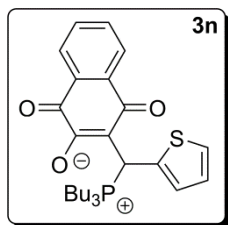
Prepared according to **TP- I** from **1** (87.1 mg, 0.5 mmol), **2m** (74.6  $\mu\text{L}$ , 1.8 equiv), trifluoroacetic acid (68.9  $\mu\text{L}$ , 1.8 equiv), and tributylphosphine (150.0  $\mu\text{L}$ , 1.2 equiv) in dry THF (0.5 mL) [reaction condition: 60-64 °C for 8.5h]. Purification by *flash*-chromatography (ethyl acetate/hexanes: 1/3; then MeOH/ $\text{CH}_2\text{Cl}_2$ : 1/125) yielded **3m** as red solid (175.3 mg, 77%). mp.: 134.9-135.9 °C;  $R_f$  0.3 (dichloromethane/methane: 30/1).

$^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 8.10-8.05 (m, 2H), 7.63 (t, 1H,  $J = 7.5$  Hz), 7.52 (t, 1H,  $J = 7.6$  Hz), 7.34-7.32 (m, 1H), 6.32-6.29 (m, 2H), 5.71 (d, 1H,  $J = 14.2$  Hz), 2.31-2.19 (m, 6H), 1.48-1.36 (m, 12H), 0.89 (t, 9H,  $J = 6.8$  Hz).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 184.8, 179.4 (d,  $J = 5.6$  Hz), 171.7, 147.6 (d,  $J = 5.9$  Hz), 141.5, 134.9, 133.3, 131.6, 130.5, 125.9, 125.6, 111.2, 110.2 (d,  $J = 6.7$  Hz), 108.8 (d,  $J = 4.4$  Hz), 29.9 (d,  $J = 50.2$  Hz), 23.8 (d,  $J = 15.4$  Hz), 23.7 (d,  $J = 5.0$  Hz), 20.3 (d,  $J = 45.8$  Hz), 13.0.  $^{31}\text{P-NMR}$  (200 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 34.7. **MS** (ESI)  $m/z$  (%): 455  $[\text{M}+\text{H}]^+$  (100). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3100 (w), 2956 (m), 1676 (s), 1580 (s), 1528 (s), 1371 (s), 1266 (s), 1223 (m), 957 (m), 761 (m). **HRMS** (ESI) for  $\text{C}_{27}\text{H}_{36}\text{O}_4\text{P}$ ,  $[\text{M}+\text{H}]^+$  (455.2351), found: 455.2331.

### Synthesis of 3n:

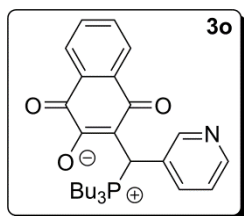
Prepared according to **TP- I** from **1** (87.1 mg, 0.5 mmol), **2n** (84.1  $\mu\text{L}$ , 1.8 equiv), trifluoroacetic acid (68.9





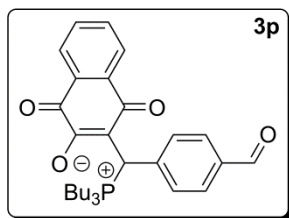
$\mu\text{L}$ , 1.8 equiv), and tributylphosphine (150.0  $\mu\text{L}$ , 1.2 equiv) in dry THF (0.5 mL) [reaction condition: 60-64 °C for 14.5h]. Purification by *flash*-chromatography (ethyl acetate/hexanes: 1/3; then MeOH/CH<sub>2</sub>Cl<sub>2</sub>: 1/125) yielded **3n** as red solid (120.1 mg, 51%). mp.: 148.1-148.9 °C;  $R_f$  0.27 (dichloromethane/methane: 30/1). <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>, 25 °C)  $\delta$ /ppm: 8.08-8.02 (m, 2H), 7.61 (t, 1H,  $J = 7.5$  Hz), 7.50 (t, 1H,  $J = 7.6$  Hz), 7.20-7.15 (m, 2H), 6.93-6.89 (m, 1H), 5.82 (d, 1H,  $J = 14.6$  Hz), 2.32-2.21 (m, 6H), 1.47-1.33 (m, 12H), 0.88 (t, 9H,  $J = 6.9$  Hz). <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>, 25 °C)  $\delta$ /ppm: 185.1, 179.5 (d,  $J = 6.3$  Hz), 171.5, 136.2 (d,  $J = 3.3$  Hz), 135.0, 133.4, 131.7, 130.6, 128.3 (d,  $J = 6.7$  Hz), 126.8 (d,  $J = 2.8$  Hz), 126.1, 126.0 (d,  $J = 4.0$  Hz), 125.7, 112.8, 31.6 (d,  $J = 51.3$  Hz), 24.0 (d,  $J = 5.6$  Hz), 23.9 (d,  $J = 15.3$  Hz), 20.3 (d,  $J = 46.1$  Hz), 13.2. <sup>31</sup>P-NMR (200 MHz, CDCl<sub>3</sub>, 25 °C)  $\delta$ /ppm: 33.4. MS (ESI)  $m/z$  (%): 471 [M+H]<sup>+</sup> (100). IR (KBr)  $\tilde{\nu}$  (cm<sup>-1</sup>): 3052 (w), 2956 (m), 1676 (m), 1580 (m), 1533 (s), 1366 (m), 1266 (m), 738 (w), 704 (w). HRMS (ESI) for C<sub>27</sub>H<sub>36</sub>O<sub>3</sub>PS, [M+H]<sup>+</sup> (471.2123), found: 471.2102.

### Synthesis of **3o**:



Prepared according to **TP- I** from **1** (87.1 mg, 0.5 mmol), **2o** (84.5  $\mu\text{L}$ , 1.8 equiv), trifluoroacetic acid (68.9  $\mu\text{L}$ , 1.8 equiv), and tributylphosphine (150.0  $\mu\text{L}$ , 1.2 equiv) in dry THF (0.5 mL) [reaction condition: 60-64 °C for 8 h]. Purification by *flash*-chromatography (ethyl acetate/hexanes: 1/3; then MeOH/CH<sub>2</sub>Cl<sub>2</sub>: 1/125) yielded **3o** as red solid (153.9 mg, 66%). mp.: 151.2-152.1 °C;  $R_f$  0.23 (dichloromethane/methane: 30/1). <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>, 25 °C)  $\delta$ /ppm: 8.66-8.62 (m, 1H), 8.55-8.50 (m, 1H), 8.09-7.95 (m, 3H), 7.62 (t, 1H,  $J = 7.3$  Hz), 7.52 (t, 1H,  $J = 7.4$  Hz), 7.26-7.21 (m, 1H), 5.42 (d, 1H,  $J = 15.0$  Hz), 2.42-2.28 (m, 3H), 2.25-2.11 (m, 3H), 1.49-1.30 (m, 12H), 0.88 (t, 9H,  $J = 6.8$  Hz). <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>, 25 °C)  $\delta$ /ppm: 185.2, 180.0, (d,  $J = 7.3$  Hz), 171.8 (d,  $J = 2.9$  Hz), 150.0 (d,  $J = 5.6$  Hz), 149.5 (d,  $J = 2.9$  Hz), 138.0 (d,  $J = 3.9$  Hz), 135.0, 133.6, 132.0 (d,  $J = 2.6$  Hz), 131.8, 130.9, 126.2, 125.8, 124.1, 112.8, 34.2 (d,  $J = 49.5$  Hz), 24.3 (d,  $J = 5.0$  Hz), 24.0 (d,  $J = 15.0$  Hz), 20.8 (d,  $J = 46.9$  Hz), 13.2. <sup>31</sup>P-NMR (200 MHz, CDCl<sub>3</sub>, 25 °C)  $\delta$ /ppm: 33.8. MS (ESI)  $m/z$  (%): 466 [M+H]<sup>+</sup> (100). IR (KBr)  $\tilde{\nu}$  (cm<sup>-1</sup>): 3043 (w), 2956 (w), 1680 (m), 1604 (w), 1580 (m), 1509 (s), 1395 (m), 1266 (w), 719 (w). HRMS (ESI) for C<sub>28</sub>H<sub>37</sub>NO<sub>3</sub>P, [M+H]<sup>+</sup> (466.2511), found: 466.2498.

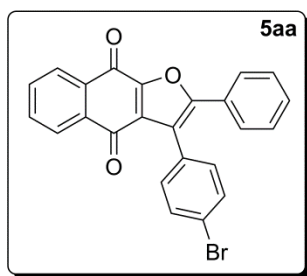
### Synthesis of **3p**:



Prepared according to **TP- I** from **1** (87.1 mg, 0.5 mmol), **2p** (72.4  $\mu\text{L}$ , 1.8 equiv), trifluoroacetic acid (68.9  $\mu\text{L}$ , 1.8 equiv), and tributylphosphine (150.0  $\mu\text{L}$ , 1.2 equiv) in dry THF (0.5 mL) [reaction condition: 60-64 °C for 8 h]. Purification by *flash*-chromatography (ethyl acetate/hexanes: 1/3; then MeOH/CH<sub>2</sub>Cl<sub>2</sub>: 1/125) yielded **3p** as red solid (158.1 mg, 64%). mp.: 166.7-167.1 °C;  $R_f$  0.28 (dichloromethane/methane: 30/1). <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>, 25 °C)  $\delta$ /ppm: 9.97 (s, 1H), 8.11-7.99 (m, 2H), 7.82 (d, 2H,  $J = 8.0$  Hz), 7.70 (d, 2H,  $J = 7.6$  Hz), 7.61 (*pseudo* t, 1H,  $J = 7.4$  Hz), 7.52 (*pseudo* t, 1H,  $J = 7.5$  Hz), 5.44 (d, 1H,  $J = 15.3$  Hz), 2.46-2.29 (m, 3H), 2.25-2.10 (m, 3H), 1.52-1.29 (m, 12H), 0.88 (t, 9H,  $J = 6.8$  Hz). <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>, 25 °C)  $\delta$ /ppm: 191.2, 185.0, 179.8 (d,  $J = 7.3$  Hz), 171.3, 142.5 (d,  $J = 2.9$  Hz), 135.7 (d,  $J = 2.7$  Hz), 134.7, 133.4, 131.5, 130.6, 130.4 (d,  $J = 4.7$  Hz), 130.0, 125.9, 125.5, 112.7, 36.8 (d,  $J = 48.8$  Hz), 24.0 (d,  $J = 5.0$  Hz), 23.7 (d,  $J = 15.2$  Hz), 20.6 (d,  $J = 46.9$  Hz), 13.0. <sup>31</sup>P (200 MHz, CDCl<sub>3</sub>, 25 °C)  $\delta$ /ppm: 33.4. MS (ESI)  $m/z$  (%): 515 [M+Na]<sup>+</sup> (100). IR (KBr)  $\tilde{\nu}$  (cm<sup>-1</sup>): 3062 (w),

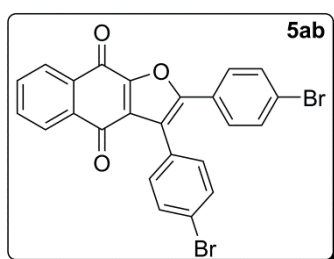
2957 (m), 2861 (m), 2727 (w), 1690 (s), 1605 (m), 1581 (m), 1519 (s), 1395 (m), 1366 (m), 1205 (m), 733 (w). **HRMS** (ESI) for  $C_{30}H_{37}NaO_4P^+$ ,  $[M+Na]^+$  (515.2322) found: 515.2314.

### Synthesis of 5aa:



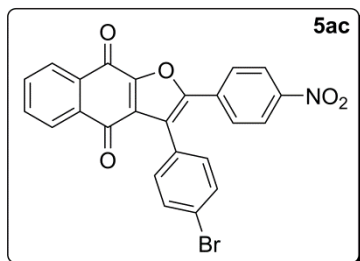
Prepared according to **TP-II** from **3a** (271.7 mg, 0.5 mmol), triethylamine (104.5  $\mu$ L, 1.5 equiv), and **4a** (75.5  $\mu$ L, 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28  $^{\circ}$ C for 1.5 h]. Purification by *flash*-chromatography ( $CH_2Cl_2$ /hexanes: 1/2.8) yielded **5aa** as pale yellow solid (197.7 mg, 92 %). mp.: 207.9-208.8  $^{\circ}$ C;  $R_f$  0.23 (dichloromethane/hexanes: 1/2.8).  $^1H$ -NMR (400 MHz,  $CDCl_3$ , 25  $^{\circ}$ C)  $\delta$ /ppm: 8.22 (d, 1H,  $J$  = 7.7 Hz), 8.08 (d, 1H,  $J$  = 7.1 Hz), 7.80-7.67 (m, 2H), 7.64-7.51 (m, 4H), 7.40-7.28 (m, 5H).  $^{13}C$ -NMR (100 MHz,  $CDCl_3$ , 25  $^{\circ}$ C)  $\delta$ /ppm: 180.7, 173.3, 155.5, 151.3, 133.9, 133.8, 133.4, 132.4, 131.9, 131.8, 130.0, 129.3, 129.2, 128.7, 128.2, 127.3, 126.9, 126.6, 122.9, 120.4. **MS** (70eV, EI)  $m/z$  (%): 430 (95), 428  $[M]^+$  (100), 262 (20), 104 (40), 77 (30). **IR** (KBr)  $\tilde{\nu}$  ( $cm^{-1}$ ): 3053 (w), 1667 (s), 1590 (m), 1214 (m), 971 (m), 681 (m). **HRMS** (FAB) for  $C_{24}H_{14}BrO_3^+$ ,  $[M+H]^+$  (429.0121) found: 429.0126.

### Synthesis of 5ab:



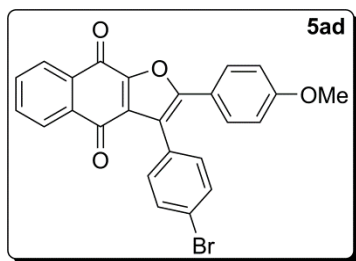
Prepared according to **TP-II** from **3a** (271.7 mg, 0.5 mmol), triethylamine (118.5  $\mu$ L, 1.7 equiv), and **4b** (164.7 mg, 1.5 equiv) in dry THF (2.5 mL) [reaction condition: 60  $^{\circ}$ C for 0.5 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 2/1) yielded **5ab** as pale yellow solid (244.9 mg, 96 %). mp.: 237.9-238.9  $^{\circ}$ C;  $R_f$  0.23 (hexanes/ dichloromethane: 2/1).  $^1H$ -NMR (400 MHz,  $CDCl_3$ , 25  $^{\circ}$ C)  $\delta$ /ppm: 8.22 (d, 1H,  $J$  = 7.5 Hz), 8.08 (d, 1H,  $J$  = 7.8 Hz), 7.80-7.68 (m, 2H), 7.61 (d, 2H,  $J$  = 8.5 Hz), 7.52-7.37 (m, 4H), 7.33 (d, 2H,  $J$  = 8.8 Hz).  $^{13}C$ -NMR (100 MHz,  $CDCl_3$ , 25  $^{\circ}$ C)  $\delta$ /ppm: 180.5, 173.3, 154.4, 151.4, 133.9, 133.4, 132.4, 132.1, 131.7, 129.3, 128.9, 128.6, 127.2, 126.9, 126.7, 124.6, 123.2, 120.9. **MS** (70eV, EI)  $m/z$  (%): 510 (55)  $[M+4]^+$ , 508  $[M+2]^+$  (100), 506 (55)  $[M]^+$ , 348 (45), 261 (40), 105 (50), 76 (60). **IR** (KBr)  $\tilde{\nu}$  ( $cm^{-1}$ ): 3072 (w), 1667 (s), 1581 (m), 1533 (m), 1214 (s), 1190 (m), 705 (m). **HRMS** (EI) for  $C_{24}H_{12}Br_2O_3$ ,  $[M]^+$  (505.9153) found: 505.9150.

### Synthesis of 5ac:



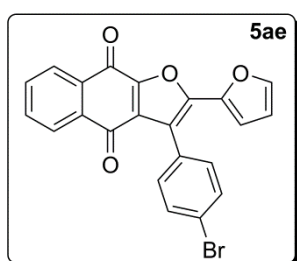
Prepared according to **TP-II** from **3a** (271.7 mg, 0.5 mmol), triethylamine (104.5  $\mu$ L, 1.5 equiv), and **4c** (120.6 mg, 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28  $^{\circ}$ C for 0.5 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 2/1) yielded **5ac** as pale yellow solid (186.5 mg, 79 %). mp.: 333.0-334.0  $^{\circ}$ C;  $R_f$  0.23 (hexanes/ dichloromethane: 2/1).  $^1H$ -NMR (400 MHz,  $CDCl_3$ , 25  $^{\circ}$ C)  $\delta$ /ppm: 8.27 (d, 1H,  $J$  = 7.3 Hz), 8.21 (d, 2H,  $J$  = 8.7 Hz), 8.13 (d, 1H,  $J$  = 7.6 Hz), 7.84-7.73 (m, 4H), 7.66 (d, 2H,  $J$  = 8.4 Hz), 7.34 (d, 2H,  $J$  = 8.4 Hz).  $^{13}C$ -NMR (100 MHz,  $CDCl_3$ , 25  $^{\circ}$ C)  $\delta$ /ppm: 180.3, 173.5, 152.5, 152.1, 148.0, 134.3, 134.2, 134.1, 133.4, 132.4, 132.3, 131.5, 129.1, 128.3, 127.8, 127.1, 126.9, 124.1, 123.8, 123.4. **MS** (70eV, EI)  $m/z$  (%): 475  $[M+2]^+$  (100), 473 (95). **IR** (KBr)  $\tilde{\nu}$  ( $cm^{-1}$ ): 3081 (w), 1676 (s), 1595 (m), 1514 (m), 1338 (s), 1214 (s), 971 (m), 690 (m). **HRMS** (ESI) for  $C_{24}H_{13}BrNO_5^+$ ,  $[M+H]^+$  (473.9972) found: 473.9993.

### Synthesis of 5ad:



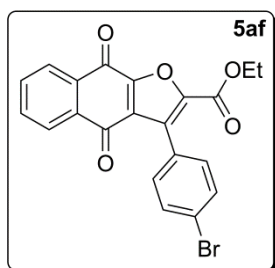
Prepared according to **TP-II** from **3a** (271.7 mg, 0.5 mmol), triethylamine (118.5  $\mu\text{L}$ , 1.7 equiv), and **4d** (101.5  $\mu\text{L}$ , 1.5 equiv) in dry THF (2.5 mL) [reaction condition: 60  $^{\circ}\text{C}$  for 0.5 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 2/1) yielded **5ad** as orange solid (205.8 mg, 90 %). mp.: 247.3-248.3  $^{\circ}\text{C}$ ;  $R_f$  0.28 (hexanes/ dichloromethane: 2/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 8.22 (d, 1H,  $J = 7.6$  Hz), 8.07 (d, 1H,  $J = 7.3$  Hz), 7.79-7.66 (m, 2H), 7.60 (d, 2H,  $J = 8.5$  Hz), 7.50 (d, 2H,  $J = 8.8$  Hz), 7.34 (d, 2H,  $J = 8.4$  Hz), 6.84 (d, 2H,  $J = 8.8$  Hz), 3.81 (s, 3H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 180.9, 173.1, 160.9, 155.9, 150.8, 133.8, 133.7, 133.4, 132.5, 131.9, 131.8, 129.5, 129.4, 128.9, 126.8, 126.6, 122.8, 120.7, 118.9, 114.2, 55.3. **MS** (70eV, EI)  $m/z$  (%): 460  $[\text{M}+2]^+$  (100), 458 (95), 135 (40), 104 (40). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3062 (w), 2919 (w), 1667 (s), 1604 (m), 1252 (s), 1214 (s), 1176 (m), 709 (m). **HRMS** (FAB) for  $\text{C}_{25}\text{H}_{15}\text{BrO}_4$ ,  $[\text{M}+\text{H}]^+$  (459.0232) found: 459.0227.

### Synthesis of 5ae:



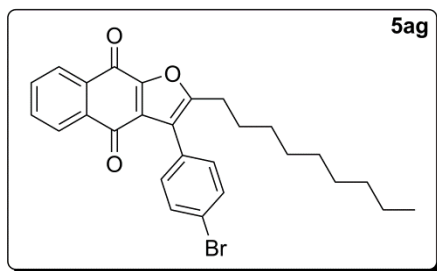
Prepared according to **TP-II** from **3a** (271.7 mg, 0.5 mmol), triethylamine (118.5  $\mu\text{L}$ , 1.7 equiv), and **4e** (73.9  $\mu\text{L}$ , 1.5 equiv) in dry THF (2.5 mL) [reaction condition: 25-28  $^{\circ}\text{C}$  for 1.5 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 2/1) yielded **5ae** as red solid (167.7 mg, 80 %). mp.: 260.1-260.8  $^{\circ}\text{C}$ ;  $R_f$  0.23 (hexanes/dichloromethane: 2/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 8.24 (d, 1H,  $J = 7.1$  Hz), 8.10 (d, 1H,  $J = 7.1$  Hz), 7.82-7.67 (m, 2H), 7.62 (d, 2H,  $J = 8.4$  Hz), 7.46 (s, 1H), 7.41 (d, 2H,  $J = 8.4$  Hz), 6.77 (d, 1H,  $J = 3.6$  Hz), 6.50-6.46 (m, 1H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 180.6, 173.1, 151.1, 147.5, 144.5, 143.8, 133.9, 133.8, 133.5, 132.5, 131.9, 131.4, 129.0, 128.1, 127.0, 126.7, 123.0, 119.6, 112.1, 111.9. **MS** (70eV, EI)  $m/z$  (%): 420  $[\text{M}+2]^+$  (100), 418 (95), 226 (100), 76 (50). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3148 (w), 1667 (s), 1590 (m), 1562 (m), 1238 (m), 1214 (m), 1195 (m), 705 (m). **HRMS** (FAB) for  $\text{C}_{22}\text{H}_{12}\text{BrO}_4^+$ ,  $[\text{M}+\text{H}]^+$  (418.9919) found: 418.9915.

### Synthesis of 5af:



Prepared according to **TP-II** from **3a** (271.7 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **4f** (72.6  $\mu\text{L}$ , 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28  $^{\circ}\text{C}$  for 0.42 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 1/1) yielded **5af** as Olivine (197.3 mg, 93 %). mp.: 243.3-244.3  $^{\circ}\text{C}$ ;  $R_f$  0.30 (hexanes/ dichloromethane: 1/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 8.33-8.21 (m, 1H), 8.19-8.10 (m, 1H), 7.85-7.76 (m, 2H), 7.6111 (d, 2H,  $J = 8.3$  Hz), 7.39 (d, 2H,  $J = 8.1$  Hz), 4.35 (q, 2H,  $J = 7.0$  Hz), 1.30 (t, 3H,  $J = 7.3$  Hz).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 179.8, 173.8, 157.8, 152.6, 144.0, 134.5, 134.2, 133.6, 132.2, 131.7, 131.1, 131.0, 127.7, 127.3, 127.1, 127.0, 123.6, 62.1, 14.0. **MS** (70eV, EI)  $m/z$  (%): 426  $[\text{M}+2]^+$  (100), 424 (95), 187 (40). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3081 (w), 2986 (w), 1719 (s), 1671 (s), 1581 (m), 1238 (m), 1162 (m), 714 (m). **HRMS** (ESI) for  $\text{C}_{21}\text{H}_{14}\text{BrO}_5^+$ ,  $[\text{M}+\text{H}]^+$  (425.0025) found: 425.0024.

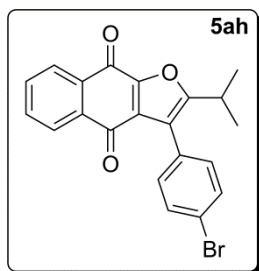
### Synthesis of 5ag:



Prepared according to **TP- II** from **3a** (271.7 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **4g** (134.9  $\mu\text{L}$ , 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28  $^{\circ}\text{C}$  for 1 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 2/1) yielded **5ag** as pale yellow solid (212.6 mg, 89 %). mp.: 93.1-93.8  $^{\circ}\text{C}$ ;  $R_f$  0.33 (hexanes/ dichloromethane: 2/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )

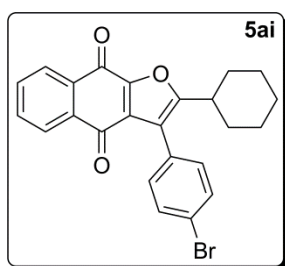
$\delta/\text{ppm}$ : 8.18 (d, 1H,  $J = 7.0$  Hz), 8.06 (d, 1H,  $J = 7.0$  Hz), 7.78-7.65 (m, 2H), 7.59 (d, 2H,  $J = 8.2$  Hz), 7.31 (d, 2H,  $J = 8.0$  Hz), 2.76 (t, 2H,  $J = 7.6$  Hz), 1.80-1.68 (m, 2H), 1.40-1.15 (m, 12H), 0.87 (t, 3H,  $J = 6.7$  Hz).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta/\text{ppm}$ : 180.7, 173.1, 160.9, 151.4, 133.6, 133.4, 132.2, 131.4, 128.8, 128.1, 126.8, 126.5, 122.4, 120.6, 31.8, 29.3, 29.2, 29.1, 29.0, 28.0, 26.4, 22.6, 14.0. **MS** (70eV, EI)  $m/z$  (%): 480  $[\text{M}+2]^+$  (100), 478 (95), 286 (35), 258 (35), 55 (20). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3062 (w), 2909 (m), 2833 (m), 1676 (s), 1538 (m), 1371 (m), 1190 (m), 538 (m). **HRMS** (FAB) for  $\text{C}_{27}\text{H}_{28}\text{BrO}_3^+$ ,  $[\text{M}+\text{H}]^+$  (479.1222) found: 479.1214.

### Synthesis of 5ah:



Prepared according to **TP- II** from **3a** (271.7 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **4h** (68.1  $\mu\text{L}$ , 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 60  $^{\circ}\text{C}$  for 0.5 h]. Purification by *flash*-chromatography (hexanes/ethyl acetate: 20/1) yielded **5ah** as yellow solid (184.3 mg, 93 %). mp.: 179.1-179.8  $^{\circ}\text{C}$ ;  $R_f$  0.3 (hexanes/ethyl acetate: 20/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta/\text{ppm}$ : 8.20 (d, 1H,  $J = 7.3$  Hz), 8.06 (d, 1H,  $J = 7.3$  Hz), 7.78-7.65 (m, 2H), 7.60 (d, 2H,  $J = 8.1$  Hz), 7.30 (d, 2H,  $J = 8.3$  Hz), 3.24-3.10 (m, 1H), 1.36 (d, 6H,  $J = 6.9$  Hz).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta/\text{ppm}$ : 180.9, 173.2, 164.9, 151.3, 133.8, 133.7, 133.4, 132.3, 131.6, 131.5, 128.9, 128.3, 126.7, 126.6, 122.4, 119.1, 26.5, 21.1. **MS** (70eV, EI)  $m/z$  (%): 396  $[\text{M}+2]^+$  (100), 394 (90), 379 (60), 272 (60), 257 (35), 215 (40), 104 (35), 76 (40). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3072 (w), 2967 (m), 2870 (w), 1671 (s), 1590 (m), 1214 (m), 1181 (m), 710 (m). **HRMS** (ESI) for  $\text{C}_{21}\text{H}_{16}\text{BrO}_3^+$ ,  $[\text{M}+\text{H}]^+$  (395.0283) found: 395.0298.

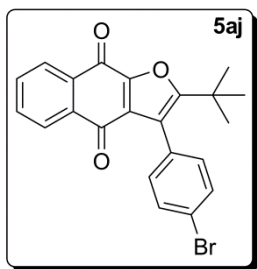
### Synthesis of 5ai:



Prepared according to **TP- II** from **3a** (271.7 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **4i** (87.0  $\mu\text{L}$ , 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28  $^{\circ}\text{C}$  for 0.25 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 2.8/1) yielded **5ai** as yellow solid (193.2 mg, 89 %). mp.: 190.2-191.2  $^{\circ}\text{C}$ ;  $R_f$  0.26 (hexanes/dichloromethane: 2.8/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta/\text{ppm}$ : 8.19 (d, 1H,  $J = 7.9$  Hz), 8.06 (d, 1H,  $J = 7.0$  Hz), 7.77-7.66 (m, 2H), 7.61 (d, 2H,  $J = 8.1$  Hz), 7.30 (d, 2H,  $J = 8.4$  Hz), 2.87-2.72 (m, 1H), 1.94-1.65 (m, 7H), 1.39-1.19 (m, 3H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta/\text{ppm}$ : 180.9, 173.2, 164.5, 151.2, 133.7, 133.6, 133.4, 132.3, 131.6, 131.5, 128.9, 128.2, 126.7, 126.65, 122.4, 119.3, 36.2, 31.2, 25.9, 25.4. **MS** (70eV, EI)  $m/z$  (%): 436  $[\text{M}+2]^+$  (100), 434 (100), 393 (20), 299 (20), 257 (20), 55 (20). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3062 (w), 2918 (m), 2842 (m), 1671 (s), 1595 (m), 1543 (m), 1200 (m), 976 (m), 705 (m). **HRMS** (ESI) for  $\text{C}_{24}\text{H}_{19}\text{BrO}_3$ ,  $[\text{M}+\text{H}]^+$  (434.0518) found: 434.0510.

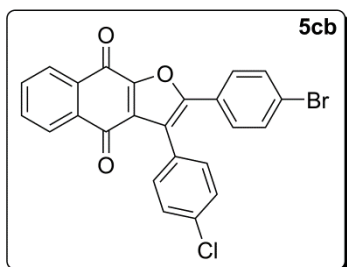


### Synthesis of 5aj:



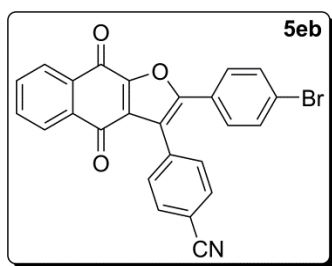
Prepared according to **TP-II** from **3a** (271.7 mg, 0.5 mmol), triethylamine (118.5  $\mu\text{L}$ , 1.7 equiv), and **4j** (92.4  $\mu\text{L}$ , 1.5 equiv) in dry THF (2.5 mL) [reaction condition: 60  $^{\circ}\text{C}$  for 4 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 1.5/1) yielded **5aj** as Olivine solid (187.4 mg, 91%). mp.: 248.7-249.7  $^{\circ}\text{C}$ ;  $R_f$  0.23 (hexanes/dichloromethane: 1.5/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 8.18 (d, 1H,  $J = 7.8$  Hz), 7.99 (d, 1H,  $J = 7.3$  Hz), 7.76-7.63 (m, 2H), 7.57 (d, 2H,  $J = 8.3$  Hz), 7.21 (d, 2H,  $J = 8.3$  Hz), 1.29 (s, 9H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 180.8, 173.2, 166.1, 149.9, 133.7, 133.6, 133.3, 132.4, 131.8, 131.2, 130.6, 129.7, 126.6, 126.5, 122.4, 118.9, 35.2, 29.7. **MS** (70eV, EI)  $m/z$  (%): 410  $[\text{M}+2]^+$  (60), 408 (60), 395 (100), 393 (70), 286 (30), 271 (20), 76 (20). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3062 (w), 2957 (m), 1671 (s), 1590 (m), 1533 (m), 1233 (m), 1210 (m), 705 (m). **HRMS** (ESI) for  $\text{C}_{22}\text{H}_{18}\text{BrO}_3^+$ ,  $[\text{M}+\text{H}]^+$  (409.0439) found: 409.0453.

### Synthesis of 5cb:



Prepared according to **TP-II** from **3c** (249.5 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **4b** (142.7 mg, 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28  $^{\circ}\text{C}$  for 0.5 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 2.6/1) yielded **5cb** as yellow solid (204.6 mg, 88 %). mp.: 256.5-257.3  $^{\circ}\text{C}$ ;  $R_f$  0.28 (hexanes/ethyl acetate: 2.6/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 8.23 (d, 1H,  $J = 6.7$  Hz), 8.09 (d, 1H,  $J = 6.9$  Hz), 7.79-7.70 (m, 2H), 7.50-7.36 (m, 8H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 180.6, 173.3, 154.4, 151.4, 134.9, 133.9, 133.4, 132.3, 131.4, 129.3, 128.6, 128.3, 127.1, 126.9, 126.7, 124.5, 120.9. **MS** (70 eV, EI)  $m/z$  (%): 462  $[\text{M}]^+$  (90), 464 (100). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3043 (w), 1671 (s), 1590 (m), 1395 (m), 1214 (m), 1080 (m), 709 (m), 676 (m). **HRMS** (ESI) for  $\text{C}_{24}\text{H}_{13}\text{BrClO}_3$ ,  $[\text{M}+\text{H}]^+$  (462.9731) found: 462.9757.

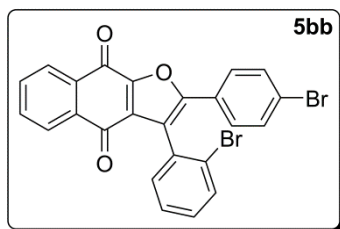
### Synthesis of 5eb:



Prepared according to **TP-II** from **3e** (244.8 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **4b** (142.7 mg, 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28  $^{\circ}\text{C}$  for 0.5 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 1/1) yielded **5eb** as yellow solid (221.1 mg, 97 %). mp.: 309.4-310.4  $^{\circ}\text{C}$ ;  $R_f$  0.3 (hexanes/ethyl acetate: 8/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 8.25 (d, 1H,  $J = 7.5$  Hz), 8.10 (d, 1H,  $J = 7.4$  Hz), 7.82-7.72 (m, 4H), 7.59 (d, 2H,  $J = 8.2$  Hz), 7.49 (d, 2H,  $J = 8.8$  Hz), 7.38 (d, 2H,  $J = 8.4$  Hz).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 180.5, 173.3, 154.7, 151.6, 135.0, 134.2, 134.1, 133.3, 132.5, 131.0, 129.0, 128.7, 127.0, 126.8, 126.7, 125.0, 120.1, 118.4, 112.7. **MS** (70eV, EI)  $m/z$  (%): 455  $[\text{M}+2]^+$  (100), 453 (100), 368 (30), 288 (50), 212 (30), 187 (20), 105 (40) 76 (60). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3062 (w), 2230 (w), 1671 (s), 1590 (m), 1219 (m), 1186 (m), 705 (m). **HRMS** (ESI) for  $\text{C}_{25}\text{H}_{13}\text{BrNO}_3^+$ ,  $[\text{M}+\text{H}]^+$  (454.0079) found: 454.0096.

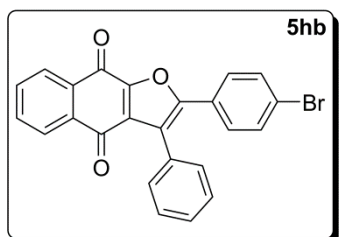
### Synthesis of 5bb:

Prepared according to **TP-II** from **3b** (271.7 mg, 0.5 mmol), triethylamine (118.5  $\mu\text{L}$ , 1.7 equiv), and **4b** (164.7 mg, 1.5 equiv) in dry THF (2.5 mL) [reaction condition: 60  $^{\circ}\text{C}$  for 12 h]. Purification by



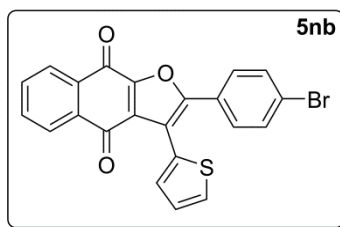
*flash*-chromatography (hexanes/dichloromethane: 2/1) yielded **5bb** as yellow solid (200.4 mg, 79%). mp.: 235.4-236.4 °C;  $R_f$  0.26 (hexanes/dichloromethane: 2/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta$ /ppm: 8.26 (d, 1H,  $J = 7.5$  Hz), 8.09 (d, 1H,  $J = 7.2$  Hz), 7.81-7.70 (m, 3H), 7.50-7.33 (m, 7H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta$ /ppm: 180.3, 173.4, 154.5, 151.0, 138.9, 133.8, 133.3, 133.2, 132.6, 132.1, 131.8, 131.3, 130.1, 128.0, 127.9, 127.4, 126.9, 126.8, 124.6, 124.5, 120.6 **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3071 (w), 1660 (s), 1598 (s), 1528 (s), 1454 (m), 1347 (m). **HRMS** (70 eV, EI) for  $\text{C}_{24}\text{H}_{12}\text{Br}_2\text{O}_3$ ,  $[\text{M}]^+$  (505.9153), found: 505.9159.

### Synthesis of **5hb**:



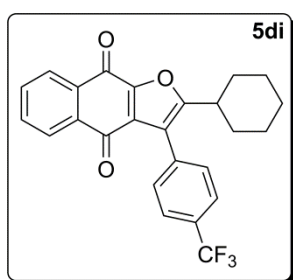
Prepared according to **TP-II** from **3h** (271.7 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **4b** (142.7 mg, 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28 °C for 1 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 1/1) yielded **5hb** as yellow solid (177.1 mg, 83 %). mp.: 259.7-260.2 °C;  $R_f$  0.30 (hexanes/ethyl acetate: 1/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta$ /ppm: 8.24 (d, 1H,  $J = 7.1$  Hz), 8.09 (d, 1H,  $J = 7.2$  Hz), 7.82-7.66 (m, 2H), 7.55-7.38 (m, 9H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta$ /ppm: 180.6, 173.4, 154.3, 151.3, 133.9, 133.8, 133.5, 132.4, 131.9, 130.0, 129.9, 129.6, 128.8, 128.6, 127.5, 126.9, 126.7, 124.2, 122.2. **MS** (70eV, EI)  $m/z$  (%): 430  $[\text{M}+2]^+$  (100), 428 (100), 345 (20), 263 (40), 187 (20), 105 (30) 77 (20). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3062 (w), 1671 (s), 1586 (m), 1214 (m), 1076 (m), 710 (m). **HRMS** (ESI) for  $\text{C}_{24}\text{H}_{14}\text{BrO}_3^+$ ,  $[\text{M}+\text{H}]^+$  (431.0106) found: 431.0103.

### Synthesis of **5nb**:



Prepared according to **TP-II** from **3n** (235.3 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **4b** (142.7 mg, 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28 °C for 0.5 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 3/1) yielded **5nb** as yellow solid (198.1 mg, 91 %). mp.: 224.3-225.3 °C;  $R_f$  0.24 (hexanes/ dichloromethane: 3/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta$ /ppm: 8.24 (d, 1H,  $J = 7.7$  Hz), 8.13 (d, 1H,  $J = 6.9$  Hz), 7.80-7.71 (m, 2H), 7.55-7.47 (m, 5H), 7.25-7.21 (m, 1H), 7.20-7.16 (m, 1H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta$ /ppm: 180.2, 173.3, 155.4, 151.2, 133.9, 133.8, 133.4, 132.3, 131.9, 129.7, 129.3, 128.7, 127.7, 127.6, 127.1, 126.9, 126.6, 124.6, 114.9. **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3100 (w), 1671 (s), 1585 (m), 1552 (m), 1209 (s), 1071 (m), 952 (m), 704 (m). **HRMS** (70 eV, EI) for  $\text{C}_{22}\text{H}_{11}\text{BrO}_3\text{S}$ ,  $[\text{M}]^+$  (433.9612), found: 433.9611.

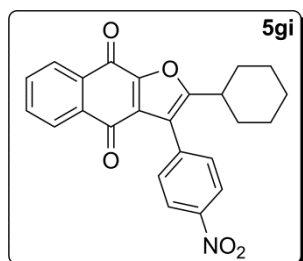
### Synthesis of **5di**:



Prepared according to **TP-II** from **3d** (266.3 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **4i** (86.9 mg, 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28 °C for 0.5 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 3.5/1) yielded **5di** as yellow solid (192.4 mg, 91%). mp.: 198.4-199.4 °C;  $R_f$  0.41 (hexanes/ dichloromethane: 2/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta$ /ppm: 8.21 (d, 1H,  $J = 7.2$  Hz), 8.07 (d, 1H,  $J = 7.4$  Hz), 7.78-7.68 (m, 4H), 7.55 (d, 2H,  $J = 8.0$  Hz), 2.87-2.72 (m, 1H), 1.93-1.70 (m, 7H),

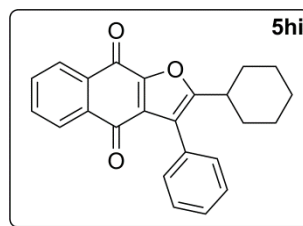
1.39-1.20 (m, 3H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 180.9, 173.3, 164.7, 151.4, 133.9, 133.8, 133.7, 133.4, 132.3, 130.3, 130.2 (q,  $J = 32.6$  Hz), 128.2, 126.8, 126.6, 125.2 (q,  $J = 3.7$  Hz), 124.1 (q,  $J = 272.0$  Hz), 119.1, 36.3, 31.3, 25.9, 25.4.  $^{31}\text{P-NMR}$  (200 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 34.7. **MS** (70 eV, EI)  $m/z$  (%): 424  $[\text{M}]^+$  (100). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3062 (w), 2928 (m), 2851 (m), 1671 (s), 1590 (m), 1328 (s), 1209 (s), 1119 (s), 842 (m), 704 (m). **HRMS** (ESI) for  $\text{C}_{25}\text{H}_{20}\text{F}_3\text{O}_3$ ,  $[\text{M}+\text{H}]^+$  (425.1365), found: 425.1381.

#### Synthesis of 5gi:



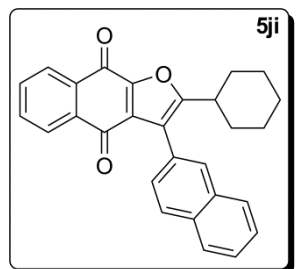
Prepared according to **TP-II** from **3g** (254.8 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **4i** (86.9 mg, 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28 °C for 1.25 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 2/1) yielded **5gi** as yellow solid (194.1 mg, 97%). mp.: 181.4-182.4 °C;  $R_f$  0.21 (hexanes/ dichloromethane: 2/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 8.34 (d, 2H,  $J = 8.5$  Hz), 8.21 (d, 1H,  $J = 7.1$  Hz), 8.07 (d, 1H,  $J = 8.5$  Hz), 7.78-7.69 (m, 2H), 7.61 (d, 2H,  $J = 8.5$  Hz), 2.85-2.74 (m, 1H), 1.92-1.71 (m, 7H), 1.37-1.22 (m, 3H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 180.8, 173.2, 164.8, 151.5, 147.6, 137.0, 133.9, 133.8, 133.3, 132.2, 130.9, 127.9, 126.7, 123.5, 118.5, 36.4, 31.2, 25.9, 25.3. **MS** (70 eV, EI)  $m/z$  (%): 401  $[\text{M}]^+$  (100). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3072 (w), 2927 (w), s847 (w), 1667 (m), 1591 (w), 1519 (m), 1346 (m), 1208 (m), 706 (w). **HRMS** (ESI) for  $\text{C}_{24}\text{H}_{20}\text{NO}_5$ ,  $[\text{M}+\text{H}]^+$  (402.1341), found: 402.1325.

#### Synthesis of 5hi:



Prepared according to **TP-II** from **3h** (232.3 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **4i** (86.9 mg, 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28 °C for 0.66 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 4/1) yielded **5hi** as yellow solid (175.1 mg, 98%). mp.: 170.2-171.2 °C;  $R_f$  0.28 (hexanes/ dichloromethane: 4/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25°C)  $\delta/\text{ppm}$ : 8.19 (d, 1H,  $J = 6.6$  Hz), 8.06 (t, 1H,  $J = 6.9$  Hz), 7.75-7.64 (m, 2H), 7.53-7.40 (m, 5H), 2.90-2.79 (m, 1H), 1.93-1.68 (m, 7H), 1.38-1.21 (m, 3H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 180.9, 173.2, 164.5, 151.1, 133.6, 133.5, 132.3, 130.0, 129.9, 128.5, 128.2, 128.1, 126.7, 126.4, 120.4, 36.1, 31.2, 25.9, 25.4. **MS** (70 eV, EI)  $m/z$  (%): 356  $[\text{M}]^+$  (100). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3052 (w), 2928 (s), 2851 (m), 1666 (s), 1590 (s), 1209 (s), 704 (s). **HRMS** (ESI) for  $\text{C}_{24}\text{H}_{21}\text{O}_3$ ,  $[\text{M}+\text{H}]^+$  (357.1491), found: 357.1492.

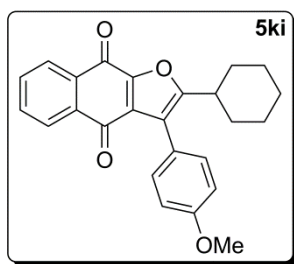
#### Synthesis of 5ji:



Prepared according to **TP-II** from **3j** (257.3 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **4i** (86.9 mg, 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28 °C for 1.5 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 2/1) yielded **5ji** as yellow solid (191.5 mg, 94%). mp.: 192.6-193.6 °C;  $R_f$  0.33 (hexanes/ dichloromethane: 2/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 8.21 (d, 1H,  $J = 7.0$  Hz), 8.06 (d, 1H,  $J = 7.1$  Hz), 7.98-7.83 (m, 4H), 7.74-7.64 (m, 2H), 7.58-7.48 (m, 3H), 2.96-2.84 (m, 1H), 1.95-1.75 (m, 7H), 1.35-1.19 (m, 3H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 180.9, 173.3, 164.7, 151.2, 133.5, 133.1, 133.0, 132.4, 128.8, 128.7, 128.1, 127.8, 127.7, 127.6, 126.8, 126.5, 126.4, 126.3, 120.5, 36.2, 31.3, 25.9, 25.4. **MS** (70 eV, EI)  $m/z$  (%): 401  $[\text{M}]^+$  (100). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3052 (w), 2981 (m), 2842 (m), 1666 (s), 1580 (m), 1538 (m),

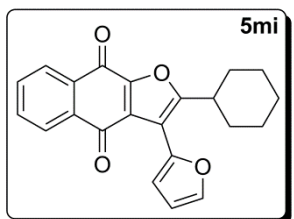
1204 (m), 747 (m). HRMS (ESI) for  $C_{28}H_{23}O_3$ ,  $[M+H]^+$  (407.1647), found: 407.1623.

### Synthesis of 5ki:



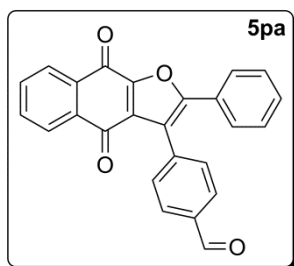
Prepared according to **TP-II** from **3k** (248.3 mg, 0.5 mmol), triethylamine (104.5  $\mu$ L, 1.5 equiv), and **4i** (86.9 mg, 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28  $^{\circ}$ C for 1 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 1.8/1) yielded **5ki** as yellow solid (143.5 mg, 74%). mp.: 174.3-175.0  $^{\circ}$ C;  $R_f$  0.28 (hexanes/ dichloromethane: 1.8/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}$ C)  $\delta$ /ppm: 8.19 (d, 1H,  $J = 7.2$  Hz), 8.07 (d, 1H,  $J = 7.2$  Hz), 7.74-7.65 (m, 4H), 7.35 (d, 2H,  $J = 8.6$  Hz), 7.01 (d, 2H,  $J = 8.6$  Hz), 3.88 (s, 1H), 2.89-2.78 (m, 1H), 1.90-1.74 (m, 7H), 1.38-1.22 (m, 3H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}$ C)  $\delta$ /ppm: 181.0, 173.2, 164.4, 159.4, 133.6, 133.5, 132.4, 131.0, 128.6, 126.7, 126.4, 122.0, 120.2, 113.7, 55.2, 36.2, 31.2, 26.0, 25.5. **MS** (70 eV, EI)  $m/z$  (%): 386  $[M]^+$  (100). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3062 (w), 2918 (m), 2842 (w), 1666 (s), 1585 (w), 1247 (m), 1204 (s), 709 (m). HRMS (ESI) for  $C_{25}H_{23}O_4$ ,  $[M+H]^+$  (387.1596), found: 387.1602.

### Synthesis of 5mi:



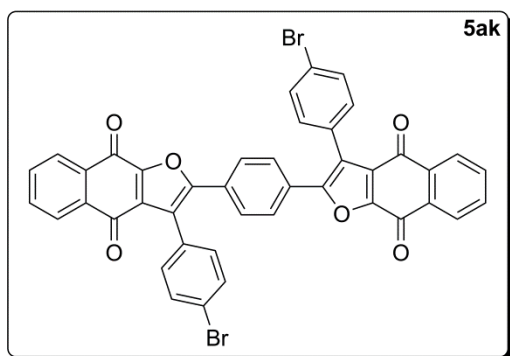
Prepared according to **TP-II** from **3m** (227.3 mg, 0.5 mmol), triethylamine (104.5  $\mu$ L, 1.5 equiv), and **4i** (86.9 mg, 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28  $^{\circ}$ C for 1.25 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 3/1) yielded **5mi** as orange solid (157.6 mg, 91%). mp.: 155.8-156.5  $^{\circ}$ C;  $R_f$  0.23 (hexanes/ dichloromethane: 3/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}$ C)  $\delta$ /ppm: 8.25-8.10 (m, 2H), 7.76-7.66 (m, 1H), 7.54 (brs, 1H), 7.36 (d, 2H,  $J = 3.2$  Hz), 6.55 (brs, 2H), 3.47-3.33 (m, 1H), 2.02-1.69 (m, 7H), 1.51-1.19 (m, 3H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}$ C)  $\delta$ /ppm: 180.4, 173.0, 164.7, 151.3, 144.7, 142.2, 133.6, 133.5, 132.0, 127.0, 126.9, 126.4, 112.1, 111.4, 110.9, 37.6, 30.9, 26.1, 25.6. **MS** (70 eV, EI)  $m/z$  (%): 346  $[M]^+$  (100), 303 (30), 105 (30), 55 (70). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3139 (w), 2928 (m), 2842 (m), 1667 (s), 1590 (m), 1224 (m), 1210 (m), 705 (m). HRMS (ESI) for  $C_{22}H_{19}O_4^+$ ,  $[M+H]^+$  (347.1283), found: 347.1283.

### Synthesis of 5pa:



Prepared according to **TP-II** from **3p** (246.3 mg, 0.5 mmol), triethylamine (118.5  $\mu$ L, 1.7 equiv), and **4a** (87.5  $\mu$ L, 1.5 equiv) in dry THF (2.5 mL) [reaction condition: 25-28  $^{\circ}$ C for 0.5 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 1/1.5) yielded **5pa** as yellow solid (181.3 mg, 96 %). mp.: 242.4-243.4  $^{\circ}$ C;  $R_f$  0.24 (hexanes/dichloromethane : 1.5/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}$ C)  $\delta$ /ppm: 10.11 (s, 1H), 8.23 (d, 1H,  $J = 7.2$  Hz), 8.08 (d, 1H,  $J = 7.1$  Hz), 7.99 (d, 2H,  $J = 8.0$  Hz), 7.81-7.69 (m, 2H), 7.66 (d, 2H,  $J = 7.9$  Hz), 7.53 (d, 2H,  $J = 7.4$  Hz), 7.41-7.28 (m, 3H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}$ C)  $\delta$ /ppm: 191.7, 180.6, 173.3, 155.8, 151.4, 136.8, 136.2, 134.0, 133.9, 133.4, 132.3, 130.9, 130.2, 129.9, 129.2, 128.8, 128.0, 127.4, 126.9, 126.7, 120.3. **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3057 (w), 2746 (w), 1693 (s), 1668 (s), 1588 (m), 1220 (s), 685 (m). HRMS (EI) for  $C_{25}H_{14}O_4$ ,  $[M]^+$  (378.0892) found: 378.0902.

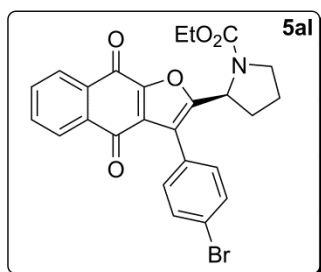
### Synthesis of 5ak:



Prepared according to **TP-II** from **3a** (597.8 mg, 2.2 eq), triethylamine (174.2  $\mu\text{L}$ , 2.5 equiv), and **4k** (101.6 mg, 0.5 equiv) in dry THF (2.5 mL) [reaction condition: 25-28  $^{\circ}\text{C}$  for 0.66 h]. Purification by *flash*-chromatography (hexanes/ dichloromethane: 1.6/1) yielded **5ak** as red solid (230.2 mg, 59 %). mp.: over 400 $^{\circ}\text{C}$ ;  $R_f$  0.26 (hexanes/ dichloromethane: 1.6/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 8.25 (d, 2H,  $J = 7.5$  Hz), 8.10 (d, 2H,  $J = 7.1$  Hz), 7.81-7.71 (m, 4H), 7.62 (d, 4H,  $J = 8.2$  Hz), 7.56 (*pseudo* s, 4H), 7.34 (d, 4H,  $J = 8.2$  Hz).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25

$^{\circ}\text{C}$ )  $\delta$ /ppm: 180.6, 173.4, 154.2, 151.6, 134.1, 134.0, 133.5, 132.4, 132.1, 131.6, 129.5, 129.3, 128.8, 127.4, 127.0, 123.3, 121.7. **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3062 (w), 1671 (s), 1595 (m), 1543 (m), 1214 (s), 971 (m), 704 (m). **HRMS** (EI) for  $\text{C}_{42}\text{H}_{20}\text{Br}_2\text{O}_6$ ,  $[\text{M}]^+$  (777.9627) found: 777.9622.

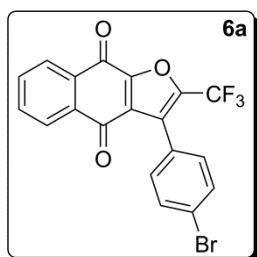
### Synthesis of 5al:



Prepared according to **TP-II** from **3a** (271.7 mg, 0.5 mmol), triethylamine (120.6  $\mu\text{L}$ , 1.7 equiv), and **4l** (1.5 equiv) in dry THF (2.5 mL) [reaction condition: 25-28  $^{\circ}\text{C}$  for 0.83 h]. Purification by *flash*-chromatography (hexanes/ethyl acetate: 8/1) yielded **5al** as Olivine solid (173.2 mg, 70 %). mp.: 163.3-163.8  $^{\circ}\text{C}$ ;  $R_f$  0.23 (hexanes/ethyl acetate: 6/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 8.26-8.15 (m, 1H), 8.13-8.03 (m, 1H), 7.80-7.67 (m, 2H),

7.60 (d, 2H,  $J = 7.0$  Hz), 7.56-7.30 (m, 2H), 5.07-4.94 (m, 1H), 4.17-3.90 (m, 2H), 3.72-3.46 (m, 2H), 2.39-1.85 (m, 4H), 1.34-0.92 (m, 3H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 180.7, 173.4, 173.2, 159.4, 159.1, 151.9, 154.3, 151.5, 133.8, 133.7, 133.4, 132.2, 131.7, 131.4, 128.3, 128.2, 127.9, 126.8, 126.6, 122.6, 121.1, 120.8, 61.3, 53.4, 52.7, 47.1, 46.8, 33.1, 32.3, 24.5, 23.9, 14.6. **MS** (70eV, EI)  $m/z$  (%): 495  $[\text{M}+2]^+$  (100), 493 (100), 419 (100), 351 (20), 311 (20), 105 (20). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3053 (w), 2967 (m), 2880 (w), 1705 (s), 1667 (s), 1586 (m), 1205 (m), 1119 (s), 714 (m). **HRMS** (EI) for  $\text{C}_{25}\text{H}_{20}\text{BrNO}_5$ ,  $[\text{M}]^+$  (493.0525) found: 493.0533.

### Synthesis of 6a:

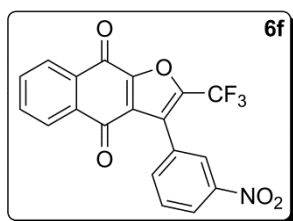


Prepared according to **TP-II** from **3a** (271.7 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **TFAA** (90.4  $\mu\text{L}$ , 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28  $^{\circ}\text{C}$  for 10 min]. Purification by *flash*-chromatography (hexanes/ dichloromethane: 2.2/1) yielded **6a** as Olivine solid (198.0 mg, 94 %). mp.: 142.3-142.9  $^{\circ}\text{C}$ ;  $R_f$  0.30 (hexanes/ dichloromethane: 2.2/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 8.31-8.19 (m, 1H), 8.18-8.08 (m, 1H), 7.87-7.74 (m, 2H), 7.63

(d, 2H,  $J = 8.3$  Hz), 7.35 (d, 2H,  $J = 8.5$  Hz).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta$ /ppm: 179.4, 173.4, 152.5, 142.2 (quart,  $J = 41.4$  Hz), 134.6, 134.3, 133.3, 131.9, 131.7, 131.5, 131.3, 127.3, 127.2, 127.0, 126.7 (quart,  $J = 2.3$  Hz), 125.3, 124.1, 118.6 (quart,  $J = 271.0$  Hz). **MS** (70eV, EI)  $m/z$  (%): 422  $[\text{M}+2]^+$  (100), 420 (95), 341 (40), 187 (20). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3052 (w), 1676 (s), 1580 (m), 1243 (s), 1200 (s), 1138 (s), 724 (m), 710 (m). **HRMS** (ESI) for  $\text{C}_{19}\text{H}_9\text{BrF}_3\text{O}_3^+$ ,  $[\text{M}]^+$  (420.9699) found: 420.9687.

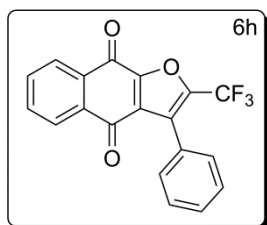


### Synthesis of 6f:



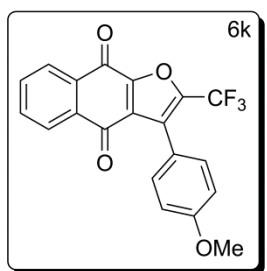
Prepared according to **TP-II** from **3f** (254.8 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **TFAA** (90.4  $\mu\text{L}$ , 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28  $^{\circ}\text{C}$  for 5 min]. Purification by *flash*-chromatography (hexanes/dichloromethane: 3/1) yielded **6f** as Olivine solid (188.1 mg, 97 %). mp.: 180.7-181.4  $^{\circ}\text{C}$ ;  $R_f$  0.31 (hexanes/ dichloromethane: 3/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta/\text{ppm}$ : 8.43-8.35 (m, 2H), 8.31-8.25 (m, 1H), 8.17-8.10 (m, 1H), 7.87-7.78 (m, 3H), 7.75-7.66 (m, 1H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta/\text{ppm}$ : 179.4, 173.3, 152.7, 148.1, 142.8 (quart,  $J = 41.8$  Hz), 135.6, 134.8, 134.5, 133.2, 131.9, 129.3, 128.3, 127.4, 127.2, 125.3 (quart,  $J = 2.2$  Hz), 125.0, 124.4, 118.5 (quart,  $J = 271.1$  Hz). **MS** (70eV, EI)  $m/z$  (%): 387 [ $\text{M}]^+$  (80), 340 (100), 215 (30), 187 (40), 76 (30). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3079 (w), 1678 (s), 1606 (m), 1527 (s), 1350 (s), 1245 (s), 1187 (s), 1126 (s), 710 (m). **HRMS** (EI) for  $\text{C}_{19}\text{H}_8\text{F}_3\text{NO}_5$ , [ $\text{M}]^+$  (387.0355) found: 387.0360.

### Synthesis of 6h:



Prepared according to **TP-II** from **3h** (254.8 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **TFAA** (90.4  $\mu\text{L}$ , 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28  $^{\circ}\text{C}$  for 15 min]. Purification by *flash*-chromatography (hexanes/dichloromethane: 3/1) yielded **6h** as Olivine solid (145.5 mg, 85 %). mp.: 108.3-109.3  $^{\circ}\text{C}$ ;  $R_f$  0.30 (hexanes/dichloromethane: 3/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta/\text{ppm}$ : 8.29-8.24 (m, 1H), 8.16-8.10 (m, 1H), 7.83-7.76 (m, 2H), 7.54-7.44 (m, 5H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta/\text{ppm}$ : 179.5, 173.6, 152.4, 142.2 (quart,  $J = 41.3$  Hz), 134.6, 134.2, 133.4, 131.9, 129.6, 129.5, 128.2, 127.8 (quart,  $J = 2.3$  Hz), 127.5, 127.3, 127.0, 126.4, 118.7 (quart,  $J = 270.9$  Hz). **MS** (70 eV, EI)  $m/z$  (%): 342 [ $\text{M}]^+$  (100). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3065 (w), 1678 (s), 1588 (m), 1241 (s), 1179 (s), 1129 (s), 710 (m), 684 (m). **HRMS** (ESI) for  $\text{C}_{19}\text{H}_{10}\text{F}_3\text{O}_3$ , [ $\text{M}+\text{H}]^+$  (343.0582), found: 343.0553.

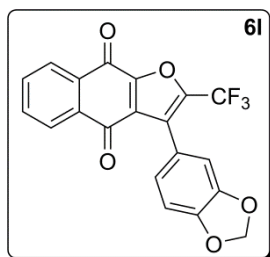
### Synthesis of 6k:



Prepared according to **TP-II** from **3k** (248.3 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **TFAA** (90.4  $\mu\text{L}$ , 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28  $^{\circ}\text{C}$  for 20 min]. Purification by *flash*-chromatography (hexanes/dichloromethane: 2.5/1) yielded **6k** as yellow solid (174.4 mg, 93 %). mp.: 147.7-148.5  $^{\circ}\text{C}$ ;  $R_f$  0.26 (hexanes/dichloromethane: 2.5/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta/\text{ppm}$ : 8.31-8.22 (m, 1H), 8.19-8.10 (m, 1H), 7.84-7.75 (m, 2H), 7.42 (d, 2H,  $J = 7.5$  Hz), 7.04-6.98 (m, 2H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta/\text{ppm}$ : 179.6, 173.6, 160.5, 152.3, 141.8 (q,  $J = 41.3$  Hz), 134.5, 134.1, 133.4, 131.8, 131.1, 127.7 (q,  $J = 2.6$  Hz), 127.4, 127.2, 126.9, 118.8 (q,  $J = 270.7$  Hz), 118.2, 113.6, 55.2. **MS** (70 eV, EI)  $m/z$  (%): 372 [ $\text{M}]^+$  (100). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3079 (w), 2971 (m), 1678 (s), 1613 (s), 1588 (s), 1528 (s), 1252 (s), 1194 (s), 1122 (s), 710 (m), 692 (m). **HRMS** (ESI) for  $\text{C}_{20}\text{H}_{12}\text{F}_3\text{O}_4$ , [ $\text{M}+\text{H}]^+$  (373.0688), found: 373.0672.

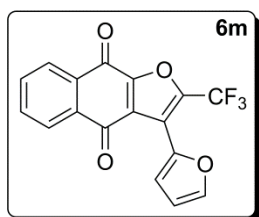
### Synthesis of 6l:

Prepared according to **TP-II** from **3l** (254.8 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **TFAA** (90.4  $\mu\text{L}$ , 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28  $^{\circ}\text{C}$  for 10 min]. Purification by



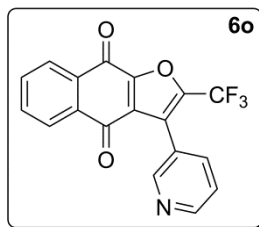
*flash*-chromatography (hexanes/dichloromethane: 2/1) yielded **6l** as orange solid (181.5 mg, 94 %). mp.: 144.3-145.3 °C;  $R_f$  0.23 (hexanes/ dichloromethane: 2/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta$ /ppm: 8.30-8.19 (m, 1H), 8.18-8.08 (m, 1H), 7.86-7.74 (m, 2H), 7.00-6.83 (m, 3H), 6.06 (*pseudo s*, 2H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta$ /ppm: 179.5, 173.5, 152.3, 148.7, 147.5, 142.1 (quart,  $J = 42.1$  Hz), 134.6, 134.2, 133.4, 131.8, 127.6 (quart,  $J = 2.3$  Hz), 127.5, 127.3, 126.9, 123.8, 119.5, 118.7 (quart,  $J = 270.7$  Hz), 110.1, 108.2, 101.5. **MS** (70eV, EI)  $m/z$  (%): 386  $[\text{M}]^+$  (60), 203 (20), 175 (20), 76 (10). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3072 (w), 2913 (w), 1689 (s), 1595 (m), 1230 (m), 1205 (m), 1143 (m), 1097 (m), 710 (m). **HRMS** (FAB) for  $\text{C}_{20}\text{H}_9\text{F}_3\text{O}_5$ ,  $[\text{M}]^+$  (386.0402) found: 386.0400.

### Synthesis of **6m**:



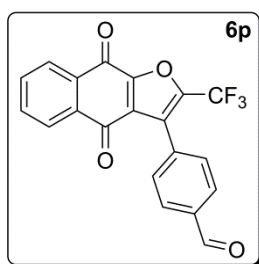
Prepared according to **TP-II** from **3m** (227.3 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **TFAA** (90.4  $\mu\text{L}$ , 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28 °C for 5 min]. Purification by *flash*-chromatography (hexanes/dichloromethane: 3.5/1) yielded **6m** as yellow solid (151.0 mg, 91 %). mp.: 156.5-157.5 °C;  $R_f$  0.38 (hexanes/ dichloromethane: 3.5/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta$ /ppm: 8.29-8.23 (m, 2H), 7.86-7.84 (m, 2H), 7.66-7.61 (m, 2H), 6.62-6.59 (m, 1H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta$ /ppm: 179.1, 173.3, 152.3, 144.4, 141.3 (q,  $J = 43.2$  Hz), 140.8, 134.6, 134.1, 133.5, 131.6, 127.5, 126.9, 125.9, 118.9 (q,  $J = 270.5$  Hz), 117.5, 115.8, 111.8. **MS** (70 eV, EI)  $m/z$  (%): 332  $[\text{M}]^+$  (100). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3152 (w), 3065 (w), 1678 (s), 1591 (m), 1248 (s), 1172 (m), 1136 (s), 1136 (s), 760 (m), 706 (m). **HRMS** (ESI) for  $\text{C}_{17}\text{H}_8\text{F}_3\text{O}_4$ ,  $[\text{M}+\text{H}]^+$  (333.0375), found: 333.0385

### Synthesis of **6o**:



Prepared according to **TP-II** from **3o** (232.8 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **TFAA** (90.4  $\mu\text{L}$ , 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28 °C for 5 min]. Purification by *flash*-chromatography (hexanes/ethyl acetate: 3/1) yielded **6o** as yellow solid (152.7 mg, 89 %). mp.: 121.1-122.1 °C;  $R_f$  0.3 (hexanes/ethyl acetate : 3/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta$ /ppm: 8.81-8.66 (m, 2H), 8.29-8.21 (m, 1H), 8.17-8.09 (m, 1H), 7.90-7.76 (m, 3H), 7.52-7.41 (m, 1H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta$ /ppm: 179.4, 173.3, 152.6, 150.5, 150.0, 142.6 (quart,  $J = 41.6$  Hz), 137.2, 134.7, 134.4, 133.2, 131.8, 127.2, 127.0, 124.3 (quart,  $J = 2.2$  Hz), 122.9, 122.8, 118.5 (quart,  $J = 271.1$  Hz). **MS** (70eV, EI)  $m/z$  (%): 343  $[\text{M}]^+$  (100), 189 (20), 163 (20), 110 (20), 76 (30). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3094 (w), 1686 (s), 1584 (m), 1583 (m), 1245 (s), 1209 (s), 1122 (s), 714 (m). **HRMS** (EI) for  $\text{C}_{18}\text{H}_8\text{F}_3\text{NO}_3$ ,  $[\text{M}]^+$  (343.0456) found: 343.0455.

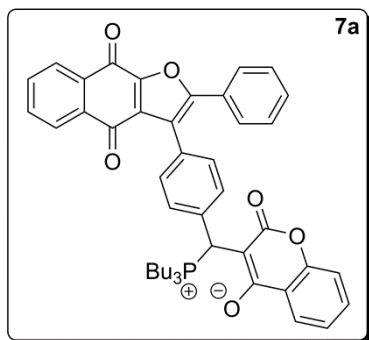
### Synthesis of **6p**:



Prepared according to **TP-II** from **3p** (246.3 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **TFAA** (90.4  $\mu\text{L}$ , 1.3 equiv) in dry THF (2.5 mL) [reaction condition: 25-28 °C for 5 min]. Purification by *flash*-chromatography (hexanes/dichloromethane: 2/1) yielded **6p** as Olivine solid (168.5 mg, 91 %). mp.: 136.4-137.4 °C;  $R_f$  0.28 (hexanes/ dichloromethane: 2/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta$ /ppm: 10.1 (s, 1H), 8.29-8.21 (m, 1H), 8.15-8.08 (m, 1H), 8.02 (d,

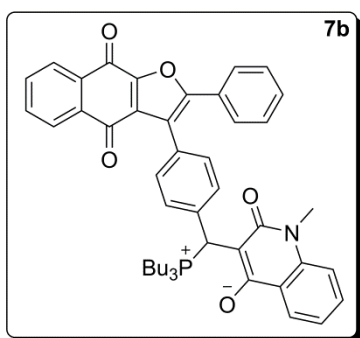
2H,  $J = 8.0$  Hz), 7.85-7.77 (m, 2H), 7.67 (d, 2H,  $J = 7.2$  Hz).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 191.5, 179.3, 173.3, 152.5, 142.3 (q,  $J = 41.7$  Hz), 136.8, 134.7, 134.4, 133.1, 132.4, 131.7, 130.5, 129.3, 127.2, 127.1, 126.4 (q,  $J = 1.7$  Hz), 118.5 (q,  $J = 271.1$  Hz). **MS** (70 eV, EI)  $m/z$  (%): 370  $[\text{M}]^+$  (100), 340 (80). **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3065 (w), 2731 (w), 1700 (s), 1682 (s), 1617 (m), 1595 (m), 1241 (s), 1205 (s), 1125 (s), 706 (m). **HRMS** (FAB) for  $\text{C}_{20}\text{H}_{10}\text{F}_3\text{O}_4$ ,  $[\text{M}+\text{H}]^+$  (371.0531), found: 371.0538.

### Synthesis of 7a:



Prepared according to **TP-III** from 4-hydroxycoumarin (81.1 mg, 0.5 mmol), **5pa** (189.2 mg, 1 equiv), tributylphosphine (150.0  $\mu\text{L}$ , 1.2 equiv), pyrrolidine (4.1  $\mu\text{L}$ , 0.1 equiv), and benzoic acid (6.1 mg, 0.1 equiv) in dry THF (0.5 mL) [reaction condition: 28-30 °C for 1 h]. Purification by *flash*-chromatography (hexanes/ethyl acetate: 3/1 and then dichloromethane/methanol: 100/1.2) yielded **7a** as orange solid (263.4 mg, 73 %). mp.: 238.4-239.2 °C;  $R_f$  0.37 (dichloromethane/methane: 30/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 8.24 (d, 1H,  $J = 7.3$  Hz), 8.06 (*pseudo t*, 2H,  $J = 8.7$  Hz), 7.79-7.62 (m, 4H), 7.56 (d, 2H,  $J = 7.9$  Hz), 7.49-7.37 (m, 3H), 7.34-7.25 (m, 4H), 7.22 (*pseudo t*, 1H,  $J = 7.5$  Hz), 5.58-5.32 (m, 1H), 2.46-2.23 (m, 6H), 1.52-1.35 (m, 12H), 0.89-0.85 (m, 9H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 180.3, 174.8 (d,  $J = 4.2$  Hz), 173.2, 166.1 (d,  $J = 6.1$  Hz), 155.4, 153.8, 151.0, 135.8 (d,  $J = 2.0$  Hz), 133.6, 133.4, 132.3, 130.8, 130.7, 130.6, 130.2 (d,  $J = 3.3$  Hz), 129.9 (d,  $J = 4.7$  Hz), 129.8, 129.5, 128.6, 128.2, 127.1, 126.6, 126.5, 124.9, 122.5, 122.3, 120.8, 116.3, 94.1, 37.2 ( $J = 46.3$  Hz), 24.1 ( $J = 4.9$  Hz), 24.0 ( $J = 15.0$  Hz), 20.5 ( $J = 46.4$  Hz), 13.2. **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3057 (w), 2956 (m), 2870 (m), 1671 (s), 1595 (s), 1538 (s), 1364 (m), 1216 (m). **HRMS** (EI) for  $\text{C}_{46}\text{H}_{46}\text{O}_6\text{P}^+$ ,  $[\text{M}]^+$  (725.3032) found: 725.3026.

### Synthesis of 7b:

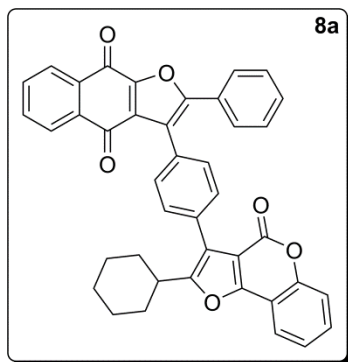


Prepared according to **TP-III** from 4-hydroxy-1-methyl-2(1*H*)-quinolone (87.6 mg, 0.5 mmol), **5pa** (189.2 mg, 1 equiv), tributylphosphine (150.0  $\mu\text{L}$ , 1.2 equiv), pyrrolidine (4.1  $\mu\text{L}$ , 0.1 equiv), and benzoic acid (6.1 mg, 0.1 equiv) in dry THF (0.5 mL) [reaction condition: 28-30 °C for 2.5 h]. Purification by *flash*-chromatography (dichloromethane/methanol: 50/1) yielded **7b** as orange solid (339.2 mg, 92 %). mp.: 137.8-138.8 °C;  $R_f$  0.29 (dichloromethane/methane: 30/1).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 8.31 (d, 1H,  $J = 7.7$  Hz), 8.23 (d, 1H,  $J = 7.0$  Hz), 8.04 (d, 1H,  $J = 6.8$  Hz), 7.81-7.66 (m, 4H), 7.59 (d, 2H,  $J = 7.1$  Hz), 7.49 (*pseudo t*, 1H,  $J = 7.5$  Hz), 7.38 (d, 2H,  $J = 7.8$  Hz), 7.19 (m, 4H), 7.17 (*pseudo t*, 1H,  $J = 7.4$  Hz), 5.95-5.45 (brs, 1H), 3.69 (s, 3H), 2.52-2.22 (m, 6H), 1.54-1.34 (m, 12H), 0.98-0.84 (m, 9H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25 °C)  $\delta/\text{ppm}$ : 180.3, 173.3, 172.3, 164.6, 155.4, 151.1, 140.2, 136.5, 133.6, 132.4, 130.6 (d,  $J = 2.3$  Hz), 130.1 (d,  $J = 4.6$  Hz), 130.0, 129.9, 129.8, 129.7, 128.7, 128.4, 127.3, 126.7, 126.6, 125.6, 123.3, 121.1, 120.2, 113.4, 29.1, 24.4 ( $J = 4.8$  Hz), 24.1 ( $J = 14.9$  Hz), 20.8 ( $J = 47.2$  Hz), 13.3. **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3065 (w), 2957 (m), 2928 (m), 2862 (m), 1675 (s), 1584 (s), 1527 (s), 1216 (m), 685 (m). **HRMS** (EI) for  $\text{C}_{47}\text{H}_{49}\text{NO}_5\text{P}^+$ ,  $[\text{M}]^+$  (738.3348) found: 738.3337.

### Synthesis of 8a:

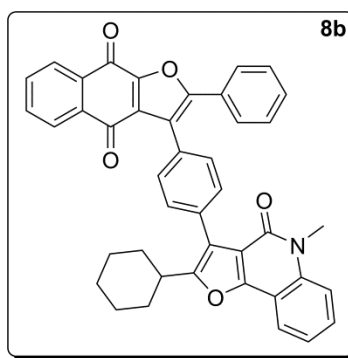
Prepared according to **TP-II** from **7a** (362.4 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **4i**



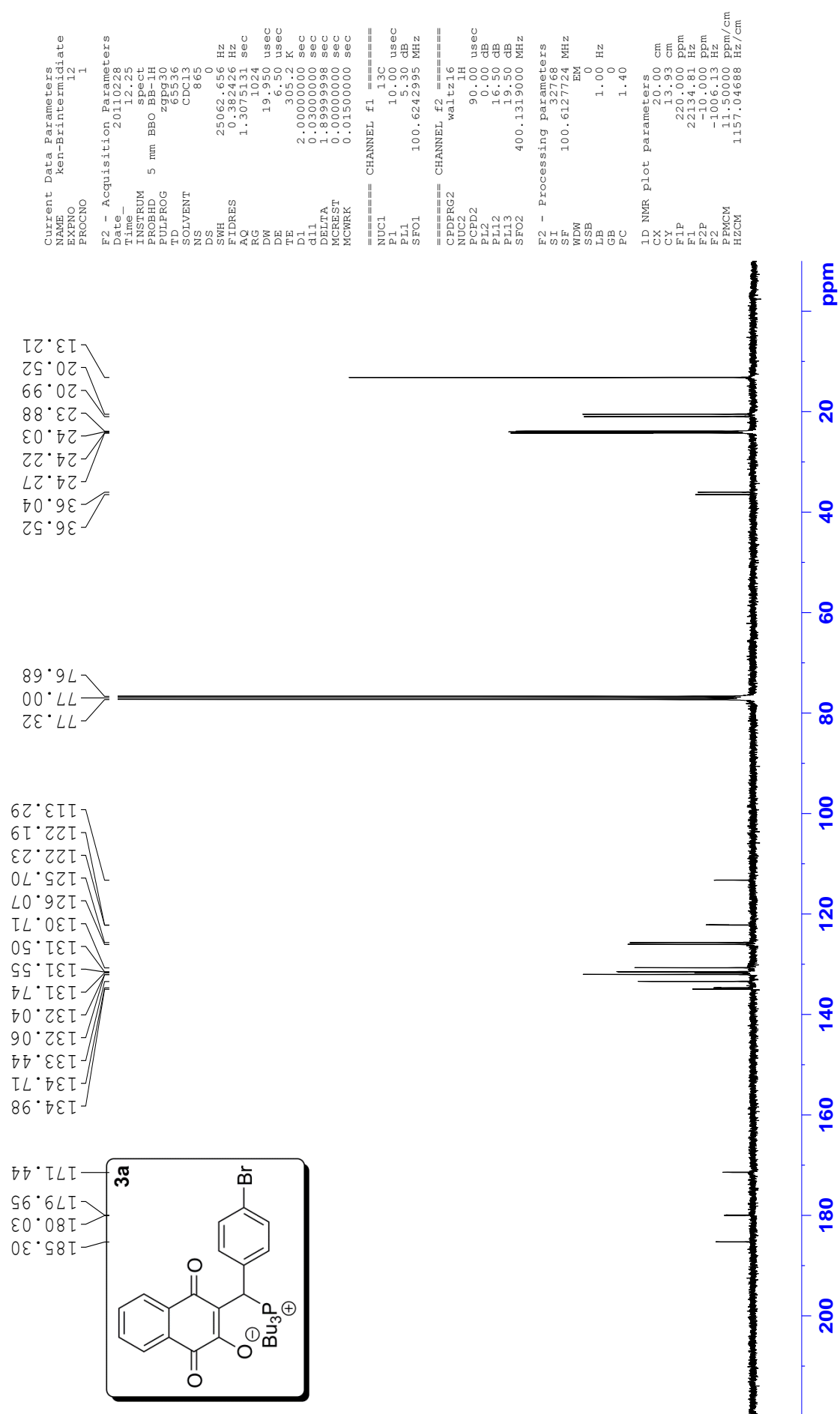


(87.0  $\mu\text{L}$ , 1.3 equiv) in dry THF (5 mL) [reaction condition: 28-30  $^{\circ}\text{C}$  for 1 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 1/1.2) yielded **8a** as pale yellow solid (206.5 mg, 67 %). mp.: 291.4-292.9  $^{\circ}\text{C}$ ;  $R_f$  0.23 (hexanes/ dichloromethane: 1/1.2).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta/\text{ppm}$ : 8.26 (d, 1H,  $J = 6.7$  Hz), 8,15 (d, 1H,  $J = 7.4$  Hz), 7.94 (d, 1H,  $J = 7.4$  Hz), 7.80-7.70 (m, 2H), 7.67-7.61 (m, 2H), 7.59 (*pseudo-s*, 4H), 7.54-7.42 (m, 2H), 7.40-7.30 (m, 4H), 3.06-2.95 (m, 1H), 2.01-1.74 (m, 7H), 1.46-1.33 (m, 3H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta/\text{ppm}$ : 180.9, 173.4, 159.7, 157.8, 156.2, 155.9, 152.4, 151.3, 133.8, 133.6, 132.5, 130.3, 130.2, 130.0, 129.9, 129.5, 129.4, 128.7, 128.4, 127.5, 126.9, 126.6, 124.2, 121.4, 120.7, 118.6, 117.0, 113.0, 109.7, 36.0, 31.8, 26.1, 25.7. **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3057 (w), 2920 (m), 2847 (m), 1747 (s), 1721 (s), 1667 (s), 1215 (s), 973 (m), 692 (m). **HRMS** (70 eV, EI) for  $\text{C}_{41}\text{H}_{28}\text{O}_6$ ,  $[\text{M}]^+$  (616.1886), found: 616.1872.

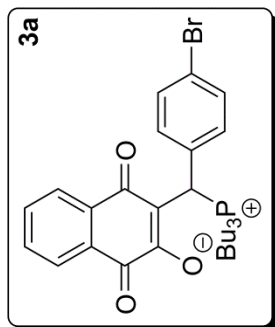
### Synthesis of **8b**:



Prepared according to **TP-II** from **7b** (368.9 mg, 0.5 mmol), triethylamine (104.5  $\mu\text{L}$ , 1.5 equiv), and **4i** (87.0  $\mu\text{L}$ , 1.3 equiv) in dry THF (5 mL) [reaction condition: 28-30  $^{\circ}\text{C}$  for 11.5 h]. Purification by *flash*-chromatography (hexanes/dichloromethane: 1/1.5) yielded **8b** as pale yellow solid (273.9 mg, 87 %). mp.: 365.4-366.4  $^{\circ}\text{C}$ ;  $R_f$  0.22 (hexanes/ dichloromethane: 1/1.5).  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta/\text{ppm}$ : 8.26 (d, 1H,  $J = 7.3$  Hz), 8,14 (d, 1H,  $J = 6.9$  Hz), 8.09 (d, 1H,  $J = 7.8$  Hz), 7.79-7.66 (m, 4H), 7.65-7.52 (m, 5H), 7.46 (d, 1H,  $J = 8.4$  Hz), 7.40-7.30 (m, 4H), 3.78 (s, 3H), 3.03-2.92 (m, 1H), 1.99-1.73 (m, 7H), 1.46-1.31 (m, 3H).  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ , 25  $^{\circ}\text{C}$ )  $\delta/\text{ppm}$ : 180.8, 173.5, 159.5, 159.0, 155.8, 153.8, 151.3, 137.9, 133.7, 133.7, 132.5, 131.9, 130.6, 129.8, 129.7, 129.6, 129.1, 128.9, 128.7, 128.6, 127.6, 127.0, 126.6, 122.0, 121.7, 121.1, 118.6, 114.8, 114.0, 113.1, 36.1, 32.0, 29.2, 26.2, 25.8. **IR** (KBr)  $\tilde{\nu}$  ( $\text{cm}^{-1}$ ): 3065 (w), 2920 (m), 2847 (m), 1663 (s), 1584 (m), 1215 (m), 684 (m). **HRMS** (FAB) for  $\text{C}_{42}\text{H}_{32}\text{NO}_5$ ,  $[\text{M}+\text{H}]^+$  (630.2280), found: 630.2288.



— 32.85



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Current Data Parameters
NAME      Ken-P
EXPNO     56
PROCNO    1

F2 - Acquisition Parameters
Date_     20120602
Time      12.35
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg50
TD        65536
SOLVENT   CDCl3
NS        96
DS        0
SWH       64724.918 Hz
FIDRES    0.987624 Hz
AQ        0.5063156 sec
RG        6502
DW        7.725 usec
DE        6.50 usec
TE        299.2 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1

===== CHANNEL f1 =====
NUC1      31P
P1        10.60 usec
PL1       12.00 dB
SFO1      161.9674942 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     90.00 usec
PL2       3.80 dB
PL12      21.60 dB
PL13      24.60 dB
SFO2      400.1316005 MHz

F2 - Processing parameters
SI        32768
SF        161.9757146 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.00
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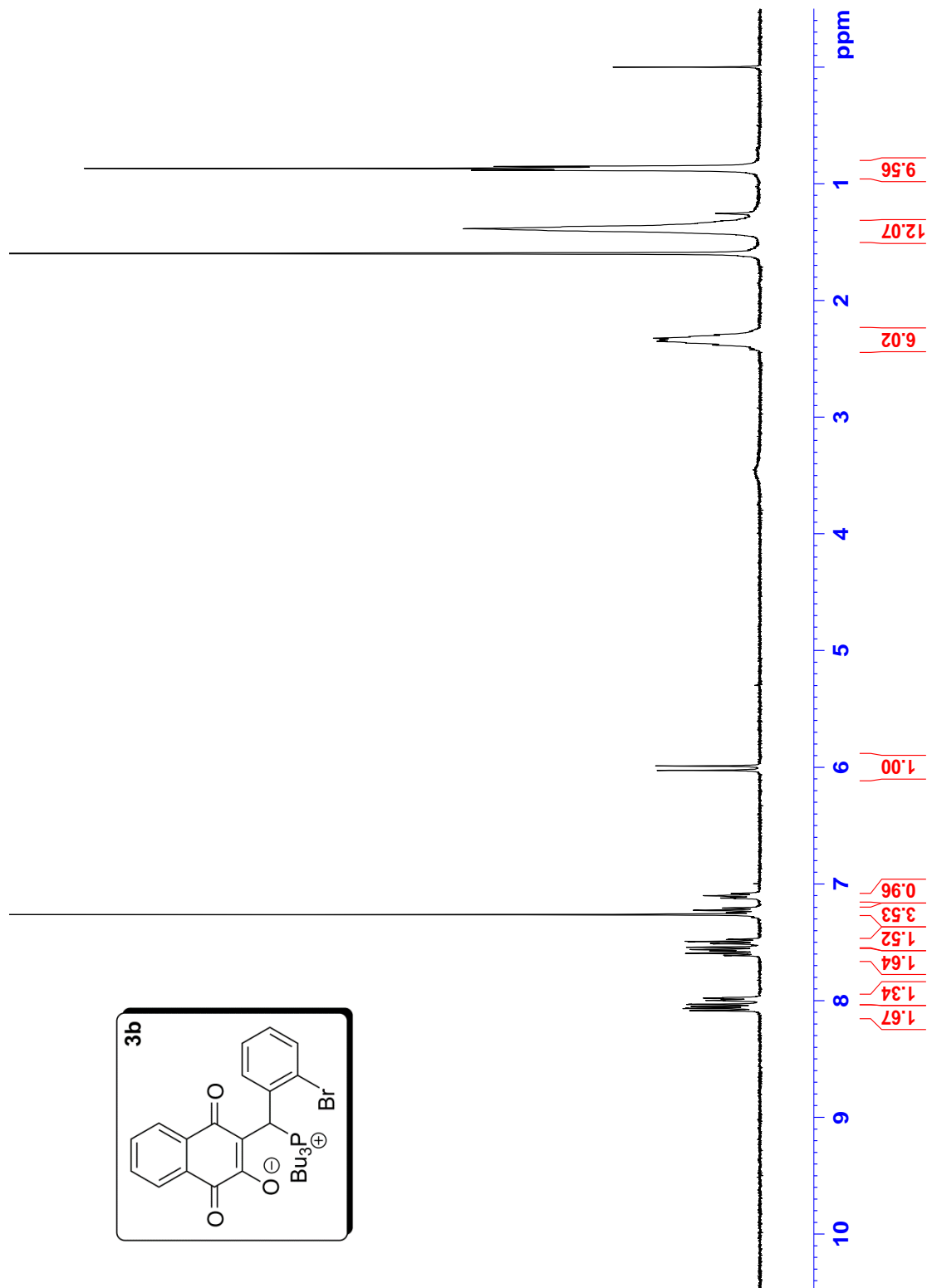
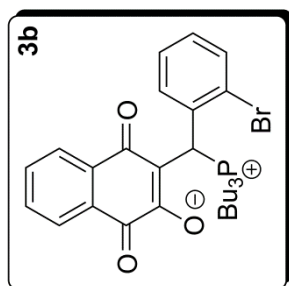
95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 ppm

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EXPNO    6
PROCNO   1

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Time     14.53
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PROBHD   5 mm BBO BB-IH
PULPROG  zg30
TD       32768
SOLVENT  CDCl3
NS       8
DS       0
SWH      7246.377 Hz
FIDRES   0.221142 Hz
AQ       2.2610421 sec
RG       322.5
DW       69.000 usec
DE       6.50 usec
TE       299.7 K
D1       2.00000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1     1H
F1       11.70 usec
FL1      4.00 dB
SFO1     400.1324008 MHz

F2 - Processing parameters
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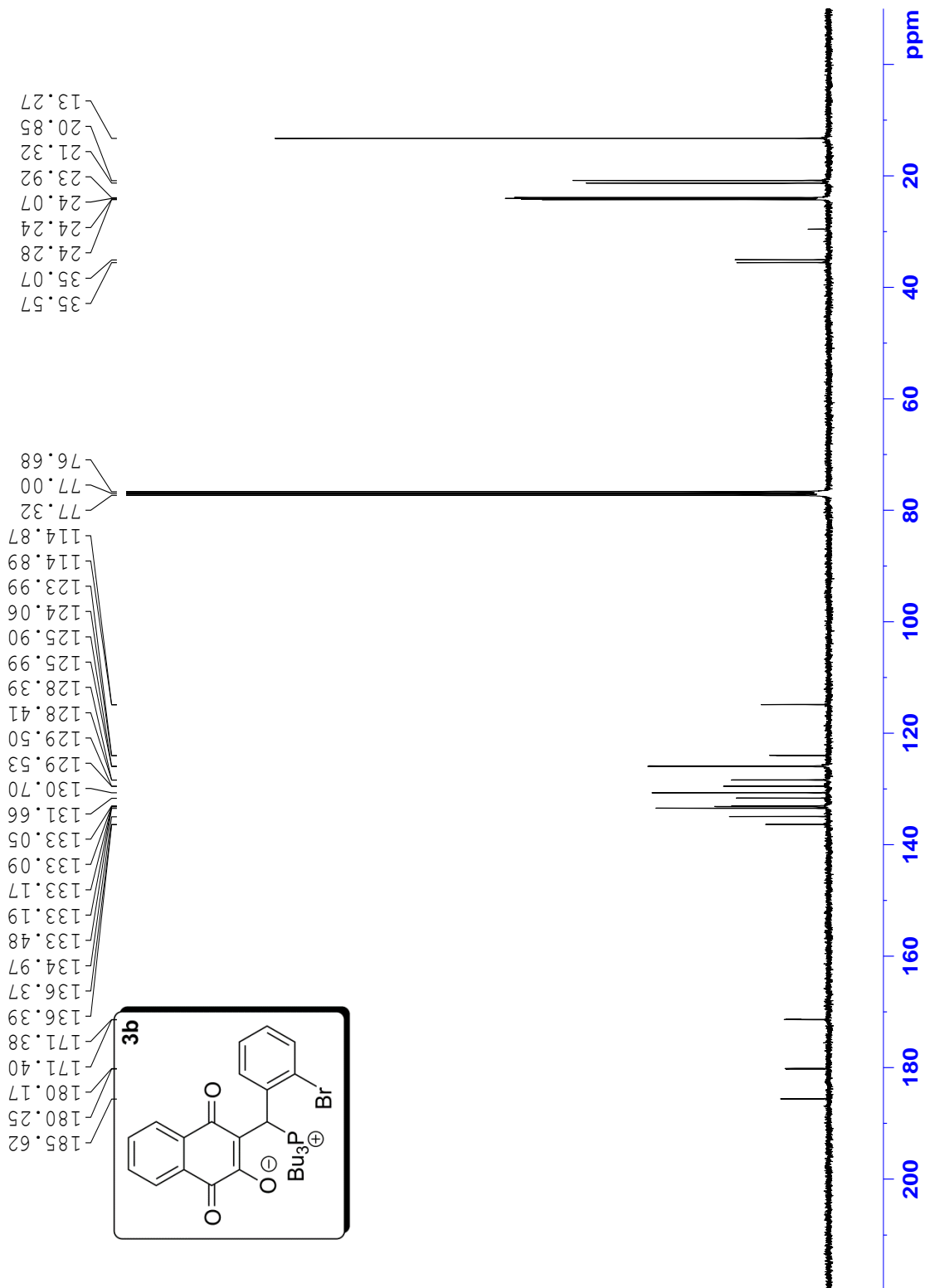
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EXPNO         9
PROCNO        1

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Time_         12.15
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PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            32768
SOLVENT       CDCl3
NS            1583
DS            4
SWH           24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6816244 sec
RG            4096
DE            20.800 usec
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D1            2.0000000 sec
D11           0.0300000 sec
TDO           1

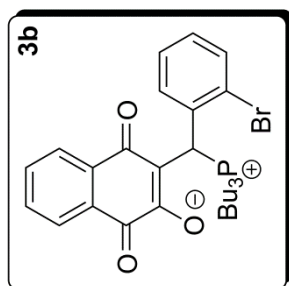
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NUC1          13C
P1            8.90 usec
PL1           7.00 dB
SFO1          100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         90.00 usec
PL2           3.80 dB
PL12          21.60 dB
PL13          24.60 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI            32768
SF            100.6127763 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.00
    
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— 36.11



```
Current Data Parameters
NAME      Ken-P
EXPNO     54
PROCNO    1

F2 - Acquisition Parameters
Date_     20120602
Time      12.25
INSTRUM   spect
PROBHD    5 mm BBO BB-IH
PULPROG   zgpg50
TD         65536
SOLVENT   CDCl3
NS         110
DS         0
SWH        64724.918 Hz
FIDRES     0.987624 Hz
AQ         0.5063156 sec
RG         5792.6
DW         7.725 usec
DE         6.50 usec
TE         299.2 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       31P
P1         10.60 usec
PL1        12.00 dB
SFO1       161.9674942 MHz

===== CHANNEL f2 =====
CPDPRG2    waitz16
NUC2       1H
PCPD2      90.00 usec
PL2        3.80 dB
PLI2       21.60 dB
PLI3       24.60 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI         32768
SF         161.9757146 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00
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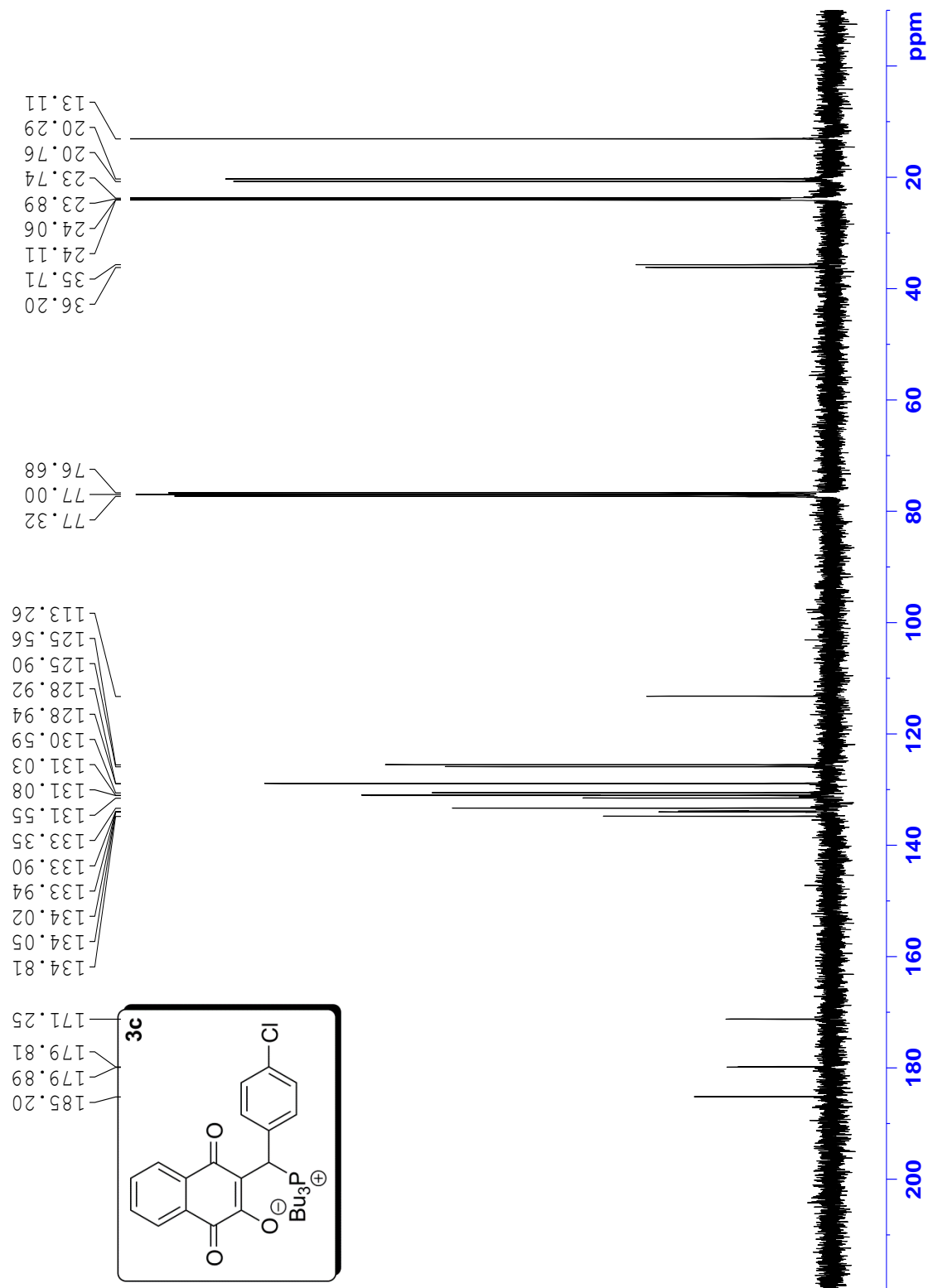
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EXPNO    15
PROCNO   1

F2 - Acquisition Parameters
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PULPROG  zgpg30
TD       32768
SOLVENT  CDCl3
NS       127
DS       1
SWH      24038.461 Hz
FIDRES   0.733536 Hz
AQ       0.6816244 sec
RG       4096
DE       20.800 usec
TE       6.50 usec
TE       299.6 K
D1       2.0000000 sec
D11      0.0300000 sec
TDO      1

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NUC1     13C
P1       8.90 usec
PL1      7.00 dB
SFO1     100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    90.00 usec
PL2      3.80 dB
PL12     21.60 dB
PL13     24.60 dB
SFO2     400.1316005 MHz

F2 - Processing parameters
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SF       100.6127877 MHz
WDW      EM
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GB       0
PC       1.00
    
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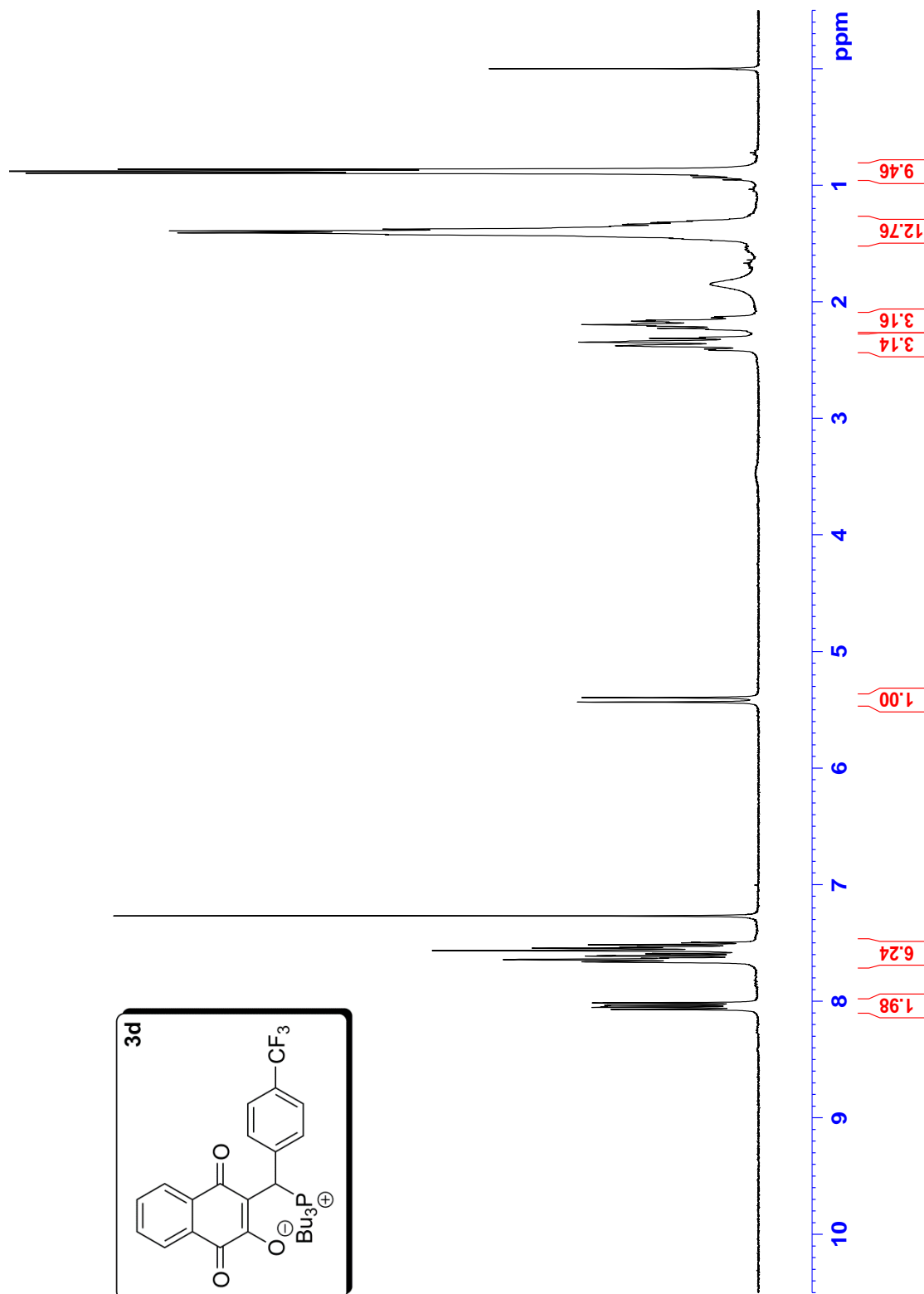


Current Data Parameters  
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EXPNO 16  
PROCNO 1

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PULPROG zg30  
TD 16384  
SOLVENT CDC13  
NS 8  
DS 0  
SWH 6009.615 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 203.2  
DW 83.200 usec  
DE 6.50 usec  
TE 299.4 K  
D1 1.50000000 sec  
MCREST 0 sec  
MCWRK 0.01500000 sec

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1326008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300057 MHz  
WDW EM  
SSB 0  
LB 0.10 Hz  
GB 0  
PC 1.00



```

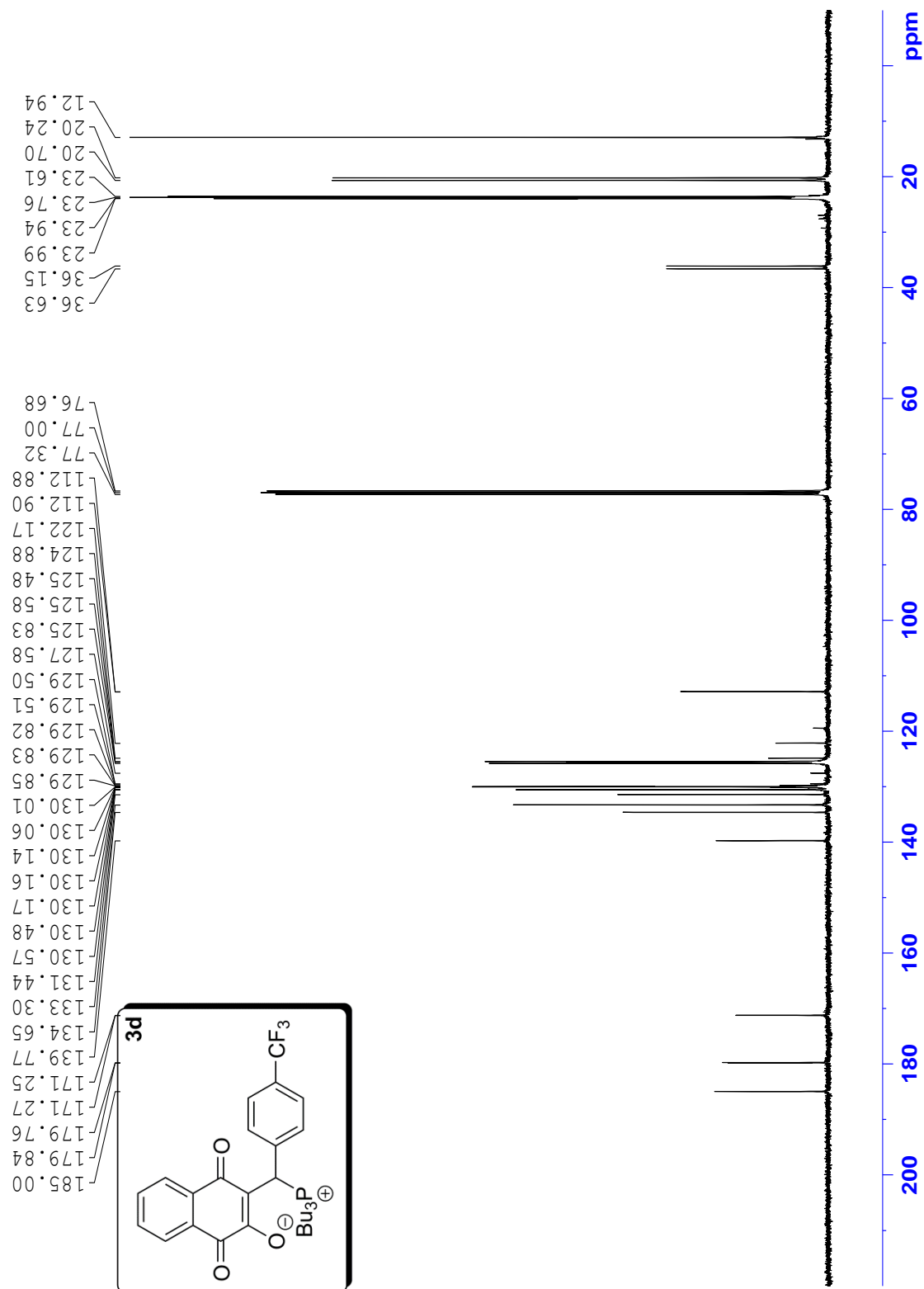
Current Data Parameters
NAME      ken282
EXPNO    6
PROCNO   1

F2 - Acquisition Parameters
Date_    20120530
Time_    12.14
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zgpg30
TD        32768
SOLVENT  CDCl3
NS        1194
DS        24038.461 Hz
SWH       0.733596 Hz
FIDRES    0.16816244 sec
AQ         4096
RG         20.800 usec
DE         6.50 usec
TE        298.8 K
D1         2.00000000 sec
D11        0.03000000 sec
TDO       1

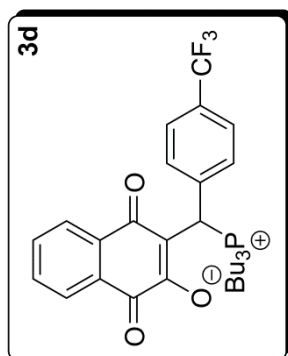
===== CHANNEL f1 =====
NUC1       13C
P1         8.90 usec
PL1        7.00 dB
SFO1       100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     90.00 usec
PL2        3.80 dB
PL12       21.60 dB
PL13       24.60 dB
SFO2       400.1316005 MHz

F2 - Processing Parameters
SI         32768
SF         100.6127932 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00
    
```



— 33.41



```
Current Data Parameters
NAME      Ken-P
EXPNO     28
PROCNO    1

F2 - Acquisition Parameters
Date_     20120310
Time      13.02
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg50
TD         65536
SOLVENT   CDCl3
NS         38
DS         0
SWH        64724.918 Hz
FIDRES     0.987624 Hz
AQ          0.5063156 sec
RG          4597.6
DW          7.725 usec
DE          6.50 usec
TE          298.2 K
D1          2.0000000 sec
D11         0.03000000 sec
TD0         1

===== CHANNEL f1 =====
NUC1       31P
P1         10.60 usec
PL1        12.00 dB
SFO1       161.9674942 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      90.00 usec
PL2        3.80 dB
PL12       21.60 dB
PL13       24.60 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI         32768
SF         161.9757133 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00
```

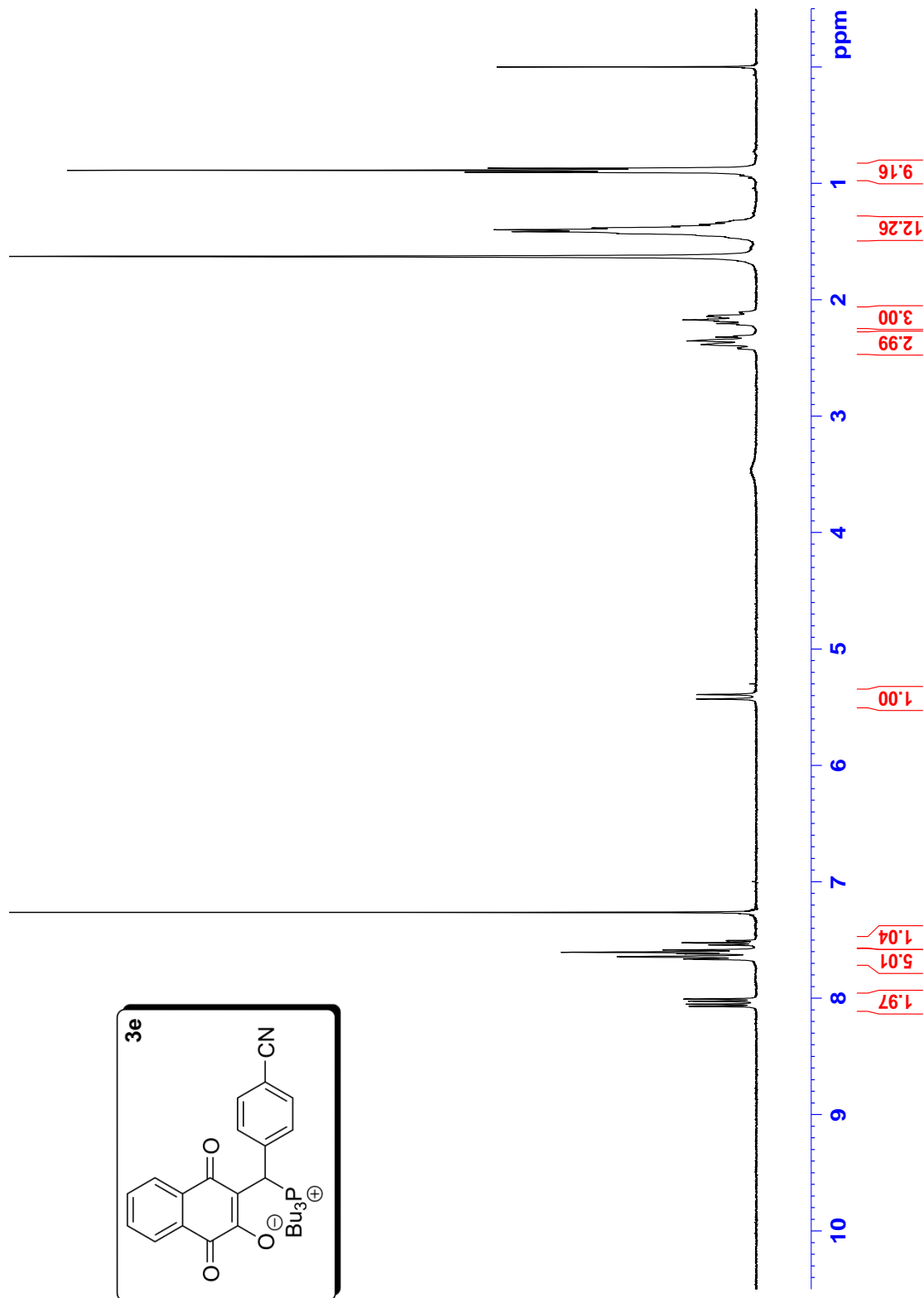


Current Data Parameters  
NAME Jimmy-11  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110829  
Time 10.33  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 16384  
SOLVENT CDC13  
NS 8  
DS 0  
SWH 6009.615 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 322.5  
DW 83.200 usec  
DE 6.50 usec  
TE 299.2 K  
D1 1.50000000 sec  
MCREST 0 sec  
MCWRK 0.01500000 sec

==== CHANNEL f1 =====  
NUC1 1H  
IH 11.70 usec  
PL1 4.00 dB  
SFO1 400.1326008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300072 MHz  
WDW EM  
SSB 0  
LB 0.10 Hz  
GB 0  
PC 1.00



```

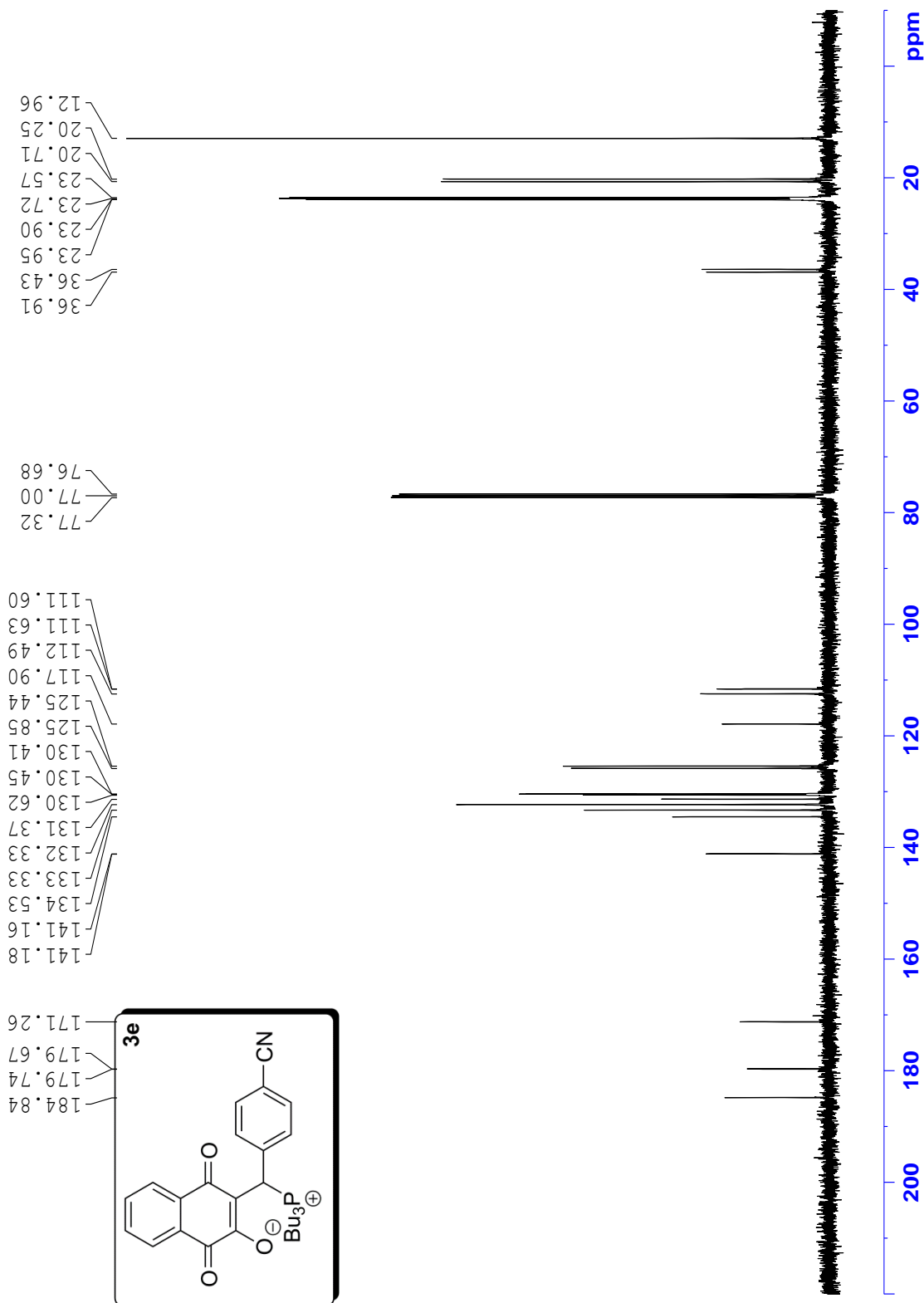
Current Data Parameters
NAME      Jimmy-11
EXPNO    6
PROCNO   1

F2 - Acquisition Parameters
Date_    20120304
Time_    19.39
INSTRUM  spect
PROBHD   5 mm BBO BB-IH
PULPROG  zgpg30
TD       32768
SOLVENT  CDCl3
NS       173
DS       1
SWH      24038.461 Hz
FIDRES   0.733536 Hz
AQ       0.16816244 sec
RG       4096
DE       20.800 usec
TE       6.50 usec
TE       299.15 K
D1       2.00000000 sec
D11      0.03000000 sec
TDO      1

===== CHANNEL f1 =====
NUC1     13C
P1       8.90 usec
PL1      7.00 dB
SFO1     100.6233325 MHz

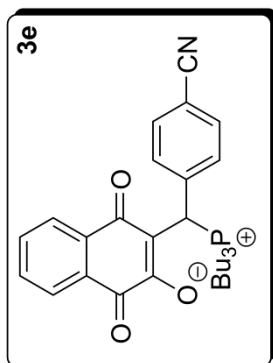
===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    90.00 usec
PL2      3.80 dB
PL12     21.60 dB
PL13     24.60 dB
SFO2     400.1316005 MHz

F2 - Processing parameters
SI       32768
SF       100.6127979 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.00
    
```





— 33.59



```
Current Data Parameters
NAME      Ken-P
EXPNO     26
PROCNO    1

F2 - Acquisition Parameters
Date_     20120310
Time      12.55
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg50
TD         65536
SOLVENT   CDCl3
NS         21
DS         0
SWH        64724.918 Hz
FIDRES     0.987624 Hz
AQ         0.5063156 sec
RG         7298.2
DW         7.725 usec
DE         6.50 usec
TE         298.6 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       131P
P1         10.60 usec
PL1        12.00 dB
SFO1       161.9674942 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      90.00 usec
PL2        3.80 dB
PL12       21.60 dB
PL13       24.60 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI         32768
SF         161.9757133 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00
```

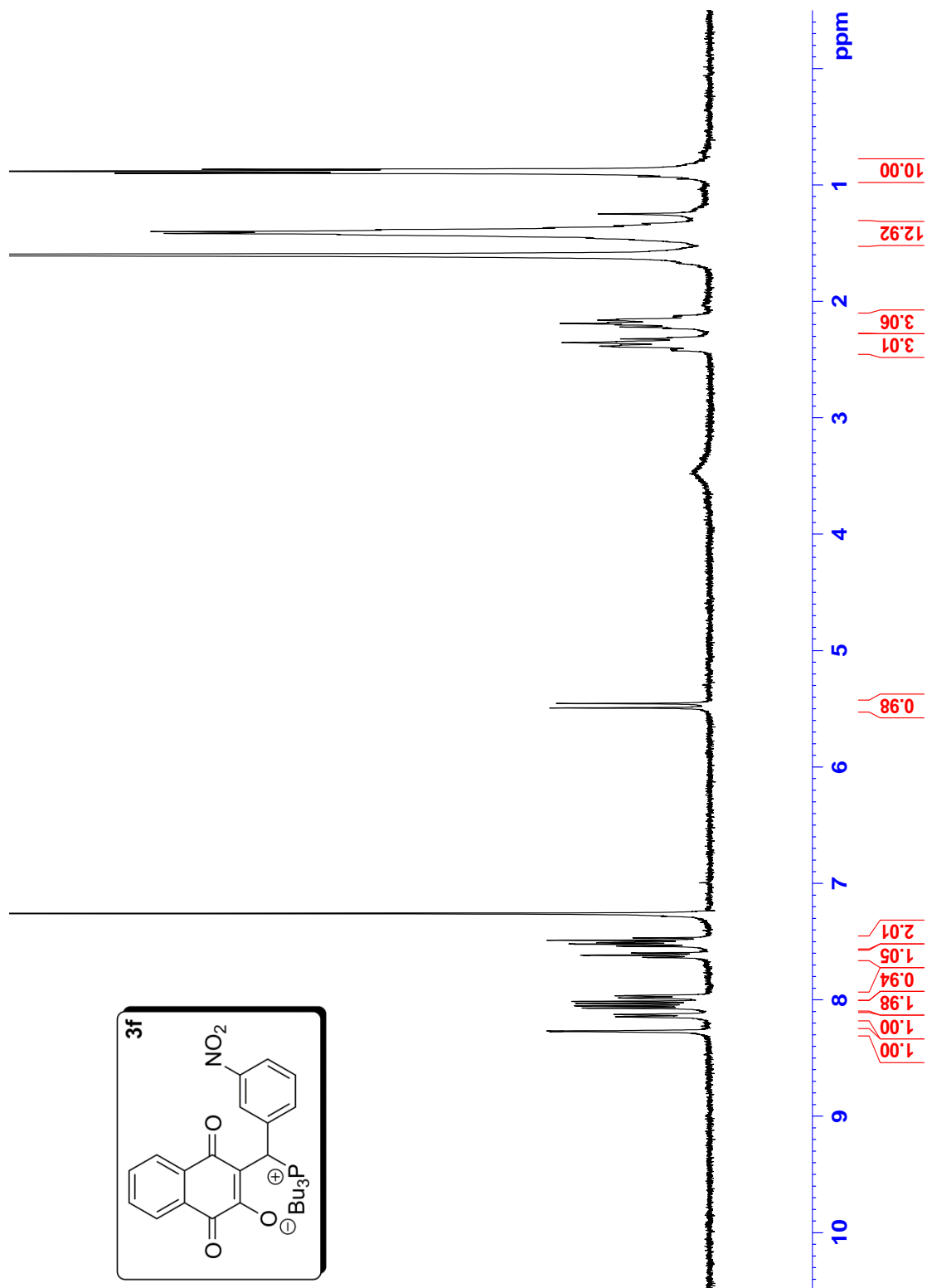


Current Data Parameters  
NAME Ken281  
EXPNO 6  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110719  
Time 20.31  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 16384  
SOLVENT CDC13  
NS 8  
DS 0  
SWH 6009.615 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 362  
DW 83.200 usec  
DE 6.50 usec  
TE 299.3 K  
D1 1.50000000 sec  
MCREST 0 sec  
MCWRK 0.01500000 sec

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1326008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300088 MHz  
WDW EM  
SSB 0  
LB 0.10 Hz  
GB 0  
PC 1.00



```

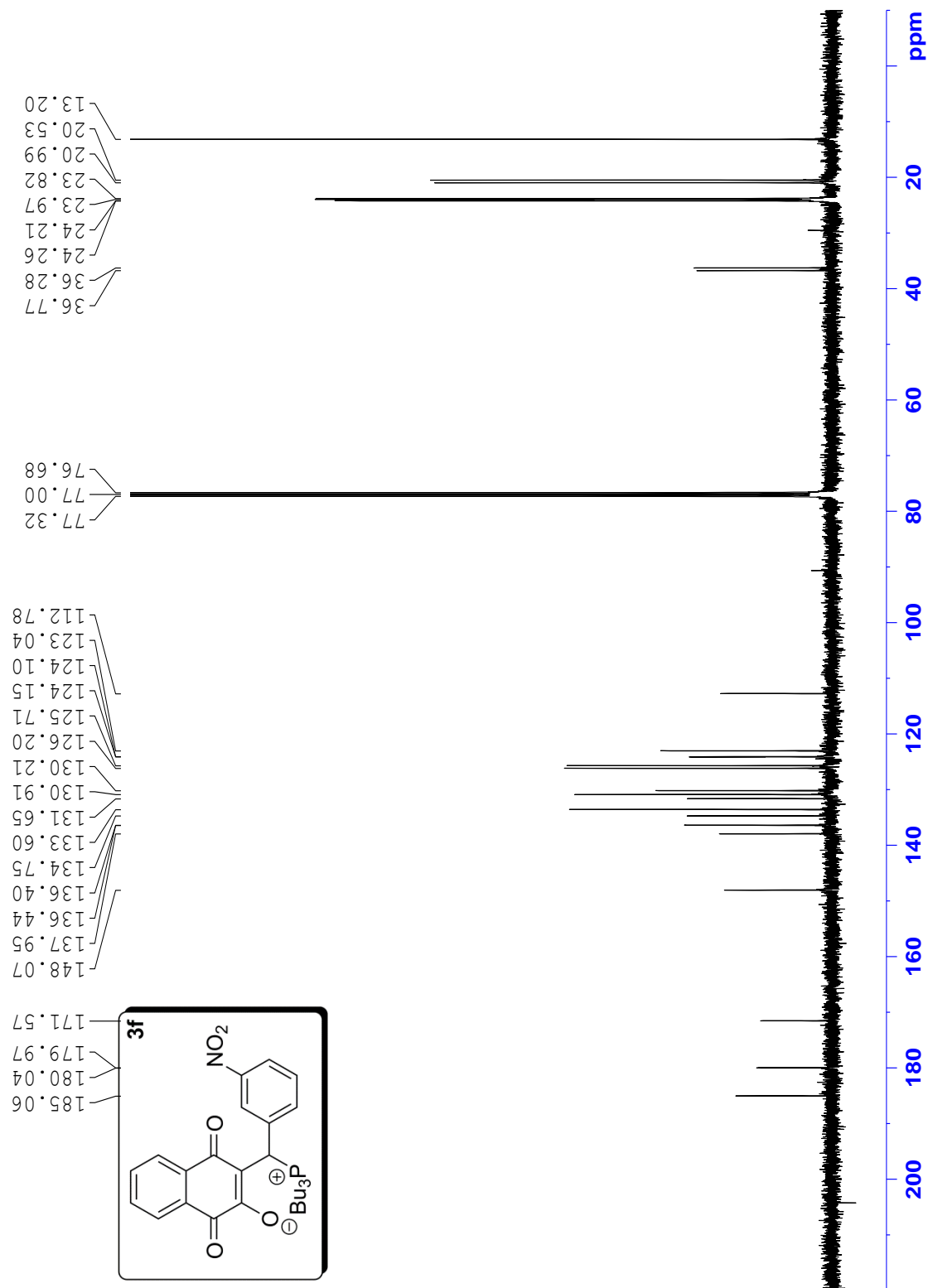
Current Data Parameters
NAME      ken281
EXPNO    8
PROCNO   1

F2 - Acquisition Parameters
Date_    20120311
Time_    17.34
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zgpg30
TD       32768
SOLVENT  CDCl3
NS       693
DS       24038.461 Hz
SWH      0.733536 Hz
FIDRES   0.16816244 sec
AQ       4096
RG       20.800 usec
DE       6.50 usec
TE       297.6 K
D1       2.00000000 sec
D11      0.03000000 sec
TDO      1

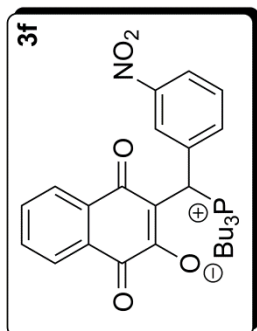
===== CHANNEL f1 =====
NUC1     13C
P1       8.90 usec
PL1      7.00 dB
SFO1     100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    90.00 usec
PL2      3.80 dB
PL12     21.60 dB
PL13     24.60 dB
SFO2     400.1316005 MHz

F2 - Processing parameters
SI       32768
SF       100.6127788 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.00
    
```



— 33.67



```
Current Data Parameters
NAME      Ken-P
EXPNO     8
PROCNO    1

F2 - Acquisition Parameters
Date_     20120315
Time      19.16
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg50
TD        65536
SOLVENT   CDCl3
NS         20
DS         0
SWH        64724.918 Hz
FIDRES     0.987624 Hz
AQ         0.5063156 sec
RG         91195.2
DW         7.725 usec
DE         6.50 usec
TE         300.6 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       131P
P1         10.60 usec
PL1        12.00 dB
SFO1       161.9674942 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      90.00 usec
PL2        3.80 dB
PL12       21.60 dB
PL13       24.60 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI         32768
SF         161.9757164 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00
```

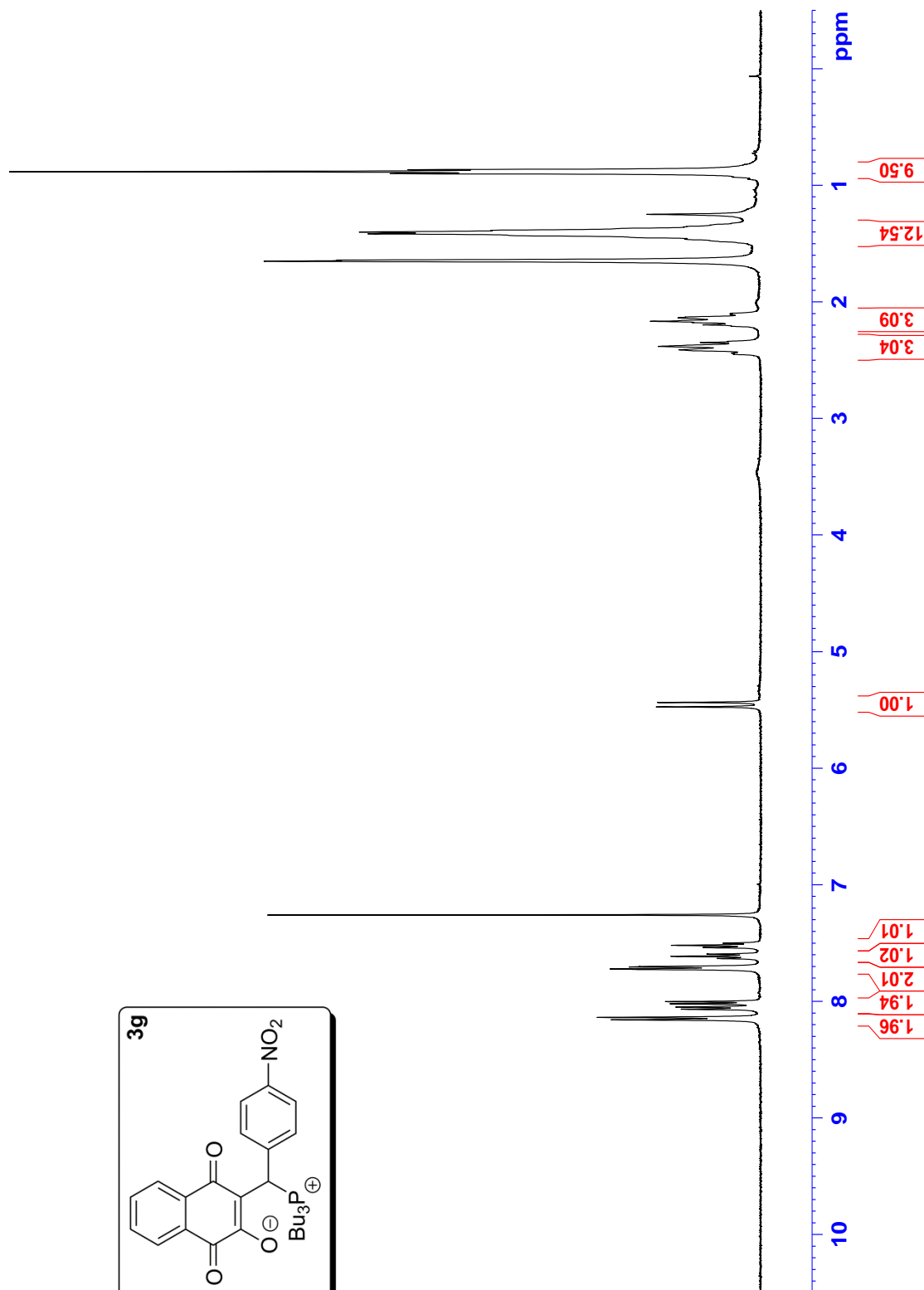
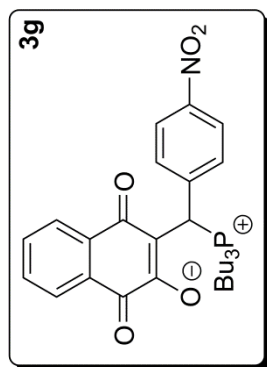


Current Data Parameters  
NAME ken278  
EXPNO 6  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110719  
Time\_ 21.06  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 16384  
SOLVENT CDC13  
NS 8  
DS 0  
SWH 6009.615 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 256  
DW 83.200 usec  
DE 6.50 usec  
TE 299.1 K  
D1 1.50000000 sec  
MCREST 0 sec  
MCWRK 0.01500000 sec

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1326008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300086 MHz  
WDW EM  
SSB 0  
LB 0.10 Hz  
GB 0  
PC 1.00



```

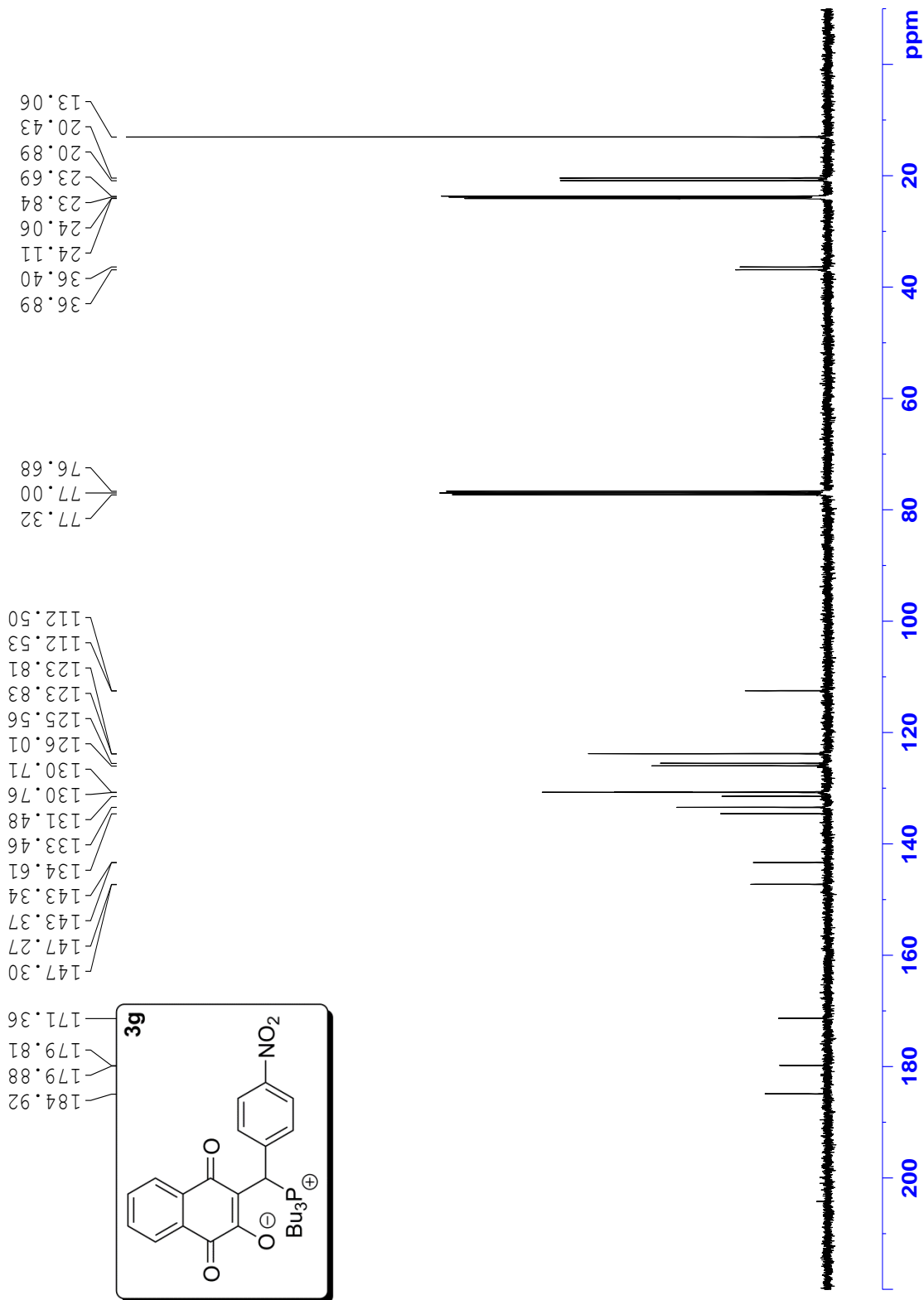
Current Data Parameters
NAME      ken278
EXPNO    8
PROCNO   1

F2 - Acquisition Parameters
Date_    20120315
Time_    9.20
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zgpg30
TD       32768
SOLVENT  CDCl3
NS       251
DS       4
SWH      24038.461 Hz
FIDRES   0.733536 Hz
AQ       0.16816244 sec
RG       4096
DW       20.800 usec
DE       6.50 usec
TE       300.1 K
D1       2.00000000 sec
D11      0.03000000 sec
TDO      1

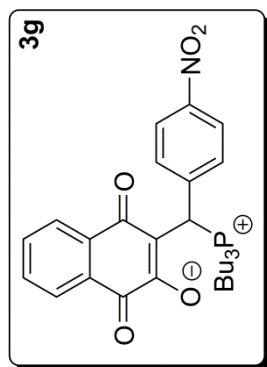
===== CHANNEL f1 =====
NUC1     13C
P1       8.90 usec
PL1      7.00 dB
SFO1     100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    90.00 usec
PL2      3.80 dB
PL12     21.60 dB
PL13     24.60 dB
SFO2     400.1316005 MHz

F2 - Processing parameters
SI       32768
SF       100.6127892 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.00
    
```



— 33.61



```
Current Data Parameters
NAME          Ken-P
EXPNO         4
PROCNO        1

F2 - Acquisition Parameters
Date_         20120315
Time_         19.00
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg50
TD            65536
SOLVENT       CDCl3
NS            24
DS            0
SWH           64724.918 Hz
FIDRES        0.987624 Hz
AQ            0.5063156 sec
RG            4597.6
DW            7.725 usec
DE            6.50 usec
TE            300.6 K
D1            2.0000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          31P
P1            10.60 usec
PL1           12.00 dB
SFO1          161.9674942 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         90.00 usec
PL2           3.80 dB
PL12          21.60 dB
PL13          24.60 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI            32768
SF            161.9757164 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.00
```



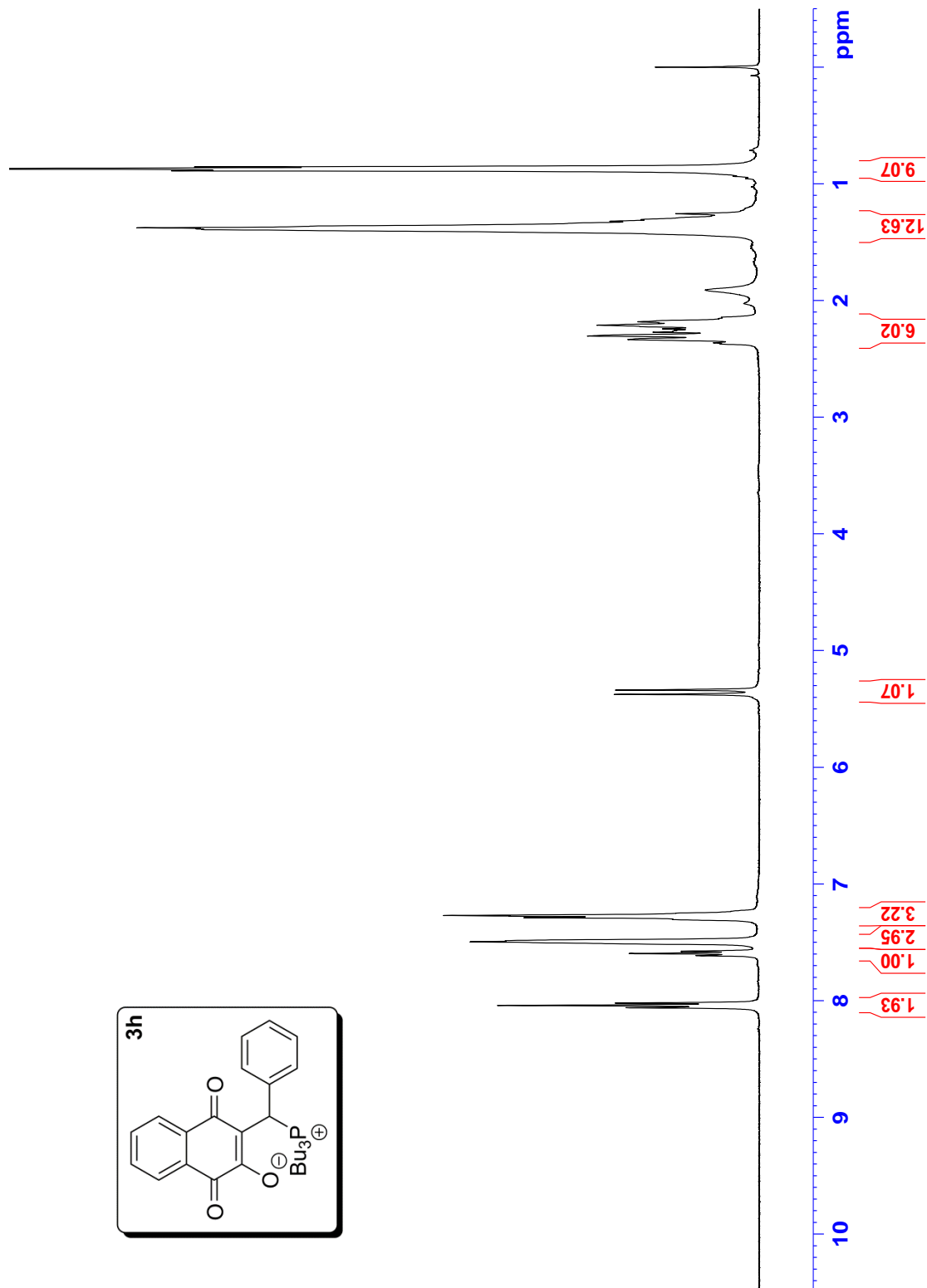
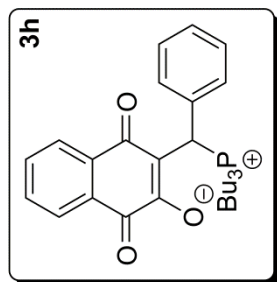


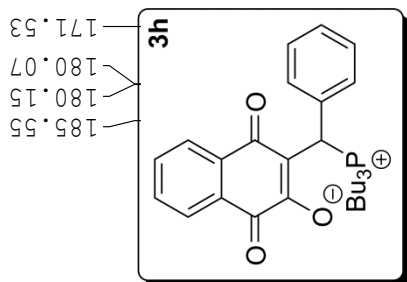
Current Data Parameters  
NAME ken280  
EXPNO 8  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120310  
Time\_ 11.07  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDC13  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 114  
DW 69.000 usec  
DE 6.50 usec  
TE 298.0 K  
D1 2.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300016 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00





185.55  
180.15  
180.07  
171.53

135.49  
135.46  
135.16  
133.44  
131.81  
130.66  
129.88  
129.84  
129.01  
128.98  
128.11  
128.08  
126.07  
125.75  
113.73

77.32  
77.00  
76.68

36.87  
36.38  
24.30  
24.25  
24.09  
23.94  
20.94  
20.47  
13.29

```

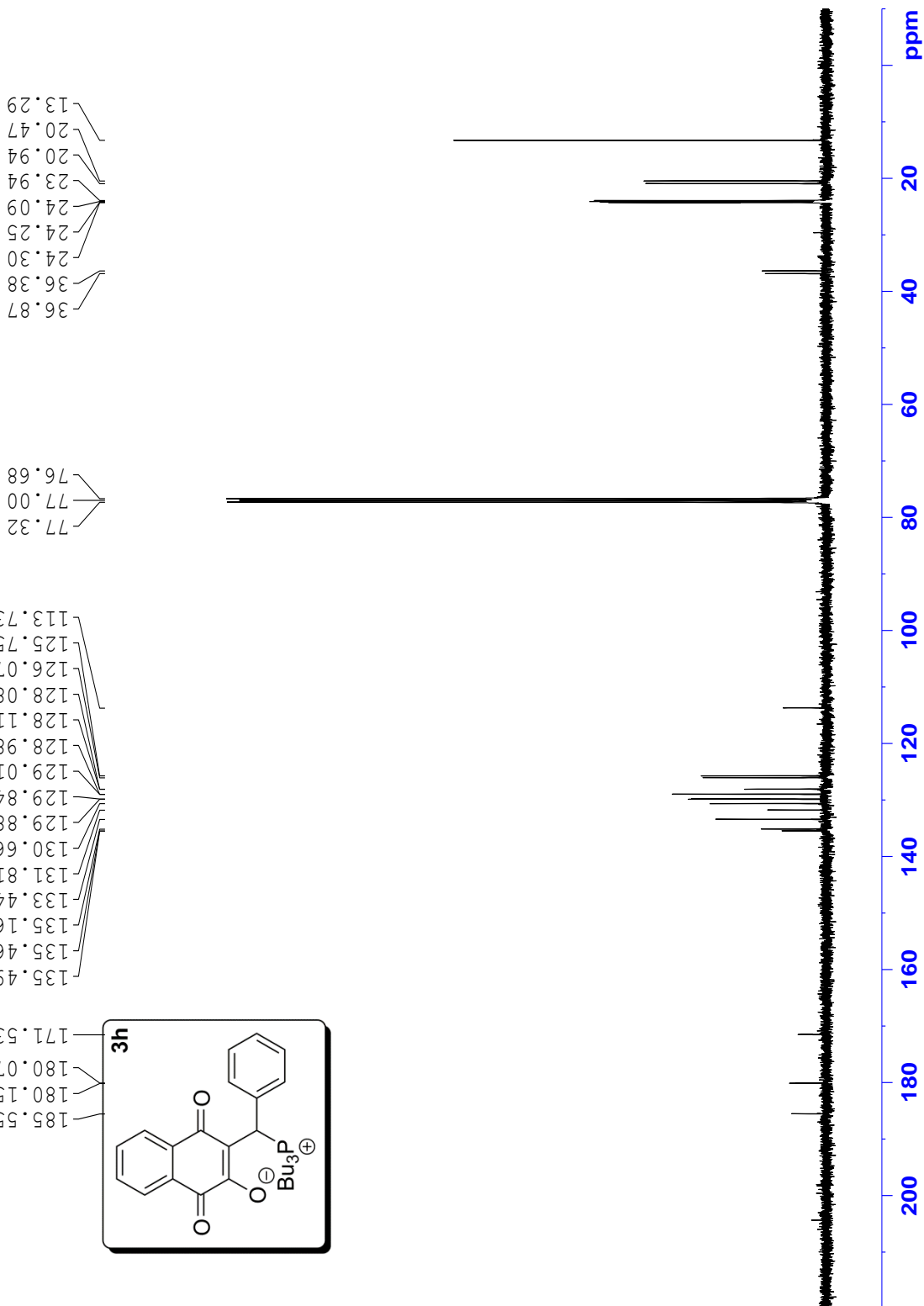
Current Data Parameters
NAME          ken280
EXPNO         7
PROCNO        1

F2 - Acquisition Parameters
Date_         20120304
Time_         18.29
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            32768
SOLVENT       CDCl3
NS            731
DS            24038.461 Hz
SWH           0.733536 Hz
FIDRES        0.16816244 sec
AQ            5792.6
RG            20.800 usec
DE            6.50 usec
TE            299.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TDO           1

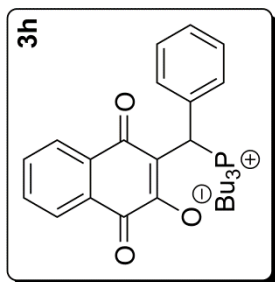
===== CHANNEL f1 =====
NUC1          13C
P1            8.90 usec
PL1           7.00 dB
SFO1          100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         90.00 usec
PL2           3.80 dB
PL12          21.60 dB
PL13          24.60 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI            32768
SF            100.6127735 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.00
    
```



— 32.92



```
Current Data Parameters
NAME      Ken-P
EXPNO     24
PROCNO    1

F2 - Acquisition Parameters
Date_     20120310
Time      12.48
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg50
TD         65536
SOLVENT   CDCl3
NS         31
DS         0
SWH        64724.918 Hz
FIDRES     0.987624 Hz
AQ          0.5063156 sec
RG          20642.5
DW          7.725 usec
DE          6.50 usec
TE          298.1 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0         1

===== CHANNEL f1 =====
NUC1       131P
P1         10.60 usec
PL1        12.00 dB
SFO1       161.9674942 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
P2         90.00 usec
PL2        3.80 dB
PL12       21.60 dB
PL13       24.60 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI          32768
SF          161.9757133 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.00
```

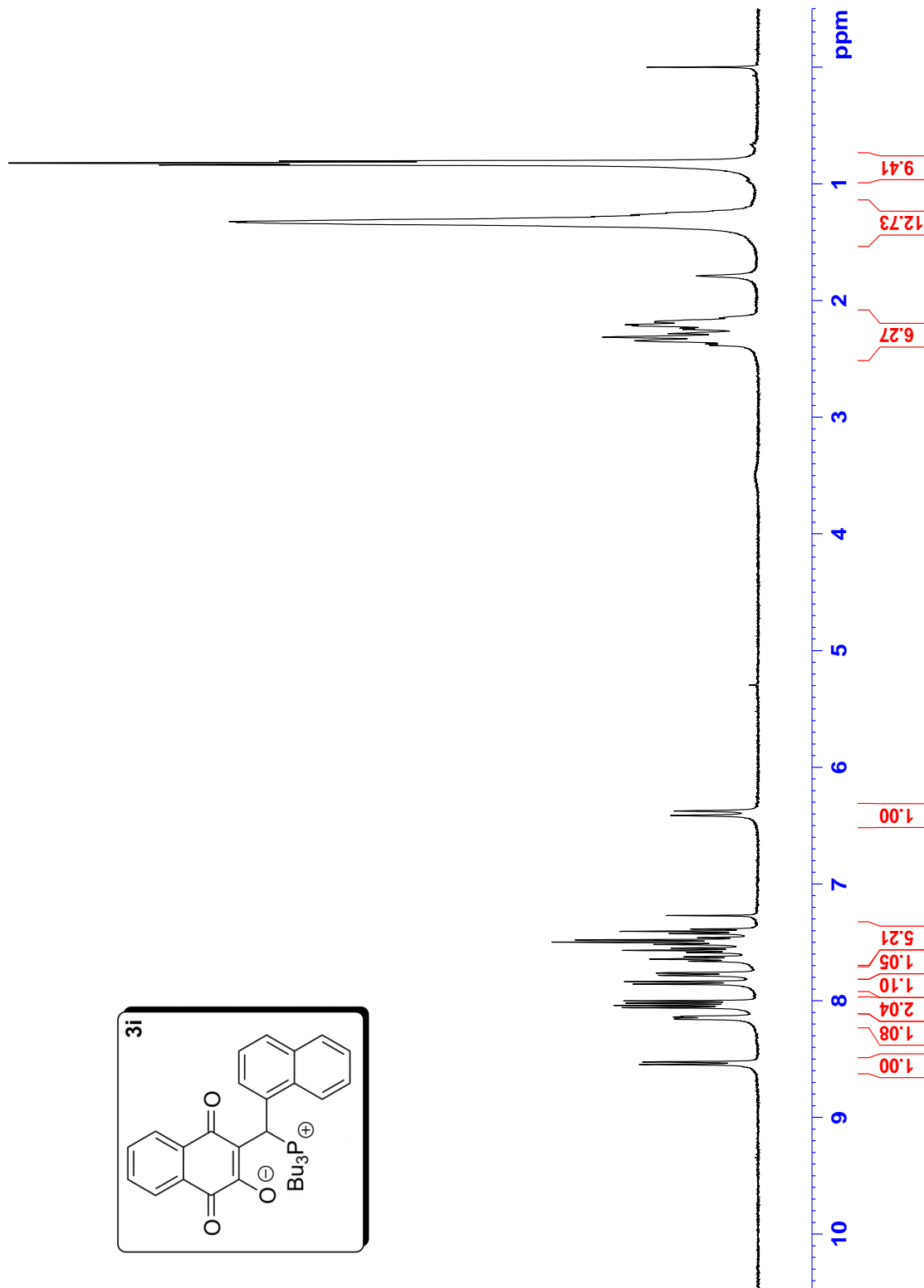
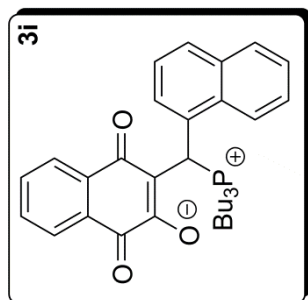


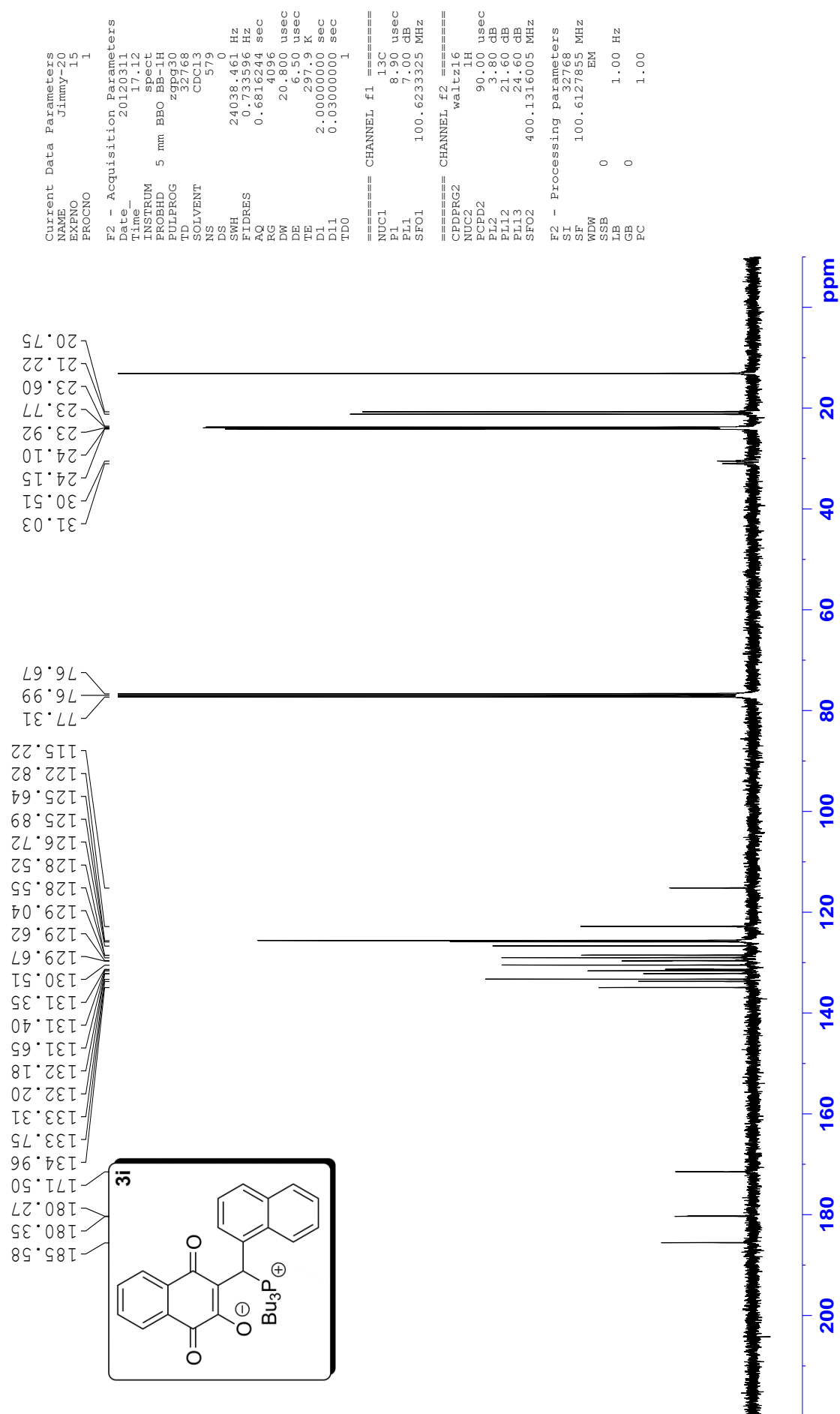
Current Data Parameters  
NAME Jimmy-20  
EXPNO 17  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120602  
Time\_ 13.02  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDC13  
NS 32  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 4  
DW 69.000 usec  
DE 6.50 usec  
TE 298.7 K  
D1 2.00000000 sec  
TD0 1

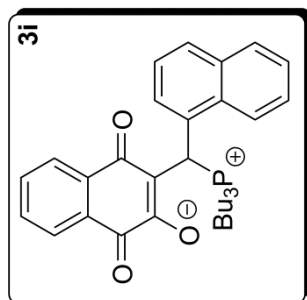
==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300039 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00





— 35.35



```
Current Data Parameters
NAME      Ken-P
EXPNO     6
PROCNO    1

F2 - Acquisition Parameters
Date_     20120315
Time      19.10
INSTRUM   spect
PROBHD    5 mm BBO BB-IH
PULPROG   zgpg50
TD         65536
SOLVENT   CDCl3
NS         17
DS         0
SWH        64724.918 Hz
FIDRES     0.987624 Hz
AQ          0.5063156 sec
RG          5792.6
DW          7.725 usec
DE          6.50 usec
TE          300.6 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0         1

===== CHANNEL f1 =====
NUC1       31P
P1         10.60 usec
PL1        12.00 dB
SFO1       161.9674942 MHz

===== CHANNEL f2 =====
CPDPRG2    waitz16
NUC2       1H
PCPD2      90.00 usec
PL2        3.80 dB
PL12       21.60 dB
PL13       24.60 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI          32768
SF          161.9757164 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.00
```

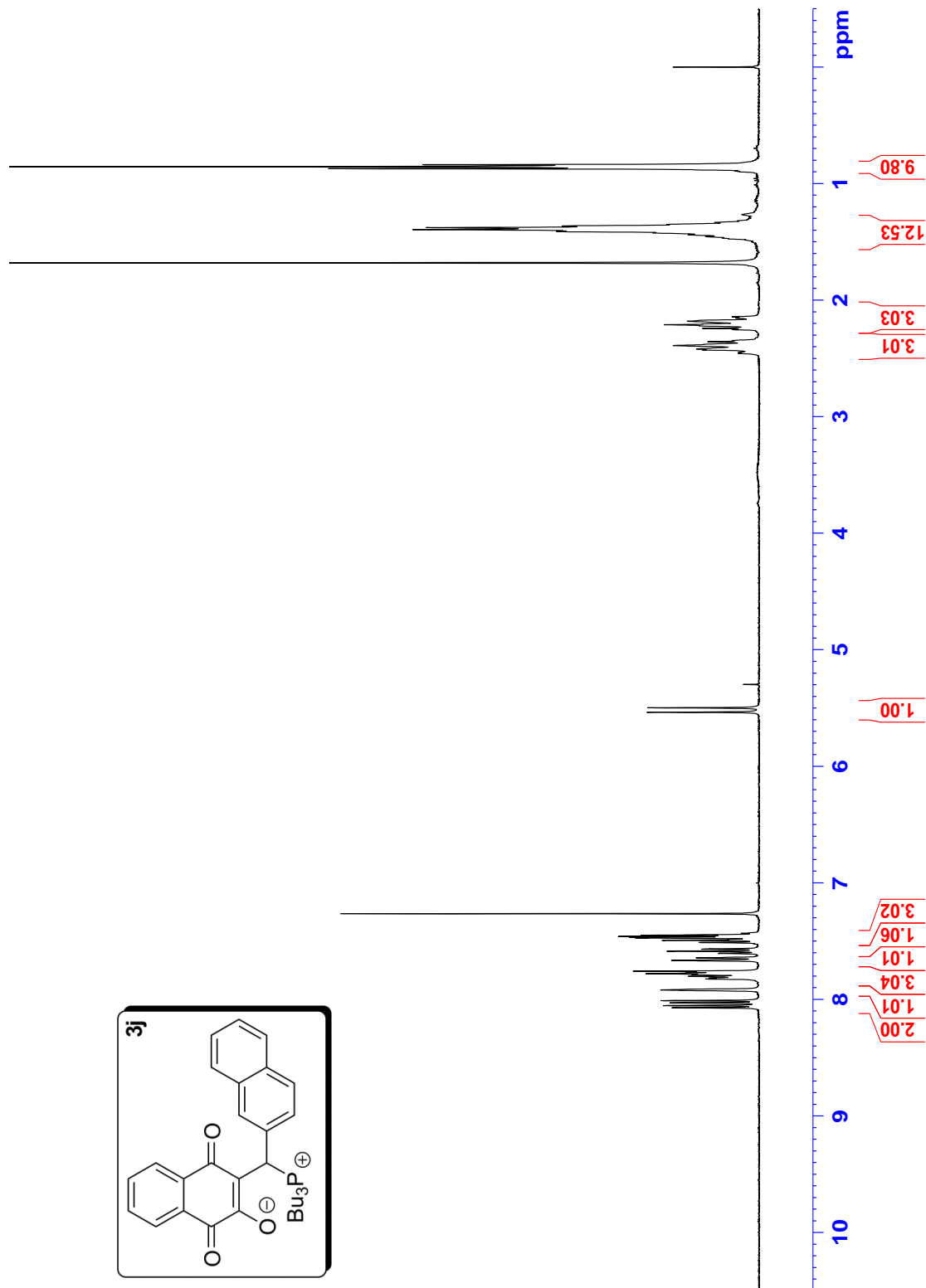
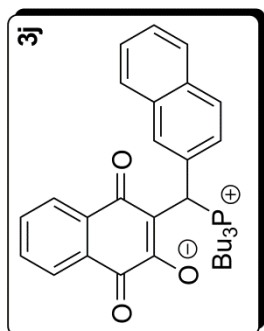


Current Data Parameters  
NAME Jimmy-13  
EXPNO 16  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110926  
Time 18.13  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 16  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 181  
DW 69.000 usec  
DE 6.50 usec  
TE 299.4 K  
D1 2.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300065 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00





```

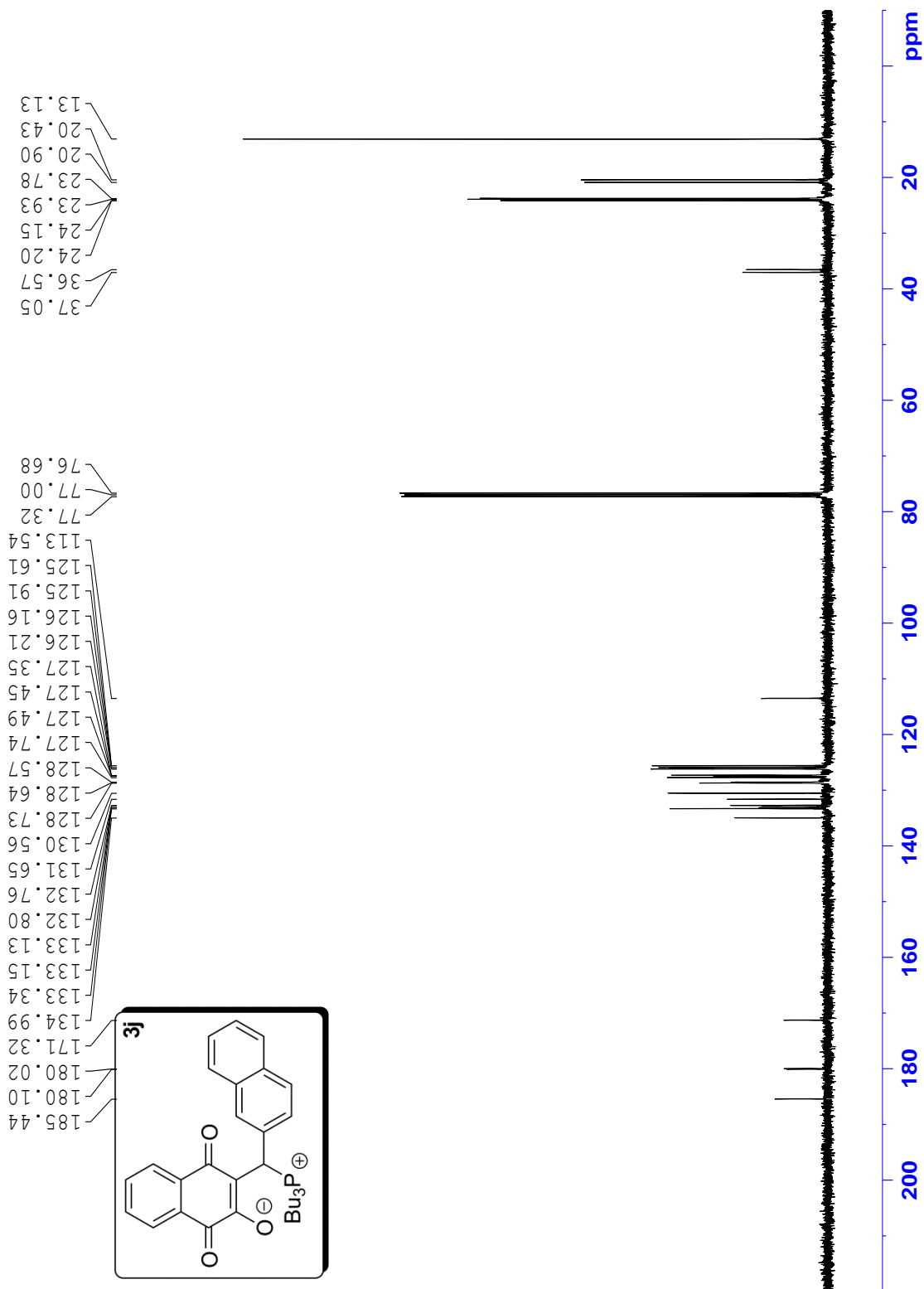
Current Data Parameters
NAME      Jimmy-13
EXPNO    18
PROCNO   1

F2 - Acquisition Parameters
Date_    20120423
Time_    12.52
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zgpg30
TD        32768
SOLVENT  CDCl3
NS        274
DS        4
SWH       24038.461 Hz
FIDRES    0.733536 Hz
AQ         0.6816244 sec
RG         4096
DE         20.800 usec
TE         6.50 usec
TE        299.7 K
D1         2.0000000 sec
D11        0.0300000 sec
TDO        1

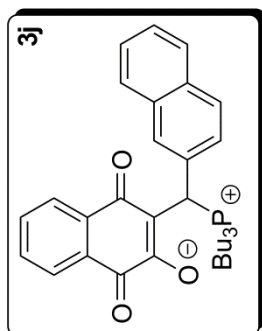
===== CHANNEL f1 =====
NUC1       13C
P1         8.90 usec
PL1        7.00 dB
SFO1       100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     90.00 usec
PL2        3.80 dB
PL12       21.60 dB
PL13       24.60 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI         32768
SF         100.6127862 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00
    
```



— 33.07



```
Current Data Parameters
NAME      Ken-P
EXPNO     38
PROCNO    1

F2 - Acquisition Parameters
Date_     20120423
Time      12.16
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg50
TD         65536
SOLVENT   CDCl3
NS         47
DS         0
SWH        64724.918 Hz
FIDRES     0.987624 Hz
AQ         0.5063156 sec
RG         20642.5
DW         7.725 usec
DE         6.50 usec
TE         299.3 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       131P
P1         10.60 usec
PL1        12.00 dB
SFO1       161.9674942 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
P2         90.00 usec
PL2        3.80 dB
PL12       21.60 dB
PL13       24.60 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI         32768
SF         161.9757192 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00
```

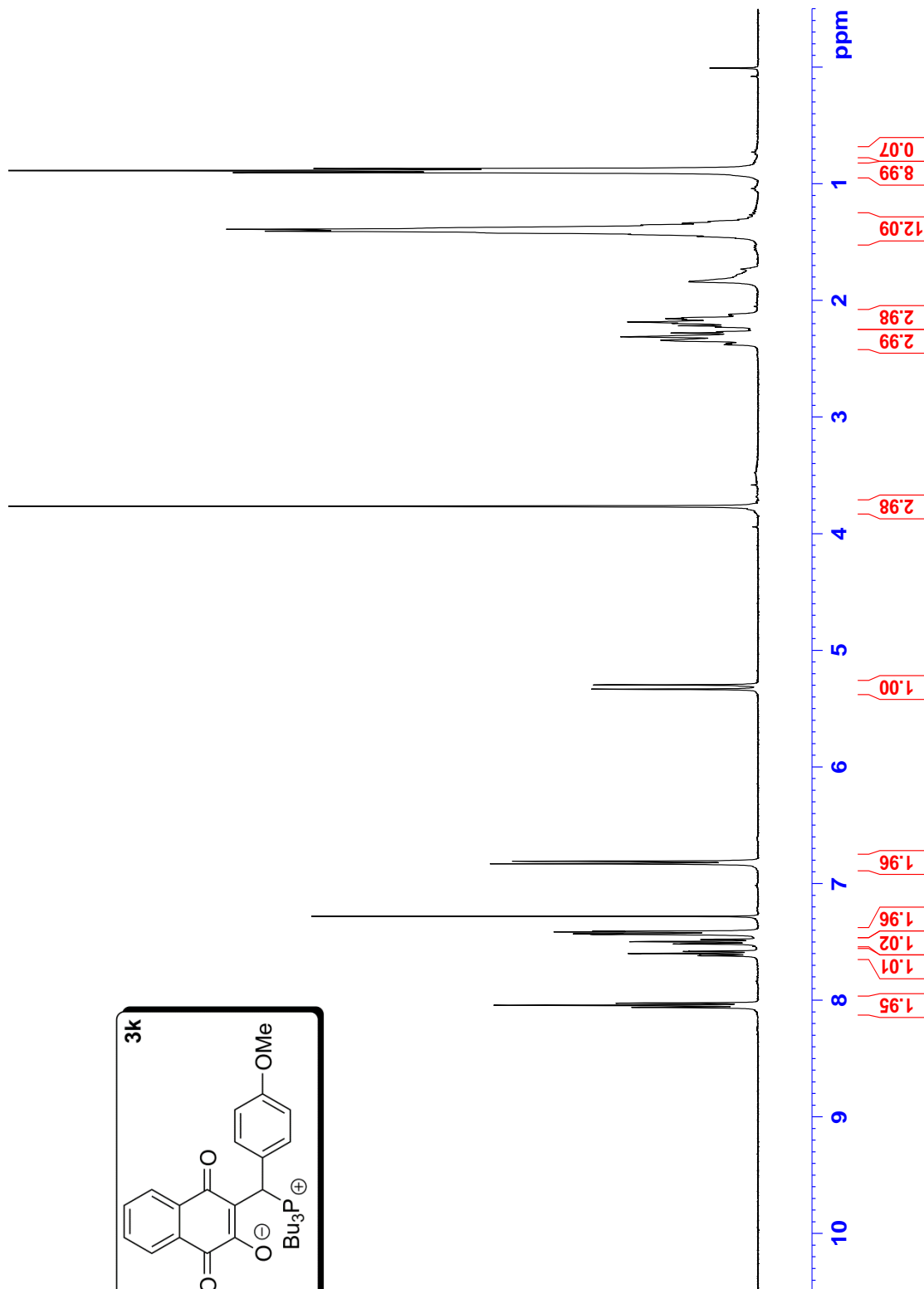
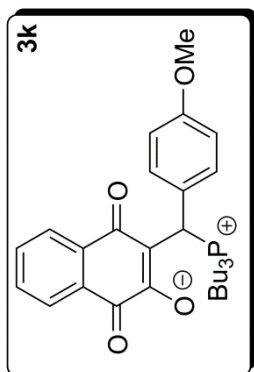


Current Data Parameters  
NAME ken284  
EXPNO 7  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20111117  
Time\_ 21.32  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDC13  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 128  
DW 69.000 usec  
DE 6.50 usec  
TE 299.3 K  
D1 2.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300000 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00



```

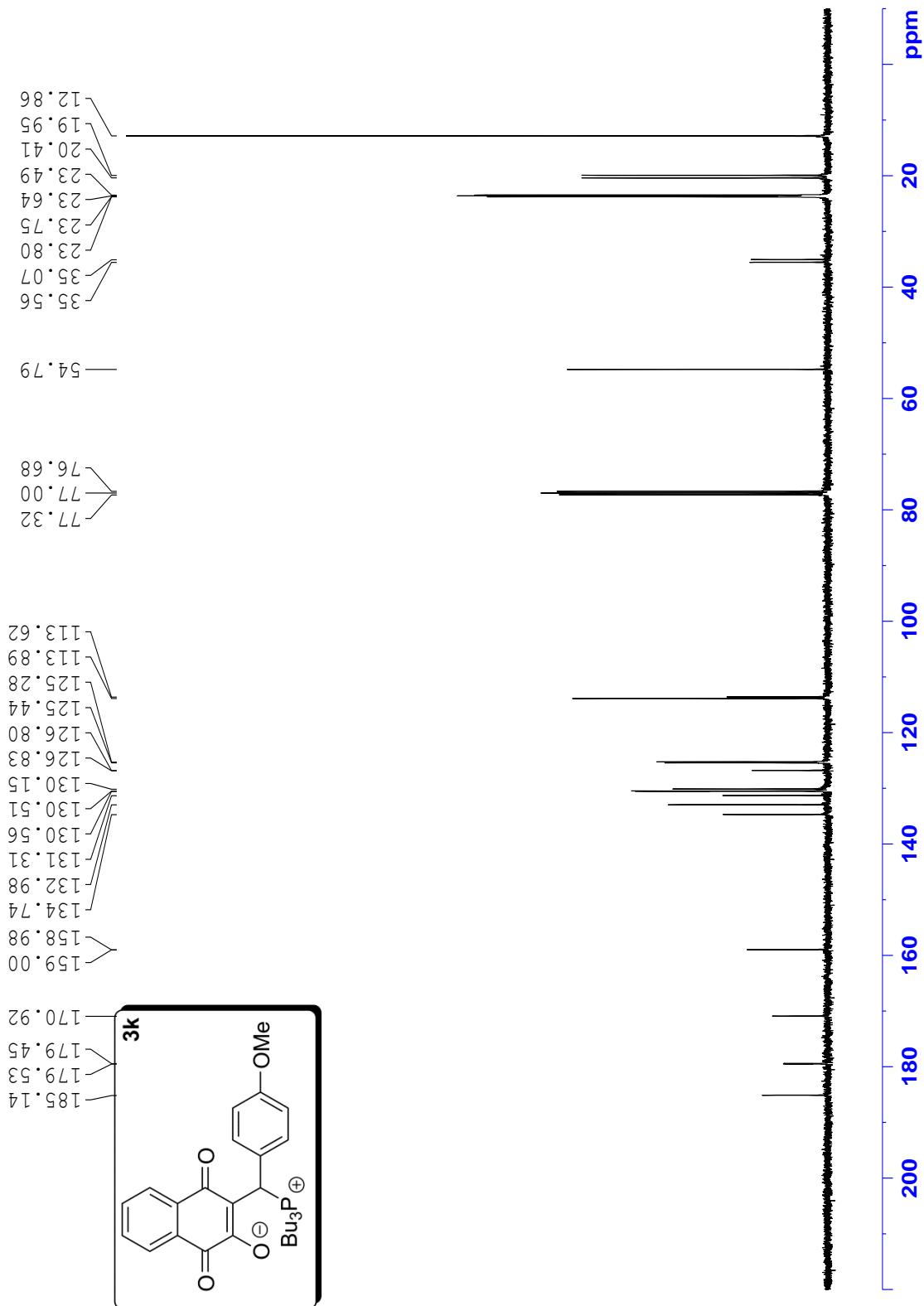
Current Data Parameters
NAME      ken284
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20120402
Time_    18.07
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zgpg30
TD       32768
SOLVENT  CDCl3
NS       150
DS       1
SWH      24038.461 Hz
FIDRES   0.733596 Hz
AQ       0.16816244 sec
RG       4096
DE       20.800 usec
TE       6.50 usec
TD0      298.4 K
D1       2.00000000 sec
D11      0.03000000 sec
TDO      1

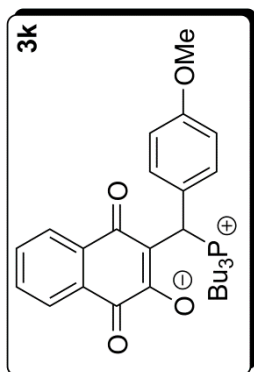
===== CHANNEL f1 =====
NUC1     13C
P1       8.90 usec
PL1      7.00 dB
SFO1     100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    90.00 usec
PL2      3.80 dB
PL12     21.60 dB
PL13     24.60 dB
SFO2     400.1316005 MHz

F2 - Processing parameters
SI       32768
SF       100.6126061 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.00
    
```



— 32.74



```
Current Data Parameters
NAME      Ken-P
EXPNO     44
PROCNO    1

F2 - Acquisition Parameters
Date_     20120423
Time      12.39
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg50
TD        65536
SOLVENT   CDCl3
NS        87
DS        0
SWH       64724.918 Hz
FIDRES    0.987624 Hz
AQ        0.5063156 sec
RG        20642.5
DW        7.725 usec
DE        6.50 usec
TE        299.6 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1

===== CHANNEL f1 =====
NUC1      131P
P1        10.60 usec
PL1       12.00 dB
SFO1      161.9674942 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     90.00 usec
PL2       3.80 dB
PL12      21.60 dB
PL13      24.60 dB
SFO2      400.1316005 MHz

F2 - Processing parameters
SI        32768
SF        161.9757192 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.00
```

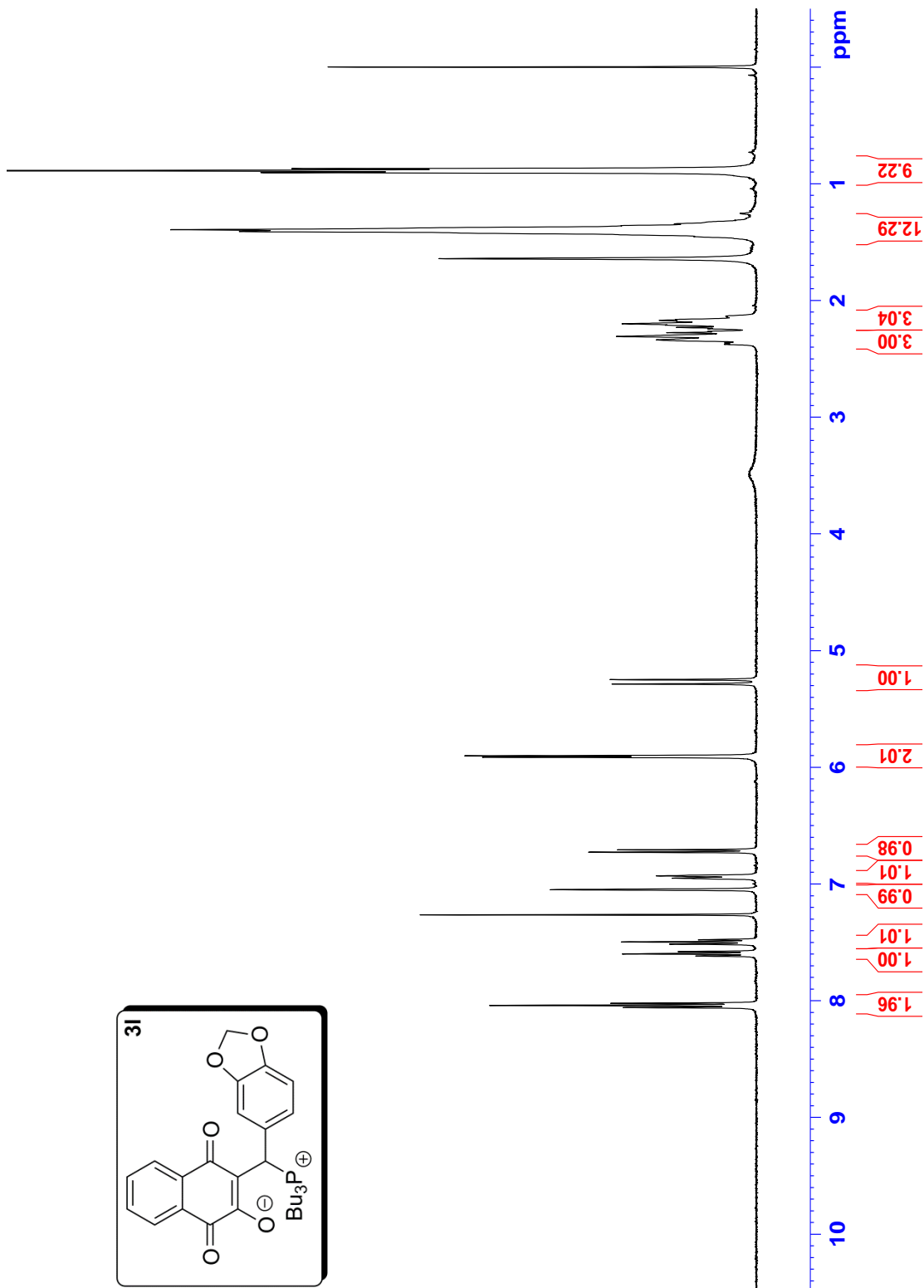
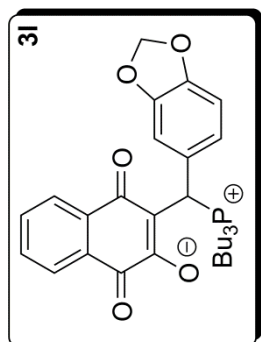


Current Data Parameters  
NAME Jimmy-30  
EXPNO 5  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120602  
Time 12.48  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDC13  
NS 32  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 181  
DW 69.000 usec  
DE 6.50 usec  
TE 298.7 K  
D1 2.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300068 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00



```

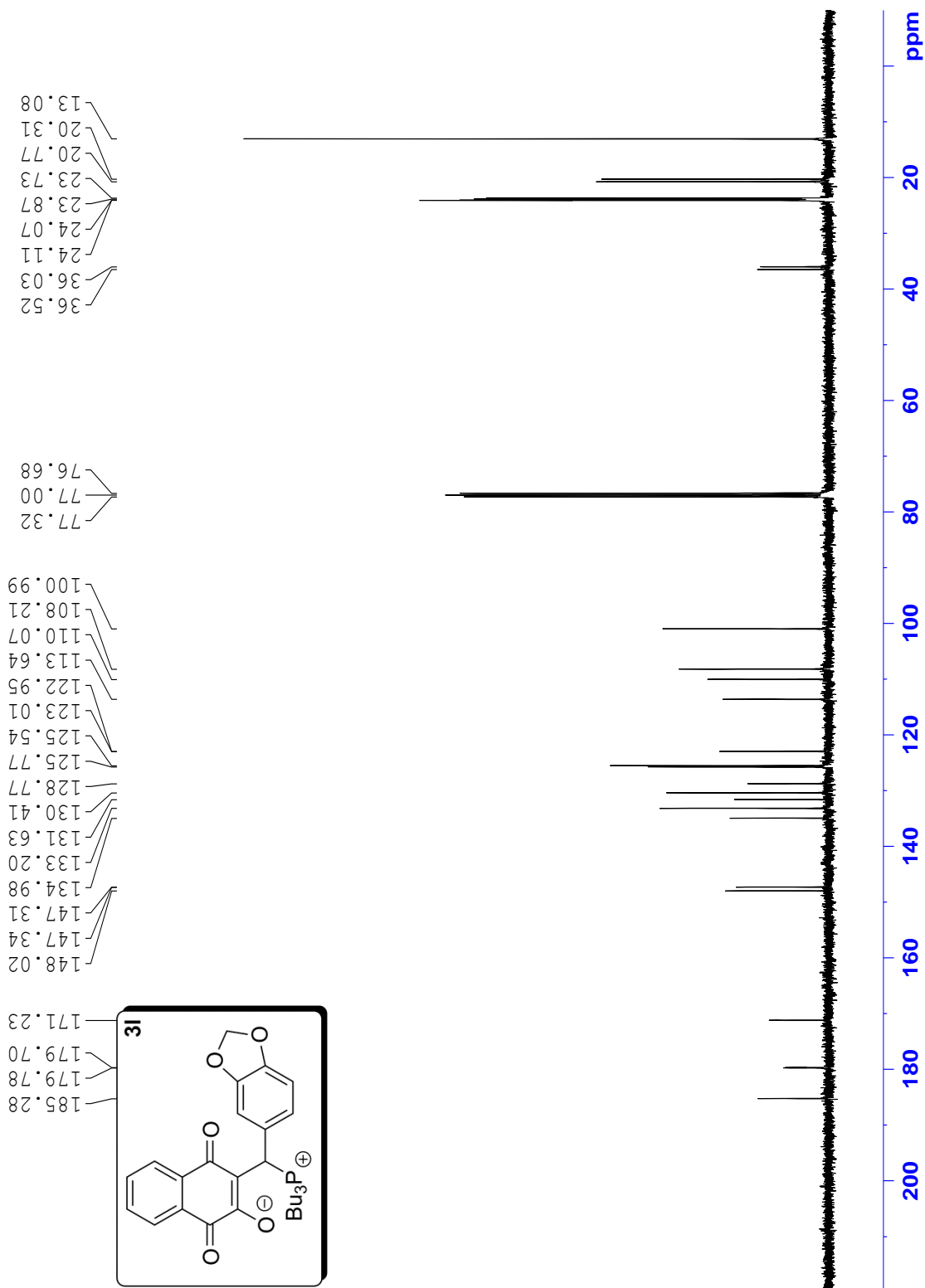
Current Data Parameters
NAME      Jimmy-30
EXPNO    4
PROCNO   1

F2 - Acquisition Parameters
Date_    20120402
Time_    19.00
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zgpg30
TD        32768
SOLVENT  CDCl3
NS        274
DS        4
SWH       24038.461 Hz
FIDRES    0.733596 Hz
AQ        0.6816244 sec
RG        4096
DE        20.800 usec
TE        301.0 K
D1        2.0000000 sec
D11       0.0300000 sec
TDO       1

===== CHANNEL f1 =====
NUC1      13C
P1        10.50 usec
PL1       7.00 dB
SFO1      100.6233325 MHz

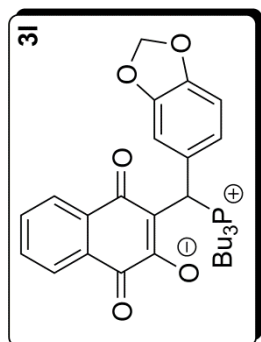
===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2      1H
PCPD2     90.00 usec
PL2       -0.60 dB
PL12      15.00 dB
PL13      18.00 dB
SFO2      400.1316005 MHz

F2 - Processing parameters
SI        32768
SF        100.6127873 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.00
    
```





— 32.79



```
Current Data Parameters
NAME      Ken-P
EXPNO     42
PROCNO    1

F2 - Acquisition Parameters
Date_     20120423
Time      12.31
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg50
TD         65536
SOLVENT   CDCl3
NS         72
DS         0
SWH        64724.918 Hz
FIDRES     0.987624 Hz
AQ         0.5063156 sec
RG         20642.5
DW         7.725 usec
DE         6.50 usec
TE         299.5 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       131P
P1         10.60 usec
PL1        12.00 dB
SFO1       161.9674942 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      90.00 usec
PL2        3.80 dB
PL12       21.60 dB
PL13       24.60 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI         32768
SF         161.9757192 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00
```

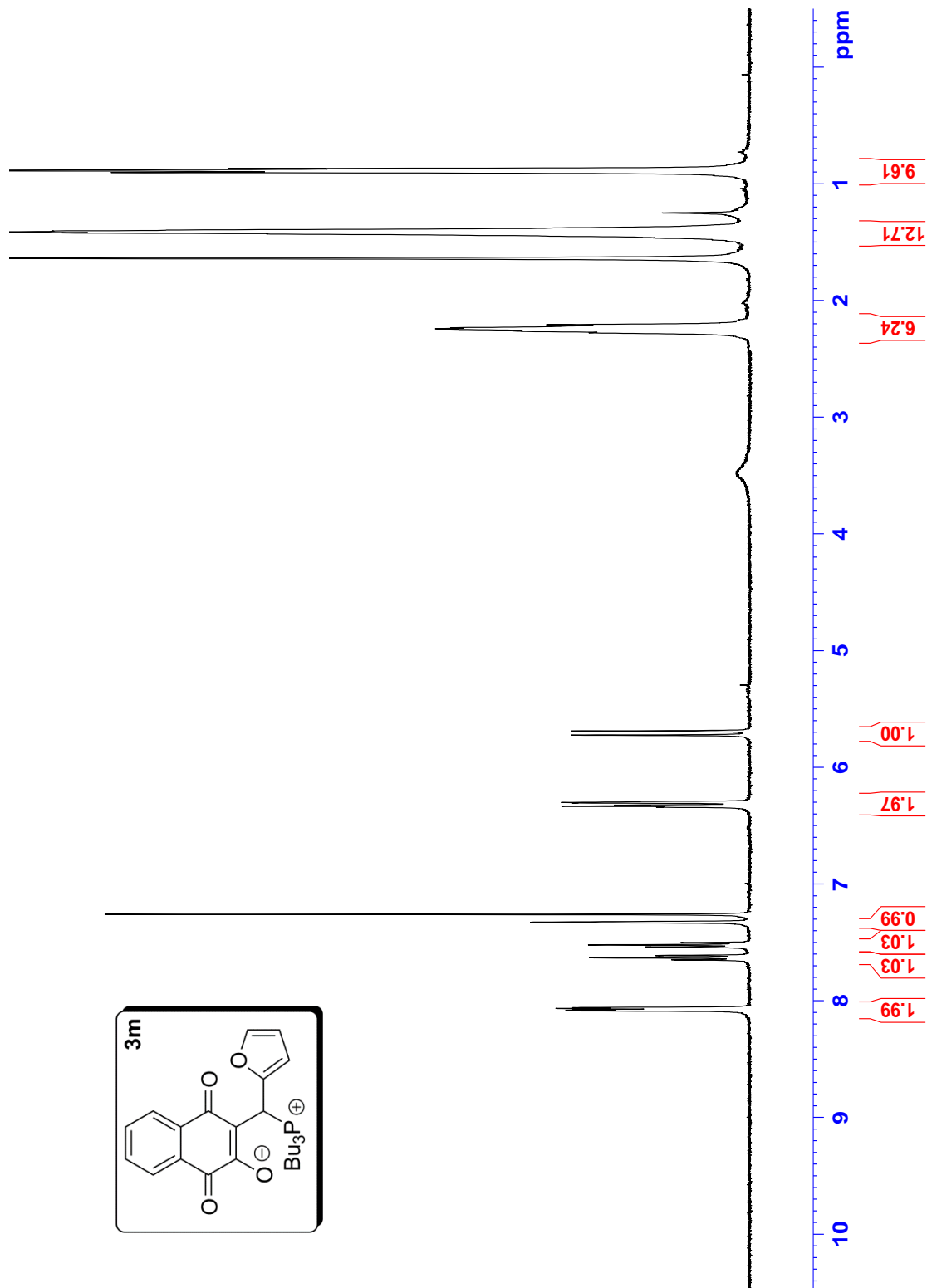
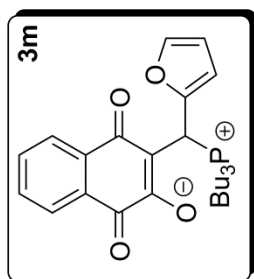


Current Data Parameters  
NAME Ken285  
EXPNO 10  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110514  
Time 16.12  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 16384  
SOLVENT CDC13  
NS 8  
DS 0  
SWH 6009.615 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 287.4  
DW 83.200 usec  
DE 6.50 usec  
TE 299.9 K  
D1 1.50000000 sec  
MCREST 0 sec  
MCWRK 0.01500000 sec

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.80 usec  
PL1 4.00 dB  
SFO1 400.1326008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300088 MHz  
WDW EM  
SSB 0  
LB 0.10 Hz  
GB 0  
PC 1.00



```

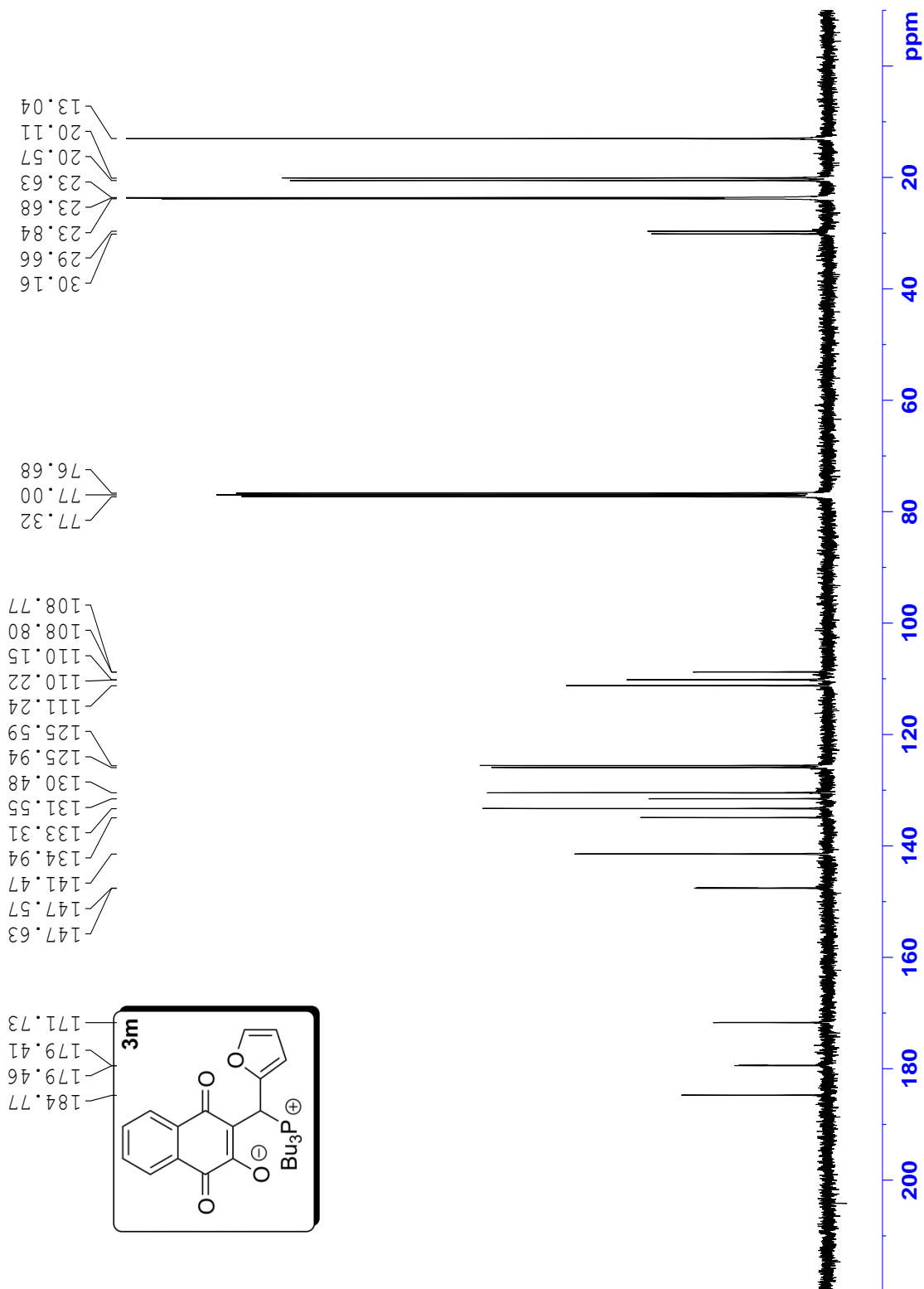
Current Data Parameters
NAME      ken285
EXPNO    12
PROCNO   1

F2 - Acquisition Parameters
Date_    20120311
Time_    16.32
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zgpg30
TD       32768
SOLVENT  CDCl3
NS       550
DS       24038.461 Hz
SWH      0.733536 Hz
FIDRES   0.16816244 sec
AQ       4096
RG       20.800 usec
DE       6.50 usec
TE       297.4 K
D1       2.00000000 sec
D11      0.03000000 sec
TDO      1

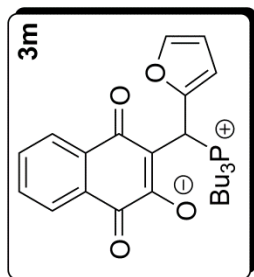
===== CHANNEL f1 =====
NUC1     13C
P1       8.90 usec
PL1      7.00 dB
SFO1     100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    90.00 usec
PL2      3.80 dB
PL12     21.60 dB
PL13     24.60 dB
SFO2     400.1316005 MHz

F2 - Processing parameters
SI       32768
SF       100.6127900 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.00
    
```



— 34.74



```
Current Data Parameters
NAME      Ken-P
EXPNO     12
PROCNO    1

F2 - Acquisition Parameters
Date_     20120315
Time      19.27
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg50
TD         65536
SOLVENT   CDCl3
NS         17
DS         0
SWH        64724.918 Hz
FIDRES     0.987624 Hz
AQ         0.5063156 sec
RG         4597.6
DW         7.725 usec
DE         6.50 usec
TE         300.6 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       131P
P1         10.60 usec
PL1        12.00 dB
SFO1       161.9674942 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
P2         90.00 usec
PL2        3.80 dB
PL12       21.60 dB
PL13       24.60 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI          32768
SF          161.9757164 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.00
```

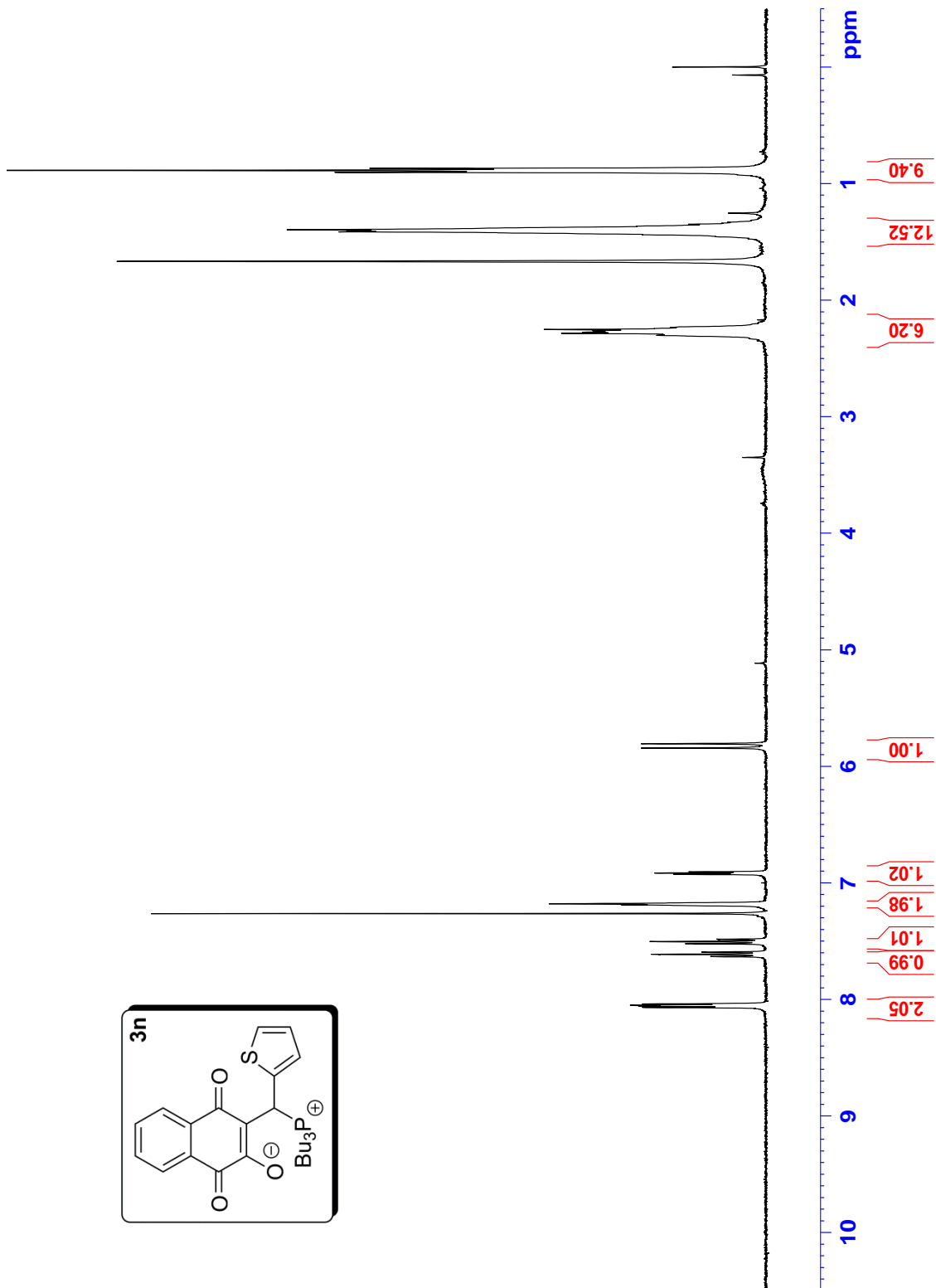
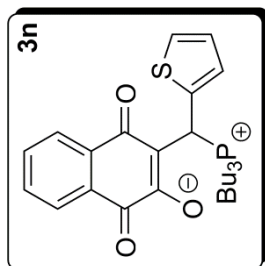


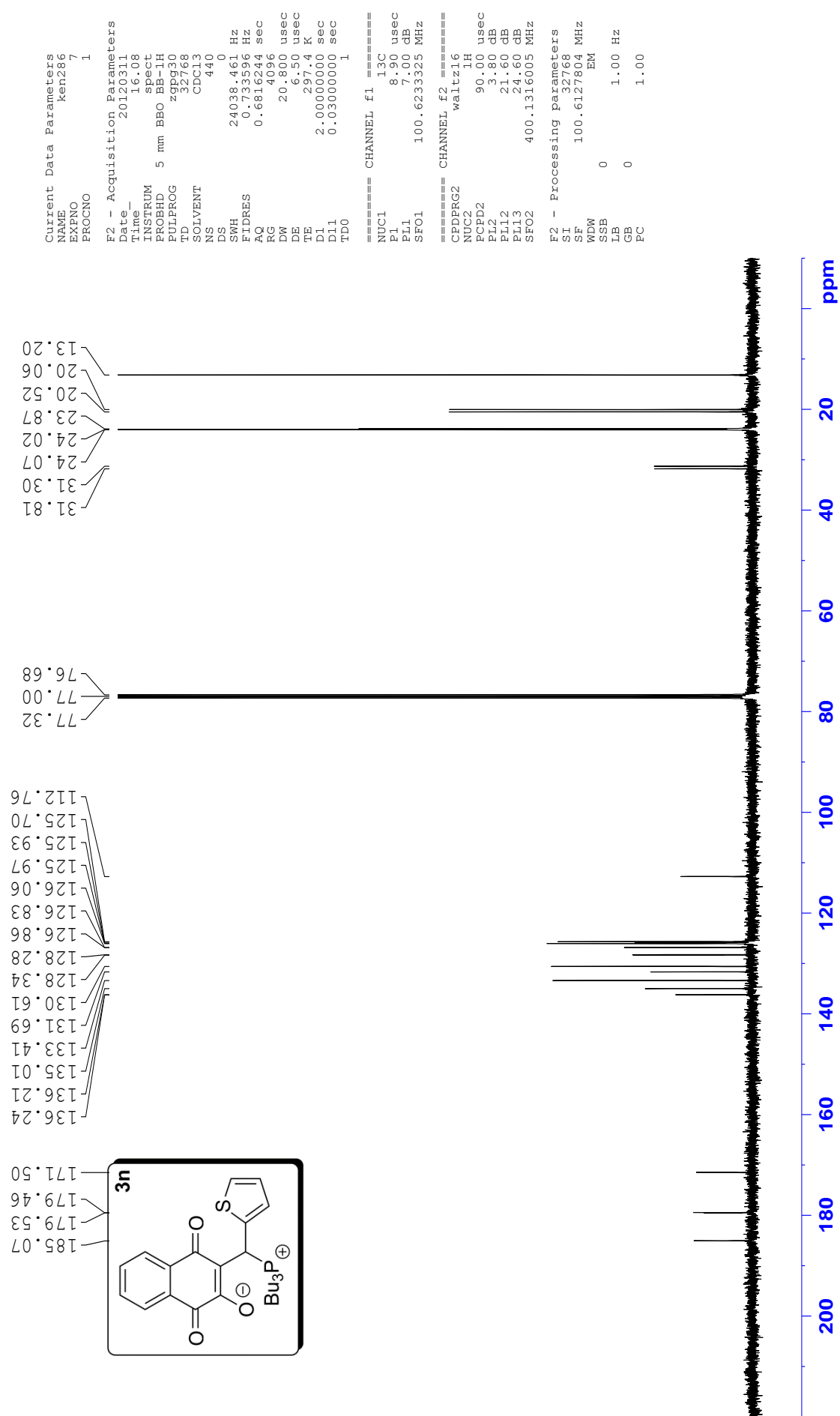
Current Data Parameters  
NAME ken286  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20111205  
Time\_ 16.58  
INSTRUM spect  
PROBHD 5 mm BBO BB-JH  
PULPROG zgpg30  
TD 32768  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 256  
DW 69.000 usec  
DE 6.50 usec  
TE 300.4 K  
D1 2.00000000 sec  
TD0 1

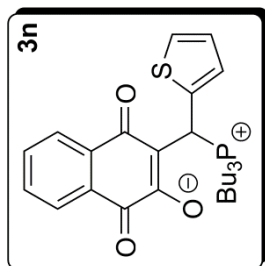
==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300070 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00





— 33.39



```
Current Data Parameters
NAME      Ken-P
EXPNO    10
PROCNO   1

F2 - Acquisition Parameters
Date_    20120315
Time     19.21
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zgpg50
TD       65536
SOLVENT  CDCl3
NS       23
DS       0
SWH      64724.918 Hz
FIDRES   0.987624 Hz
AQ       0.5063156 sec
RG       6502
DW       7.725 usec
DE       6.50 usec
TE       300.7 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1      31P
P1       10.60 usec
PL1      12.00 dB
SFO1     161.9674942 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2      1H
PCPD2    90.00 usec
PL2      3.80 dB
PL12     21.60 dB
PL13     24.60 dB
SFO2     400.1316005 MHz

F2 - Processing parameters
SI        32768
SF        161.9757164 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB         0
PC         1.00
```

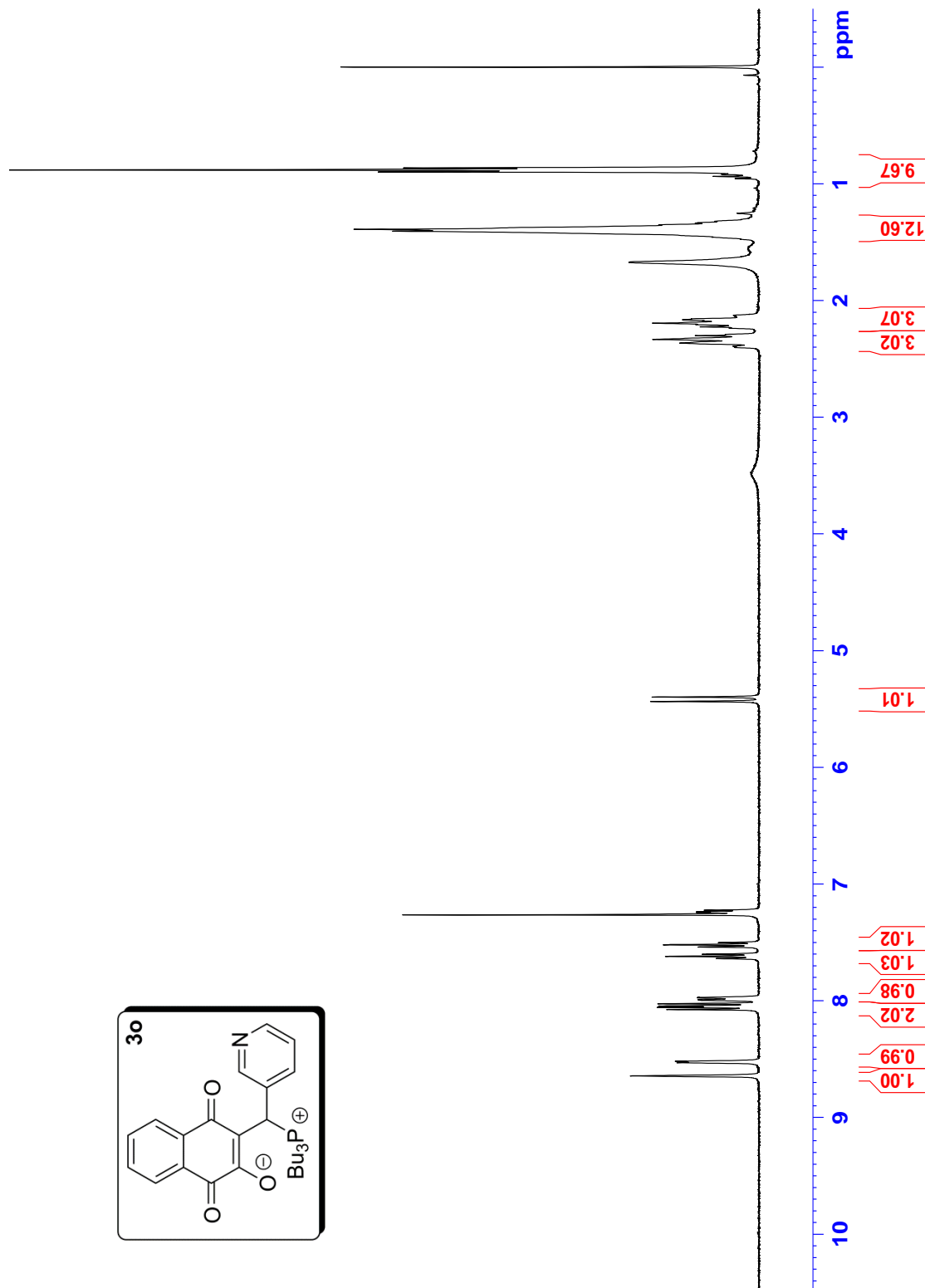
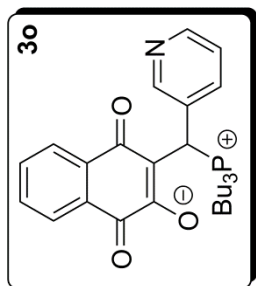


Current Data Parameters  
NAME ken385  
EXPNO 12  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120602  
Time\_ 12.53  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDC13  
NS 32  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 181  
DE 69.000 usec  
WE 6.50 usec  
TE 298.7 K  
D1 2.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300068 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00





```

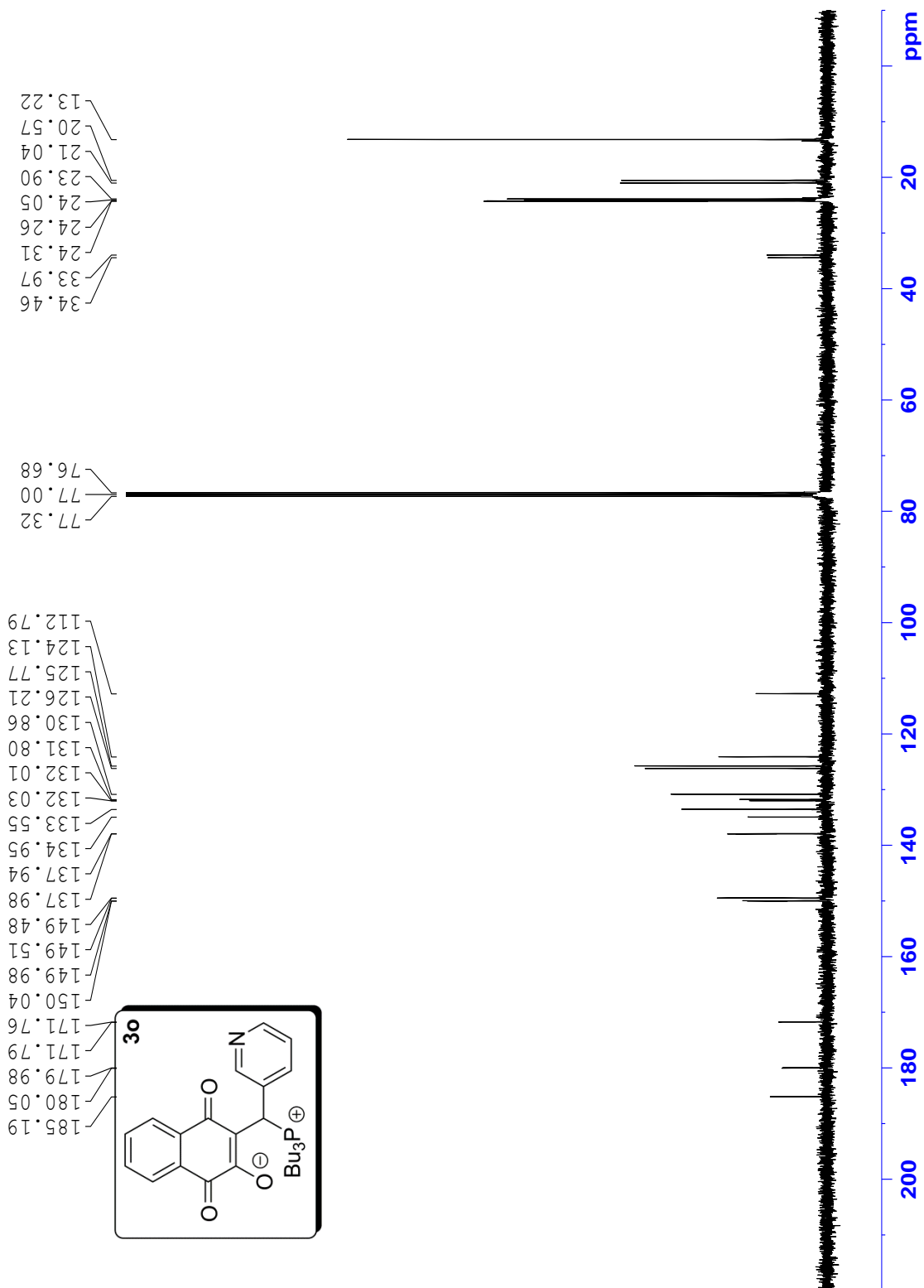
Current Data Parameters
NAME      ken385
EXPNO    11
PROCNO   1

F2 - Acquisition Parameters
Date_    20120402
Time_    18.26
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zgpg30
TD        32768
SOLVENT  CDCl3
NS        524
DS        24038.461 Hz
SWH       0.733536 Hz
FIDRES    0.16816244 sec
AQ        4096
RG        20.800 usec
DE        6.50 usec
TE        300.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TDO       1

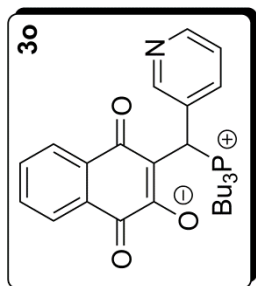
===== CHANNEL f1 =====
NUC1      13C
P1        10.50 usec
PL1       7.00 dB
SFO1      100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2      1H
PCPD2     90.00 usec
PL2       -0.60 dB
PL12      15.00 dB
PL13      18.00 dB
SFO2      400.1316005 MHz

F2 - Processing parameters
SI        32768
SF        100.6127716 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.00
    
```



— 33.85



```
Current Data Parameters
NAME      Ken-P
EXPNO     40
PROCNO    1

F2 - Acquisition Parameters
Date_     20120423
Time      12.21
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg50
TD         65536
SOLVENT   CDCl3
NS         66
DS         0
SWH        64724.918 Hz
FIDRES     0.987624 Hz
AQ         0.5063156 sec
RG         20642.5
DW         7.725 usec
DE         6.50 usec
TE         299.2 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       131P
P1         10.60 usec
PL1        12.00 dB
SFO1       161.9674942 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      90.00 usec
PL2        3.80 dB
PL12       21.60 dB
PL13       24.60 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI         32768
SF         161.9757192 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00
```

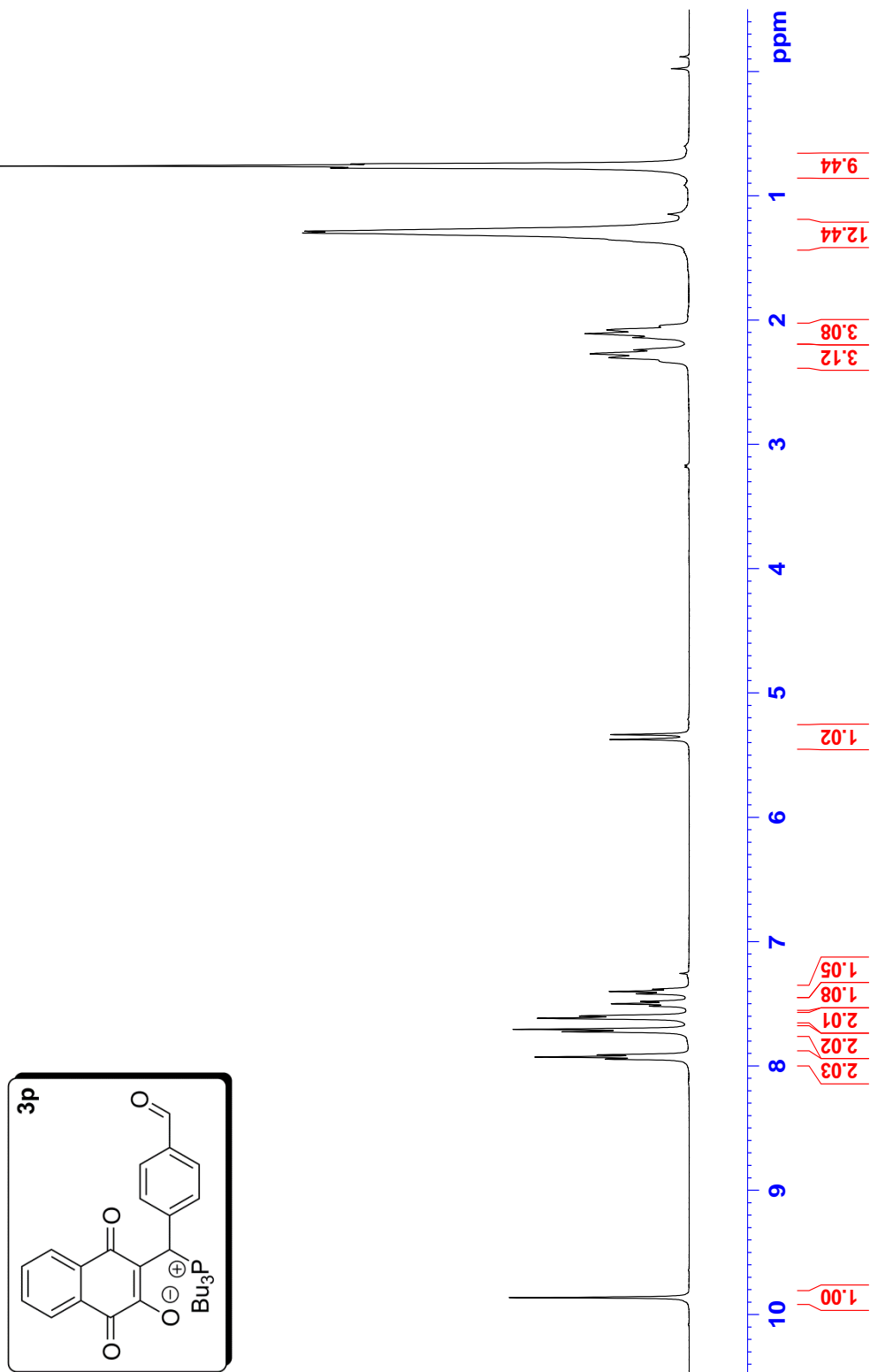


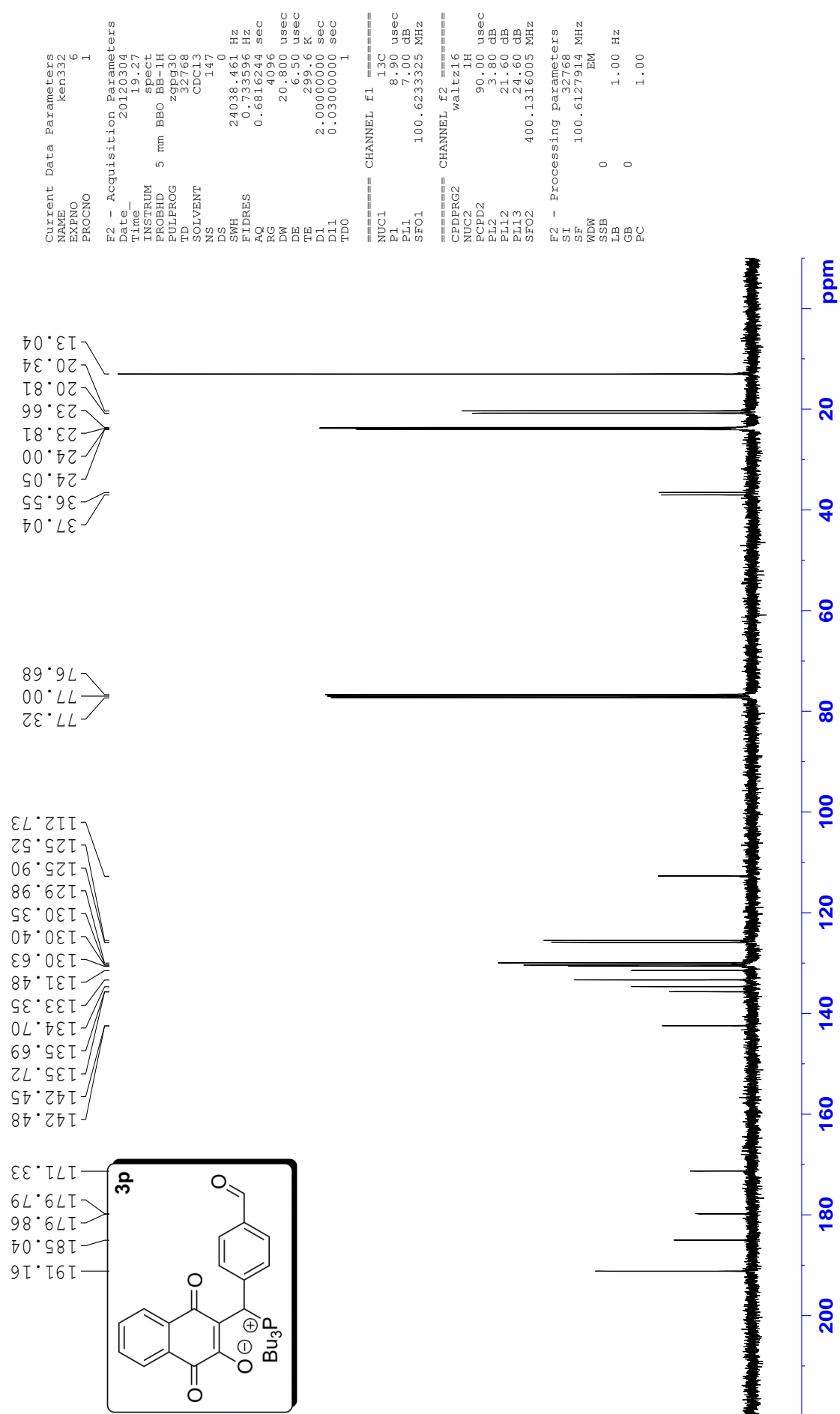
Current Data Parameters  
NAME ken332  
EXPNO 5  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120304  
Time 19.22  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDC13  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 32  
DM 69.000 usec  
DE 6.50 usec  
TE 299.2 K  
D1 2.00000000 sec  
TD0 1

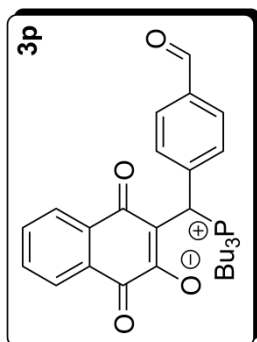
===== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300095 MHz  
WDW EM  
SSB 0 Hz  
LB 0  
GB 0  
PC 1.00





— 33.41



```
Current Data Parameters
NAME      Ken-P
EXPNO     30
PROCNO    1

F2 - Acquisition Parameters
Date_     20120310
Time      13.09
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg50
TD         65536
SOLVENT   CDCl3
NS         21
DS         0
SWH        64724.918 Hz
FIDRES     0.987624 Hz
AQ          0.5063156 sec
RG          5160.6
DW          7.725 usec
DE          6.50 usec
TE          298.3 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       31P
P1         10.60 usec
PL1        12.00 dB
SFO1       161.9674942 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      90.00 usec
PL2        3.80 dB
PL12       21.60 dB
PL13       24.60 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI         32768
SF         161.9757133 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00
```

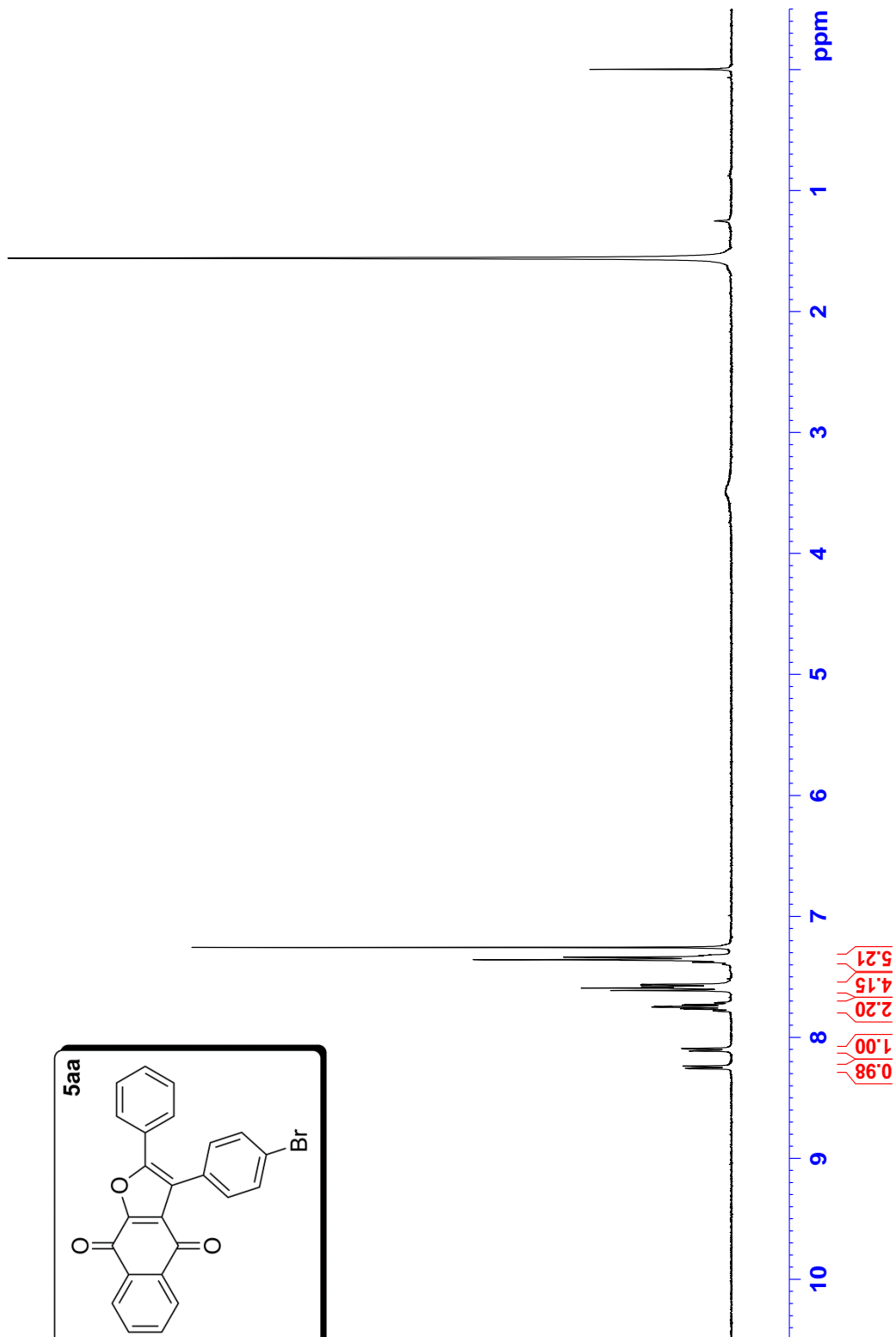
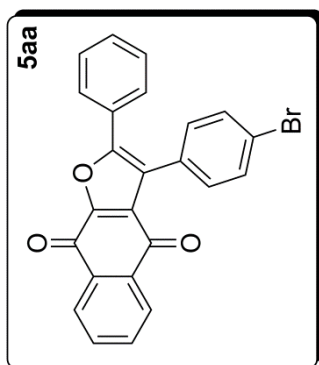


```
Current Data Parameters
NAME          ken340
EXPNO         5
PROCNO        1

F2 - Acquisition Parameters
Date_         20110922
Time          14.44
INSTRUM       spect
PROBHD        5 mm BBO BB-IH
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
NS            8
DS            0
SWH           7246.377 Hz
FIDRES        0.221142 Hz
AQ            2.2610421 sec
RG            181
DW            69.000 usec
DE            6.50 usec
TE            299.5 K
D1            2.0000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.00 usec
PL1           0 dB
SFO1          400.1324008 MHz

F2 - Processing parameters
SI            16384
SF            400.1300093 MHz
WDW           EM
SSB           0
LB            0 Hz
GB            0
PC            1.00
```



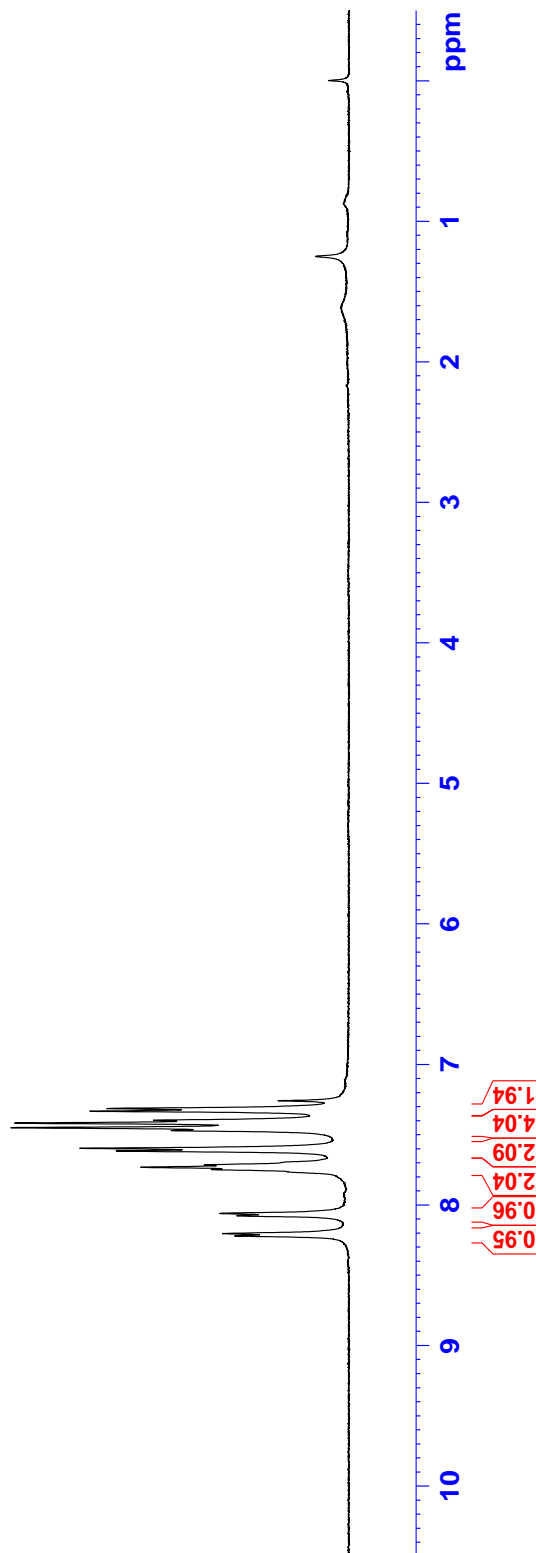
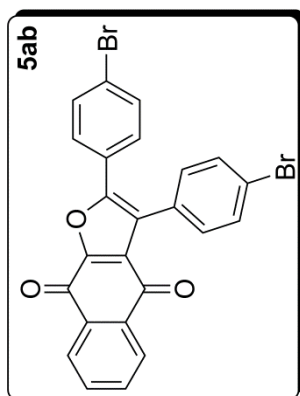


Current Data Parameters  
NAME ken373  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20111224  
Time 16.26  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
DS 4  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 161.3  
DW 69.000 usec  
DE 6.50 usec  
TE 297.4 K  
D1 2.0000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300081 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00

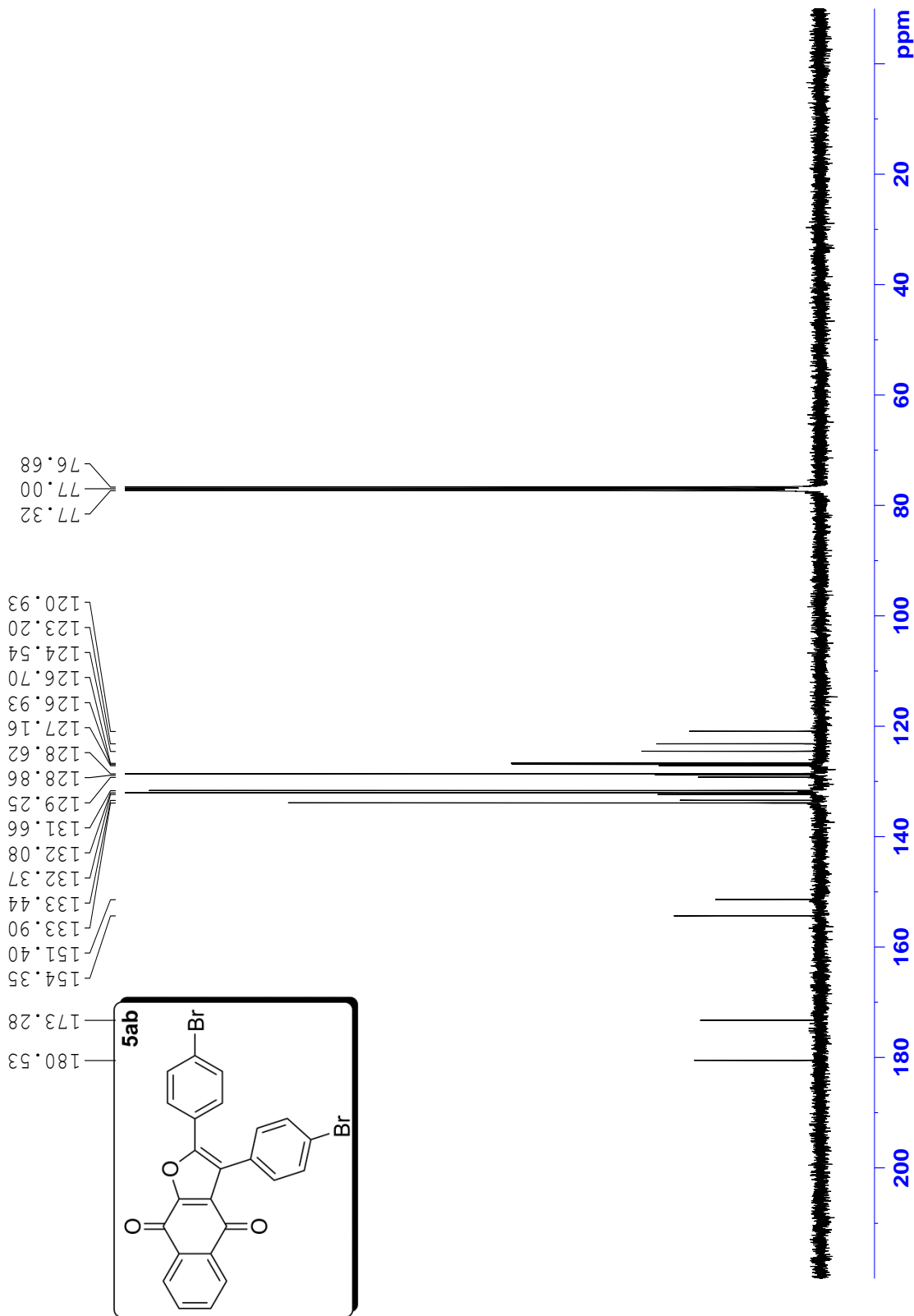




```
NAME          ken373
EXPNO         8
PROCNO        1
Date_         20111225
Time_         0.18
INSTRUM       spect
PROBHD        5 mm BBO BB-IH
PULPROG       zgpg30
TD            32768
SOLVENT       CDCl3
NS            752
DS            0
SMH           24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.681642 sec
RG            612
DE            20.800 usec
TE            300.3 K
D1            2.0000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            10.50 usec
PL1           7.00 dB
SFO1          100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2       waltzi6
NUC2          1H
PCPD2         90.00 usec
PL2           -0.60 dB
PLI2          15.00 dB
PLI3          18.00 dB
SFO2          400.1316005 MHz
SI            32768
SF            100.6127697 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.00
```

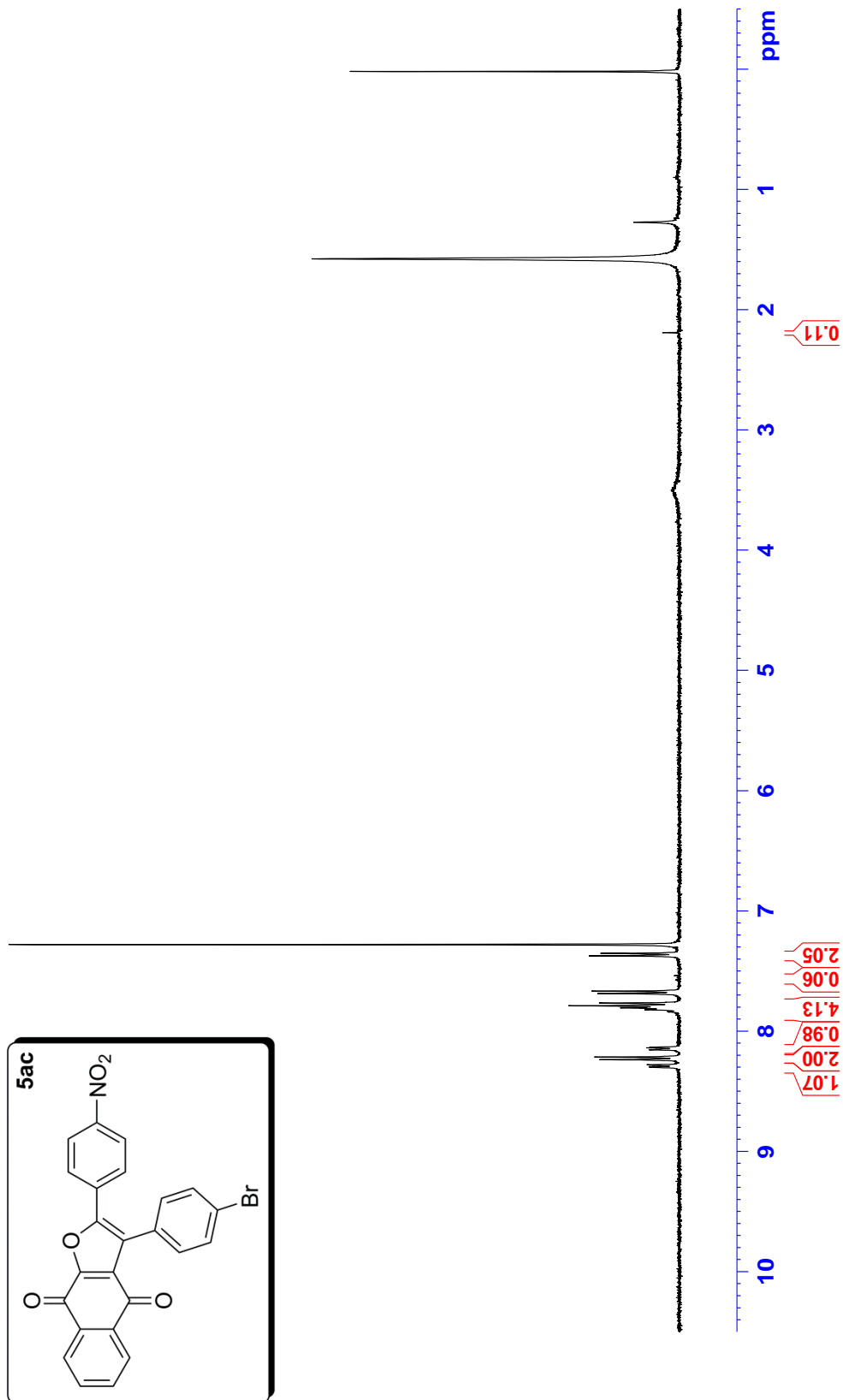


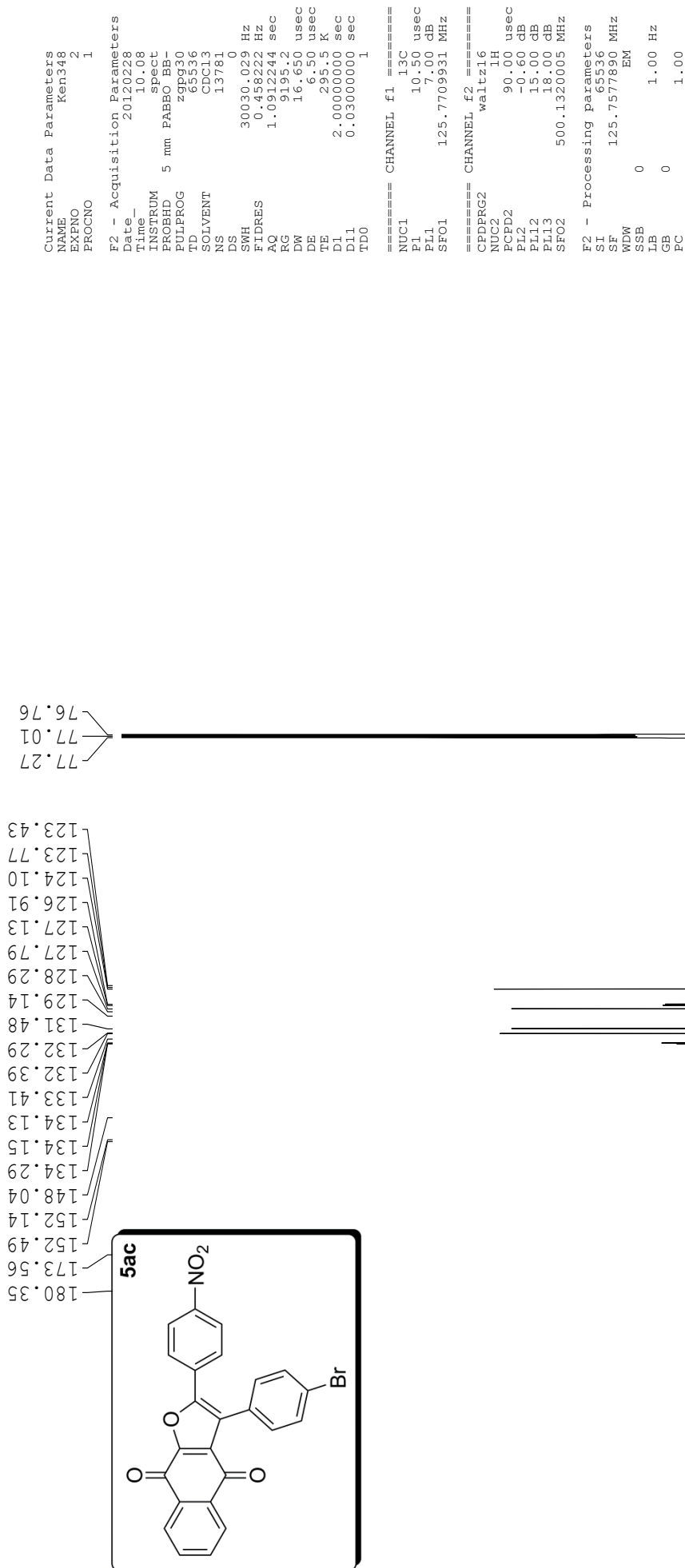
Current Data Parameters  
NAME ken348  
EXPNO 14  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120102  
Time 22.43  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDC13  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 362  
DW 69.000 usec  
DE 6.50 usec  
TE 298.5 K  
D1 2.0000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300000 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00



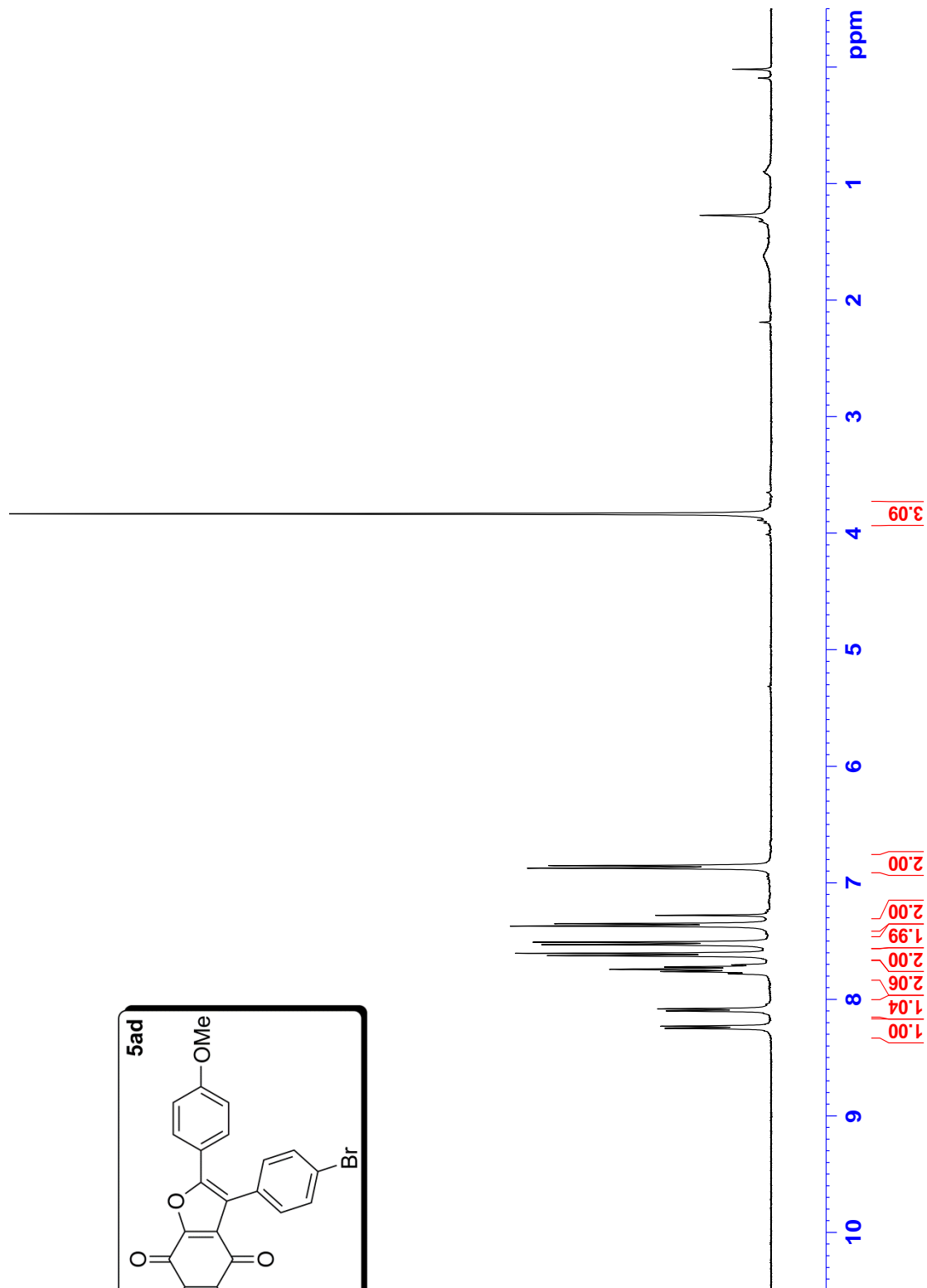
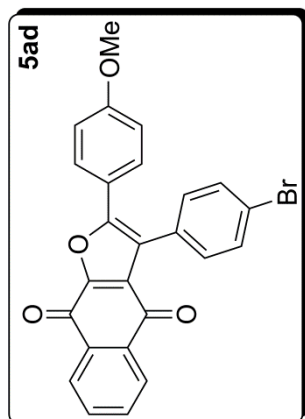


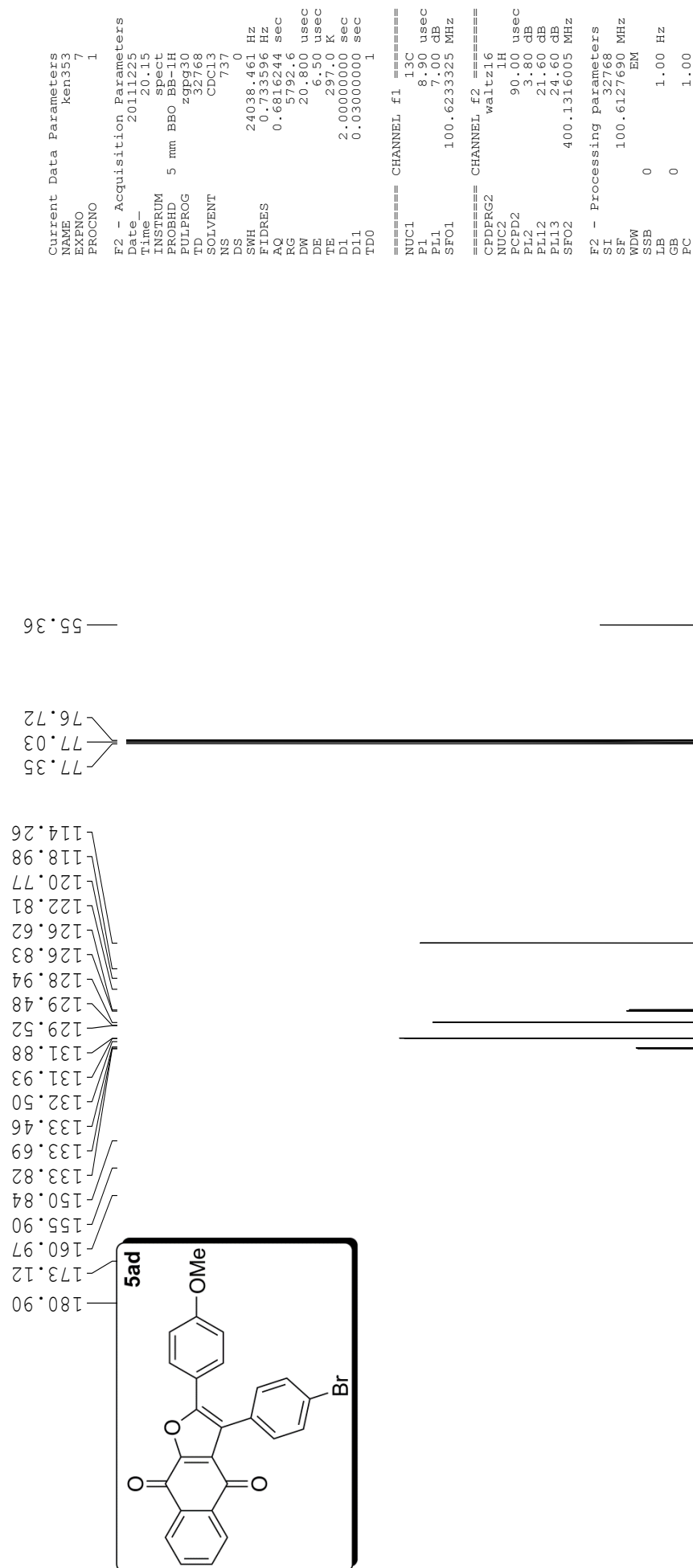
```
Current Data Parameters
NAME          Ken353
EXPNO        4
PROCNO       1

F2 - Acquisition Parameters
Date_        20111224
Time_        22.16
INSTRUM      spect
PROBHD       5 mm BBO BB-IH
PULPROG      zg30
TD           32768
SOLVENT      CDCl3
NS           4
DS           0
SWH          7246.377 Hz
FIDRES       0.221142 Hz
AQ           2.2610421 sec
RG           161
RG           161
DW           69.000 usec
DE           6.50 usec
TE           297.4 K
D1           2.00000000 sec
TD0          1

===== CHANNEL f1 =====
NUC1         1H
P1           11.70 usec
PL1         4.00 dB
SFO1        400.1324008 MHz

F2 - Processing parameters
SI           16384
SF          400.1300000 MHz
WDW          EM
SSB          0 Hz
LB           0
GB           0
PC           1.00
```



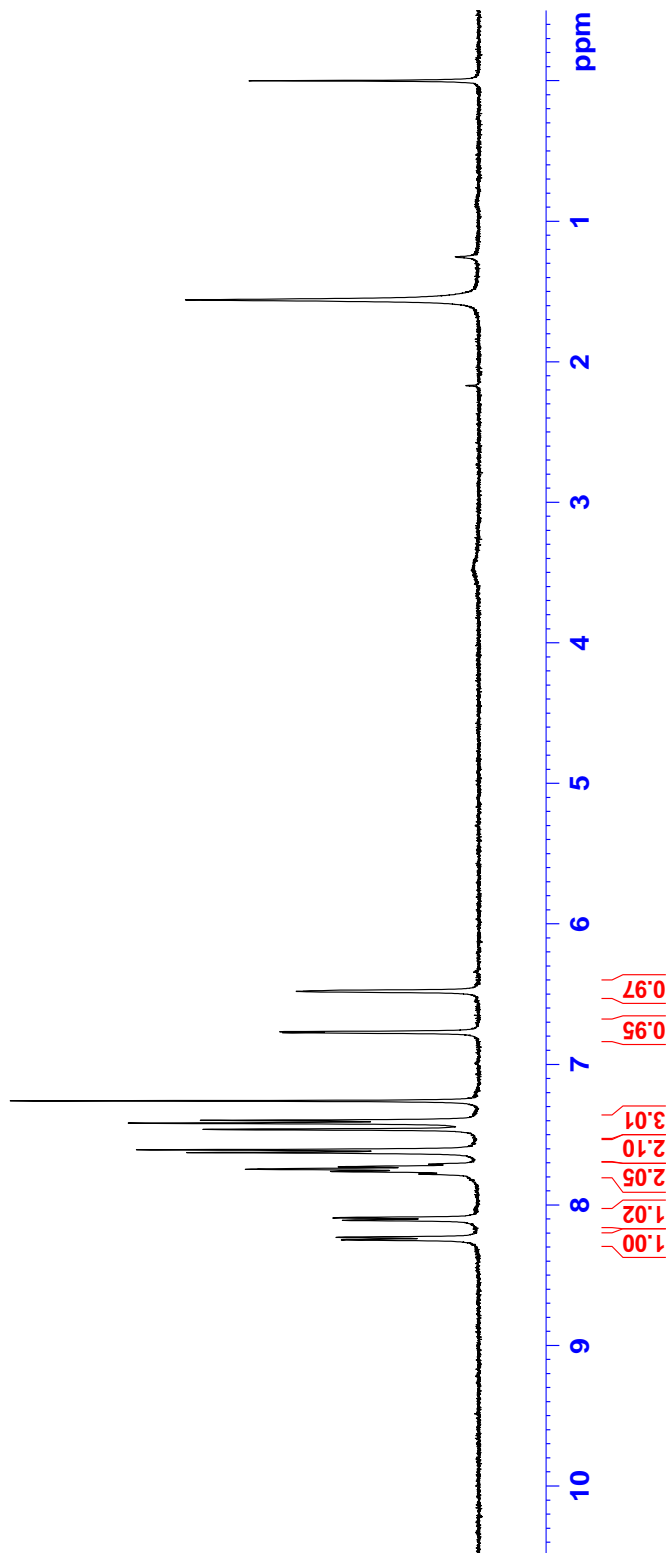
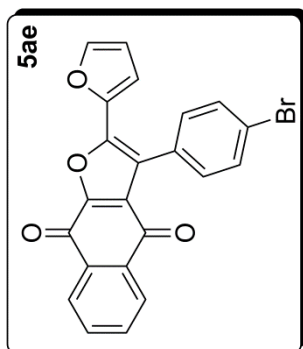


Current Data Parameters  
NAME ken374  
EXPNO 12  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20111230  
Time 23.00  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDC13  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 322.5  
DW 69.000 usec  
DE 6.50 usec  
TE 300.0 K  
D1 2.0000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300080 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00



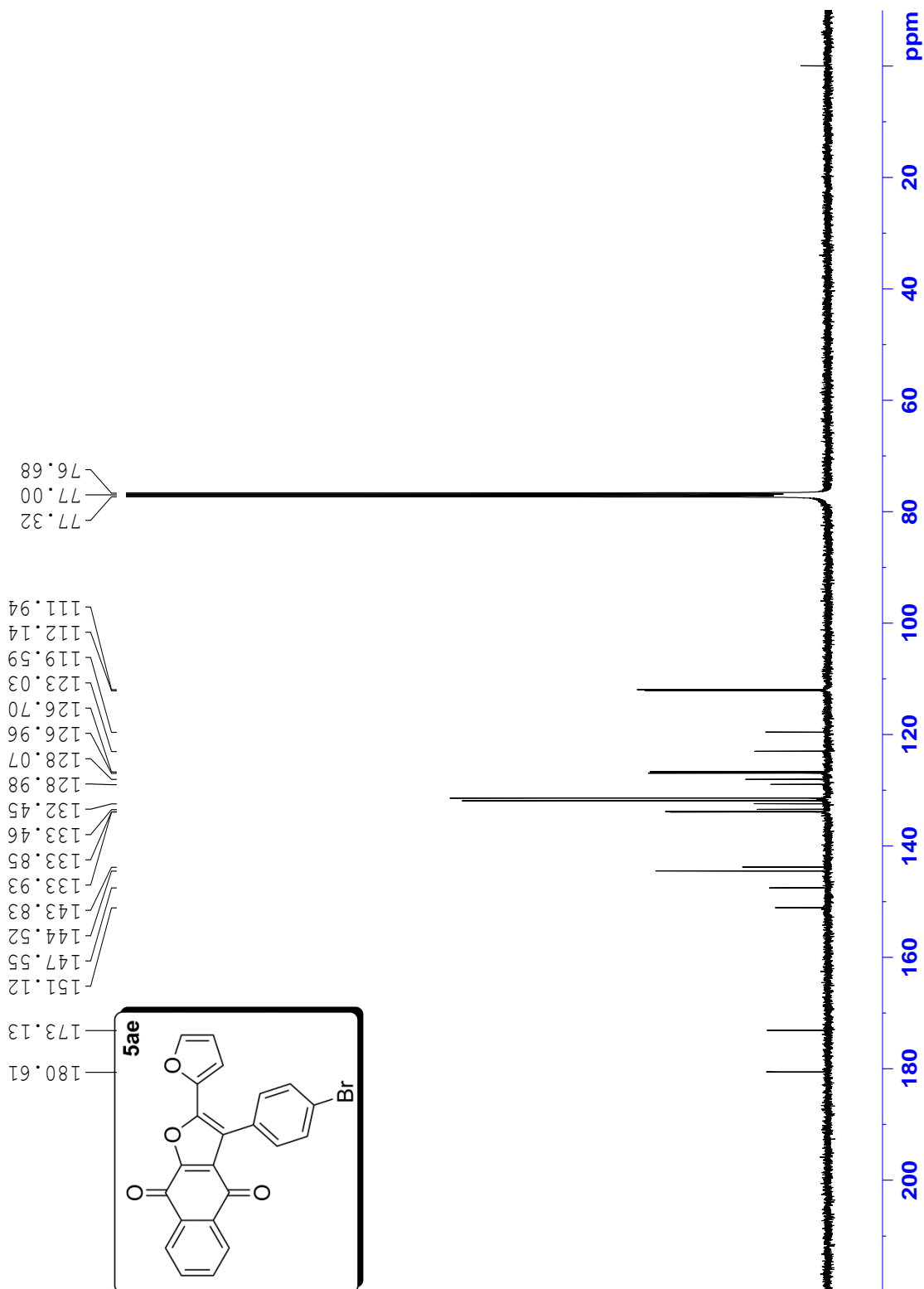
```
Current Data Parameters
NAME      ken374
EXPNO    13
PROCNO   1

F2 - Acquisition Parameters
Date_    20111230
Time_    23.50
INSTRUM  spect
PROBHD   5 mm BBO BB-IH
PULPROG  zgpg30
TD       32768
SOLVENT  CDCl3
NS       11746
DS       0
SWH      24038.461 Hz
FIDRES   0.733536 Hz
AQ       0.6816244 sec
RG       9195.2
DW       20.800 usec
DE       6.50 usec
TE       300.8 K
D1       2.00000000 sec
D11      0.03000000 sec
TDO      1

===== CHANNEL f1 =====
NUC1     13C
P1       8.90 usec
PL1      7.00 dB
SFO1     100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    90.00 usec
PL2      3.80 dB
PL12     21.60 dB
PL13     24.60 dB
SFO2     400.1316005 MHz

F2 - Processing Parameters
SI       32768
SF       100.6127650 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.00
```

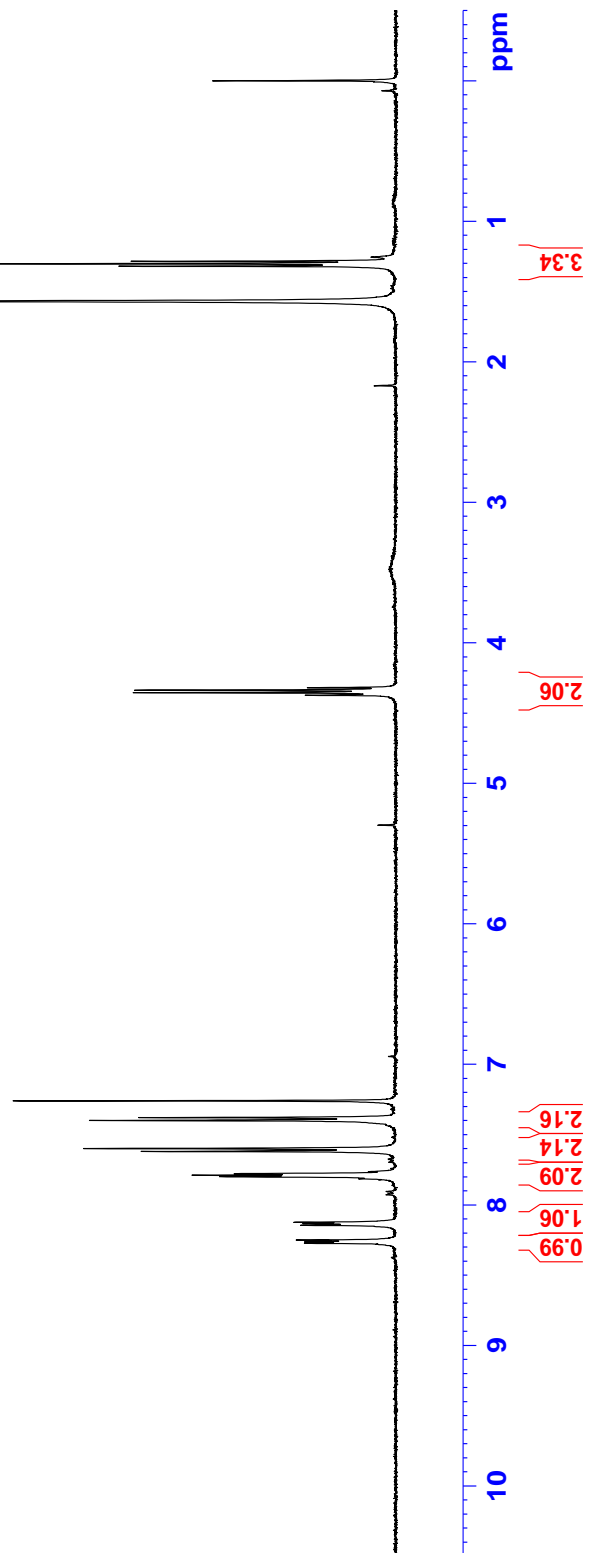
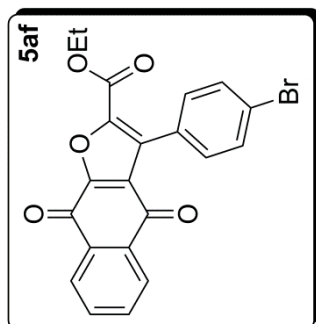


Current Data Parameters  
NAME ken375  
EXPNO 12  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120221  
Time 21.42  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 228.1  
DW 69.000 usec  
DE 6.50 usec  
TE 299.5 K  
D1 2.0000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300082 MHz  
WDW EM  
SSB 0 Hz  
LB 0  
GB 0  
PC 1.00





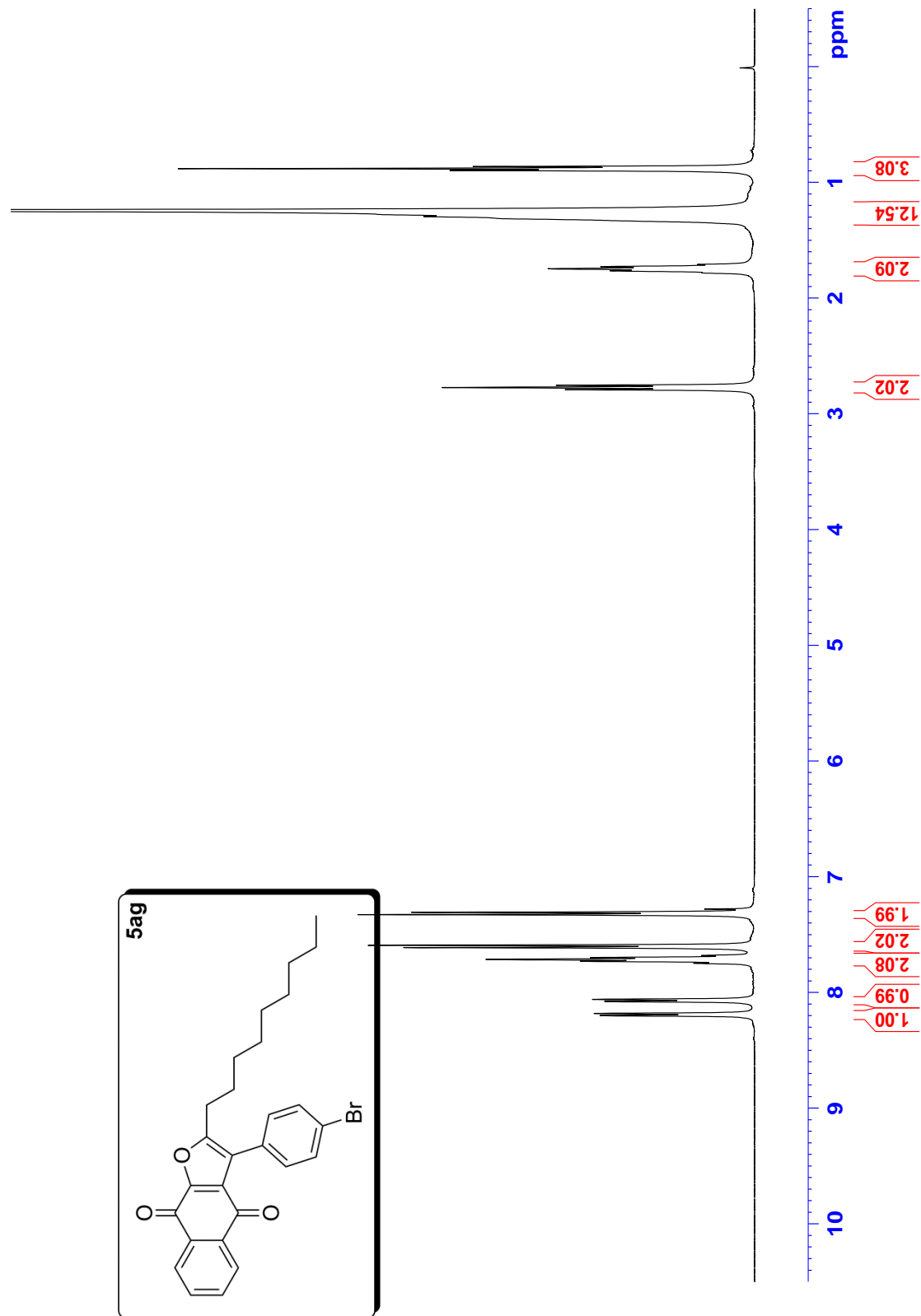


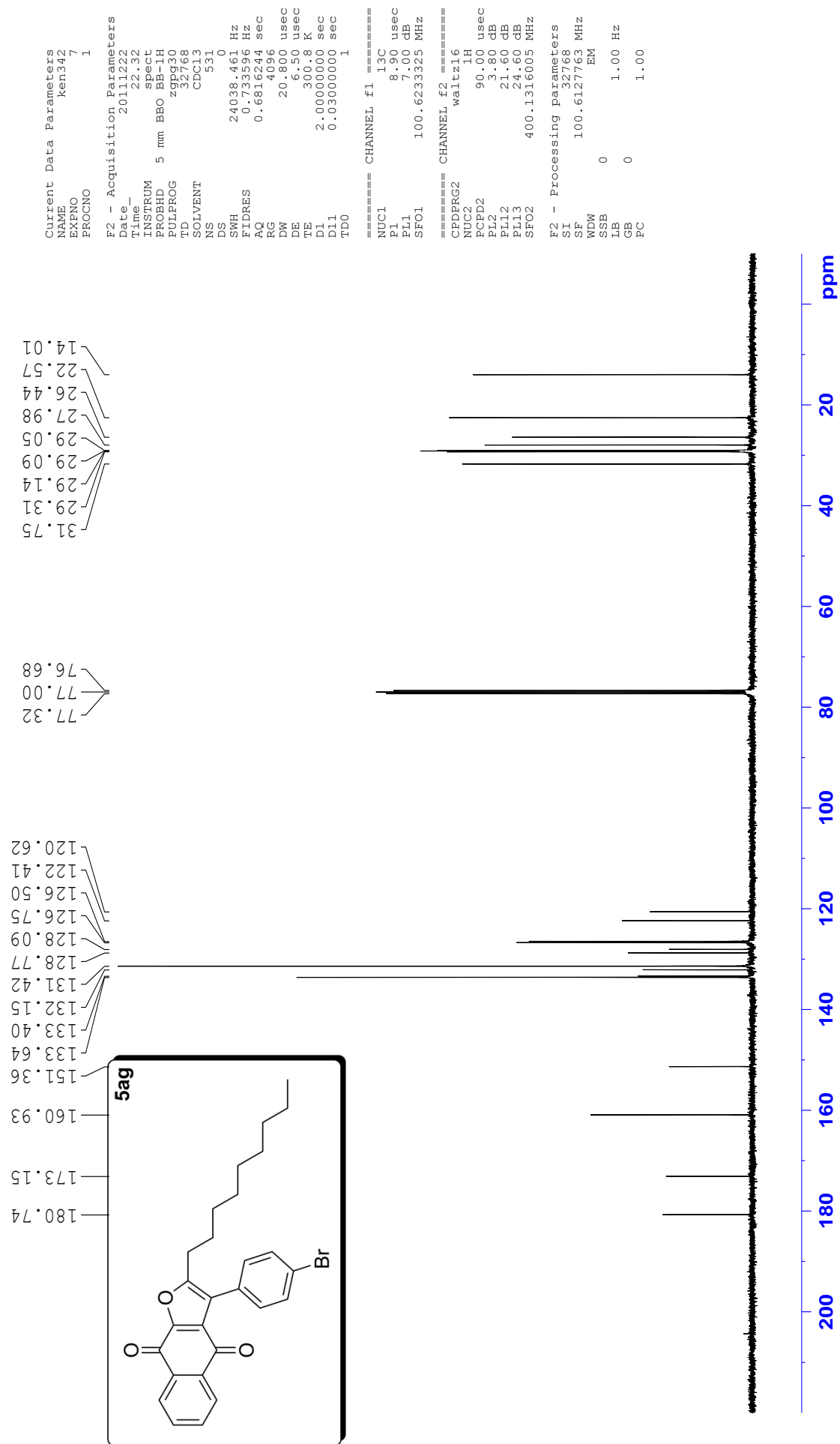
Current Data Parameters  
NAME ken342  
EXPNO 6  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20111222  
Time 22.14  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDC13  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 40.3  
DW 69.000 usec  
DE 6.50 usec  
TE 300.2 K  
D1 2.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300000 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00



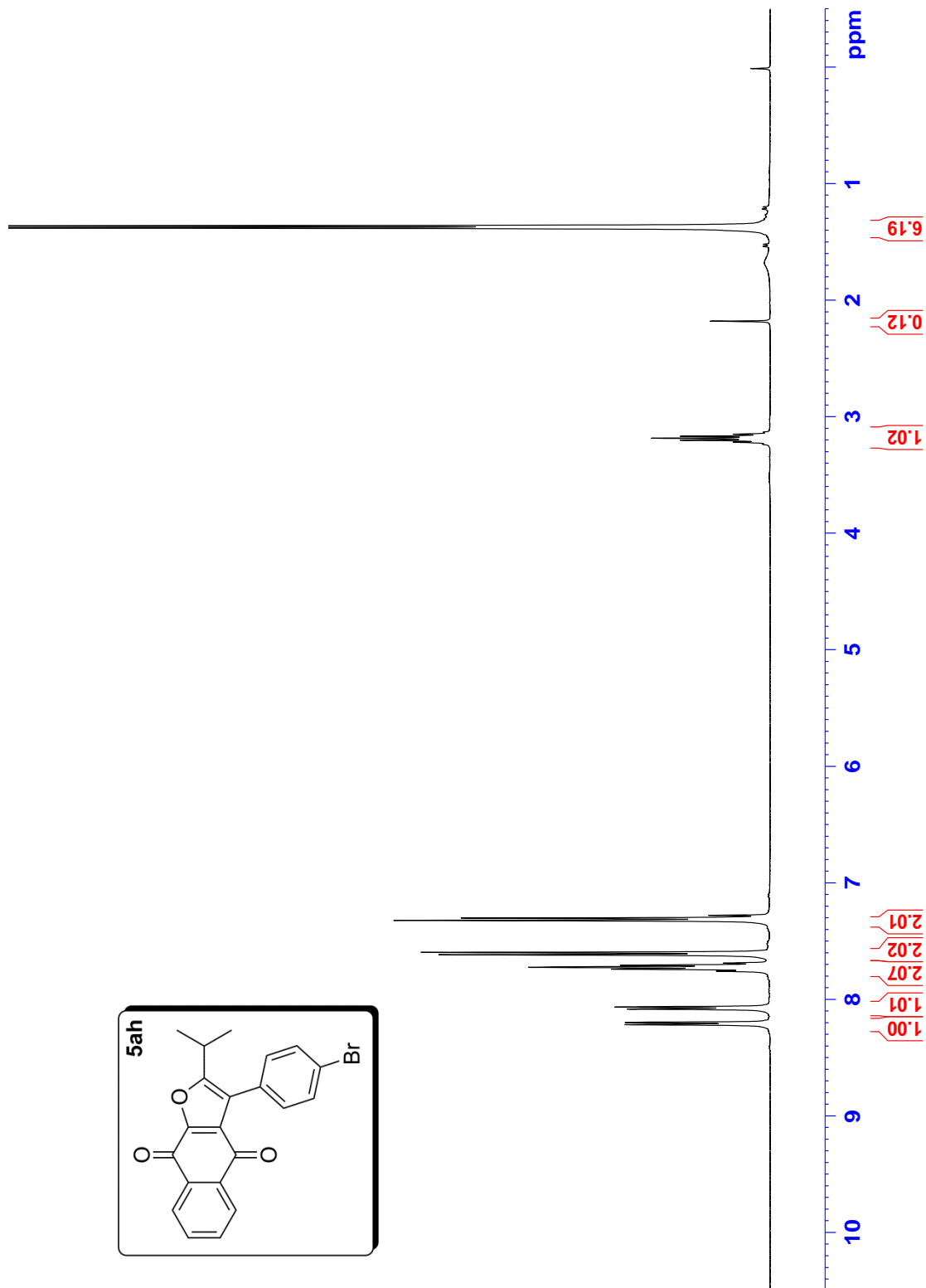
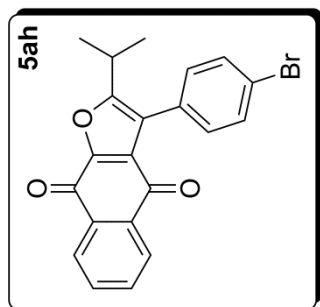


Current Data Parameters  
NAME Ken346  
EXPNO 7  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20111222  
Time\_ 23.16  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 90.5  
DW 69.000 usec  
DE 6.50 usec  
TE 300.3 K  
D1 2.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300000 MHz  
WDW EM  
SSB 0 Hz  
LB 0  
GB 0  
PC 1.00



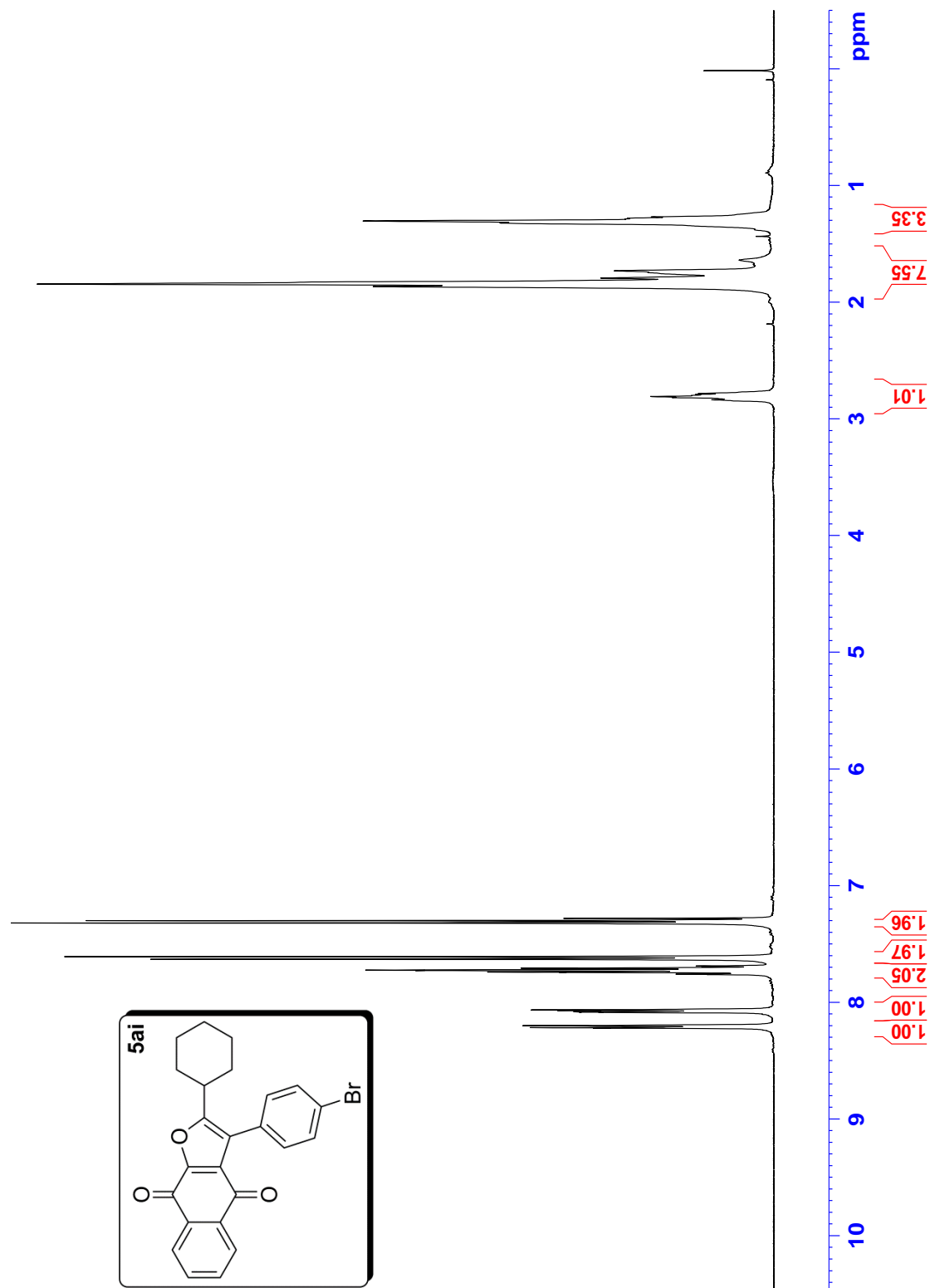


Current Data Parameters  
NAME Ken395  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20111225  
Time 17.19  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 90.5  
DW 69.000 usec  
DE 6.50 usec  
TE 296.5 K  
D1 2.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SF01 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300000 MHz  
WDW EM  
SSB 0 Hz  
LB 0  
GB 0  
PC 1.00



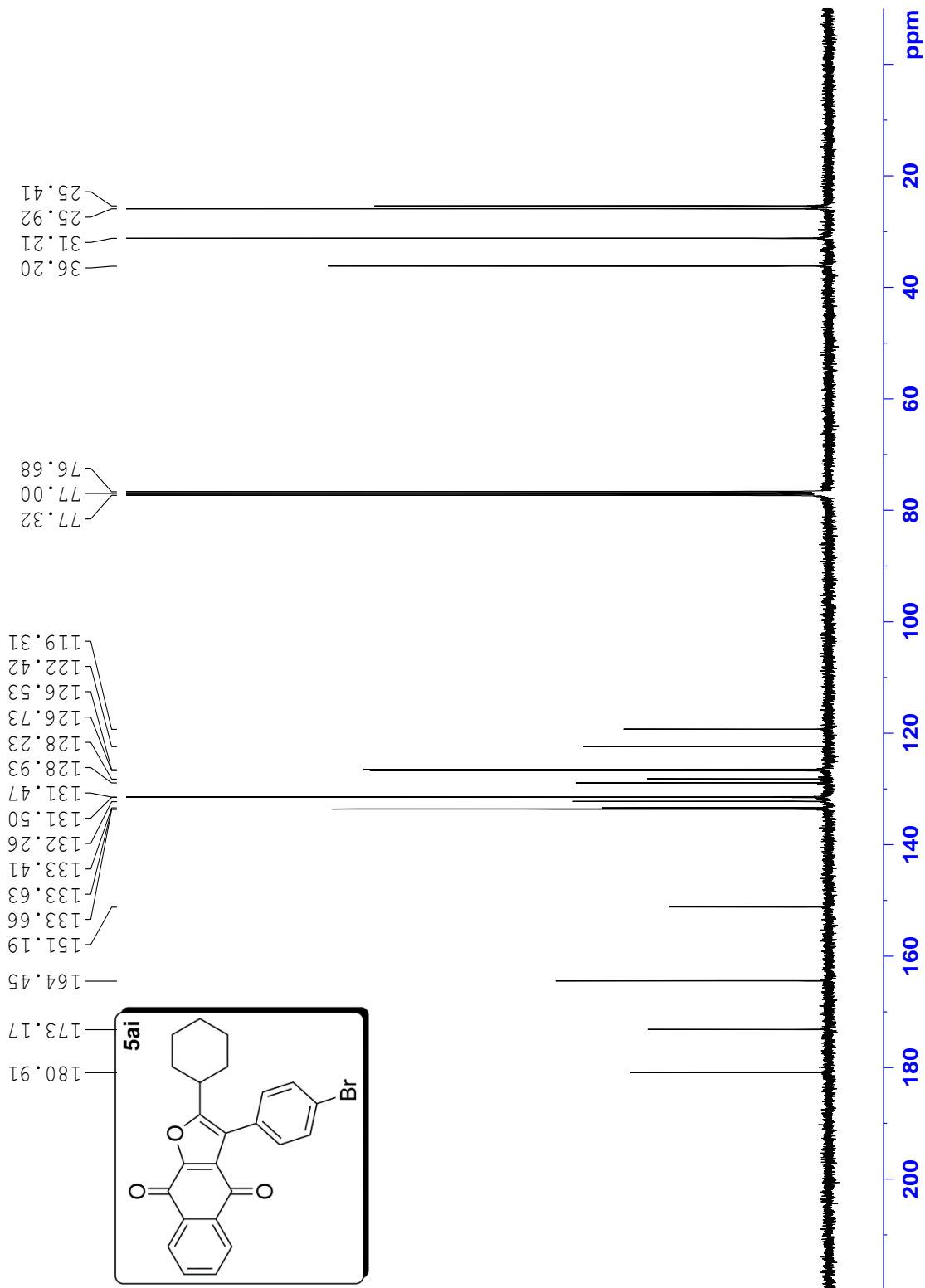
Current Data Parameters  
NAME ken395  
EXPNO 7  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120520  
Time\_ 20.10  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 332768  
SOLVENT CDCl3  
NS 1328  
DS 24038.461 Hz  
SWH 0.733536 Hz  
FIDRES 0.16816244 sec  
AQ 4096  
RG 20.800 usec  
DE 6.50 usec  
TE 299.0 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TDO 1

=====  
CHANNEL f1  
NUC1 13C  
P1 8.90 usec  
PL1 7.00 dB  
SFO1 100.6233325 MHz

=====  
CHANNEL f2  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL2 3.80 dB  
PL12 21.60 dB  
PL13 24.60 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127739 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.00

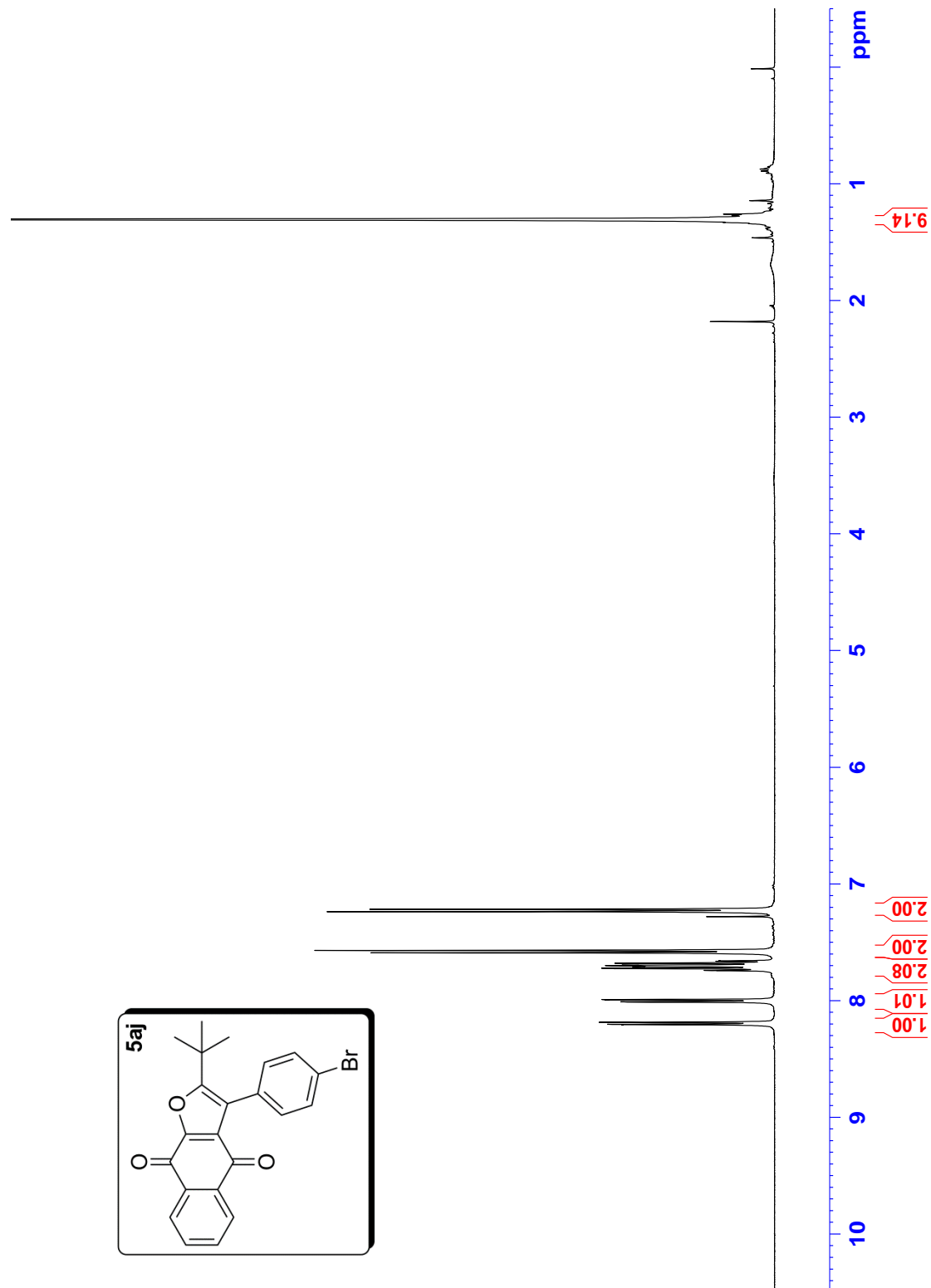
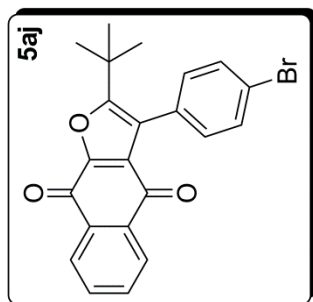


```
Current Data Parameters
NAME      Ken372
EXPNO    11
PROCNO   1

F2 - Acquisition Parameters
Date_    20111222
Time     22.47
INSTRUM  spect
PROBHD   5 mm BBO BB-IH
PULPROG  zg30
TD       32768
SOLVENT  CDCl3
NS       8
DS       0
SWH      7246.377 Hz
FIDRES   0.221142 Hz
AQ       2.2610421 sec
RG       71.8
DW       69.000 usec
DE       6.50 usec
TE       300.3 K
D1       2.00000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1     1H
P1       11.70 usec
PL1      4.00 dB
SFO1     400.1324008 MHz

F2 - Processing parameters
SI       16384
WDW      EM
SSB      0 Hz
LB       0
GB       0
PC       1.00
```





```

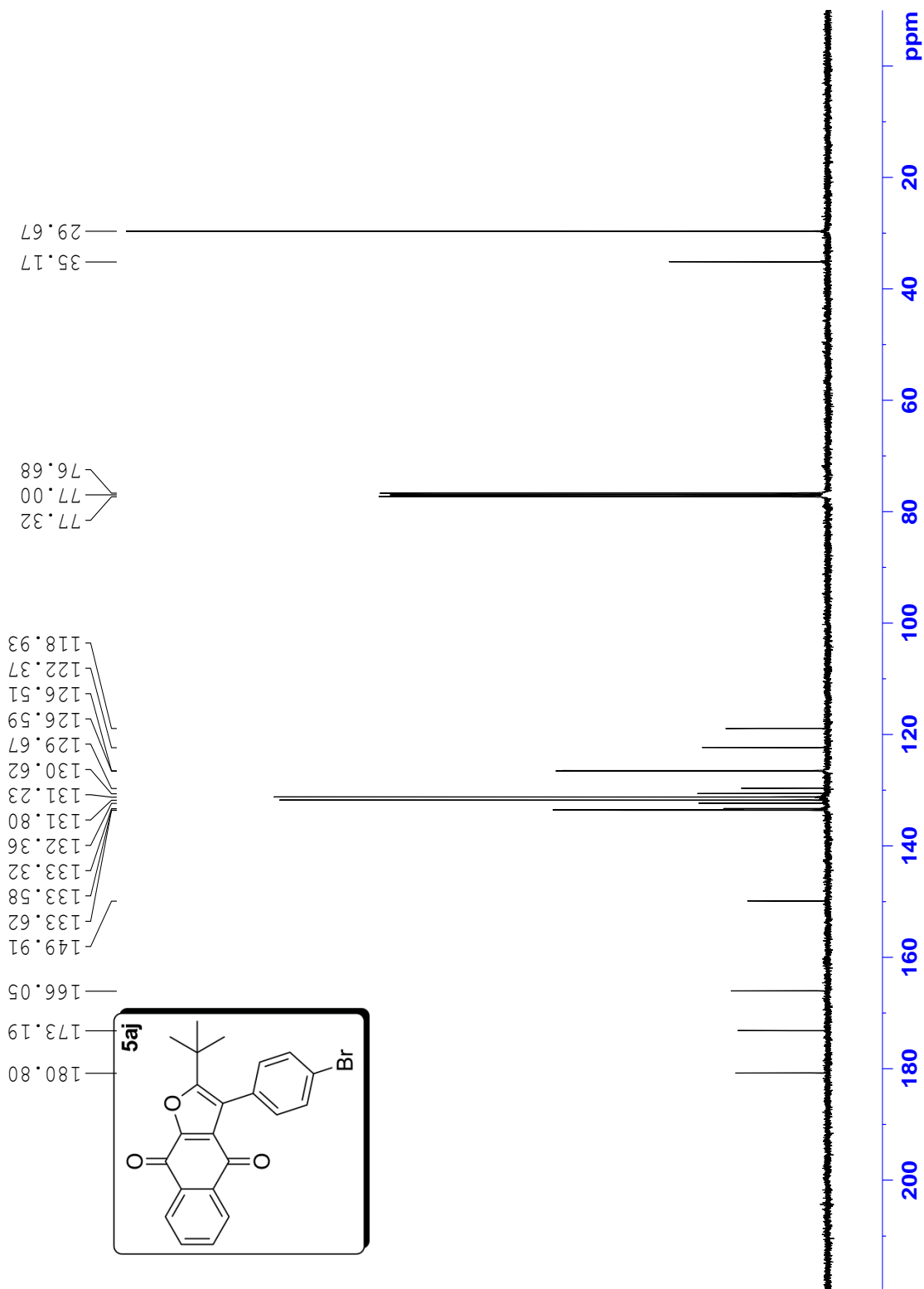
Current Data Parameters
NAME      ken372
EXPNO    12
PROCNO   1

F2 - Acquisition Parameters
Date_    20111222
Time_    22.51
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zgpg30
TD       32768
SOLVENT  CDCl3
NS       509
DS       0
SWH      24038.461 Hz
FIDRES   0.733536 Hz
AQ       0.16816244 sec
RG       4096
DE       20.800 usec
TE       300.16 K
D1       2.00000000 sec
D11      0.03000000 sec
TDO      1

===== CHANNEL f1 =====
NUC1     13C
P1       8.90 usec
PL1      7.00 dB
SFO1     100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    90.00 usec
PL2      3.80 dB
PL12     21.60 dB
PL13     24.60 dB
SFO2     400.1316005 MHz

F2 - Processing parameters
SI       32768
SF       100.6127748 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.00
    
```

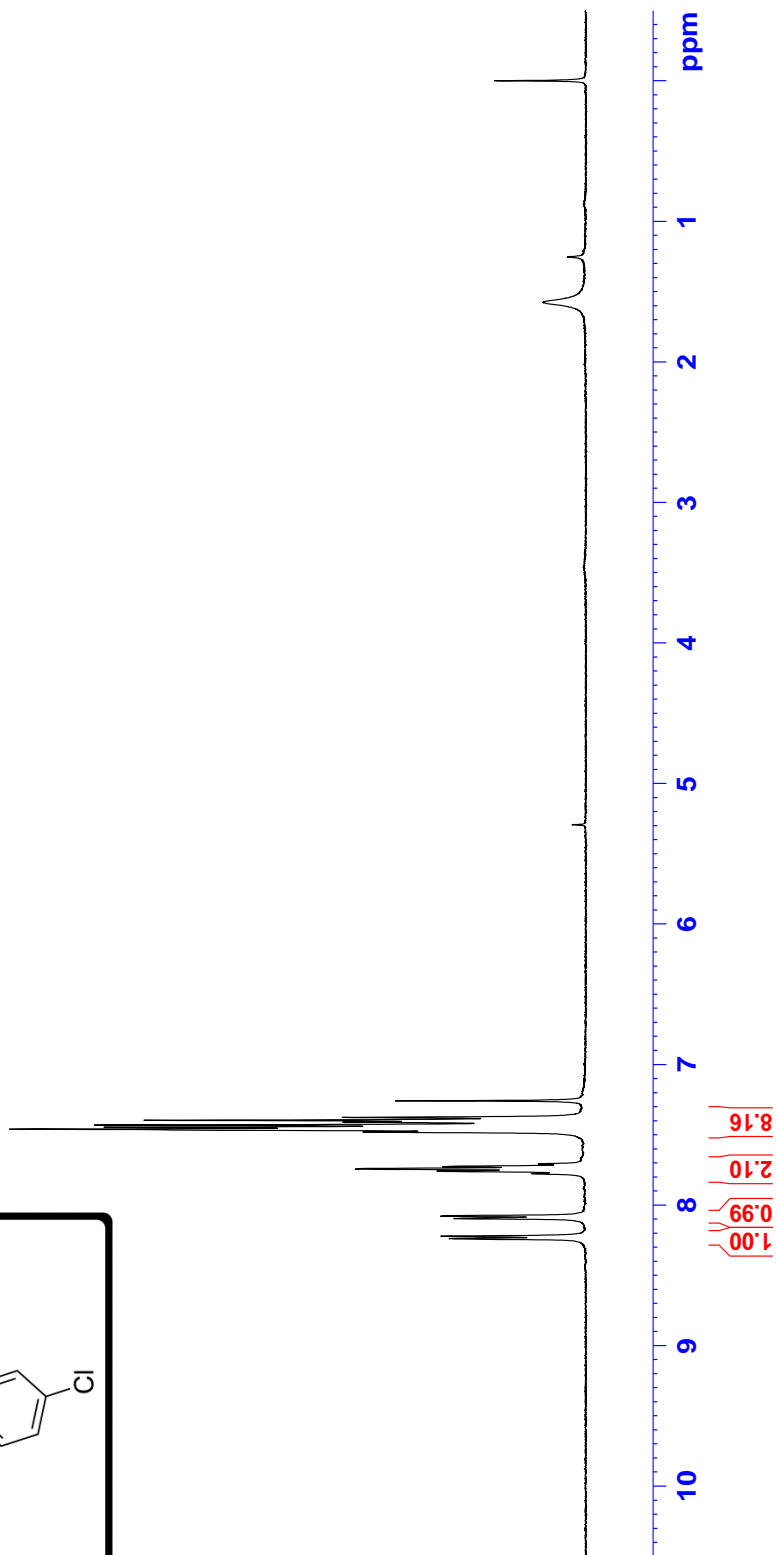
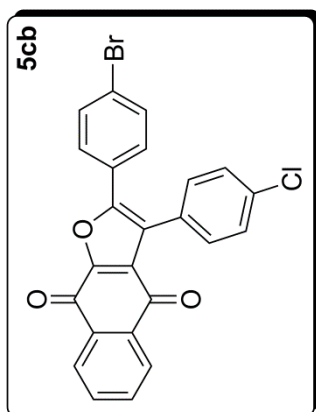


Current Data Parameters  
NAME ken356  
EXPNO 6  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20111230  
Time 11.19  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDC13  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 256  
DW 69.000 usec  
DE 6.50 usec  
TE 300.4 K  
D1 2.0000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300084 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00



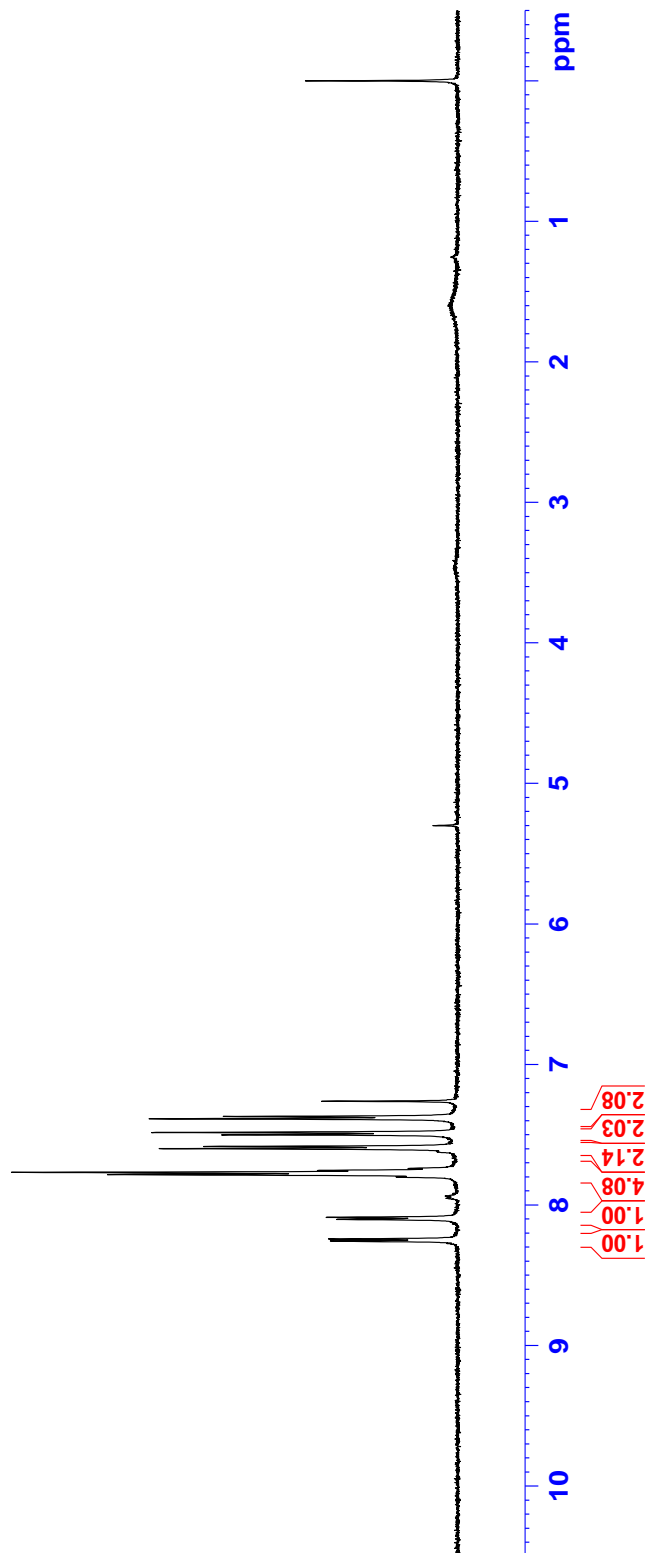
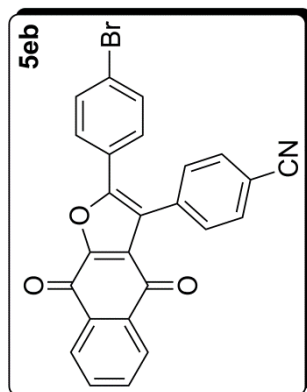


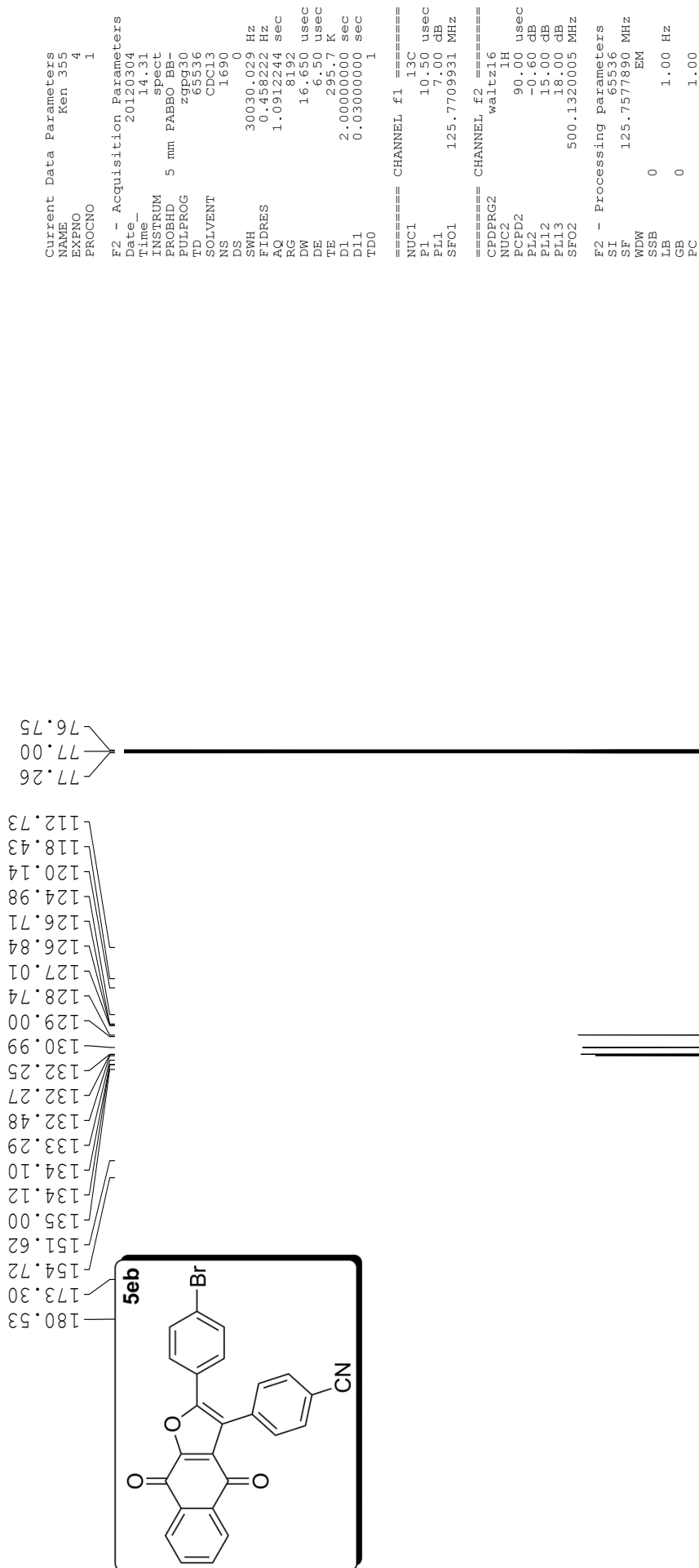
Current Data Parameters  
NAME Ken 355  
EXPNO 3  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120304  
Time 14.28  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 32768  
SOLVENT CDC13  
DS 1  
NS 0  
SWH 9057.971 Hz  
FIDRES 0.276427 Hz  
AQ 1.8088436 sec  
RG 228.1  
DW 55.200 usec  
DE 6.50 usec  
TE 295.3 K  
D1 2.0000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 0 dB  
SFO1 500.1330008 MHz

F2 - Processing parameters  
SI 16384  
SF 500.1300108 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00



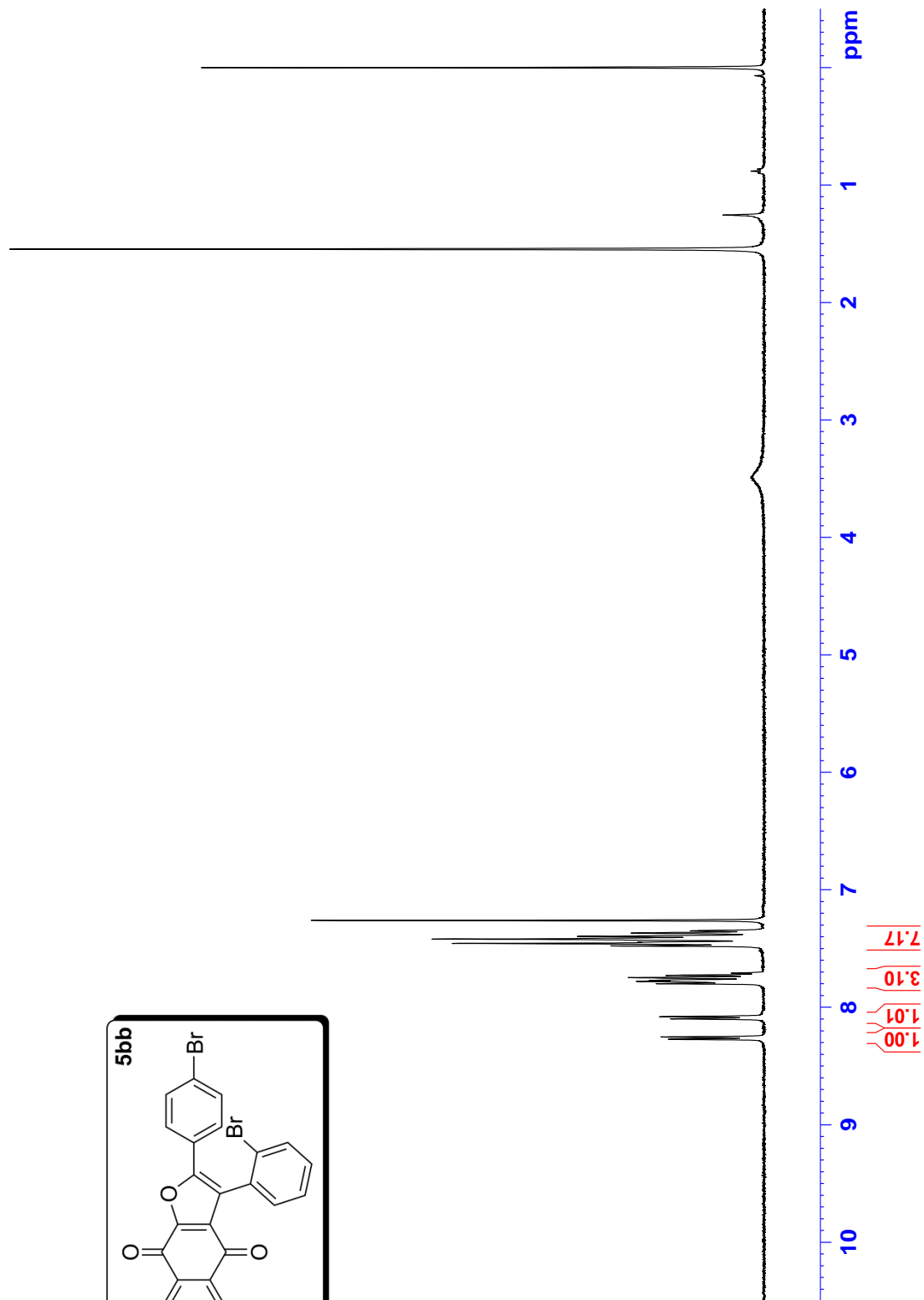
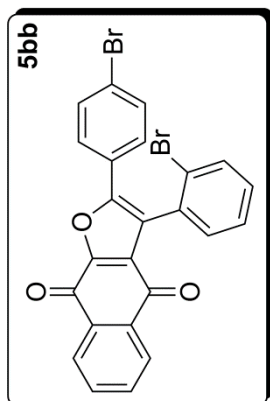


Current Data Parameters  
NAME ken394  
EXPNO 22  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120602  
Time 12.42  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 32  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 256  
DW 69.000 usec  
DE 6.50 usec  
TE 298.8 K  
D1 2.0000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300082 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00



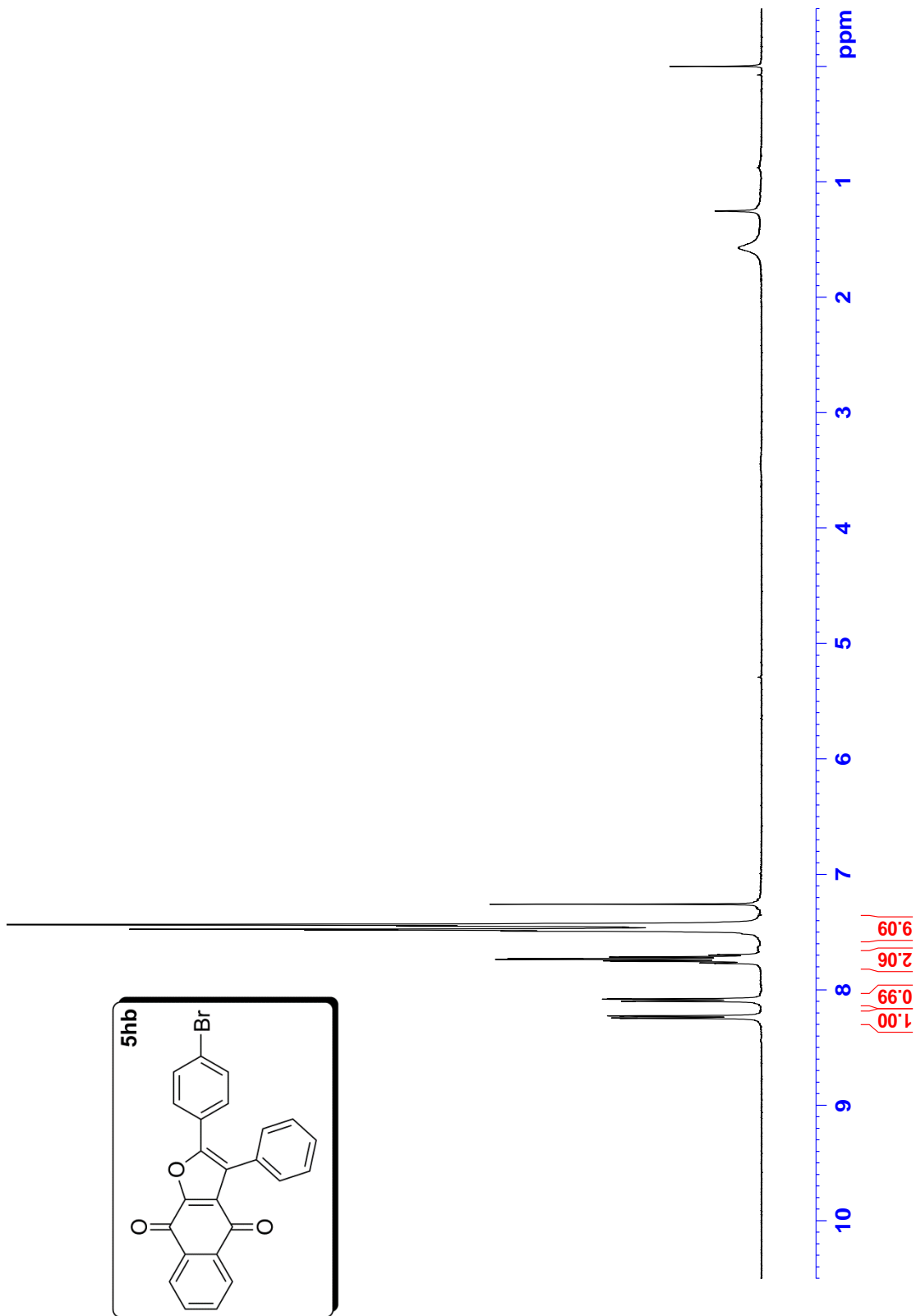


Current Data Parameters  
NAME Ken354  
EXPNO 6  
PROCNO 1

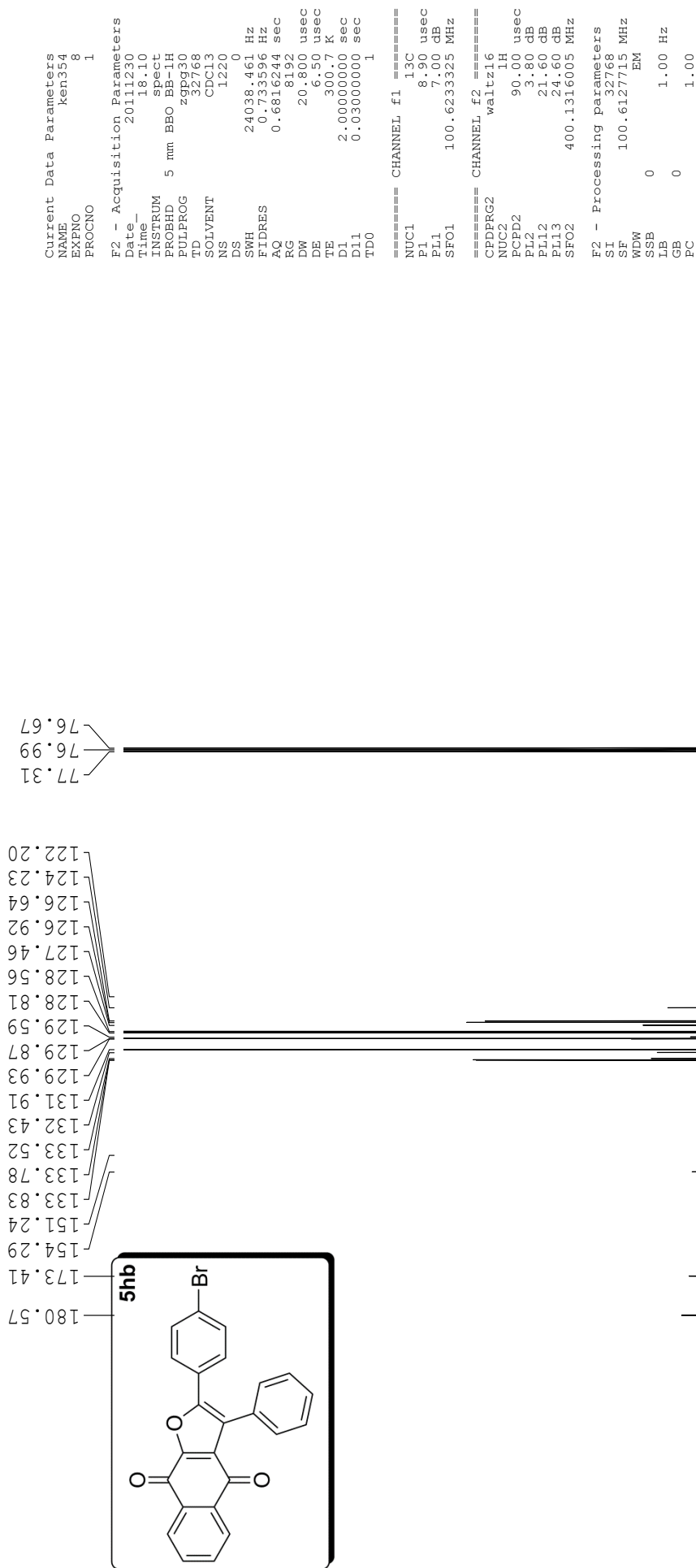
F2 - Acquisition Parameters  
Date\_ 20111229  
Time\_ 20.52  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 256  
DW 69.000 usec  
DE 6.50 usec  
TE 301.1 K  
D1 2.00000000 sec  
TDO 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300092 MHz  
WDW EM  
SSB 0 Hz  
LB 0  
GB 0  
PC 1.00





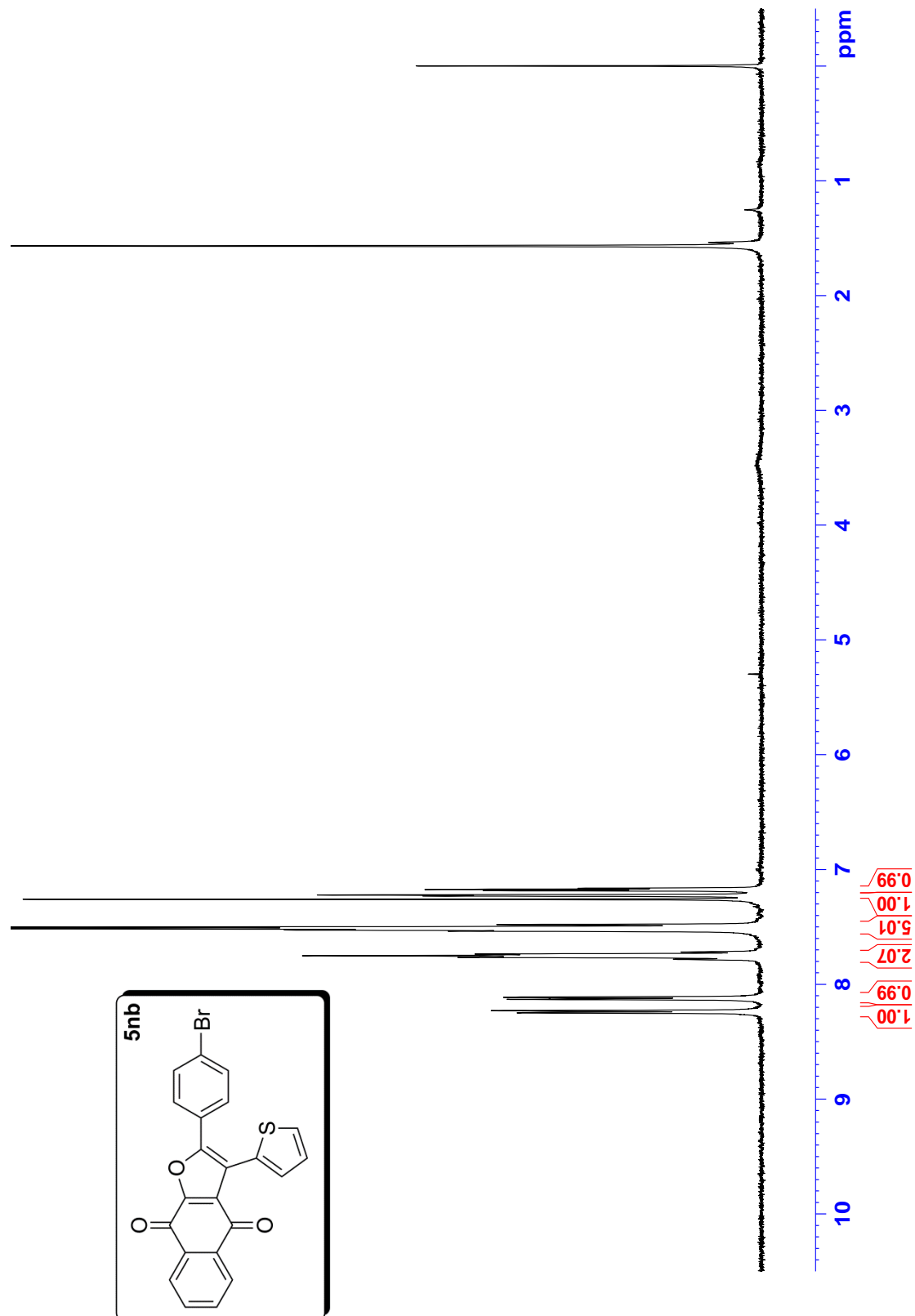


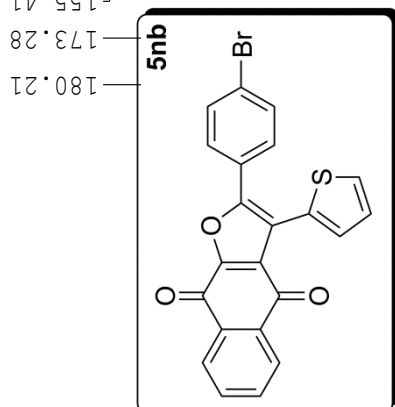
```
Current Data Parameters
NAME      Ken402
EXPNO     3
PROCNO    1

F2 - Acquisition Parameters
Date_     20120104
Time      15.14
INSTRUM   spect
PROBHD    5 mm BBO BB-IH
PULPROG   zg30
TD         32768
SOLVENT   CDCl3
NS         8
DS         0
SWH        7246.377 Hz
FIDRES     0.221142 Hz
AQ         2.2610421 sec
RG         328.1
WDW        69.000 usec
DE         6.50 usec
TE         297.8 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         11.70 usec
PL1        4.00 dB
SFO1       400.1324008 MHz

F2 - Processing parameters
SI         16384
SF         400.1300081 MHz
WDW        EM
SSB        0 Hz
LB         0 Hz
GB         0
PC         1.00
```





180.21  
173.28  
155.41  
151.17  
133.88  
133.81  
133.39  
132.25  
131.94  
129.70  
129.30  
128.71  
127.73  
127.58  
127.08  
126.94  
126.62  
124.64  
114.88

77.31  
77.00  
76.68

```

Current Data Parameters
NAME          ken402
EXPNO        5
PROCNO       1

F2 - Acquisition Parameters
Date_        20120429
Time_        16.14
INSTRUM      spect
PROBHD       5 mm BBO BB-1H
PULPROG      zgpg30
TD           32768
SOLVENT      CDCl3
NS           1493
DS           0
SWH          24038.461 Hz
FIDRES       0.733536 Hz
AQ           0.6816244 sec
RG           5792.6
DW           20.800 usec
DE           6.50 usec
TE           300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TDO         1

===== CHANNEL f1 =====
NUC1         13C
P1           8.90 usec
PL1          7.00 dB
SFO1         100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2         1H
PCPD2        90.00 usec
PL2          3.80 dB
PL12         21.60 dB
PL13         24.60 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI           32768
SF           100.6127739 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.00
    
```

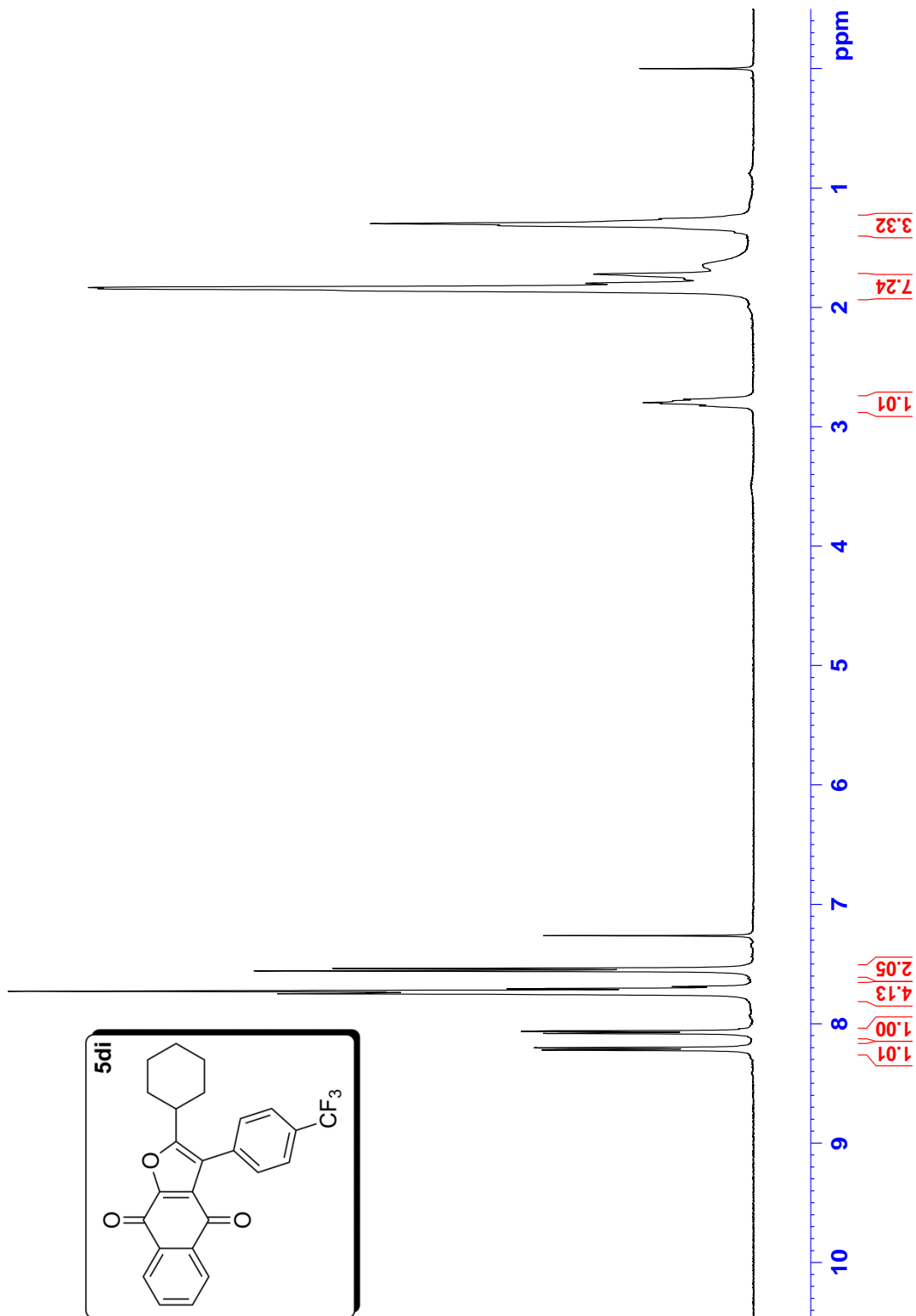
200 180 160 140 120 100 80 20 ppm

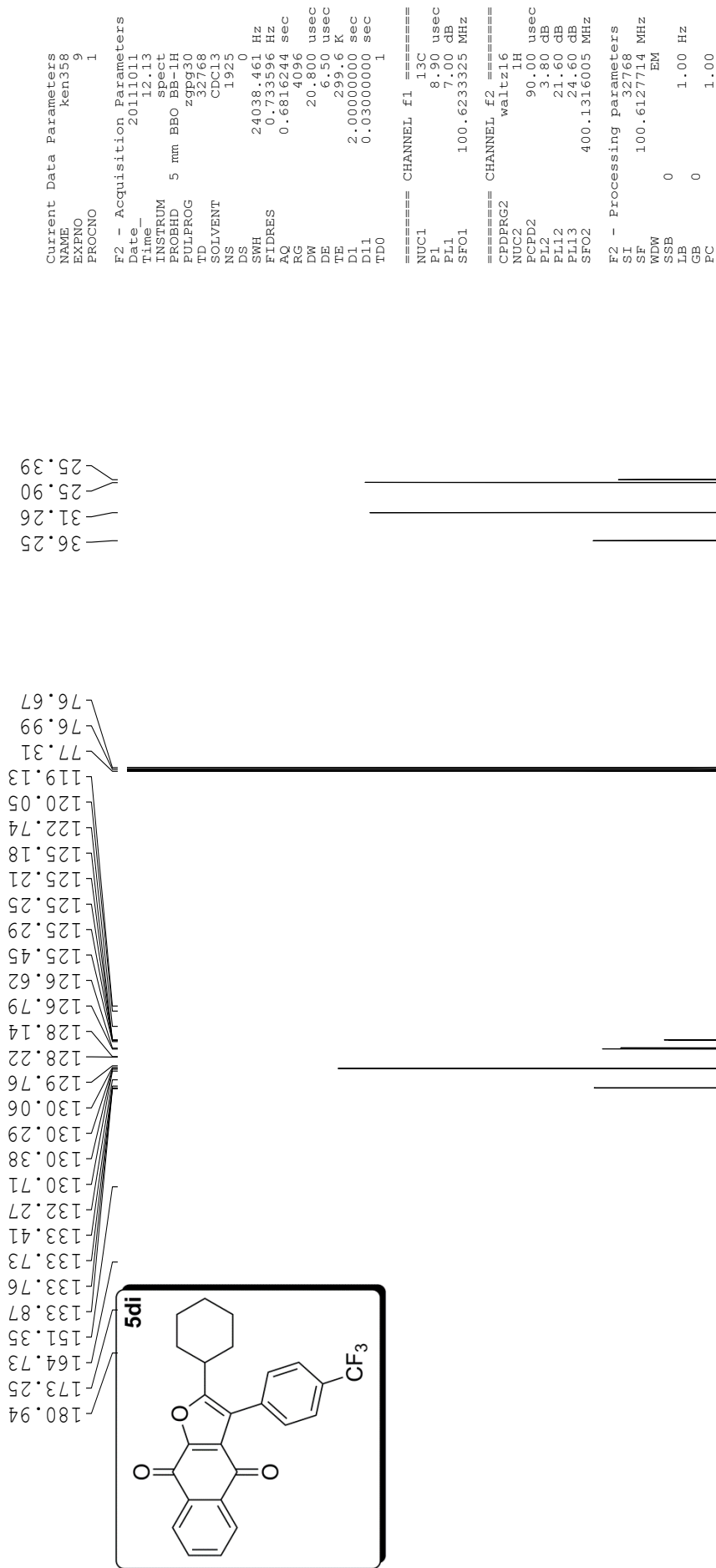
Current Data Parameters  
NAME ken358  
EXPNO 8  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20111011  
Time 12.08  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 128  
DW 69.000 usec  
DE 6.50 usec  
TE 299.2 K  
D1 2.0000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300067 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00



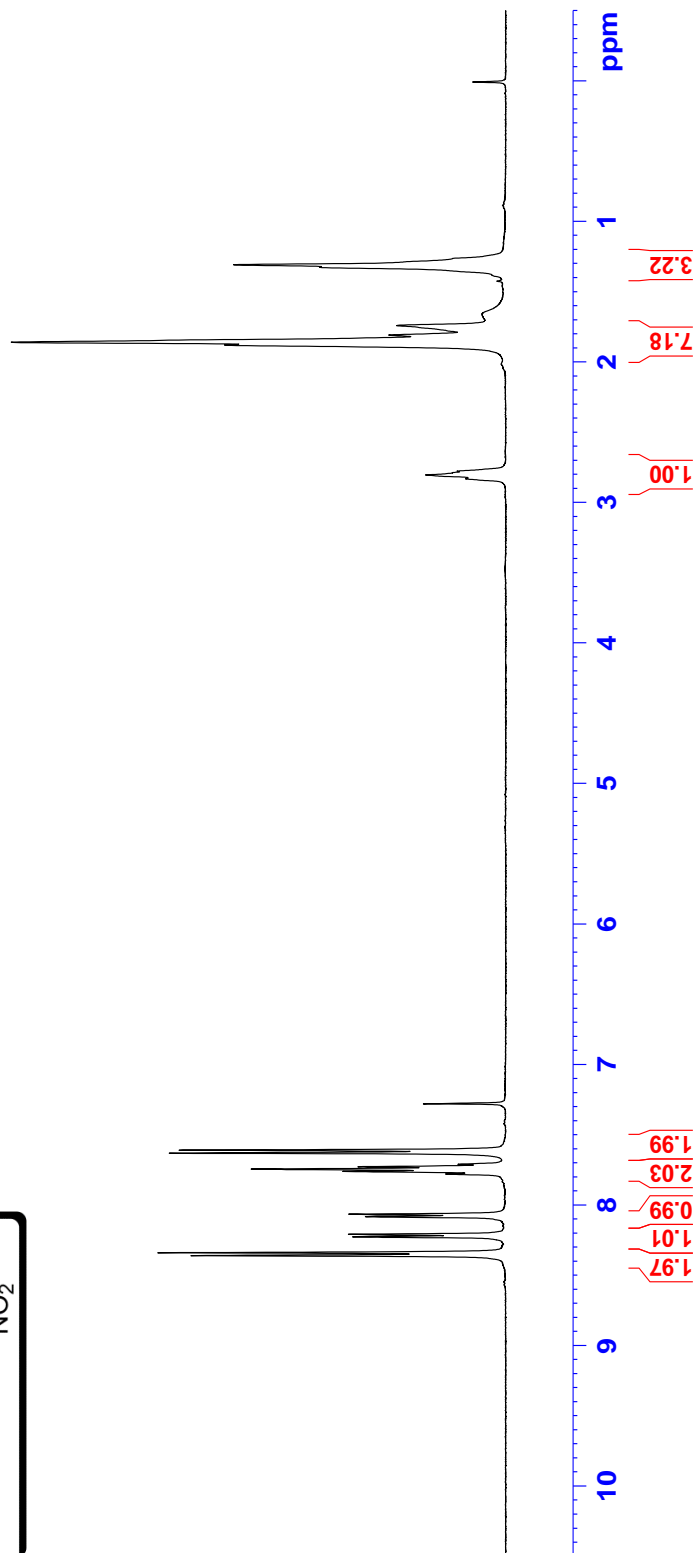
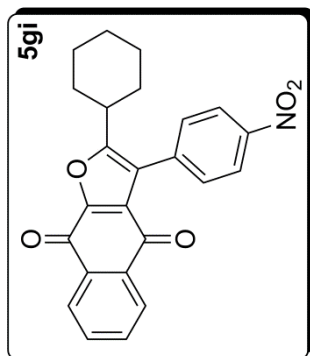


Current Data Parameters  
NAME ken357  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120202  
Time 16.16  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDC13  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 114  
DW 69.000 usec  
DE 6.50 usec  
TE 298.5 K  
D1 2.0000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300000 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00



```

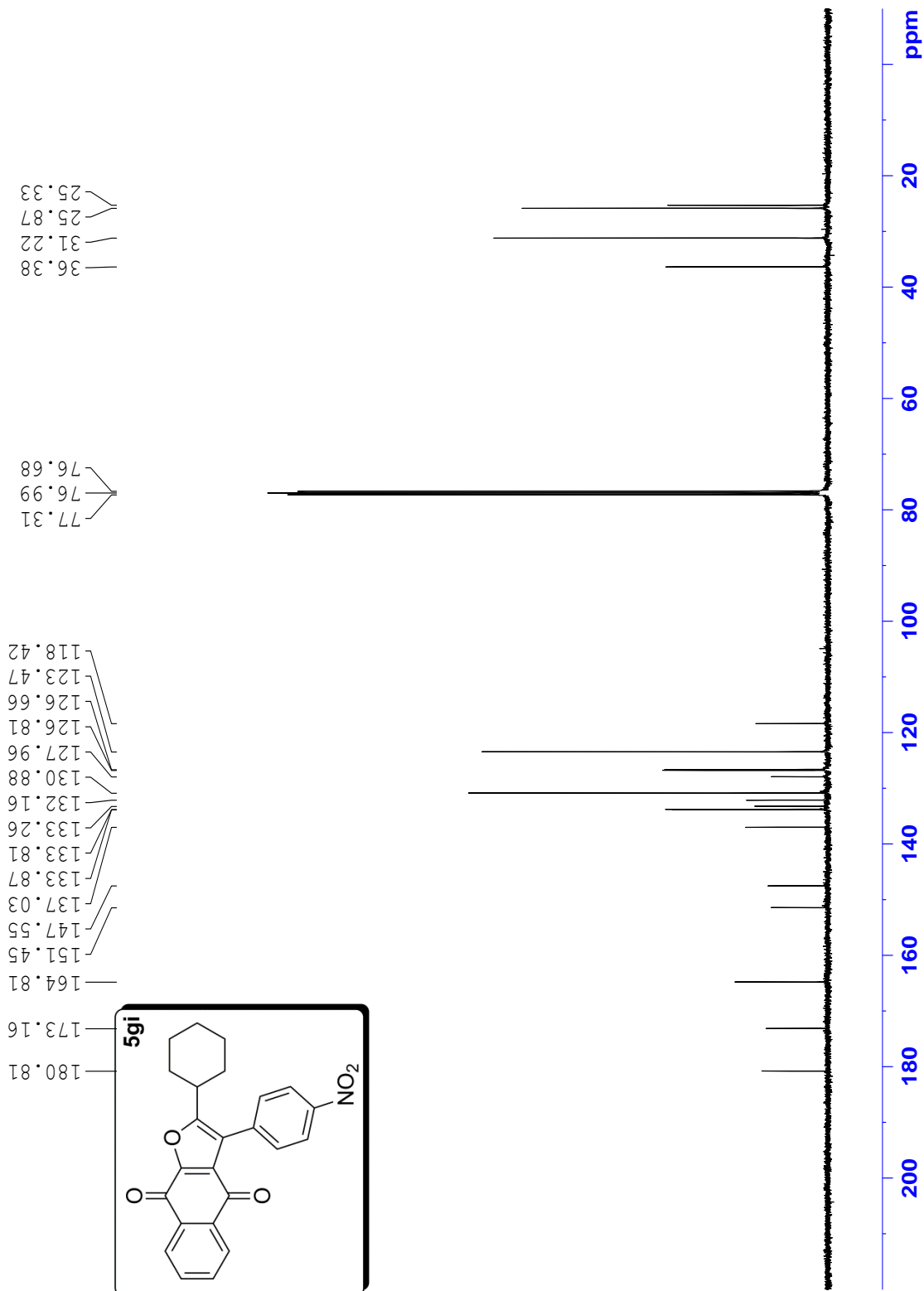
Current Data Parameters
NAME          ken357
EXPNO         6
PROCNO        1

F2 - Acquisition Parameters
Date_         20120202
Time_         18.24
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            32768
SOLVENT       CDCl3
NS            833
DS            24038.461 Hz
SWH           0.733536 Hz
FIDRES        0.16816244 sec
AQ            5792.6
RG            20.800 usec
DE            6.50 usec
TE            298.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TDO           1

===== CHANNEL f1 =====
NUC1          13C
P1            8.90 usec
PL1           7.00 dB
SFO1          100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         90.00 usec
PL2           3.80 dB
PL12          21.60 dB
PL13          24.60 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI            32768
SF            100.6127738 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.00
    
```

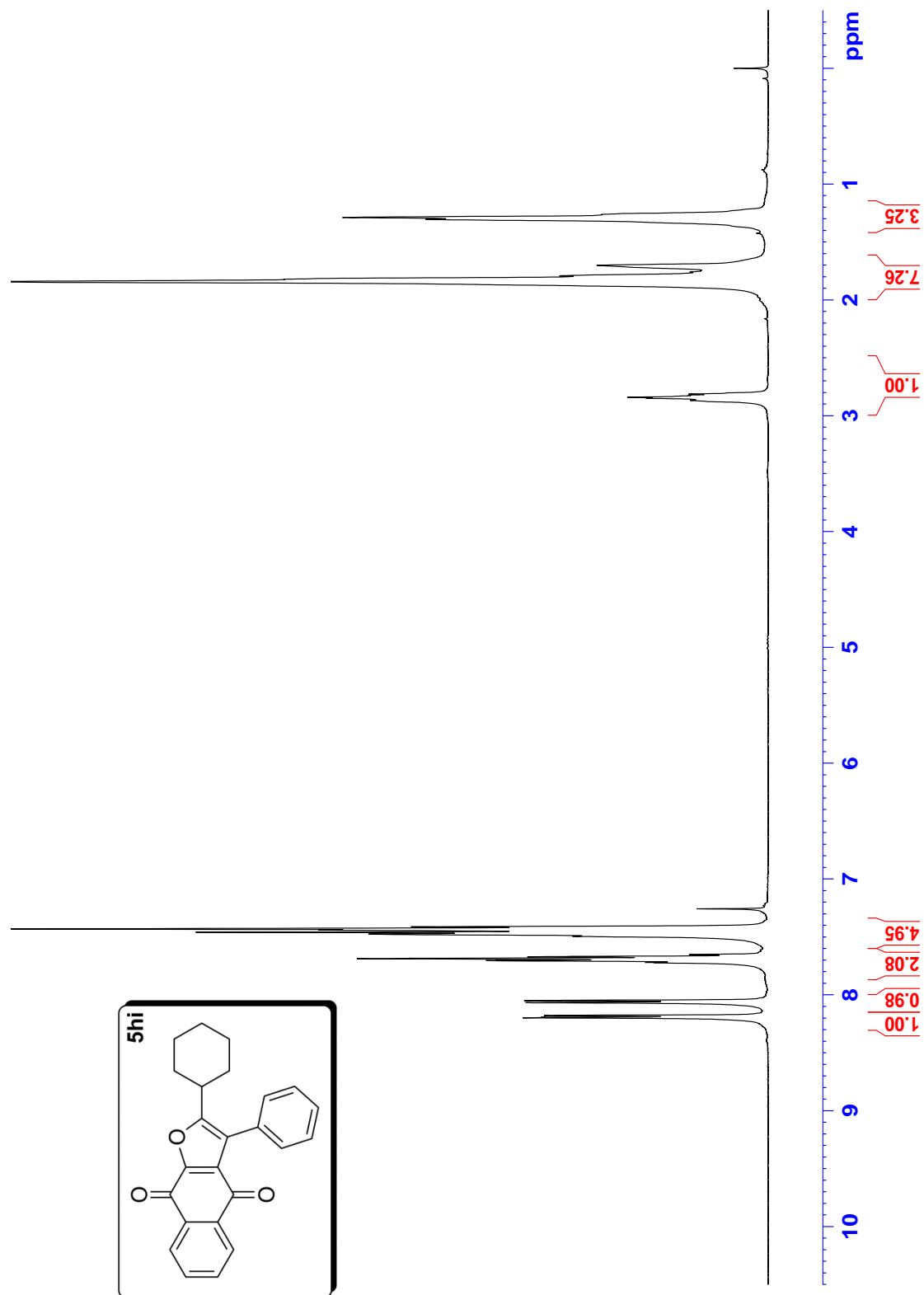


Current Data Parameters  
NAME Ken360  
EXPNO 7  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20111230  
Time\_ 17.33  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 64  
DW 69.000 usec  
DE 6.50 usec  
TE 300.2 K  
D1 2.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300082 MHz  
WDW EM  
SSB 0 Hz  
LB 0  
GB 0  
PC 1.00





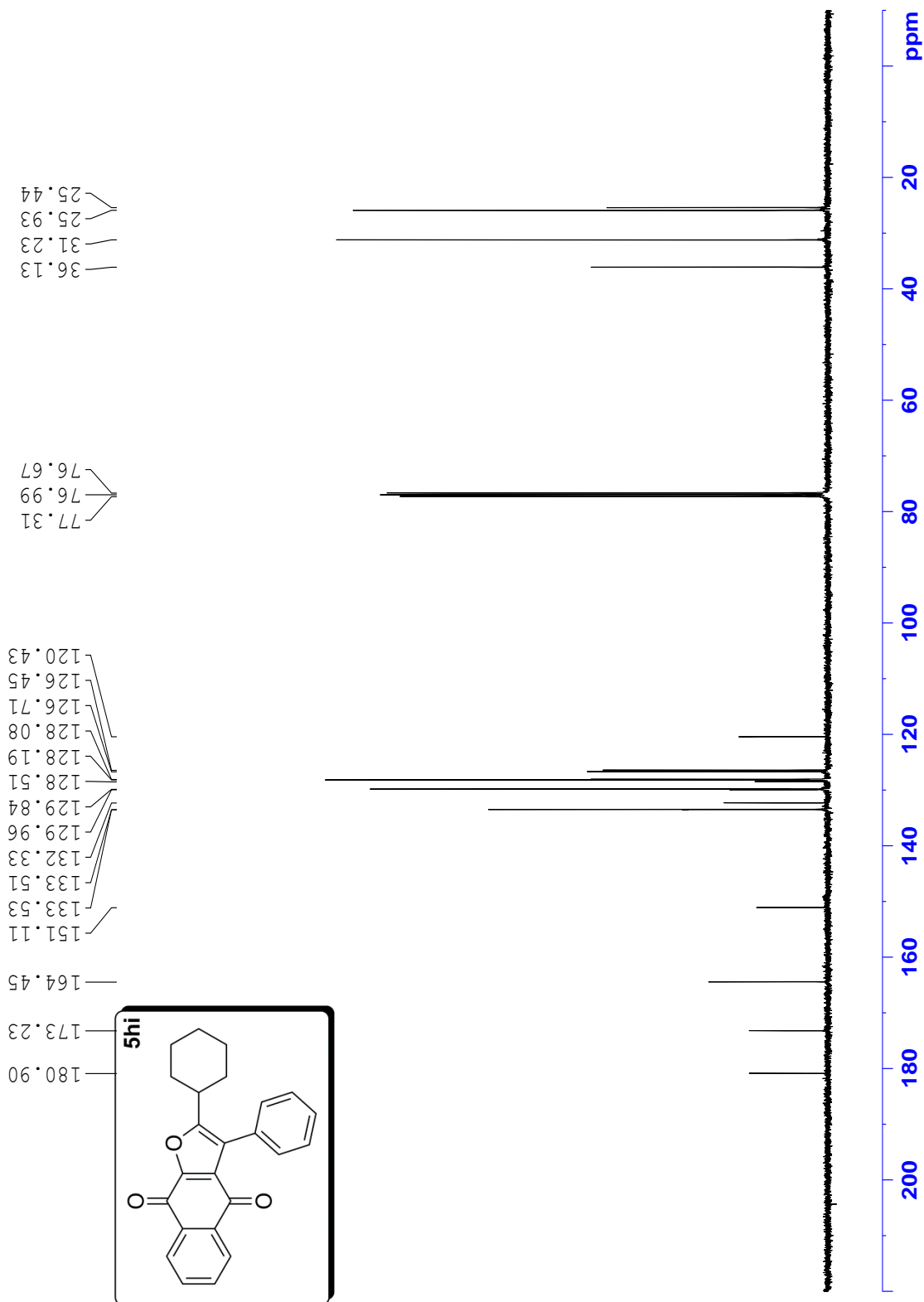
```
Current Data Parameters
NAME      ken360
EXPNO     9
PROCNO    1

F2 - Acquisition Parameters
Date_     20111231
Time_     12.16
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD        32768
SOLVENT   CDCl3
NS         527
DS         0
SWH        24038.461 Hz
FIDRES     0.733536 Hz
AQ         0.6816244 sec
RG         8192
DW         20.800 usec
DE         6.50 usec
TE         299.7 K
D1         2.00000000 sec
D11        0.03000000 sec
TDO        1

===== CHANNEL f1 =====
NUC1       13C
P1         8.90 usec
PL1        7.00 dB
SFO1       100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     90.00 usec
PL2        3.80 dB
PL12       21.60 dB
PL13       24.60 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI         32768
SF         100.6127759 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00
```

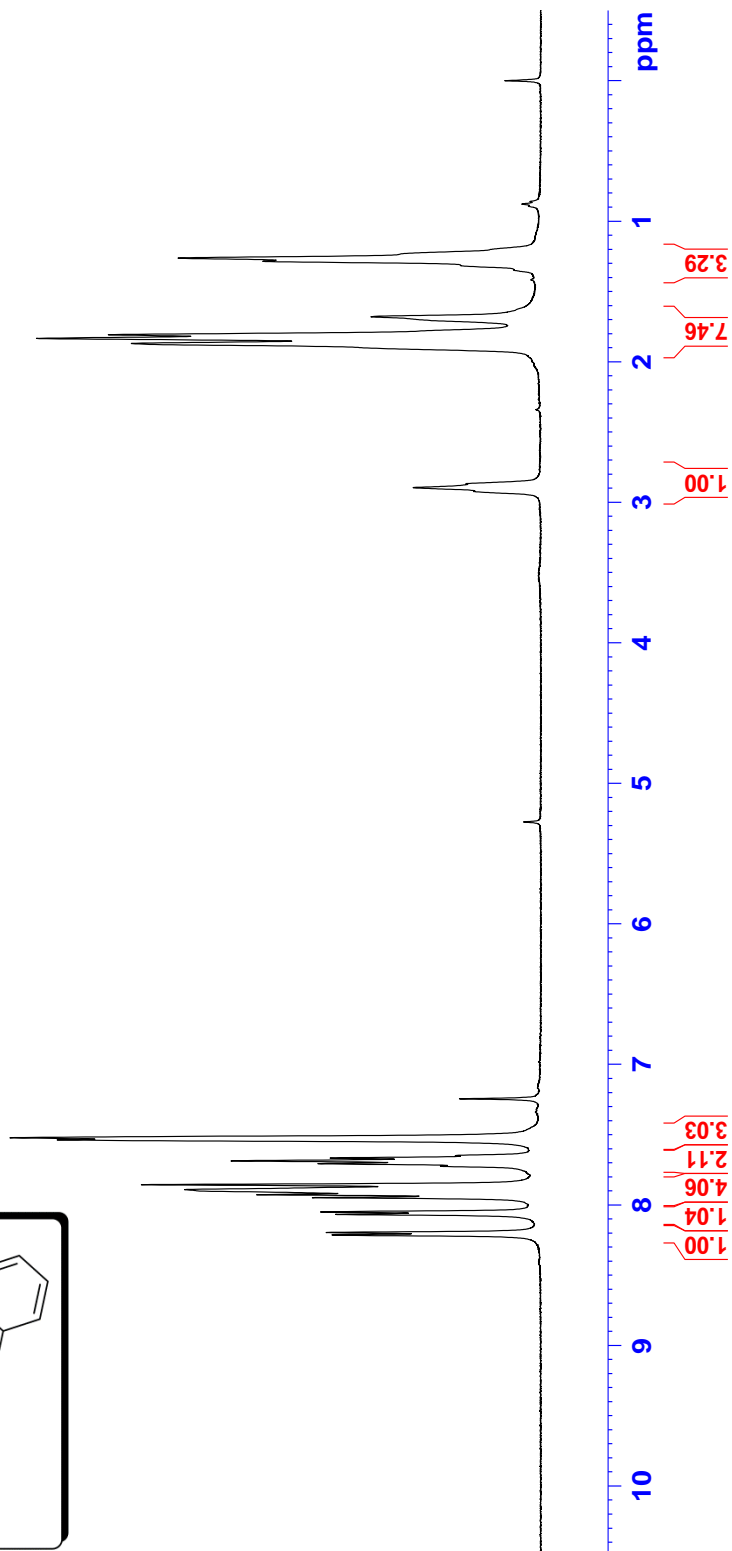
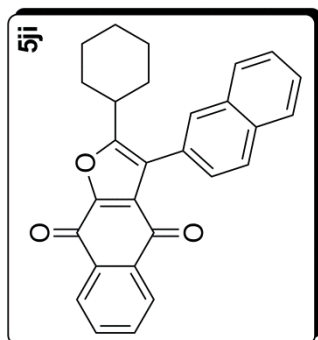


Current Data Parameters  
NAME ken361  
EXPNO 5  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120112  
Time 12.37  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDC13  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 90.5  
DW 69.000 usec  
DE 6.50 usec  
TE 299.6 K  
D1 2.0000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300133 MHz  
WDW EM  
SSB 0 Hz  
LB 0  
GB 0  
PC 1.00



```

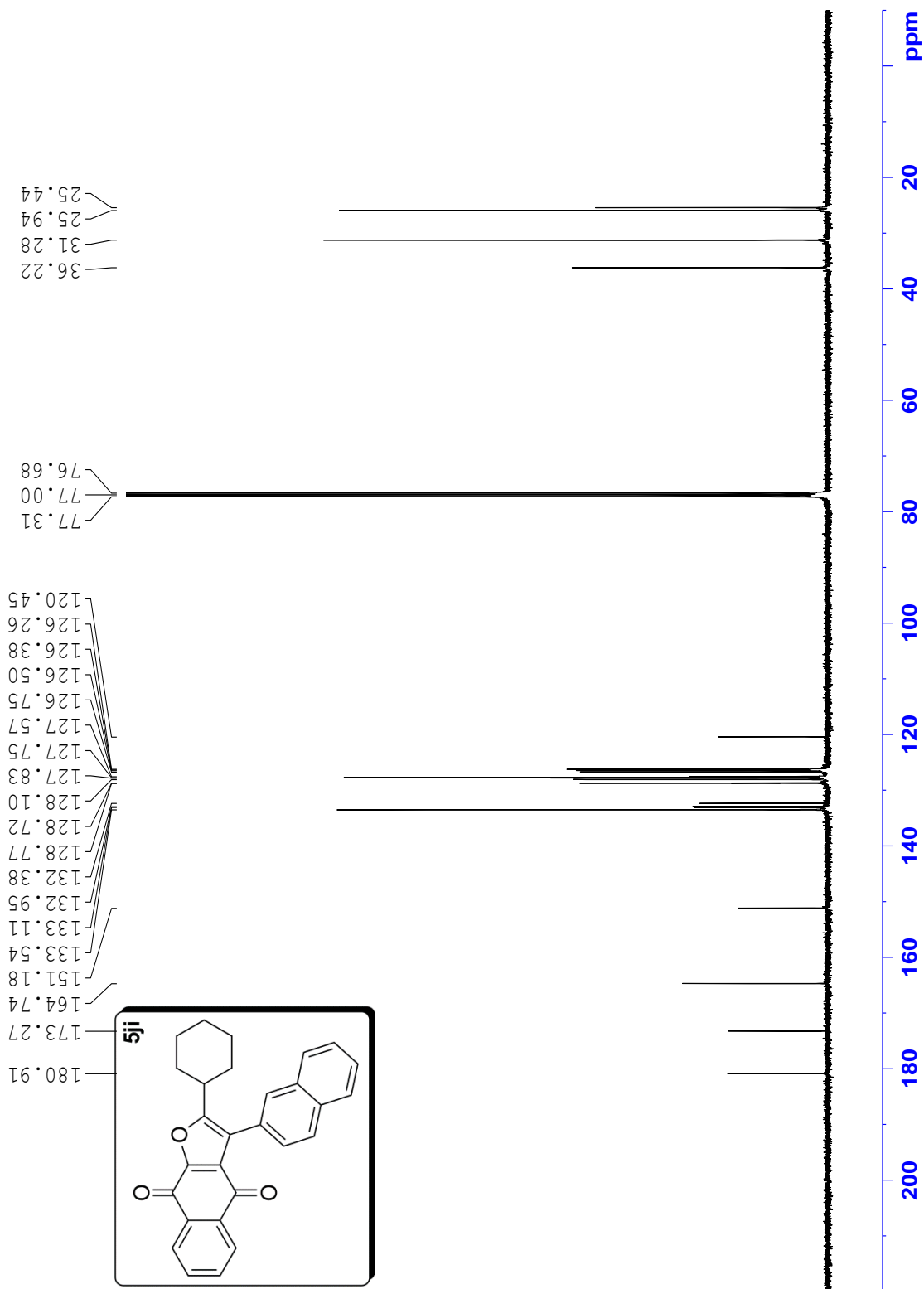
Current Data Parameters
NAME      ken361
EXPNO    6
PROCNO   1

F2 - Acquisition Parameters
Date_    20120112
Time_    12.50
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zgpg30
TD       32768
SOLVENT  CDCl3
NS       1778
DS       1
SWH      24038.461 Hz
FIDRES   0.733536 Hz
AQ       0.16816244 sec
RG       4096
DE       20.800 usec
TE       300.2 K
D1       2.00000000 sec
D11      0.03000000 sec
TDO      1

===== CHANNEL f1 =====
NUC1     13C
P1       8.90 usec
PL1      7.00 dB
SFO1     100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
P2       90.00 usec
PL2      3.80 dB
PL12     21.60 dB
PL13     24.60 dB
SFO2     400.1316005 MHz

F2 - Processing parameters
SI       32768
SF       100.6127751 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.00
    
```

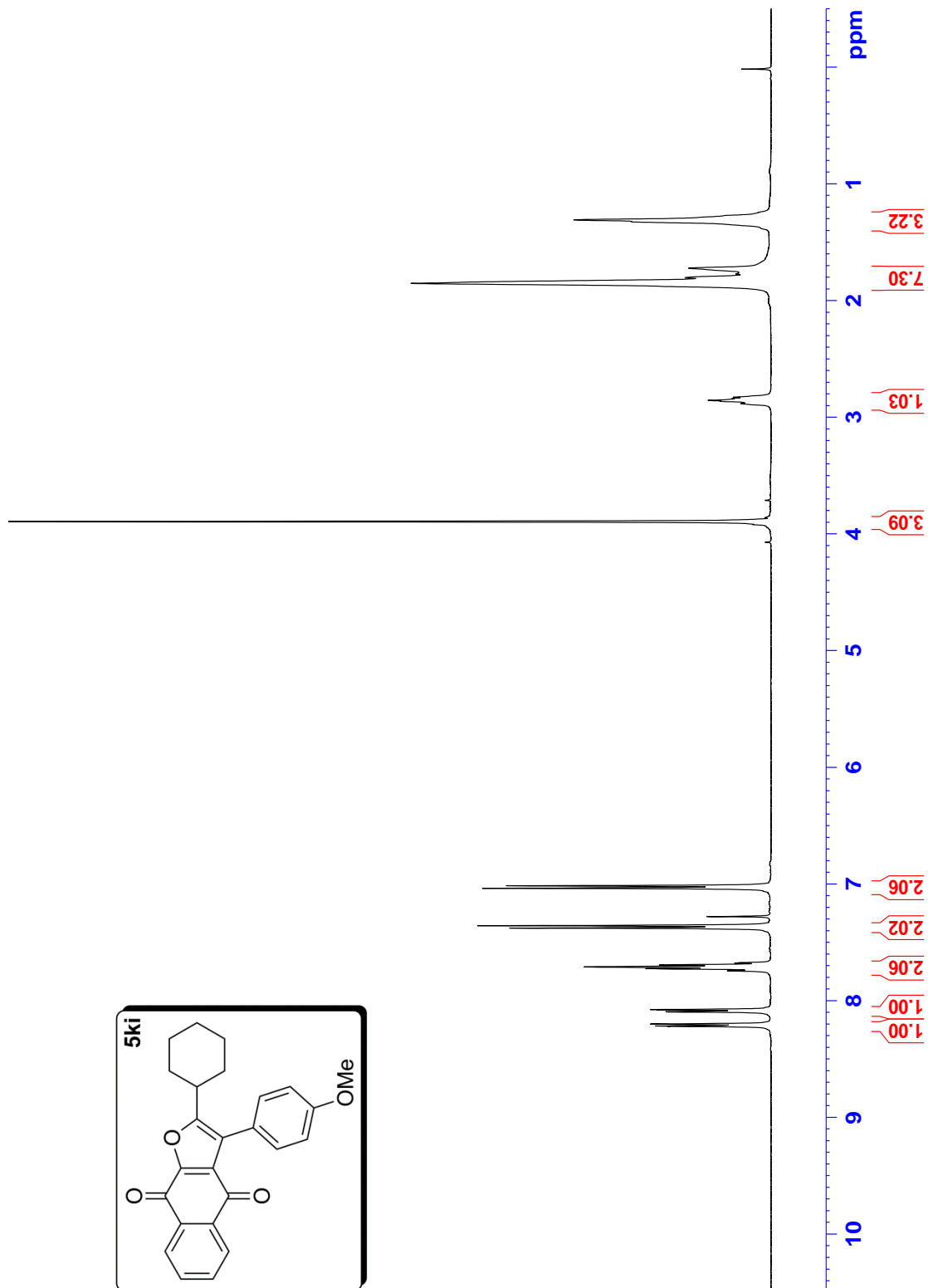


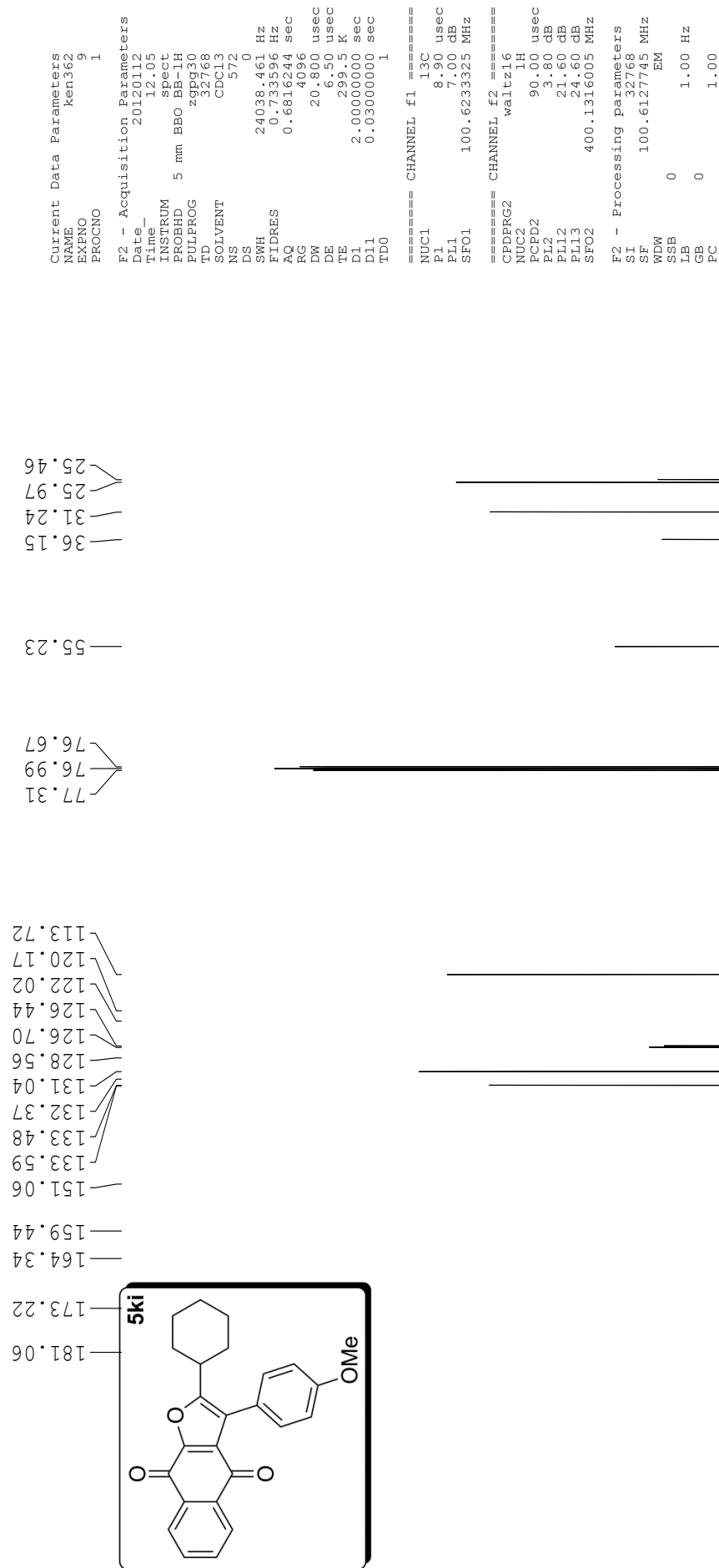
```
Current Data Parameters
NAME      Ken362
EXPNO     8
PROCNO    1

F2 - Acquisition Parameters
Date_     20120112
Time      12.03
INSTRUM   spect
PROBHD    5 mm BBO BB-IH
PULPROG   zg30
TD         32768
SOLVENT   CDCl3
NS         8
DS         0
SWH        7246.377 Hz
FIDRES     0.221142 Hz
AQ         2.2610421 sec
RG         90.5
Sf         69.000 usec
DE         6.50 usec
TE         299.3 K
D1         2.00000000 sec
TD0        1

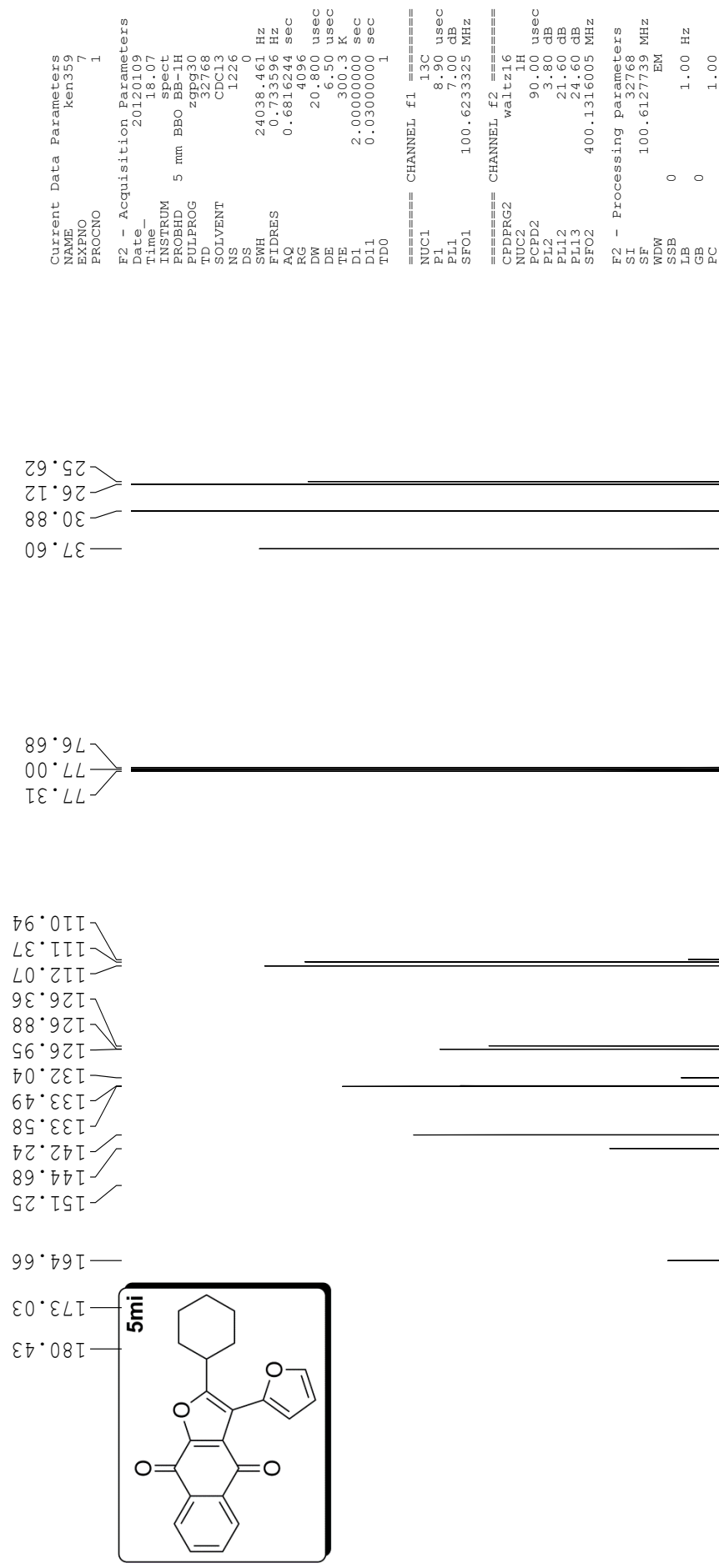
===== CHANNEL f1 =====
NUC1       1H
P1         11.70 usec
PL1        4.00 dB
SFO1       400.1324008 MHz

F2 - Processing parameters
SI         16384
SF         400.1300000 MHz
WDW        EM
SSB        0 Hz
LB         0
GB         0
PC         1.00
```







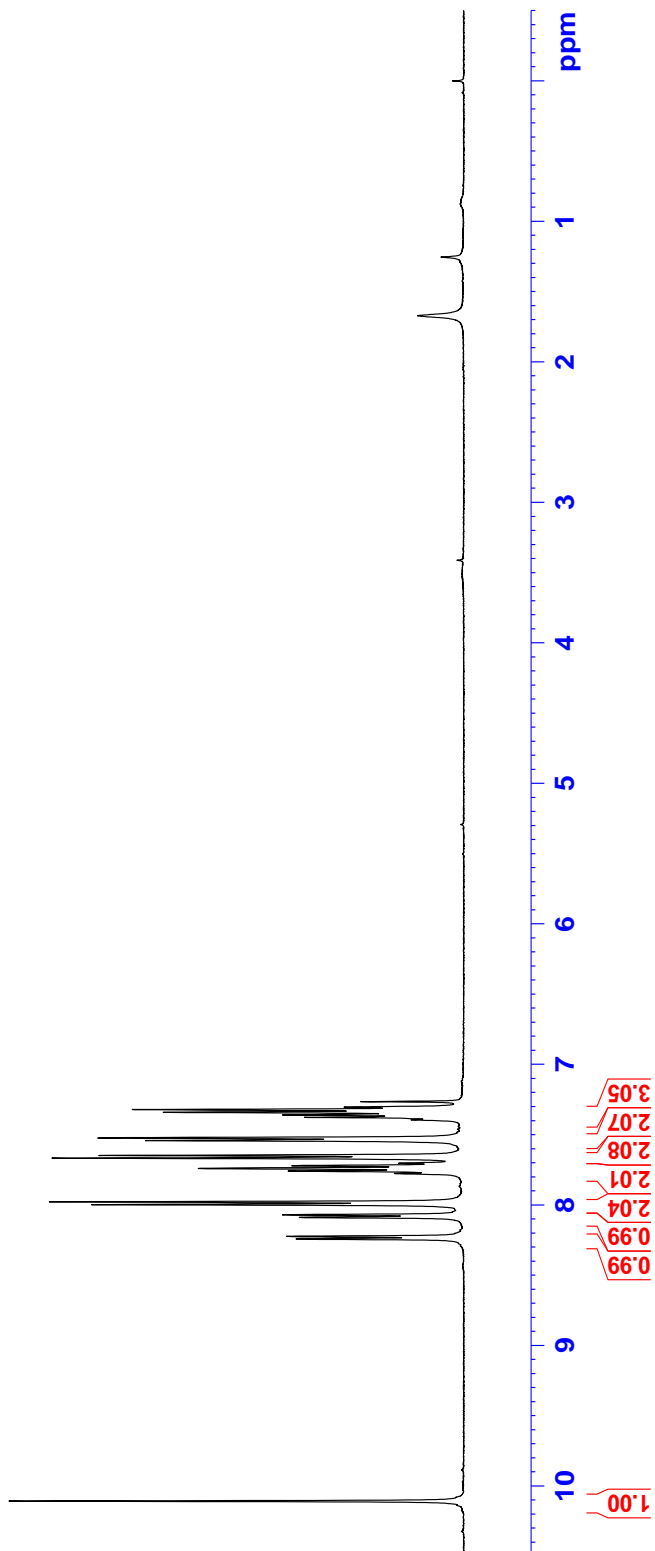
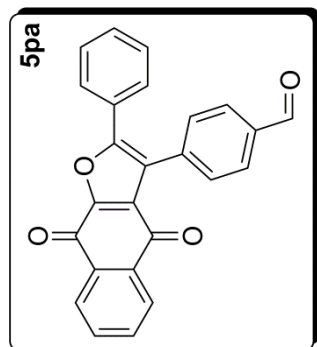


Current Data Parameters  
NAME ken396  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20111219  
Time 18.10  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDC13  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 128  
DW 69.000 usec  
DE 6.50 usec  
TE 299.8 K  
D1 2.0000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300056 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00





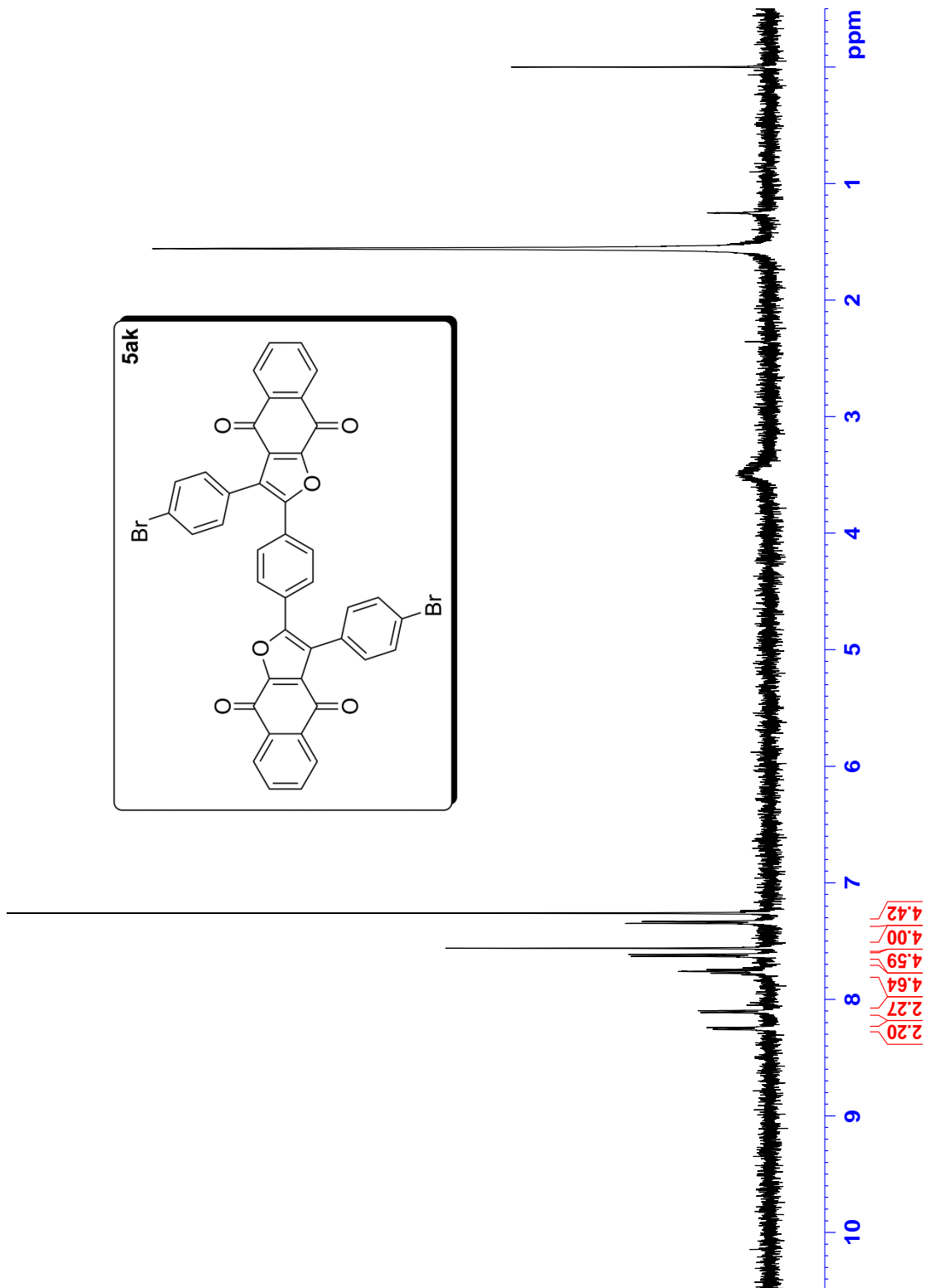
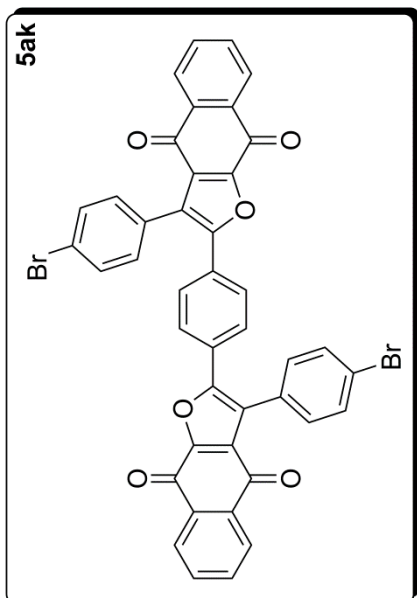


Current Data Parameters  
NAME Ren 301  
EXNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120531  
Time\_ 20.56  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 1  
DS 0  
SWH 9057.971 Hz  
FIDRES 0.276427 Hz  
AQ 1.8088436 sec  
RG 382  
DW 55.200 usec  
DE 6.50 usec  
TE 294.17 K  
D1 2.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 0 dB  
SFO1 500.1330008 MHz

F2 - Processing parameters  
SI 16384  
SF 500.1300118 MHz  
WDW EM  
SSB 0 Hz  
LB 0  
GB 0  
PC 1.00



```

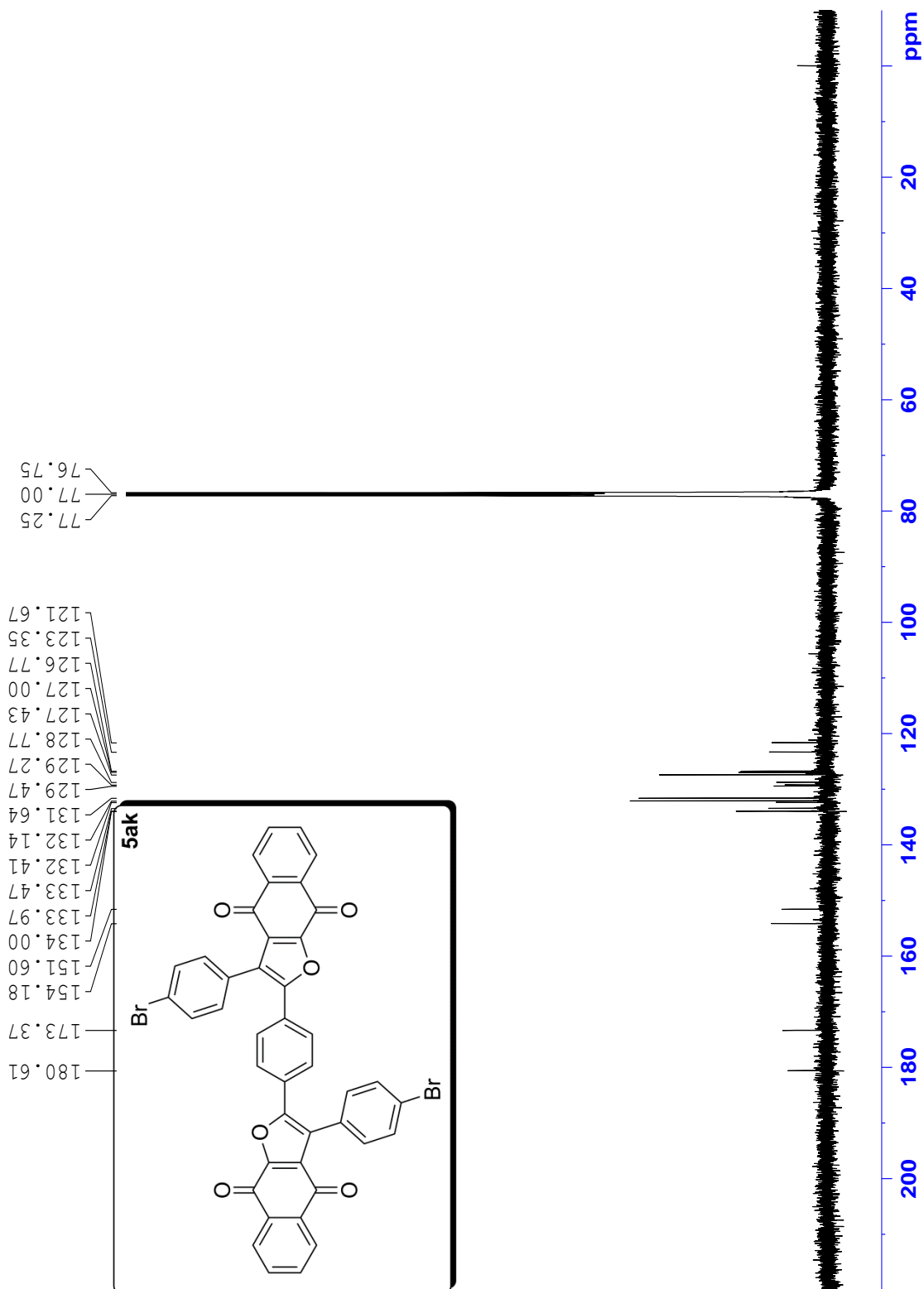
Current Data Parameters
NAME      Ken 381
EXPNO    2
PROCNO   1

F2 - Acquisition Parameters
Date_    20120531
Time_    20.58
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       32768
SOLVENT  CDCl3
NS       40550
DS       30030.029 Hz
SWH      0.458222 Hz
FIDRES   1.0912244 sec
AQ       9195.2
RG       16.650 usec
DE       6.50 usec
TE       295.2 K
D1       2.0000000 sec
D11      0.0300000 sec
TDO      1

===== CHANNEL f1 =====
NUC1     13C
P1       10.50 usec
PL1      7.00 dB
SFO1     125.7709931 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    90.00 usec
PL2      -0.60 dB
PL12     15.00 dB
PL13     18.00 dB
SFO2     500.1320005 MHz

F2 - Processing parameters
SI       65596
SF       125.7577831 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.00
    
```

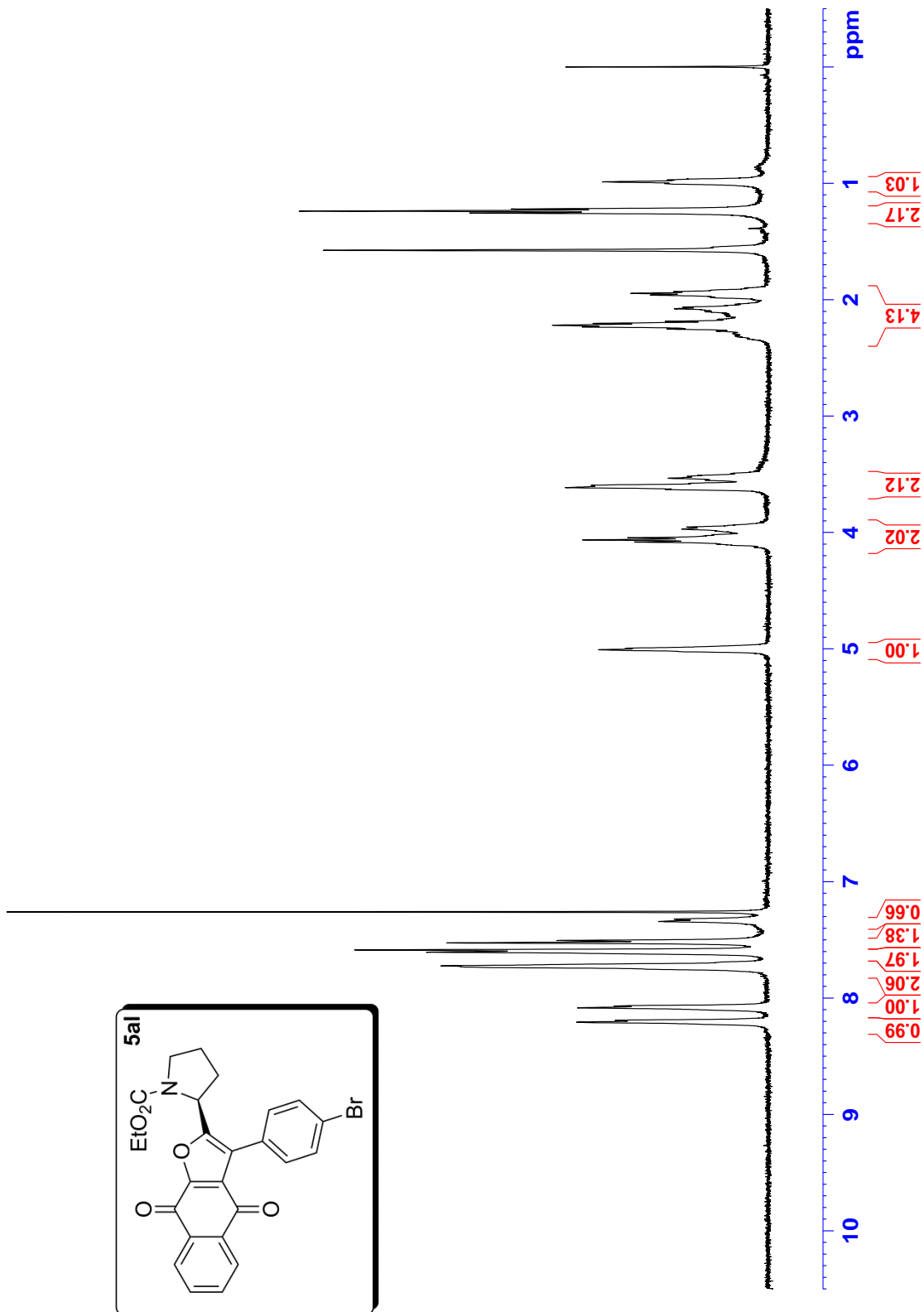


Current Data Parameters  
NAME Ken360  
EXNO 6  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20111108  
Time\_ 20.31  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
DS 8  
NS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 256  
DW 69.000 usec  
DE 6.50 usec  
TE 299.2 K  
D1 2.00000000 sec  
TDO 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

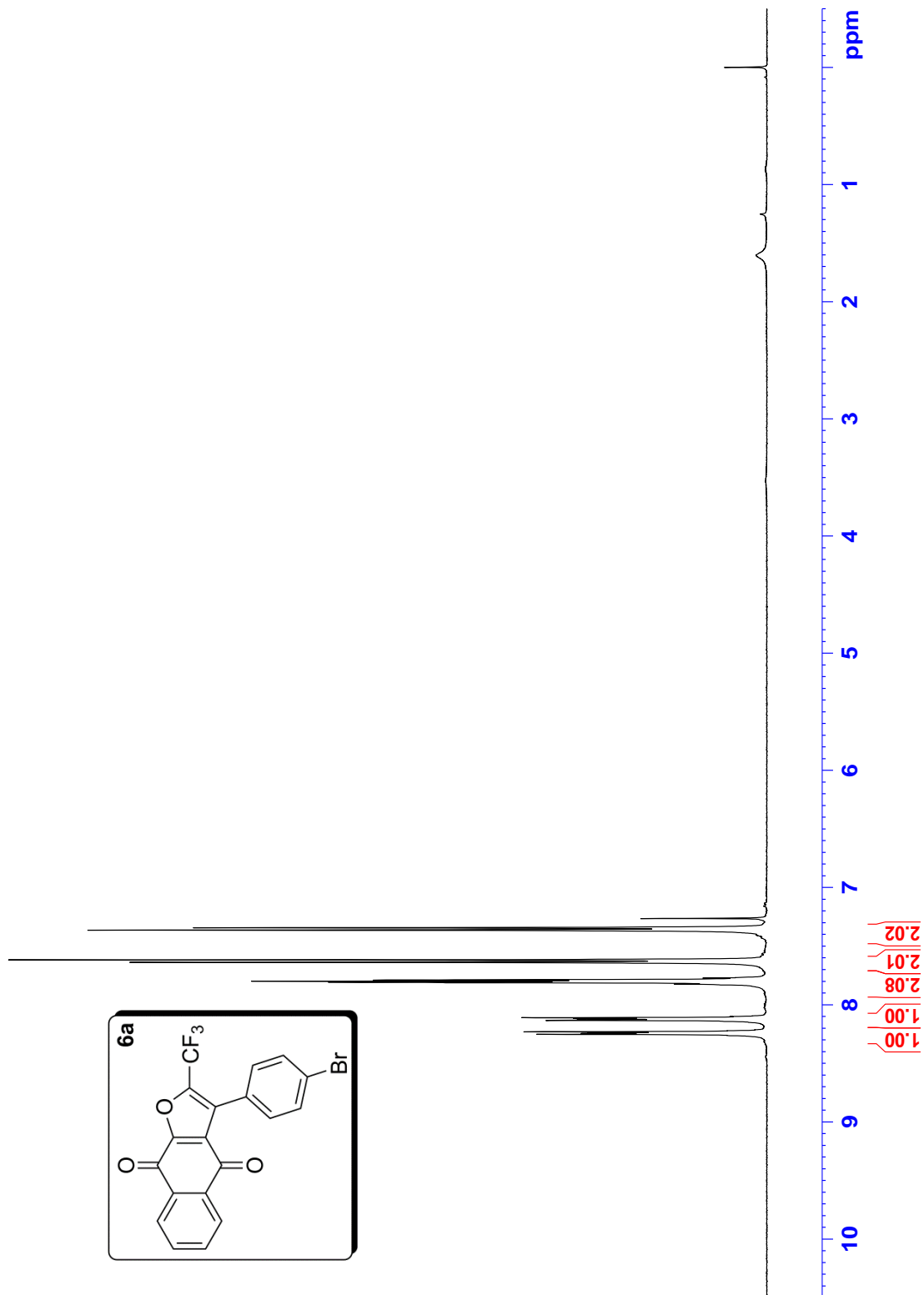
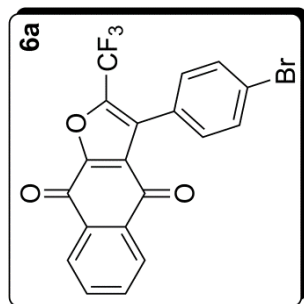
F2 - Processing parameters  
SI 16384  
WDW EM  
SSB 0 Hz  
LB 0 Hz  
GB 0  
PC 1.00



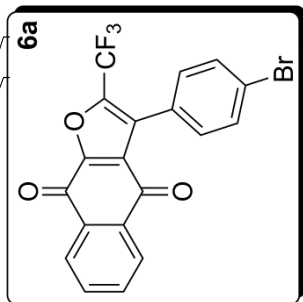


```
NAME          ken363
EXPNO         3
PROCNO        1
Date_         20111225
Time_         18.04
INSTRUM       spect
PROBHD        5 mm BBO BB-LH
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
NS            8
DS            0
SWH           7246.377 Hz
FIDRES        0.221142 Hz
AQ            2.261110 sec
RG            128
DW            69.000 usec
DE            6.50 usec
TE            296.8 K
D1            2.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            11.70 usec
PL1           4.00 dB
SFO1          400.1324008 MHz
SI            16384
SF            400.1300000 MHz
WDW           EM
SSB           0
LB            0.00 Hz
GB            0
PC            1.00
```



179.44  
173.37  
152.52  
142.82  
142.41  
141.99  
141.58  
134.63  
134.28  
133.33  
131.86  
131.66  
131.51  
131.29  
127.27  
127.24  
127.01  
126.70  
126.67  
125.31  
124.10  
122.65  
119.96  
117.27  
114.57  
77.32  
77.00  
76.68



```

NAME          ken363
EXPNO         6
PROCNO        1
Date_         20111225
Time_        22.33
INSTRUM       spect
PROBHD        5 mm BBO BB-IH
PULPROG       zgpg30
TD            32768
SOLVENT       CDCl3
NS            2064
DS            0
SMH           24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6816432 sec
RG            4096
DW            20.800 usec
DE            6.50 usec
TE            300.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            10.50 usec
PL1           7.00 dB
SFO1          100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         90.00 usec
PL2           -0.60 dB
PLI2          15.00 dB
PLI3          18.00 dB
SFO2          400.1316005 MHz
SI            32768
SF            100.6127690 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.00
    
```

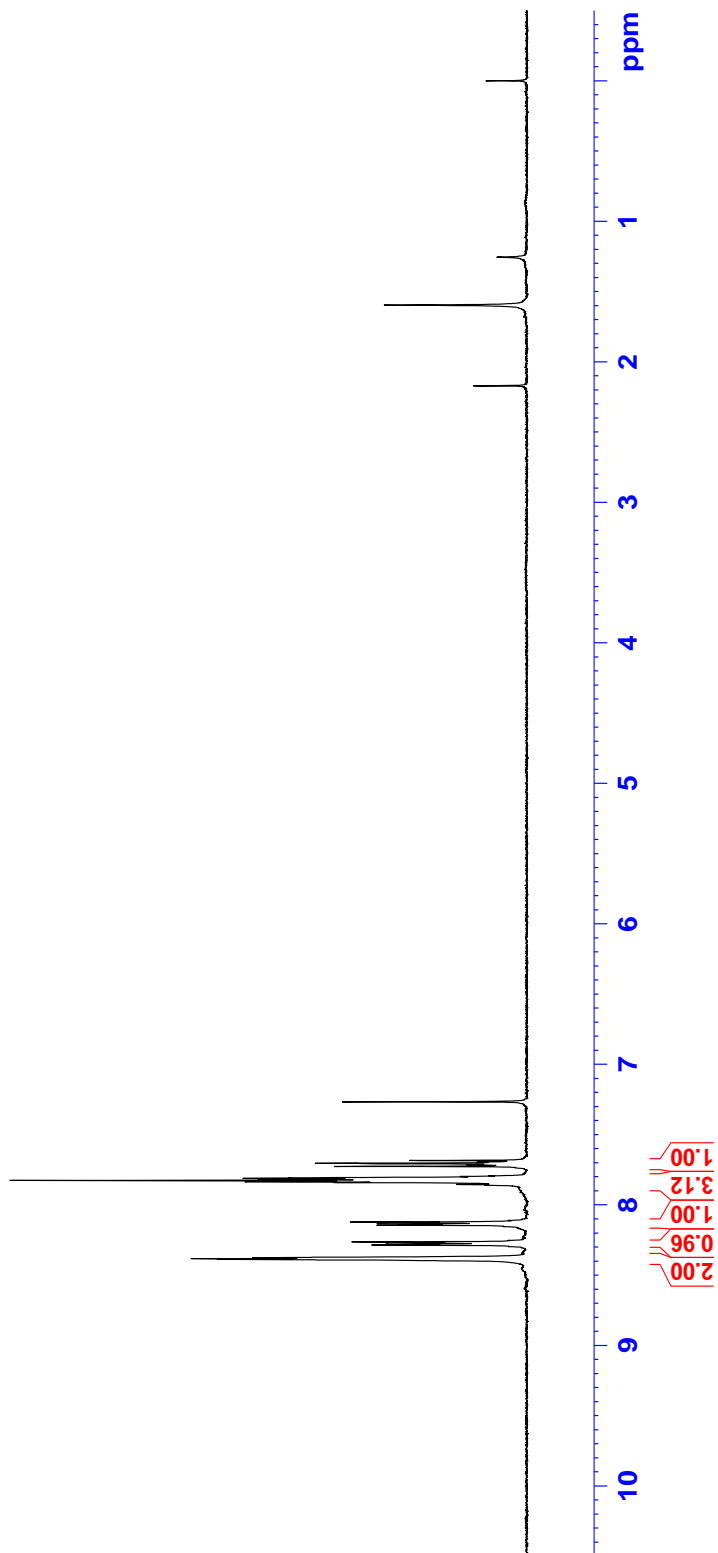
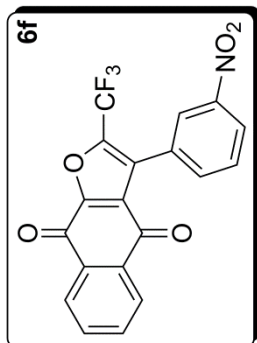


Current Data Parameters  
NAME ken377  
EXPNO 5  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20111105  
Time 21.13  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDC13  
NS 1  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 228.1  
DW 69.000 usec  
DE 6.50 usec  
TE 299.2 K  
D1 2.0000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300055 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00





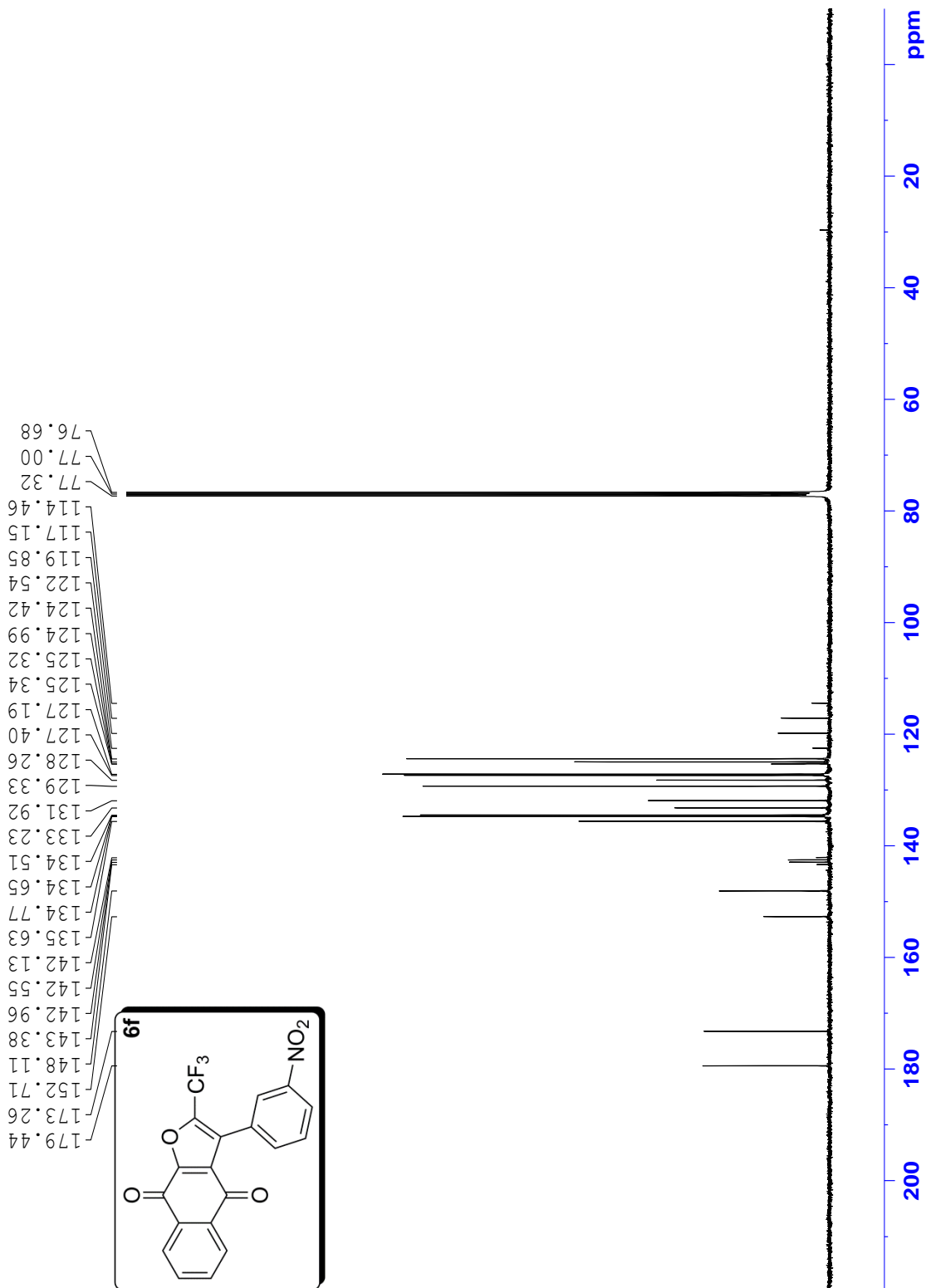
```
Current Data Parameters
NAME      ken377
EXPNO     8
PROCNO    1

F2 - Acquisition Parameters
Date_     20111106
Time_     0.04
INSTRUM   spect
PROBHD    5 mm BBO BB-IH
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         13509
DS         0
SWH        24038.461 Hz
FIDRES     0.733536 Hz
AQ         0.16816244 sec
RG         4096
DE         20.800 usec
TE         301.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TDO        1

===== CHANNEL f1 =====
NUC1       13C
P1         10.50 usec
PL1        7.00 dB
SFO1       100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      90.00 usec
PL2        -0.60 dB
PL12       15.00 dB
PL13       18.00 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI         32768
SF         100.6127668 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00
```

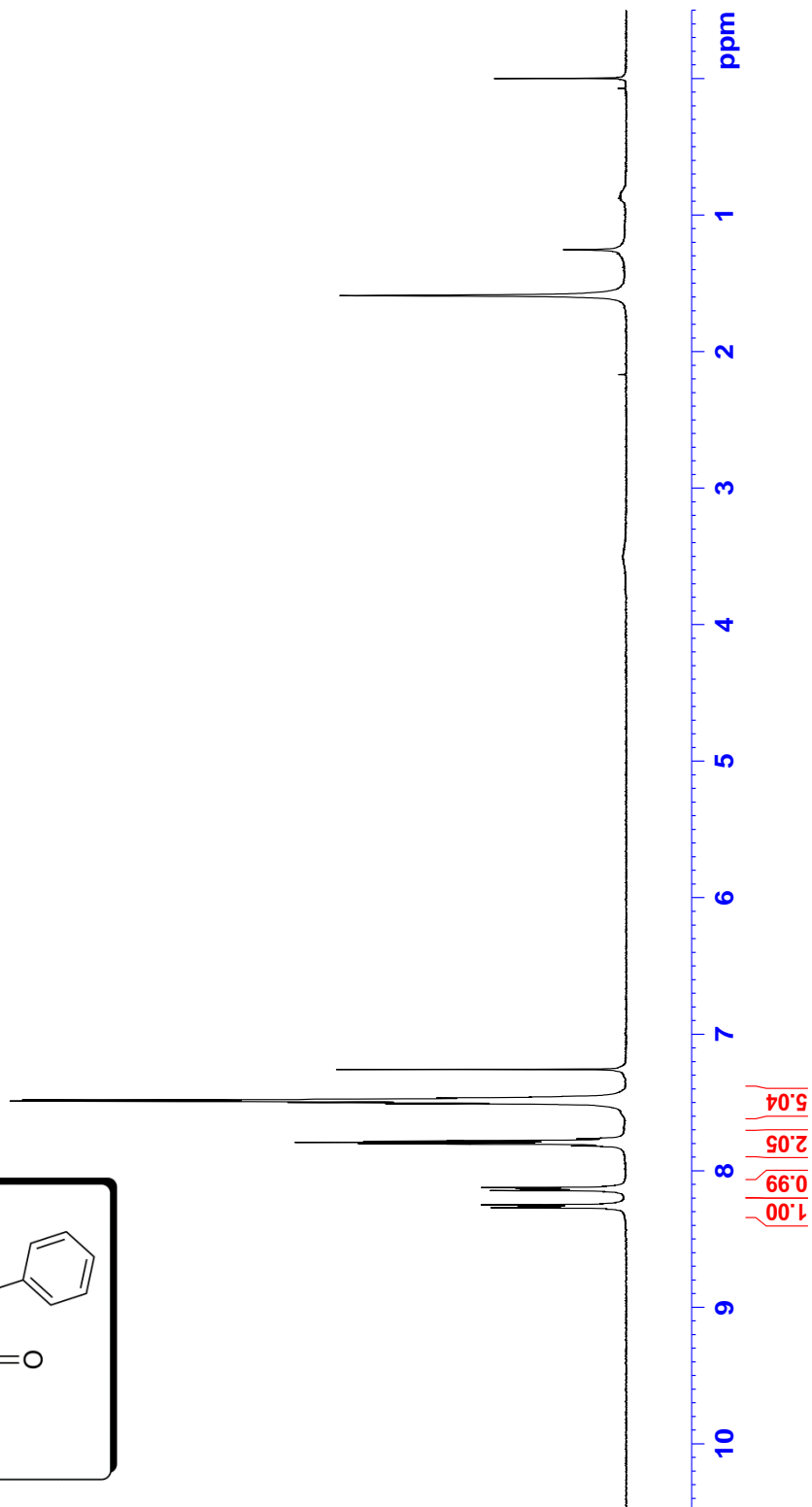
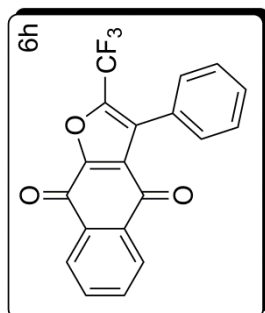


Current Data Parameters  
NAME ken365  
EXPNO 3  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20111026  
Time 12.20  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDC13  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 228.1  
DW 69.000 usec  
DE 6.50 usec  
TE 299.3 K  
D1 2.0000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300088 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00



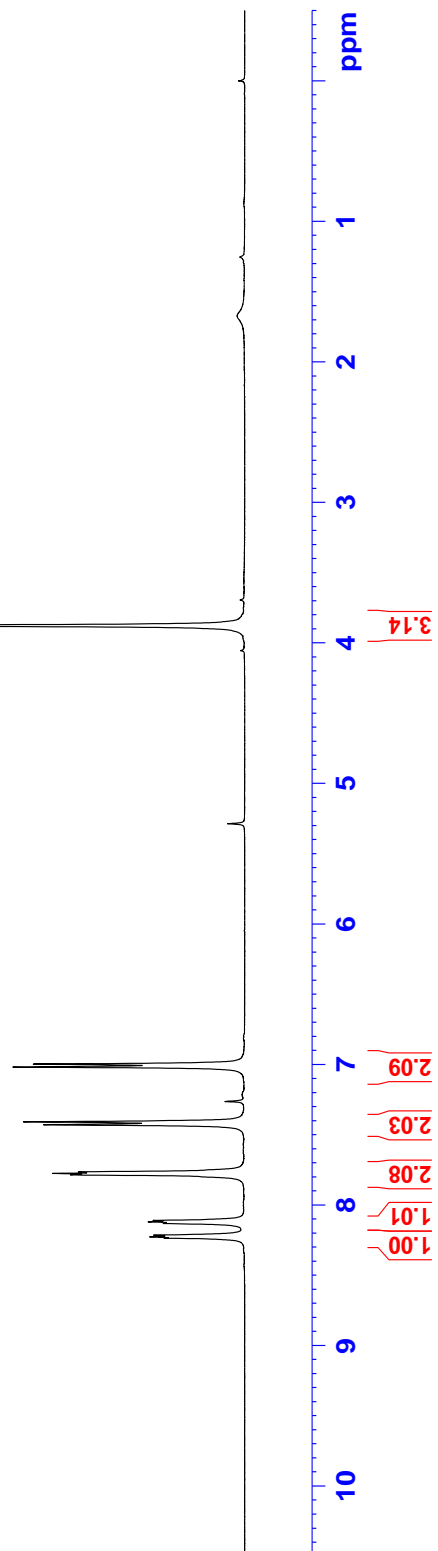
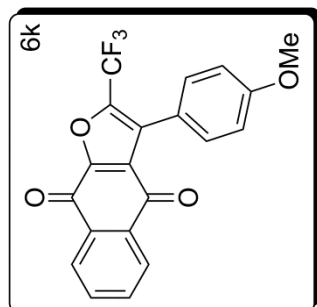


Current Data Parameters  
NAME ken366  
EXPNO 4  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120228  
Time 10.39  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDC13  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 90.5  
DW 69.000 usec  
DE 6.50 usec  
TE 297.2 K  
D1 2.0000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300062 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00



```

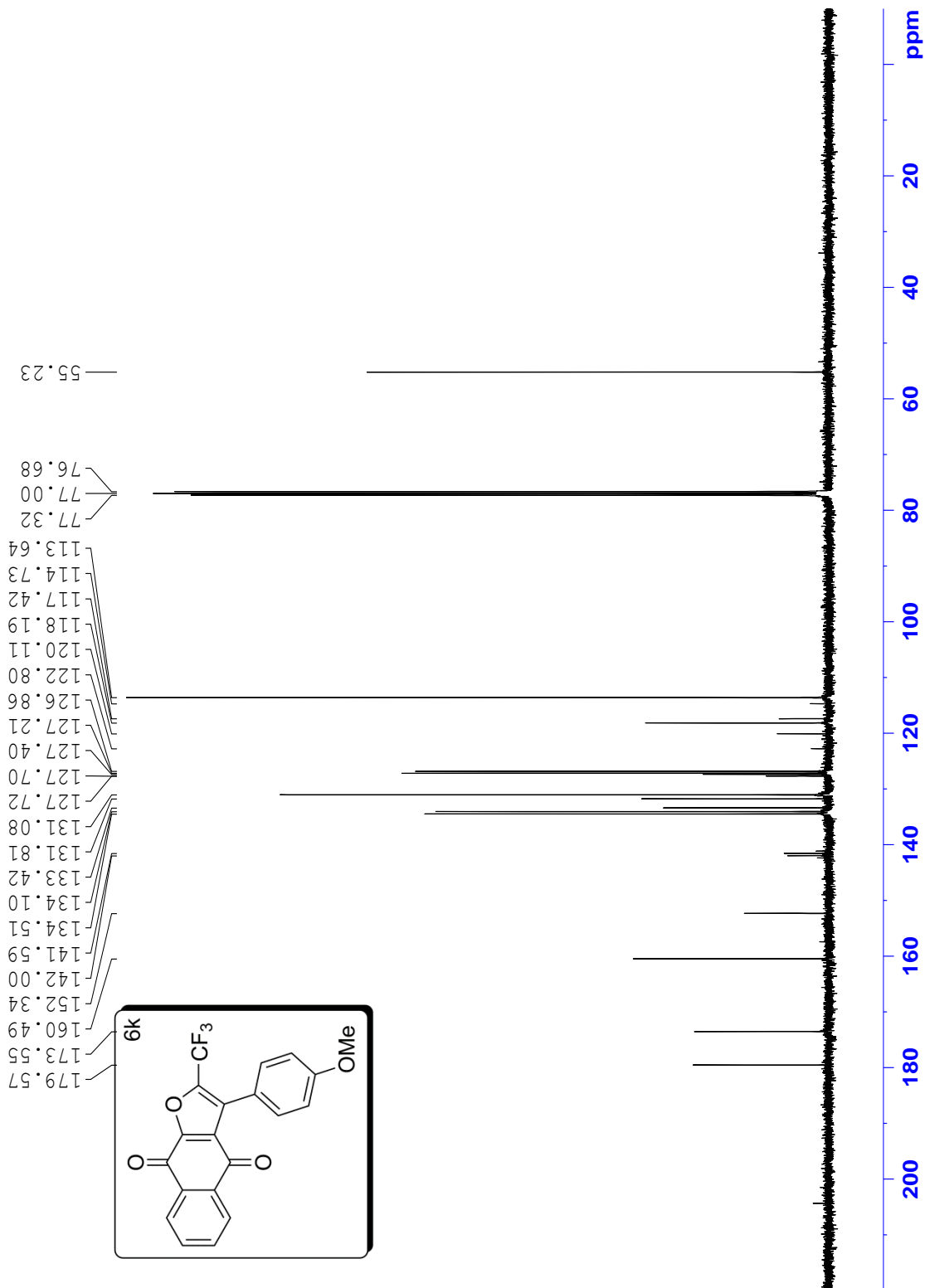
Current Data Parameters
NAME      ken366
EXPNO    5
PROCNO   1

F2 - Acquisition Parameters
Date_    20120228
Time_    10.41
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zgpg30
TD       32768
SOLVENT  CDCl3
NS       567
DS       0
SWH      24038.461 Hz
FIDRES   0.733536 Hz
AQ       0.6816244 sec
RG       5792.6
DW       20.800 usec
DE       6.50 usec
TE       297.4 K
D1       2.0000000 sec
D11      0.0300000 sec
TDO      1

===== CHANNEL f1 =====
NUC1     13C
P1       8.90 usec
PL1      7.00 dB
SFO1     100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    90.00 usec
PL2      3.80 dB
PL12     21.60 dB
PL13     24.60 dB
SFO2     400.1316005 MHz

F2 - Processing parameters
SI       32768
SF       100.6127746 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.00
    
```

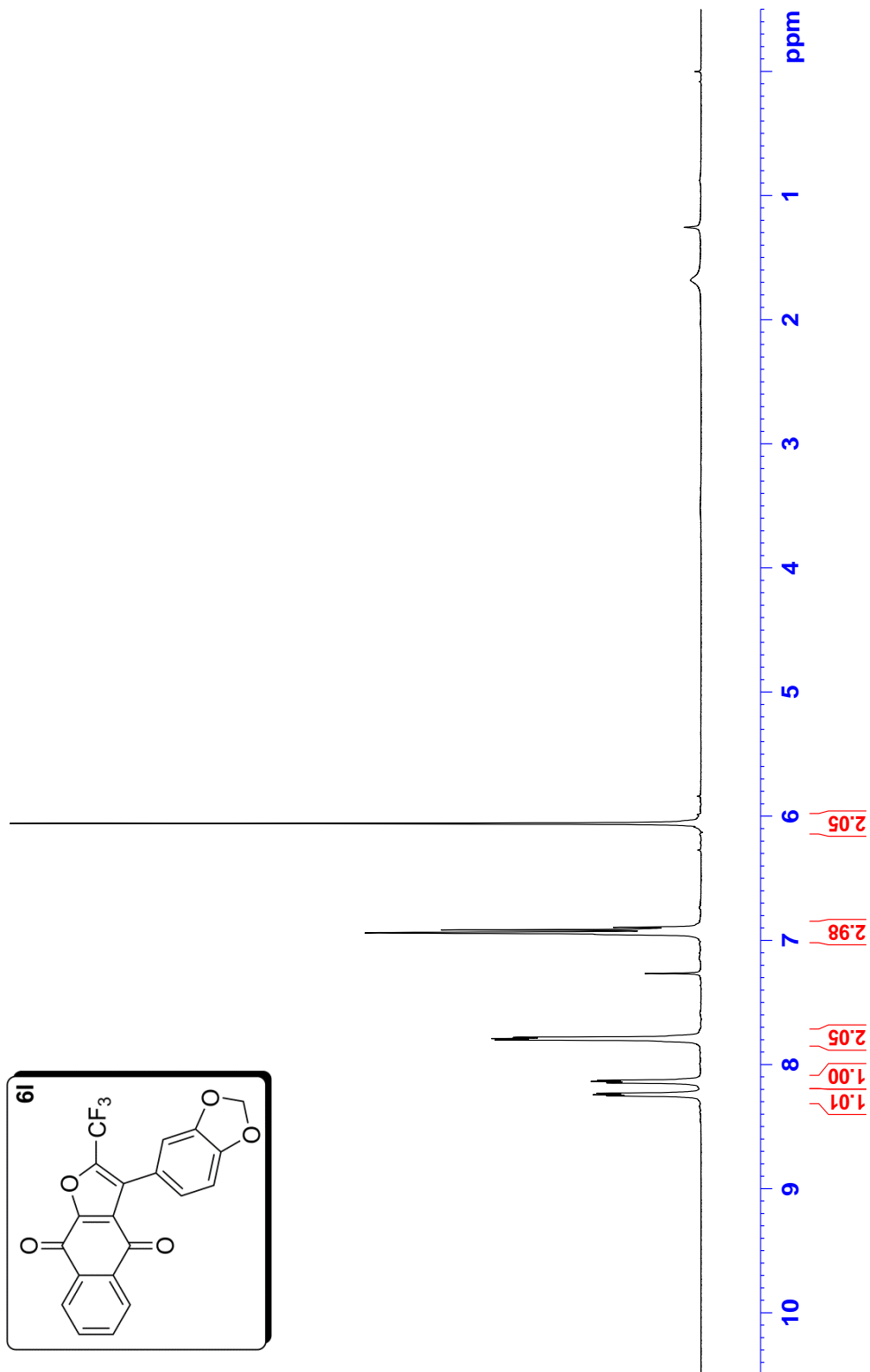
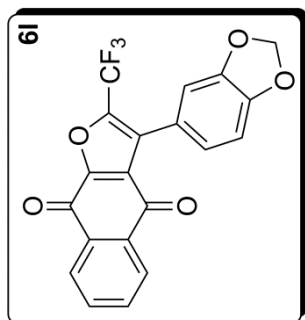


```
Current Data Parameters
NAME          ken382
EXPNO         12
PROCNO        1

F2 - Acquisition Parameters
Date_         20111219
Time         12.06
INSTRUM      spect
PROBHD       5 mm BBO BB-IH
PULPROG      zg30
TD           32768
SOLVENT      CDCl3
NS           8
DS           0
SWH          7246.377 Hz
FIDRES       0.221142 Hz
AQ           2.2610421 sec
RG           161.3
DW           69.000 usec
DE           6.50 usec
TE           299.1 K
D1           2.0000000 sec
TD0          1

===== CHANNEL f1 =====
NUC1         1H
P1           11.70 usec
PL1         4.00 dB
SFO1         400.1324008 MHz

F2 - Processing parameters
SI           16384
SF           400.1300057 MHz
WDW          EM
SSB          0
LB           0 Hz
GB           0
PC           1.00
```



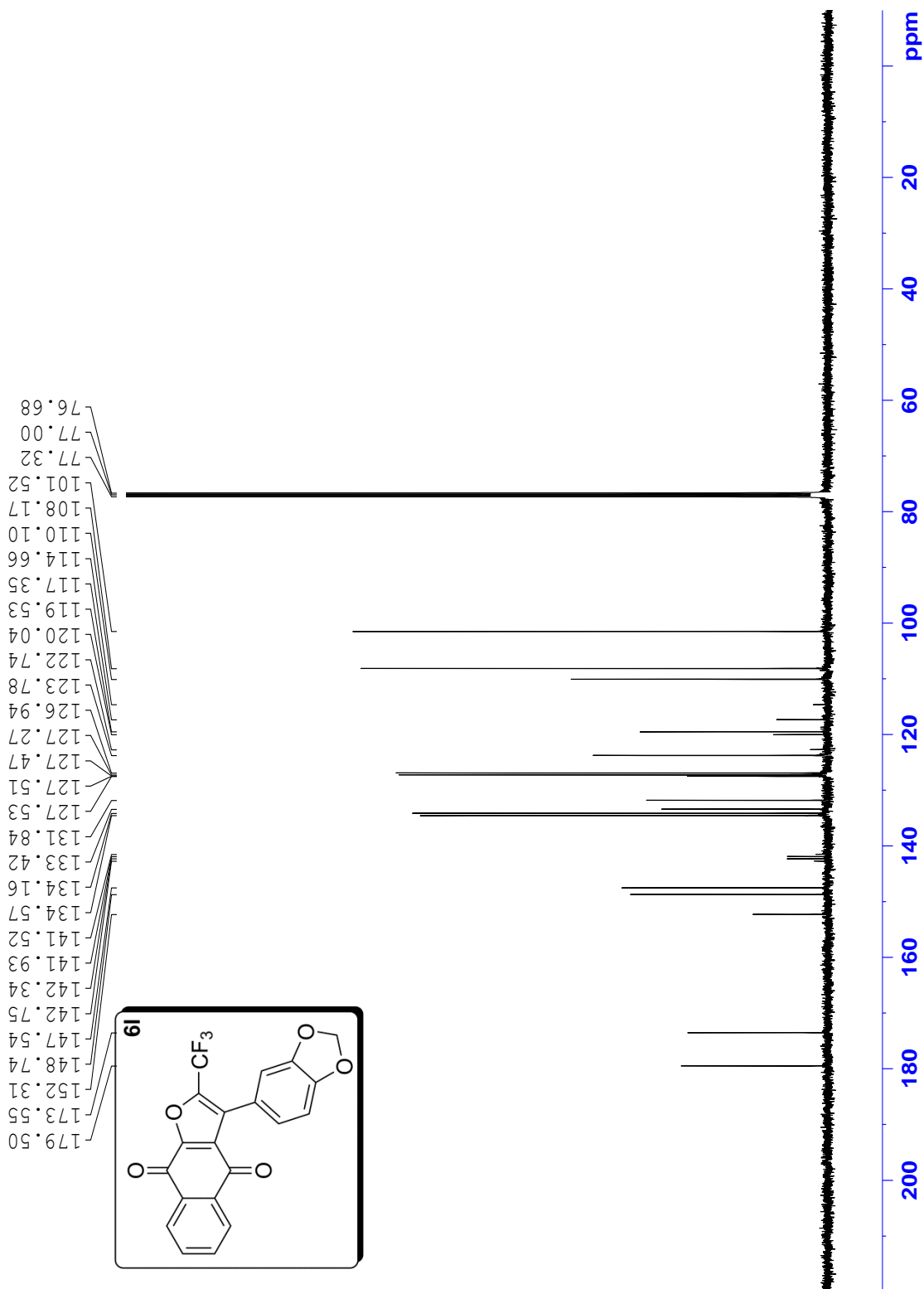
Current Data Parameters  
NAME ken382  
EXPNO 13  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20111219  
Time\_ 12.15  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 32768  
SOLVENT CDCl3  
NS 1159  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.733536 Hz  
AQ 0.6816244 sec  
RG 8192  
DW 20.800 usec  
DE 6.50 usec  
TE 299.6 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
TDO 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 8.90 usec  
PL1 7.00 dB  
SFO1 100.6233325 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL2 3.80 dB  
PL12 21.60 dB  
PL13 24.60 dB  
SFO2 400.1316005 MHz

F2 - Processing Parameters  
SI 32768  
SF 100.6127720 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.00

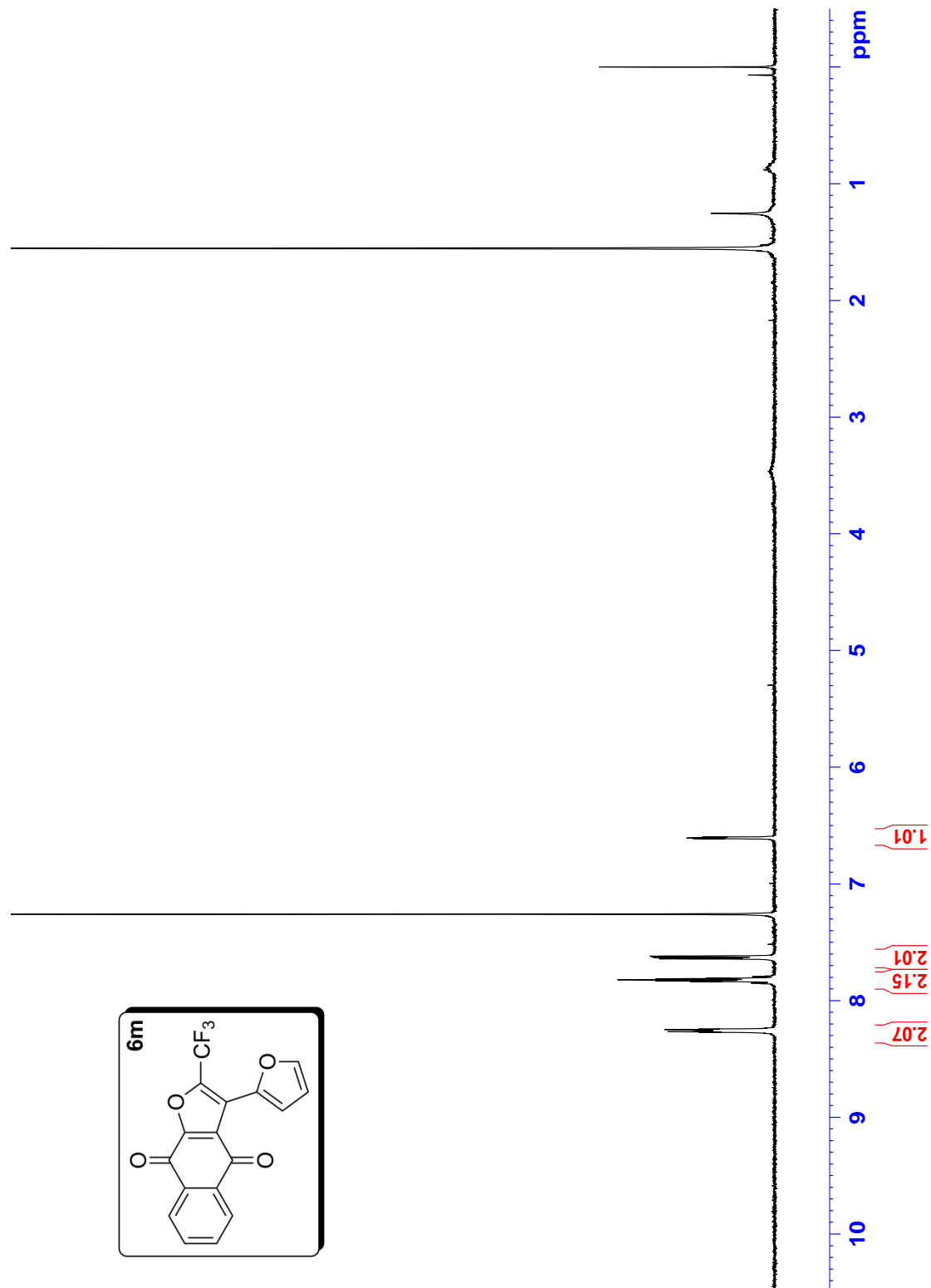


Current Data Parameters  
NAME Ken367  
EXNO 4  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20111125  
Time\_ 11.32  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 382  
DW 69.000 usec  
DE 6.50 usec  
TE 300.5 K  
D1 2.00000000 sec  
TDO 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
WDW EM  
SSB 0 Hz  
LB 0  
GB 0  
PC 1.00





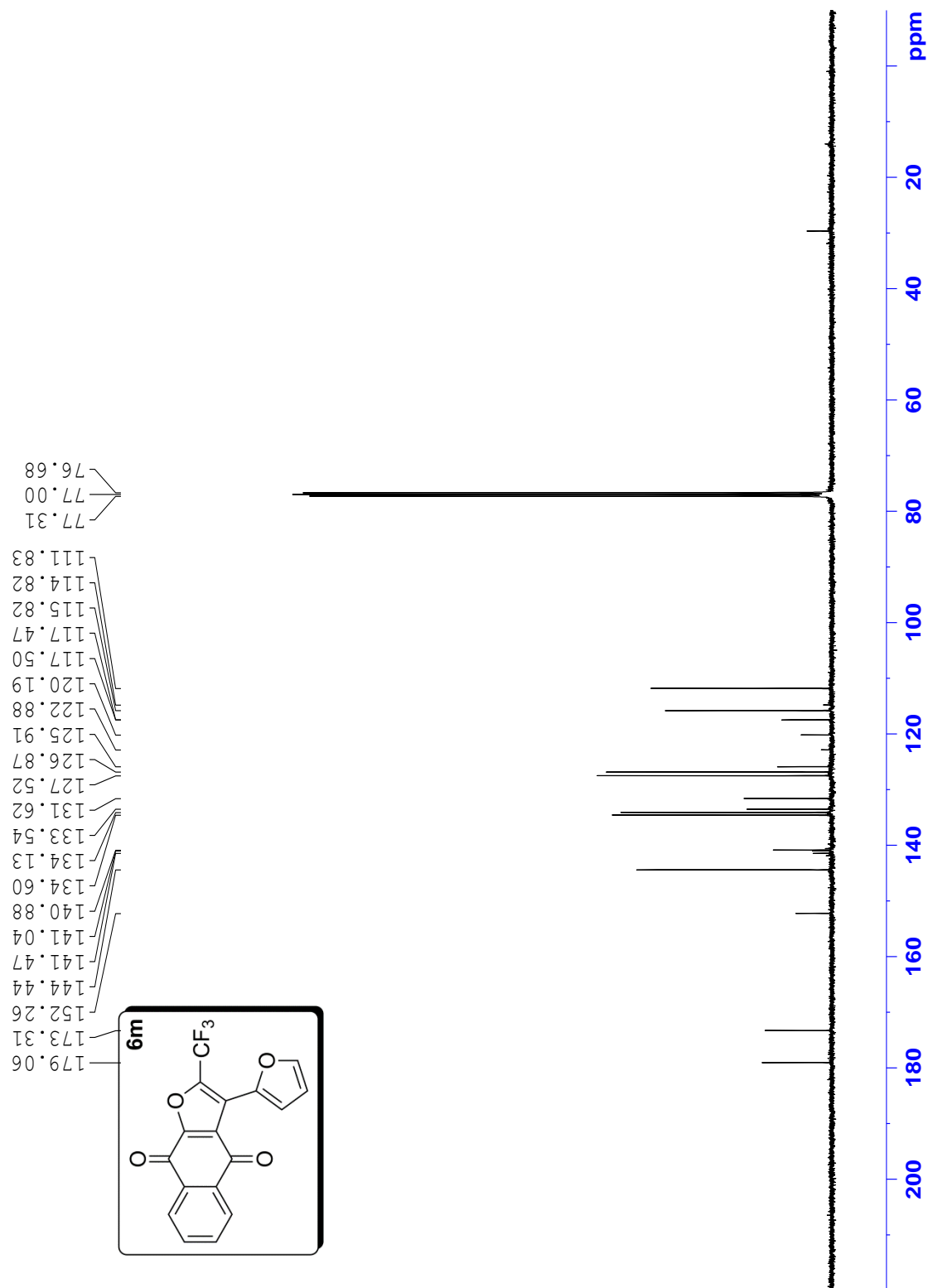
Current Data Parameters  
NAME ken367  
EXPNO 8  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20111126  
Time\_ 17.07  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 32768  
SOLVENT CDCl3  
NS 1309  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.733536 Hz  
AQ 0.16816244 sec  
RG 8192  
DW 20.800 usec  
DE 6.50 usec  
TE 299.8 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
TDO 1000

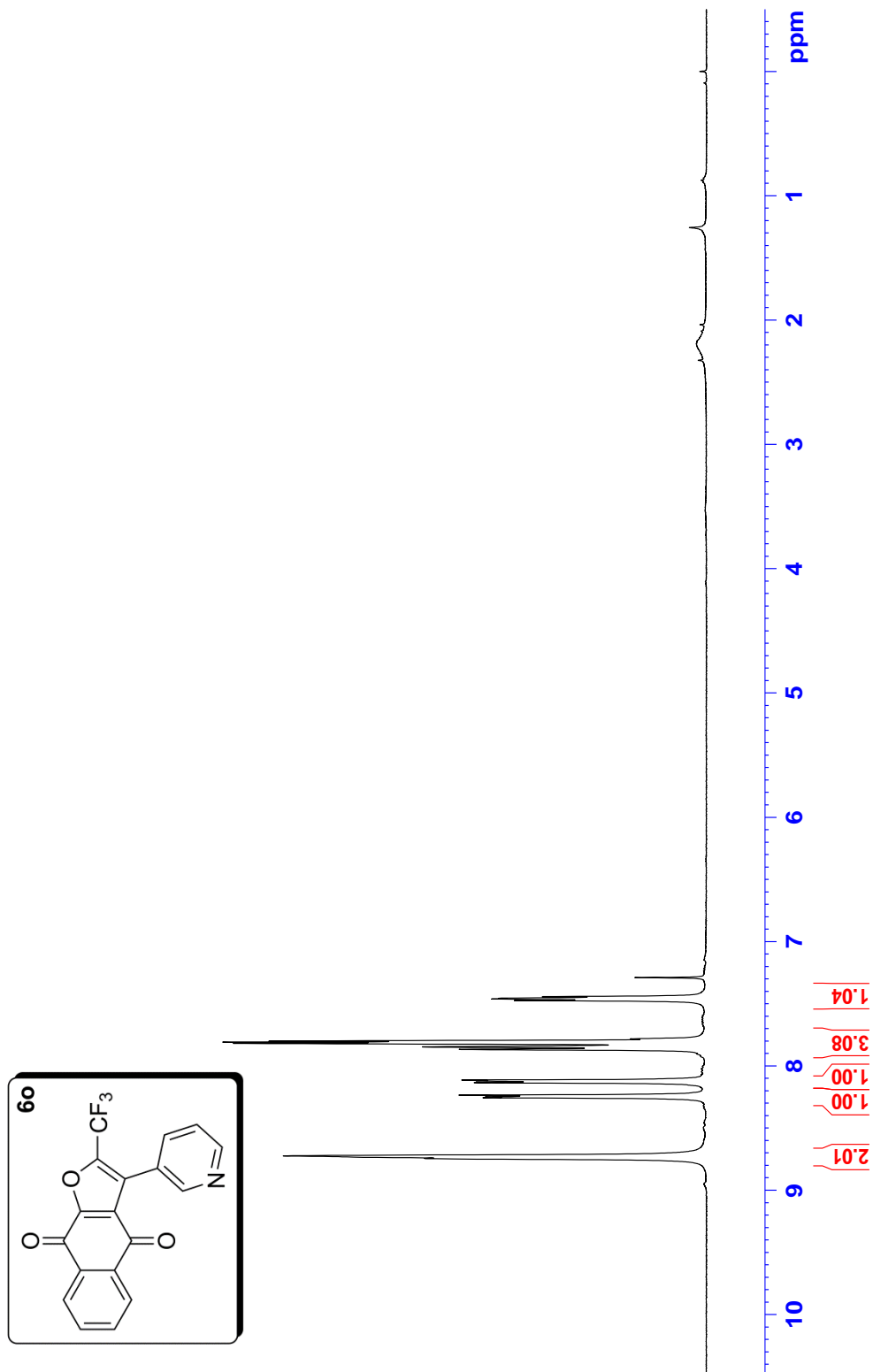
=====  
CHANNEL f1  
NUC1 13C  
P1 10.50 usec  
PL1 7.00 dB  
SFO1 100.6233325 MHz

=====  
CHANNEL f2  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL2 -0.60 dB  
PL12 15.00 dB  
PL13 18.00 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127681 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.00



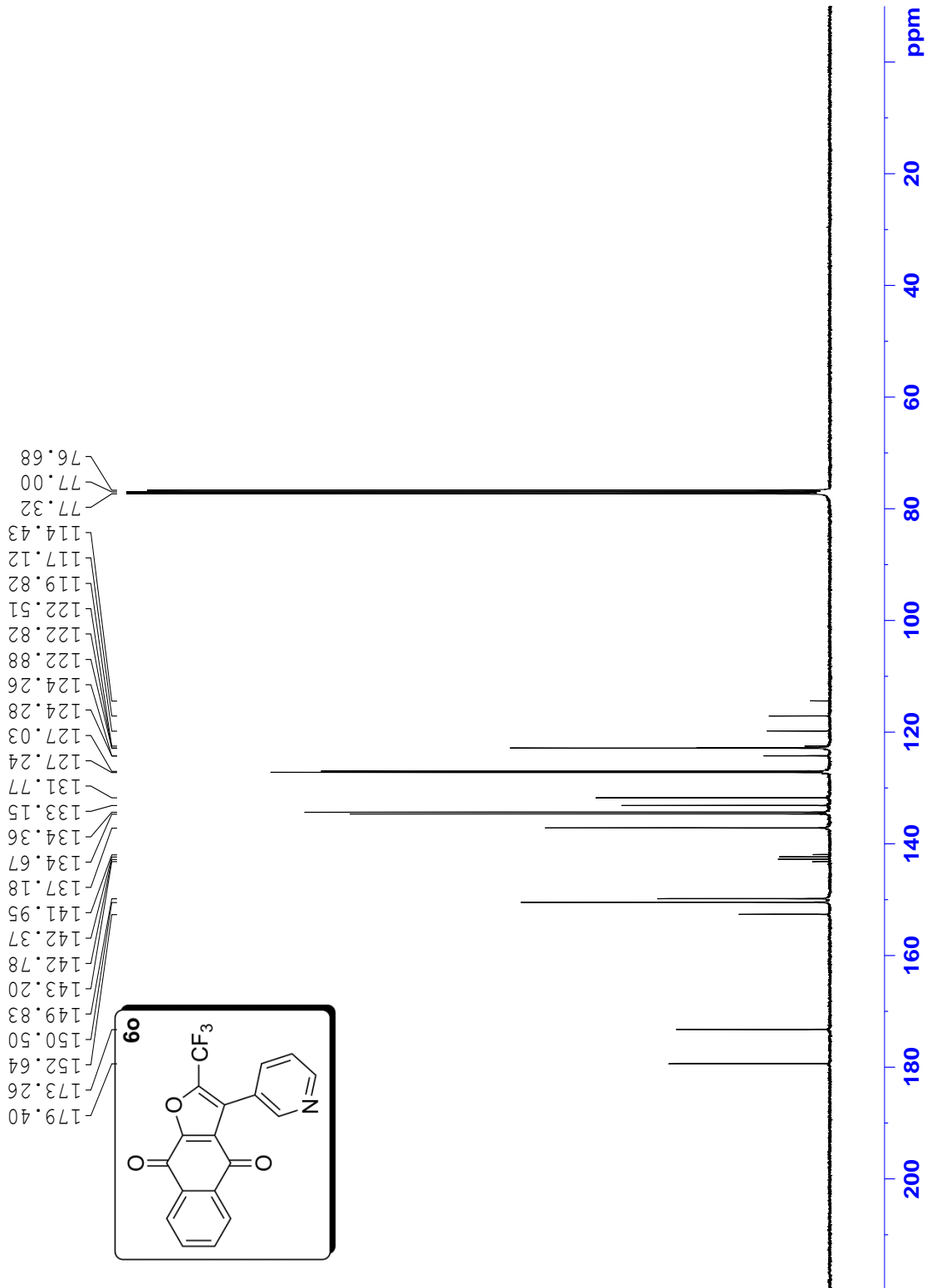
```
NAME          ken393
EXPNO         7
PROCNO        1
Date_         20111223
Time_         1.43
INSTRUM       spect
PROBHD        5 mm BBO BB-JH
PULPROG       zg30
TD            32768
SOLVENT       CDC13
NS            8
DS            0
SWH           7246.377 Hz
FIDRES        0.221142 Hz
AQ            2.261110 sec
RG            114
DW           69.000 usec
DE           6.50 usec
TE            299.9 K
D1            2.0000000 sec
TD0           1
===== CHANNEL f1 =====
NUC1          1H
P1            11.70 usec
PL1           4.00 dB
SFO1          400.1324008 MHz
SI            16384
SF            400.1300000 MHz
WDW           EM
SSB           0
LB            0.00 Hz
GB            0
PC            1.00
```



```
NAME          ken393
EXPNO         8
PROCNO        1
Date_         20111223
Time_         1.49
INSTRUM       spect
PROBHD        5 mm BBO BB-IH
PULPROG       zgpg30
TD            32768
SOLVENT       CDCl3
NS            6000
DS            0
SMH           24038.461 Hz
FIDRES        0.733596 Hz
AQ            0.6816432 sec
RG            4096
DE            20.800 usec
TE            300.3 K
D1            2.00000000 sec
D11          0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            8.90 usec
PL1           7.00 dB
SFO1          100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         90.00 usec
PL2           3.80 dB
PLI2          21.60 dB
PLI3          24.60 dB
SFO2          400.1316005 MHz
SI            32768
SF            100.6127746 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.00
```

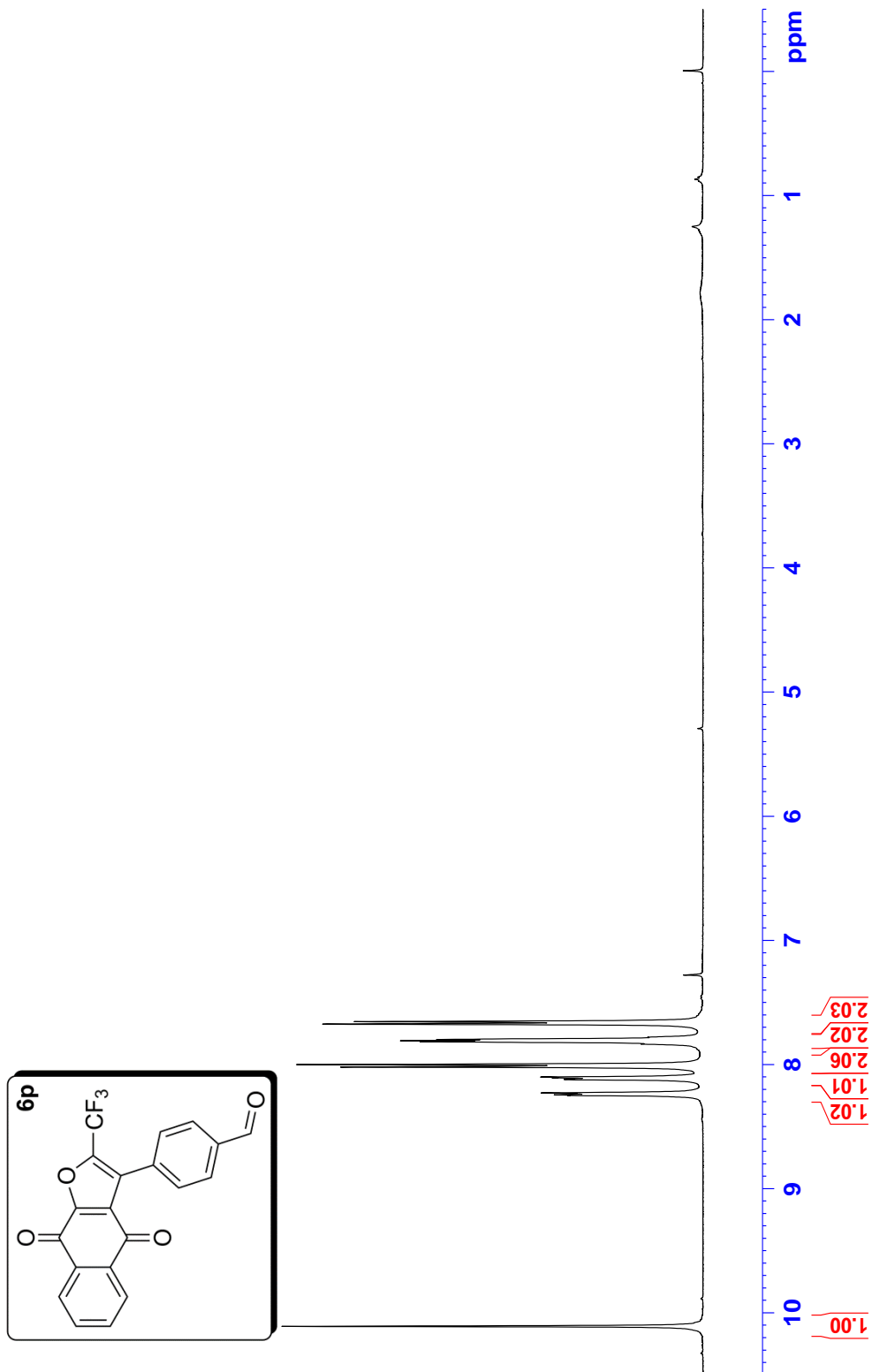


```
Current Data Parameters
NAME          ken370
EXPNO         4
PROCNO        1

F2 - Acquisition Parameters
Date_         20120228
Time         13.19
INSTRUM       spect
PROBHD        5 mm BBO BB-IH
PULPROG       zg30
TD            32768
SOLVENT       CDCl3
DS            4
NS            0
SWH           7246.377 Hz
FIDRES        0.221142 Hz
AQ            2.2610421 sec
RG            128
DE            69.000 usec
TE            297.3 K
D1            2.0000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            11.70 usec
PL1           4.00 dB
SFO1          400.1324008 MHz

F2 - Processing parameters
SI            16384
SF            400.1300000 MHz
WDW           EM
SSB           0
LB            0 Hz
GB            0
PC            1.00
```



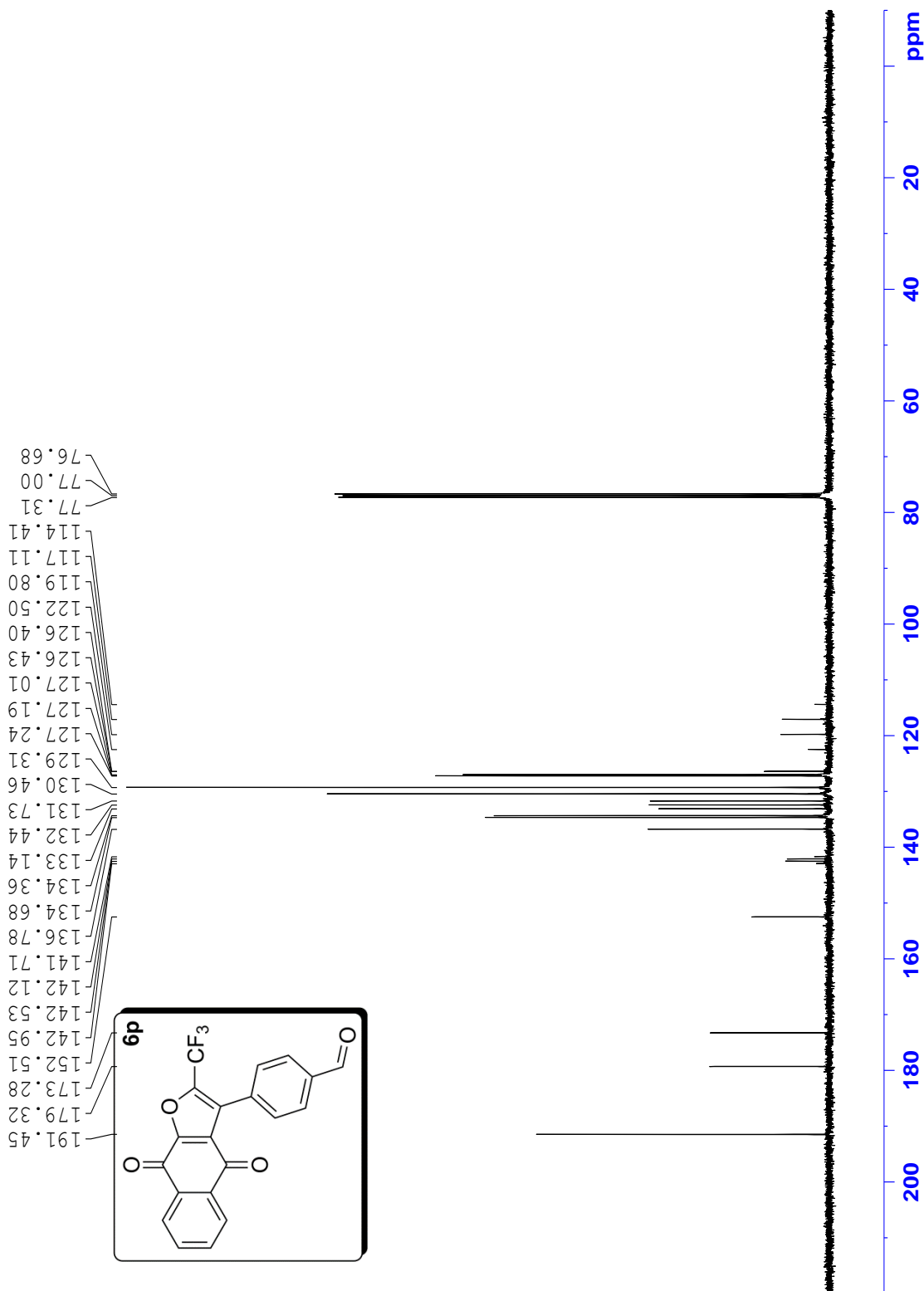
Current Data Parameters  
NAME ken370  
EXPNO 5  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120228  
Time\_ 13.50  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zgpg30  
TD 32768  
SOLVENT CDCl3  
NS 592  
DS 0  
SWH 24038.461 Hz  
FIDRES 0.733536 Hz  
AQ 0.6816244 sec  
RG 4096  
DW 20.800 usec  
DE 6.50 usec  
TE 297.4 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
TDO 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 8.90 usec  
PL1 7.00 dB  
SFO1 100.6233325 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL2 3.80 dB  
PL12 21.60 dB  
PL13 24.60 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127767 MHz  
WDW EM  
SSB 0 1.00 Hz  
LB 0 1.00 Hz  
GB 0  
PC 1.00

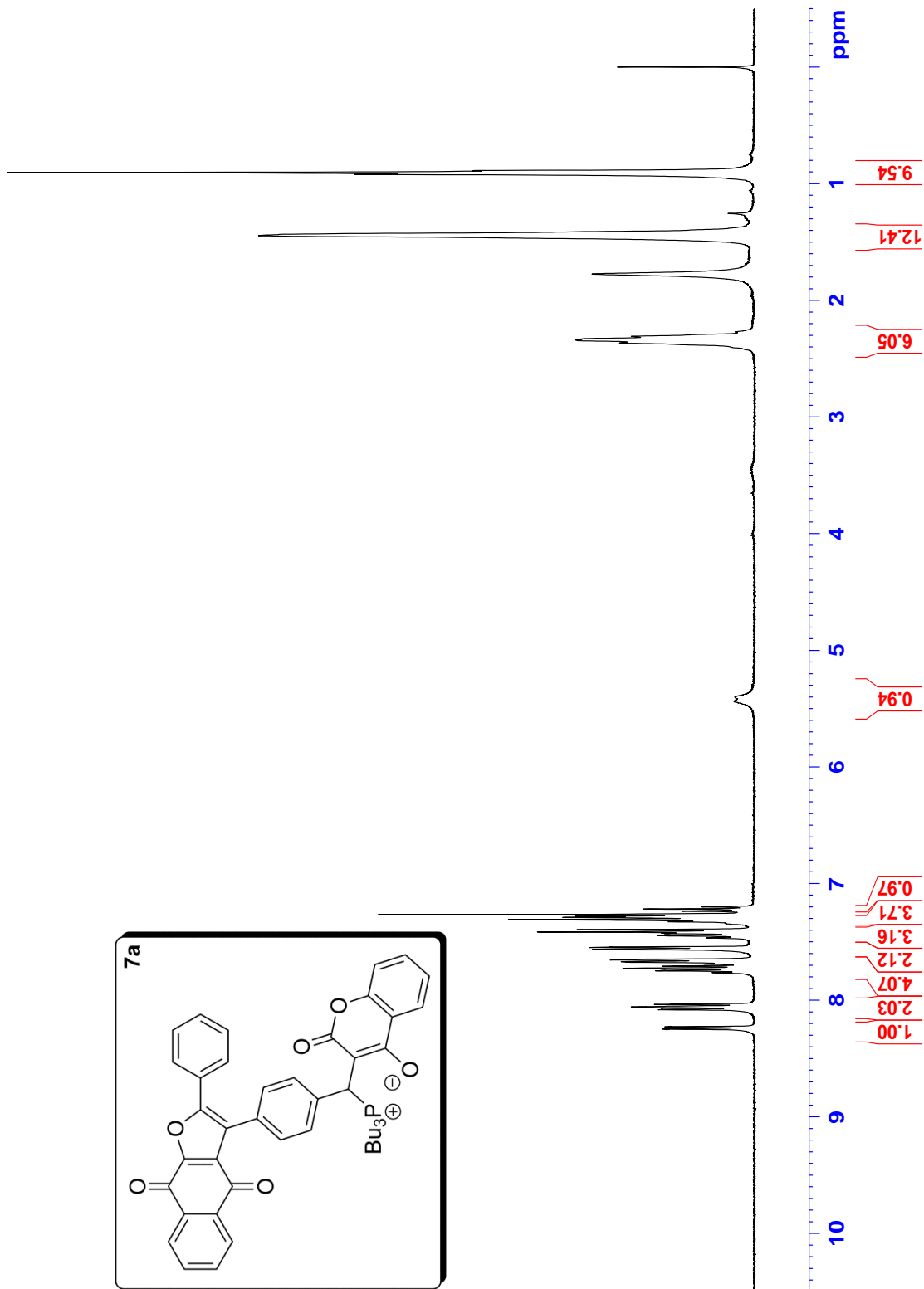
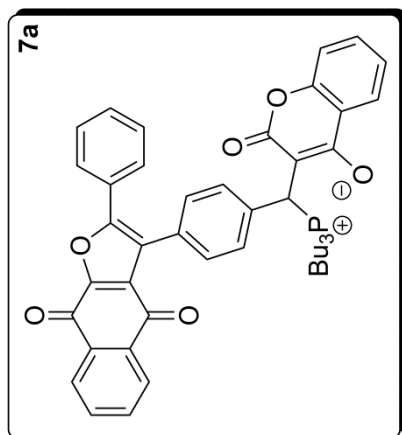


Current Data Parameters  
NAME ken405  
EXPNO 11  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120130  
Time\_ 16.05  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 161  
DW 69.000 usec  
DE 6.50 usec  
TE 299.6 K  
D1 2.0000000 sec  
TDO 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SF01 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300057 MHz  
WDW EM  
SSB 0 Hz  
LB 0  
GB 0  
PC 1.00



```

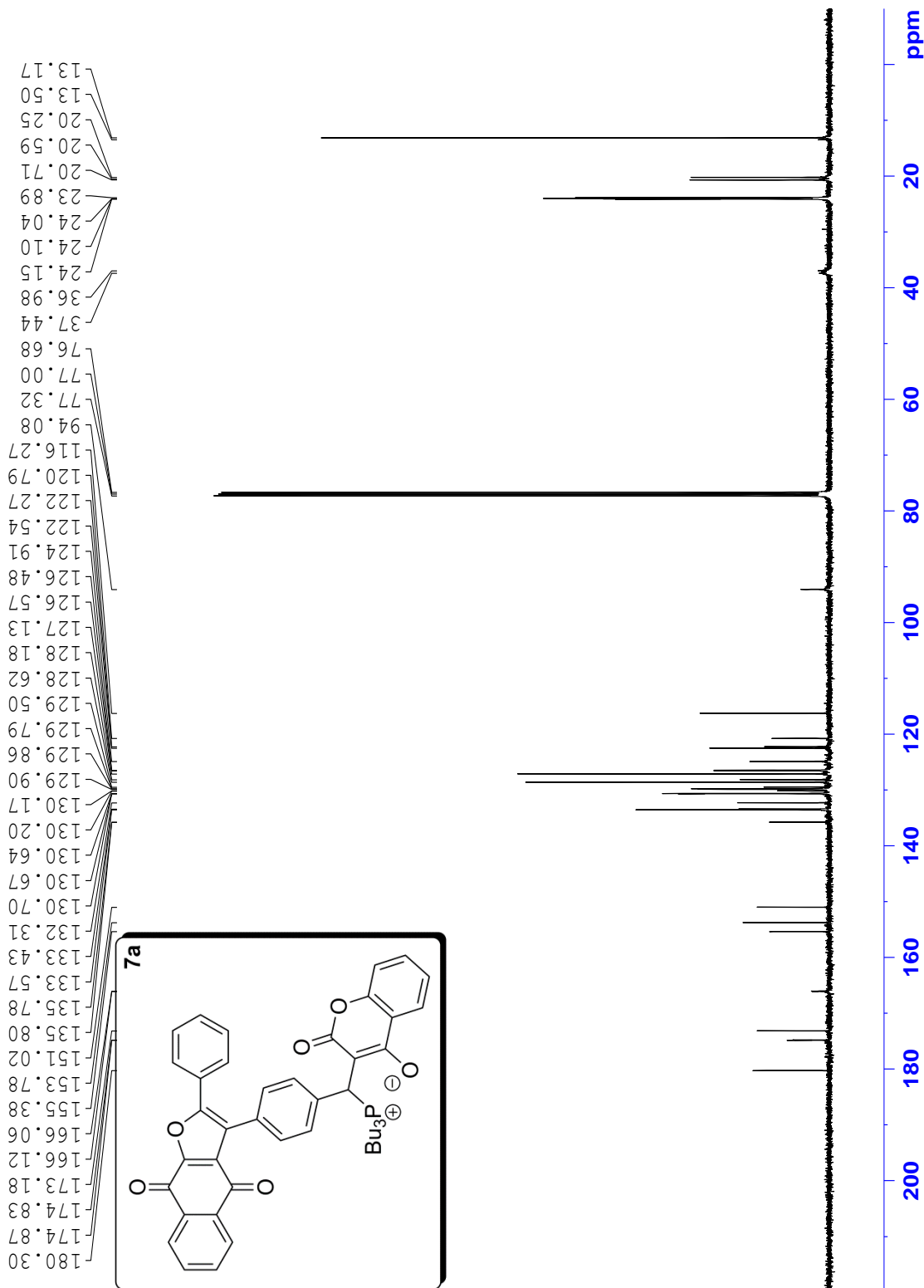
Current Data Parameters
NAME      ken405
EXPNO    14
PROCNO   1

F2 - Acquisition Parameters
Date_    20120314
Time     12.12
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zgpg30
TD       32768
SOLVENT  CDCl3
NS       1404
DS       4
SWH      24038.461 Hz
FIDRES   0.733536 Hz
AQ       0.16816244 sec
RG       4096
DE       20.800 usec
TE       6.50 usec
TE       298.8 K
D1       2.00000000 sec
D11      0.03000000 sec
TDO      1

===== CHANNEL f1 =====
NUC1     13C
P1       8.90 usec
PL1      7.00 dB
SFO1     100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    90.00 usec
PL2      3.80 dB
PL12     21.60 dB
PL13     24.60 dB
SFO2     400.1316005 MHz

F2 - Processing parameters
SI       32768
SF       100.6127799 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.00
    
```





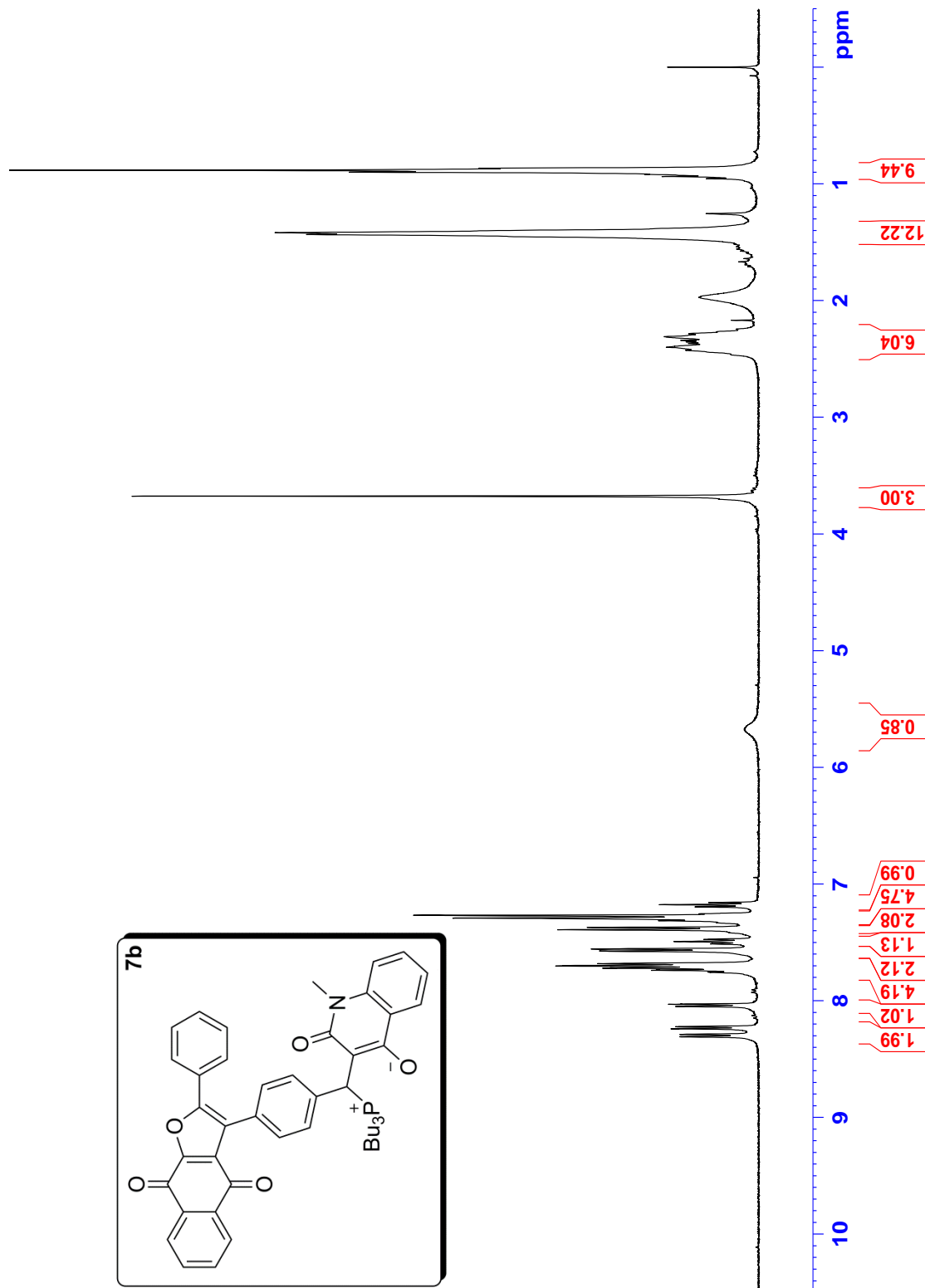
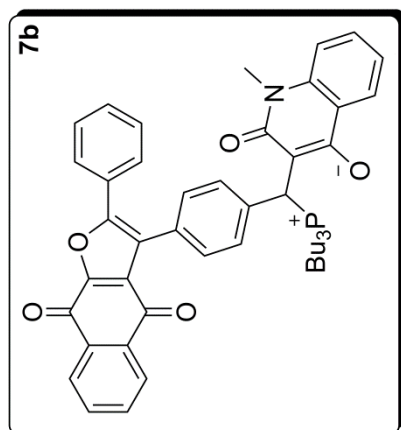


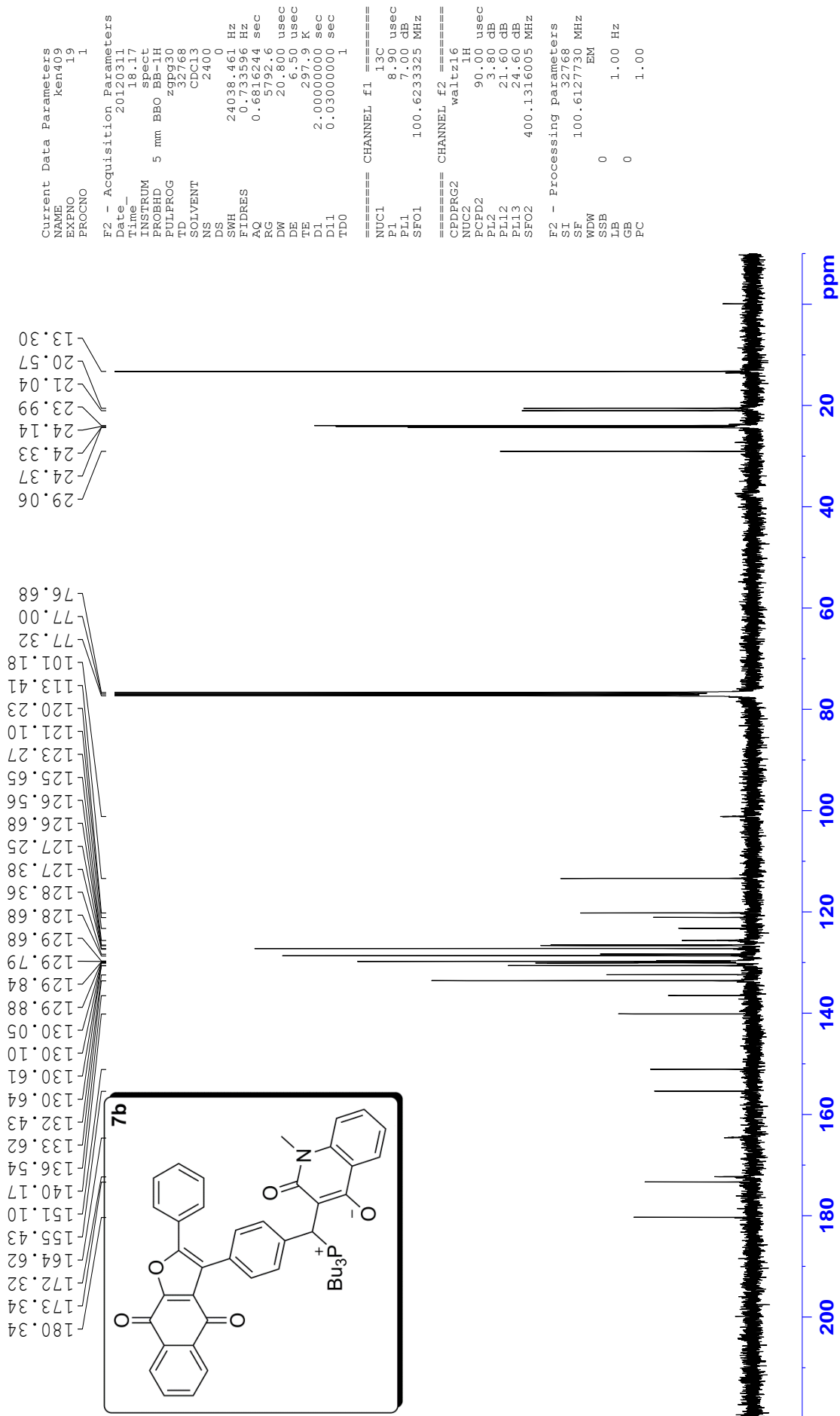
Current Data Parameters  
NAME Ken409  
EXPNO 14  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120213  
Time\_ 15.32  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDC13  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 161.3  
DW 69.000 usec  
DE 6.50 usec  
TE 299.1 K  
D1 2.00000000 sec  
TD0 1

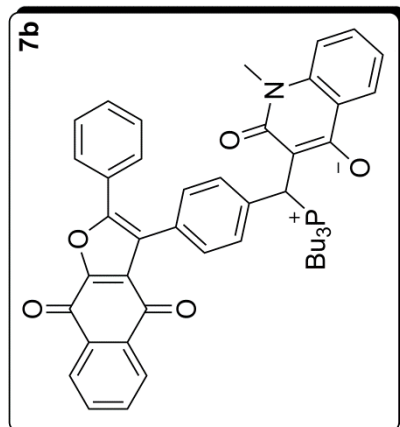
==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300053 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00





— 33.61



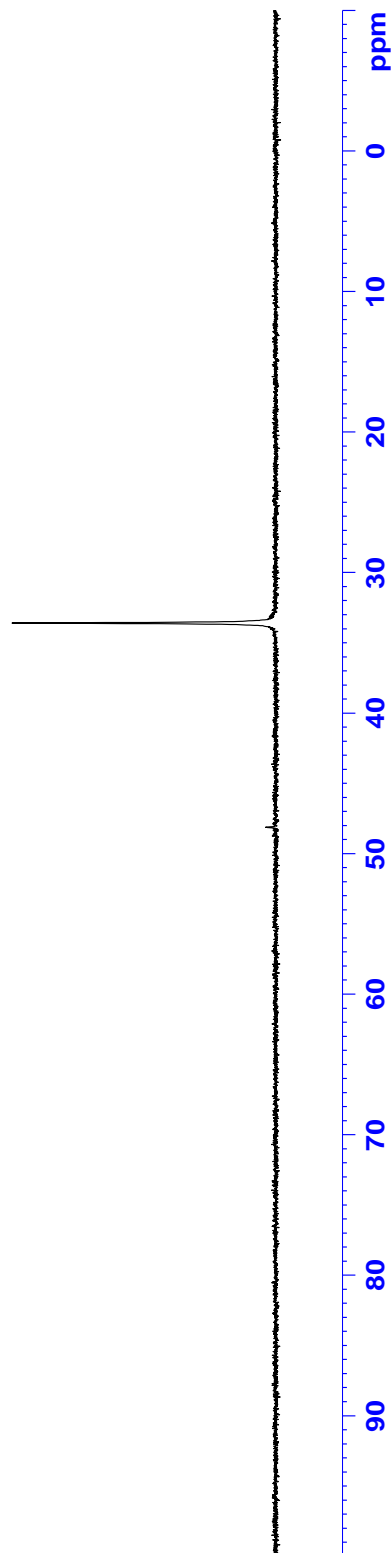
```
Current Data Parameters
NAME      Ken-P
EXPNO     20
PROCNO    1

F2 - Acquisition Parameters
Date_     20120310
Time      13.19
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg50
TD         65536
SOLVENT   CDCl3
NS         73
DS         0
SWH        64724.918 Hz
FIDRES     0.987624 Hz
AQ         0.5063156 sec
RG         20642.5
DW         7.725 usec
DE         6.50 usec
TE         298.2 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       131P
P1         10.60 usec
PL1        12.00 dB
SFO1       161.9674942 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      90.00 usec
PL2        3.80 dB
PL12       21.60 dB
PL13       24.60 dB
SFO2       400.1316005 MHz

F2 - Processing parameters
SI          32768
SF          161.9757133 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.00
```

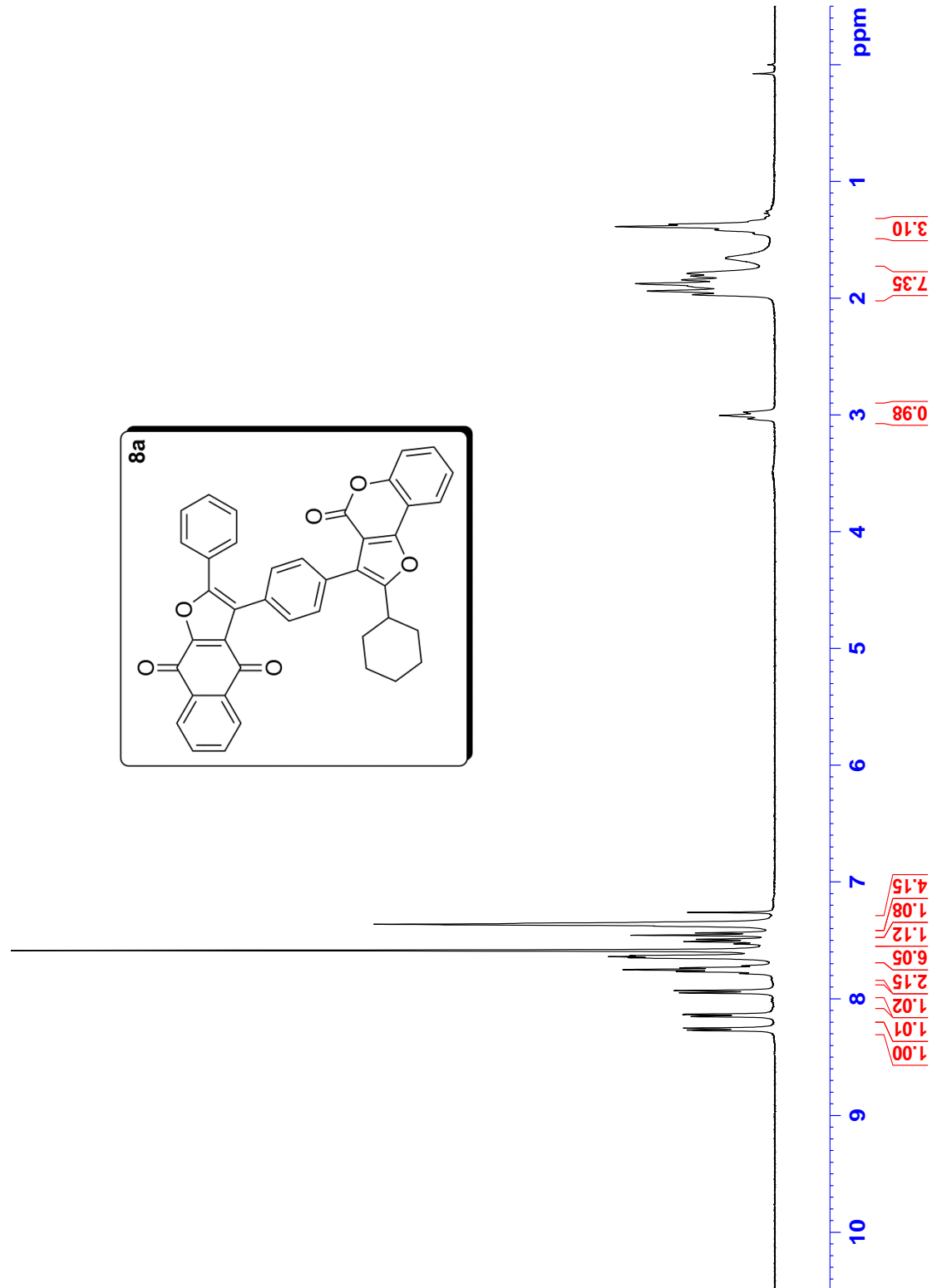
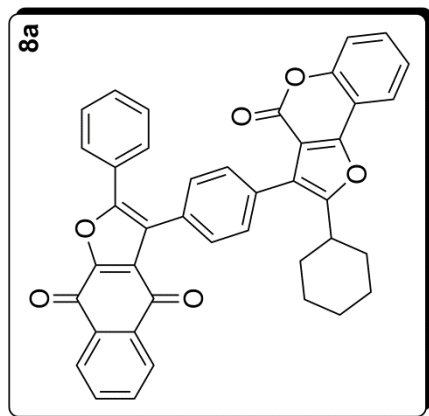


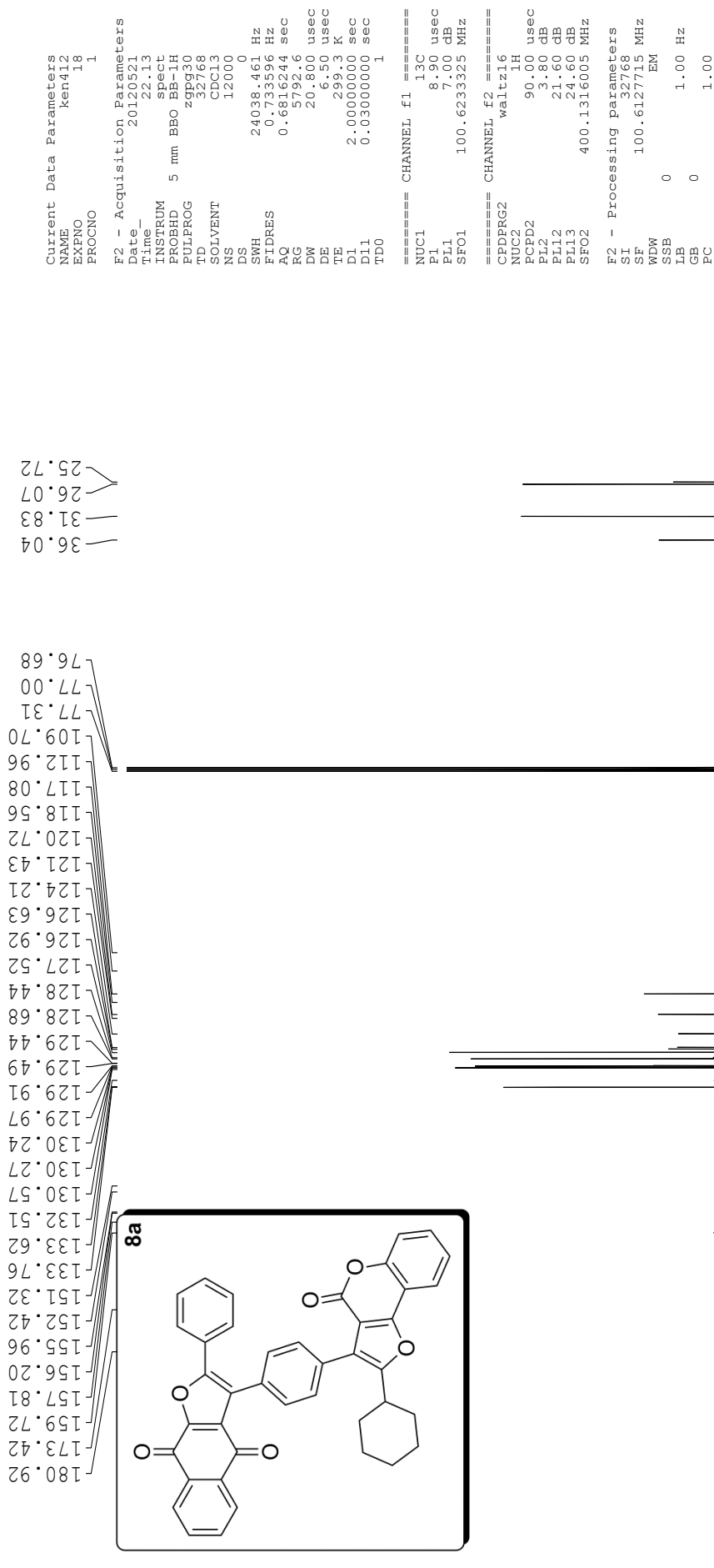
Current Data Parameters  
NAME Ken412  
EXPNO 17  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120521  
Time\_ 21.48  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 128  
RG 128  
DW 69.000 usec  
DE 6.50 usec  
TE 298.7 K  
D1 2.00000000 sec  
TD0 1

=====  
CHANNEL f1  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SF01 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300078 MHz  
WDW EM  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00



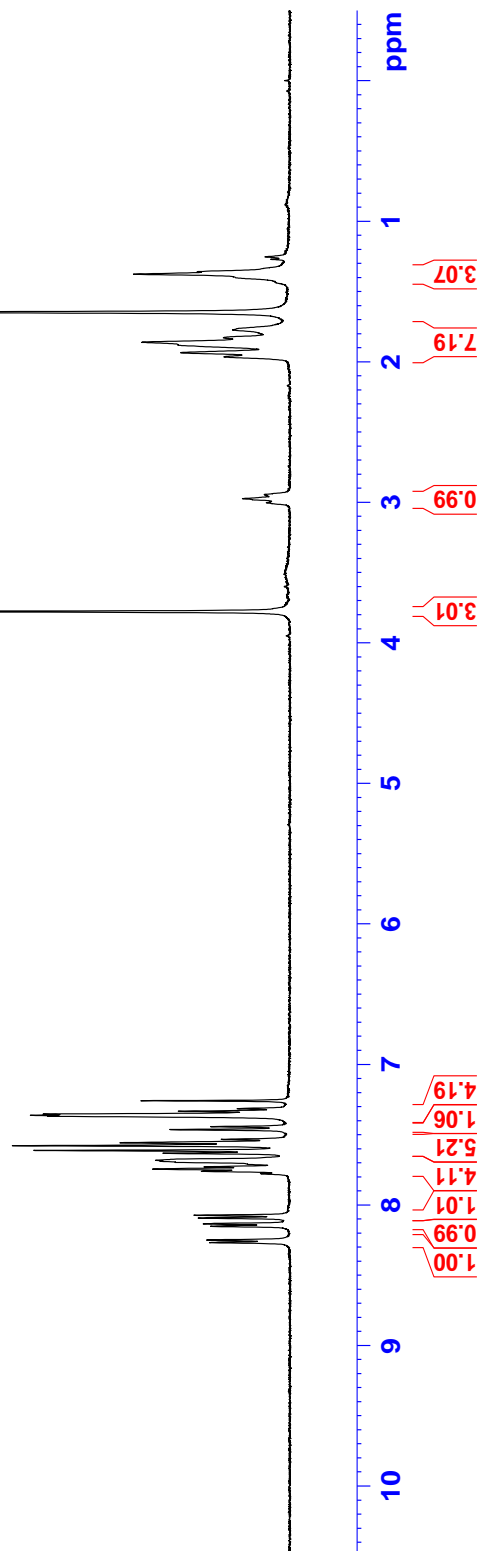
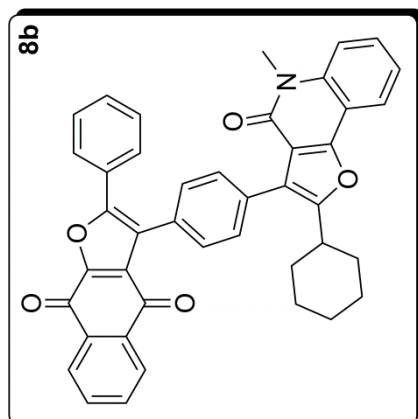


Current Data Parameters  
NAME ken415  
EXPNO 13  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120525  
Time 22.04  
INSTRUM spect  
PROBHD 5 mm BBO BB-IH  
PULPROG zg30  
TD 32768  
SOLVENT CDC13  
NS 8  
DS 0  
SWH 7246.377 Hz  
FIDRES 0.221142 Hz  
AQ 2.2610421 sec  
RG 161.3  
DW 69.000 usec  
DE 6.50 usec  
TE 298.7 K  
D1 2.0000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.70 usec  
PL1 4.00 dB  
SFO1 400.1324008 MHz

F2 - Processing parameters  
SI 16384  
SF 400.1300084 MHz  
EM  
WDW 0  
SSB 0 Hz  
LB 0  
GB 0  
PC 1.00



```

Current Data Parameters
NAME      ken415
EXPNO    14
PROCNO   1

F2 - Acquisition Parameters
Date_    20120525
Time_    22.11
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zgpg30
TD       32768
SOLVENT  CDCl3
NS       13395
DS       4
SWH      24038.461 Hz
FIDRES   0.733536 Hz
AQ       0.16816244 sec
RG       8192
DW       20.800 usec
DE       6.50 usec
TE       299.0 K
D1       2.00000000 sec
D11      0.03000000 sec
TDO      1

===== CHANNEL f1 =====
NUC1     13C
P1       8.90 usec
PL1      7.00 dB
SFO1     100.6233325 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    90.00 usec
PL2      3.80 dB
PL12     21.60 dB
PL13     24.60 dB
SFO2     400.1316005 MHz

F2 - Processing parameters
SI       32768
SF       100.6127700 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.00
    
```

