

## Base-Mediated Cyclization Reaction of 2-Alkynylphenylphosphine Oxides: Synthesis and Photophysical Properties of Benzo[*b*]phosphole Oxides

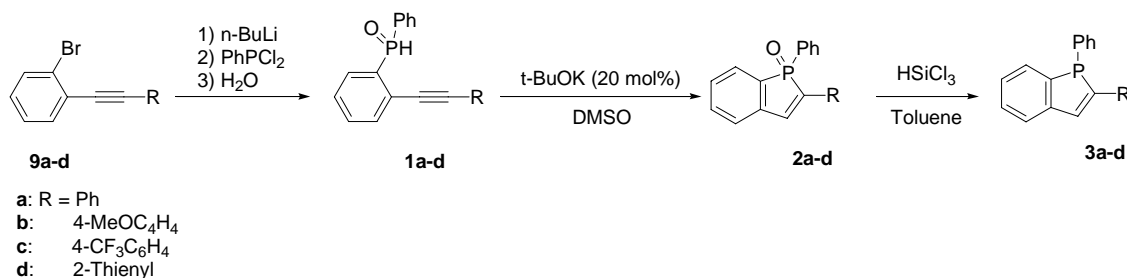
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### Experimental Section

**Measurements.**  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded using a Bruker DPX 300 FT-NMR spectrometer at 300 and 75.4 MHz, respectively.  $^{31}\text{P}\{^1\text{H}\}$  NMR (162 MHz) spectra were recorded on a JEOL JNM-Ex400 spectrometer.  $^1\text{H}$  and  $^{13}\text{C}$  chemical shifts were referenced to solvent residues.  $^{31}\text{P}\{^1\text{H}\}$  NMR chemical shift was referenced to external aq. 85%  $\text{H}_3\text{PO}_4$ . GCMS was run using a Shimadzu GC-MS 17A/QP-5000 mass spectrometer. High resolution and FAB (matrix: nitrobenzylalcohol, NBA) mass spectra were obtained with JEOL JMS-700 mass spectrometer at an ionization potential of 70 eV. Preparative GPC was performed on a JAI LC-908 equipped with JAIGEL 1H and 2H columns with toluene as an eluent. Melting points were measured on Micro melting point apparatus As-one IA9100. UV-vis spectra were recorded using an Agilent 8453 spectrometer. Fluorescence spectra were recorded using a Hitachi F 4500 spectrometer.

**Materials.** All manipulations involving air and moisture-sensitive compounds were carried out under atmosphere of dry argon or nitrogen. All solvents and reagents were of reagent quality, purchased from commercial sources and used without further purification, unless otherwise noted below. Toluene,  $\text{Et}_2\text{O}$ , and THF were dried and distilled from sodium/benzophenone just before use. DMSO and triethylamine were distilled from calcium hydride. 1-Bromo-2-ethynylbenzene,<sup>1</sup> (2-bromophenylethynyl)benzene **9a**,<sup>2</sup> 4-(2-bromophenylethynyl)anisole **9b**,<sup>2</sup> 1-(2-bromophenylethynyl)-4-trifluorobenzene **9c**,<sup>3</sup> 1,4-di(4'-*t*-butylphenylethynyl)-2,5-dibromobenzene **10**<sup>2</sup> and 1,4-di(2-bromophenylethynyl)benzene **11**<sup>4</sup> were prepared according to the previously reported procedure, respectively.

### Synthesis of 1.



- (1) Odera, A.; Wu, C.; Pratap, T. B.; Huang, C.; Ran, Y.; Liu, R. *J. Am. Chem. Soc.* **2005**, *127*, 3406.
- (2) Wang, C. H.; Hu, R. R.; Liang, S.; Chen, J. H.; Yang, Z.; Pei, J. *Tetrahedron Lett.* **2005**, *46*, 8153.
- (3) Campeau, L. C.; Parisien, M.; Jean, A.; Fagnou, K. *J. Am. Chem. Soc.* **2006**, *128*, 581.
- (4) Matsuda, T.; Kadowaki, S.; Goya, T.; Murakami, M. *Org. Lett.* **2007**, *9*, 133.

A typical example is as follows.

To a solution of 1-bromo-2-phenylethynylbenzene **9a** (3.02 g, 11.7 mmol) in Et<sub>2</sub>O (15 mL) was added a solution of 1.65 M n-butyllithium in hexane (7.1 mL, 11.7 mmol) at -50 °C. After stirring at room temperature for 3 h, the mixture was added to a solution of dichlorophenylphosphine (2.14 g, 11.9 mmol) in Et<sub>2</sub>O (15 mL) at -78 °C. The reaction mixture was allowed to room temperature and stirred for 17 h. After addition of H<sub>2</sub>O (10 mL), the product was extracted with ether and the organic layer was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. After filtration and removal of the solvent, the residue was chromatographed over silica gel (hexane/ethyl acetate = 1/2) to give **1a** (1.74 g, 5.76 mmol, 49% yield).

**1a**: pale yellow oil; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.28-7.49 (m, 11H), 7.67-7.74 (m, 2H), 7.91-8.00 (m, 1H), 8.40 (d, *J*<sub>PH</sub> = 496.4 Hz, 1H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 85.8 (d, *J*<sub>PC</sub> = 8.6 Hz), 96.7, 121.8, 124.7 (d, *J*<sub>PC</sub> = 10.0 Hz), 128.2, 128.4 (d, *J*<sub>PC</sub> = 11.2 Hz), 128.5 (d, *J*<sub>PC</sub> = 12.8 Hz), 128.8, 130.5 (d, *J*<sub>PC</sub> = 11.6 Hz), 130.8, 131.2, 132.0, 132.0 (d, *J*<sub>PC</sub> = 5.7 Hz), 132.1 (d, *J*<sub>PC</sub> = 1.8 Hz), 132.4 (d, *J*<sub>PC</sub> = 31.1 Hz), 132.7 (d, *J*<sub>PC</sub> = 8.3 Hz); <sup>31</sup>P{<sup>1</sup>H} NMR (162 MHz, CDCl<sub>3</sub>) δ 17.3; HRMS Calcd. for C<sub>20</sub>H<sub>15</sub>OP 302.0861, Found: 302.0851.

**1b**: pale yellow oil; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 3.63 (s, 3H), 6.70-6.73 (m, 2H), 7.15-7.17 (m, 2H), 7.29-7.44 (m, 6H), 7.63-7.68 (m, 2H), 7.88-7.92 (m, 1H), 8.32 (d, 1H, *J*<sub>PH</sub> = 496.2 Hz); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 54.8, 84.6 (d, *J*<sub>PC</sub> = 8.5 Hz), 96.8, 113.6, 113.7, 124.9 (d, *J*<sub>PC</sub> = 9.8 Hz), 127.8 (d, *J*<sub>PC</sub> = 11.3 Hz), 128.2 (d, *J*<sub>PC</sub> = 12.8 Hz), 130.3 (d, *J*<sub>PC</sub> = 11.5 Hz), 130.7 (d, *J*<sub>PC</sub> = 4.3 Hz), 131.7 (d, *J*<sub>PC</sub> = 3.5 Hz), 131.8 (d, *J*<sub>PC</sub> = 4.3 Hz), 131.8, 132.1 (d, *J*<sub>PC</sub> = 3.6 Hz), 132.3 (d, *J*<sub>PC</sub> = 8.3 Hz), 132.6, 159.8; <sup>31</sup>P{<sup>1</sup>H} NMR (162 MHz, CDCl<sub>3</sub>) δ 17.5; HRMS Calcd. for C<sub>21</sub>H<sub>17</sub>O<sub>2</sub>P 332.0966, Found: 332.0961.

**1c**: a white solid; mp 102.3-103.7 °C; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.40-7.48 (m, 4H), 7.52-7.62 (m, 6H), 7.68-7.75 (m, 2H), 7.99-8.06 (m, 1H), 8.42 (d, 1H, *J*<sub>PH</sub> = 481.6 Hz); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 88.2 (d, *J*<sub>PC</sub> = 8.4 Hz), 95.0, 123.7 (quart, *J*<sub>FC</sub> = 270.6 Hz), 125.3 (quart, *J*<sub>FC</sub> = 3.8 Hz), 125.8, 128.2, 128.7 (d, *J*<sub>PC</sub> = 13.0 Hz), 129.1 (d, *J*<sub>PC</sub> = 11.3 Hz), 130.7 (quart, *J*<sub>FC</sub> = 33.8 Hz), 130.8 (d, *J*<sub>PC</sub> = 11.6 Hz), 130.8, 131.7, 132.1, 132.3 (d, *J*<sub>PC</sub> = 2.3 Hz), 132.4 (d, *J*<sub>PC</sub> = 2.9 Hz), 132.6 (d, *J*<sub>PC</sub> = 8.8 Hz), 133.1 (d, *J*<sub>PC</sub> = 8.6 Hz); <sup>31</sup>P{<sup>1</sup>H} NMR (162 MHz, CDCl<sub>3</sub>) δ 17.5; HRMS Calcd. for C<sub>21</sub>H<sub>14</sub>F<sub>3</sub>OP 370.0734, Found: 370.0727.

For synthesis of **1d**, the starting material **9d** was prepared as the following.

A mixture of 2-iodothiophene (2.93 g, 14.0 mmol), 1-bromo-2-ethynylbenzene (2.51 g, 13.9 mmol), Pd(PPh<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub> (0.31 g, 0.44 mmol), CuI (0.13 g, 0.69 mmol), and triethylamine (40 mL) was stirred at room temperature for 5 h. After addition of sat. NH<sub>4</sub>Cl aq. (10 mL), the product was extracted with ether and washed with sat. NaCl aq. and then the organic layer was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. After filtration and removal of the solvent, the residue was chromatographed over silica gel (hexane) to give **9d** (3.24 g, 12.3 mmol, 89% yield).

**9d**: colorless oil; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.02-7.05 (m, 1H), 7.15-7.21 (m, 1H), 7.27-7.36 (m, 3H), 7.53-7.56 (m, 1H), 7.60-7.63 (m, 1H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 87.1, 91.6, 122.7, 125.3, 125.6, 127.0, 127.2, 128.9, 129.5, 132.36, 132.42, 133.0; Anal. Calcd. for C<sub>12</sub>H<sub>7</sub>BrS: C, 54.77; H,

2.68. Found: C, 54.92; H, 2.94.

**1d** was synthesized by the similar procedure as noted above.

**1d**: pale yellow oil;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  6.88-7.04 (m, 1H), 7.08-7.10 (m, 1H), 7.21-7.23 (m, 1H), 7.35-7.50 (m, 6H), 7.65-7.72 (m, 2H), 7.97-8.03 (m, 1H), 8.11 (d, 1H,  $J_{\text{PH}} = 619.8$  Hz);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  89.3 (d,  $J_{\text{PC}} = 8.6$  Hz), 90.0, 121.4, 124.1 (d,  $J_{\text{PC}} = 9.7$  Hz), 124.9, 126.9, 128.1, 128.2 (d,  $J_{\text{PC}} = 60.1$  Hz), 128.4 (d,  $J_{\text{PC}} = 26.9$  Hz), 128.4 (d,  $J_{\text{PC}} = 8.2$  Hz), 130.4 (d,  $J_{\text{PC}} = 11.6$  Hz), 130.8 (d,  $J_{\text{PC}} = 27.0$  Hz), 131.9 (d,  $J_{\text{PC}} = 4.4$  Hz), 132.0, 132.2 (d,  $J_{\text{PC}} = 18.6$  Hz), 132.4;  $^{31}\text{P}\{^1\text{H}\}$  NMR (162 MHz,  $\text{CDCl}_3$ )  $\delta$  17.6; HRMS Calcd. for  $\text{C}_{18}\text{H}_{13}\text{OPS}$  308.0425, Found: 308.0422.

**Synthesis of 2.** A typical example is as follows.

A mixture of **1a** (1.12 g, 3.71 mmol),  $\text{KO}^t\text{Bu}$  (0.09 g, 0.74 mmol) in DMSO (50 mL) was stirred at 70 °C for 22 h.  $\text{H}_2\text{O}$  (10 mL) was added to the mixture, and then the product was extracted with ether. After removal of the solvent, the residue was chromatographed over silica gel ( $\text{CH}_2\text{Cl}_2$ ) to give **2a** (0.89 g, 2.93 mmol, 79% yield).

**2a**: a white solid; mp 162.4-163.8 °C;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.28-7.72 (m, 15H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  124.6 (d,  $J_{\text{PC}} = 9.5$  Hz), 126.5 (d,  $J_{\text{PC}} = 6.2$  Hz), 128.8 (d,  $J_{\text{PC}} = 2.5$  Hz), 128.9, 128.9, 128.9, 129.1 (d,  $J_{\text{PC}} = 2.6$  Hz), 129.8 (d,  $J_{\text{PC}} = 97.9$  Hz), 130.7 (d,  $J_{\text{PC}} = 10.7$  Hz), 132.1 (d,  $J_{\text{PC}} = 32.7$  Hz), 132.2 (d,  $J_{\text{PC}} = 2.7$  Hz), 133.0 (d,  $J_{\text{PC}} = 65.1$  Hz), 133.2 (d,  $J_{\text{PC}} = 2.0$  Hz), 136.5 (d,  $J_{\text{PC}} = 20.0$  Hz), 138.7 (d,  $J_{\text{PC}} = 94.1$  Hz), 141.6 (d,  $J_{\text{PC}} = 28.0$  Hz);  $^{31}\text{P}\{^1\text{H}\}$  NMR (162 MHz,  $\text{CDCl}_3$ )  $\delta$  39.6; HRMS (FAB, matrix: 2-nitorobenzyl alcohol) Calcd. For  $\text{C}_{20}\text{H}_{16}\text{OP}$  [ $\text{M} + \text{H}^+$ ] 303.0939, Found: 303.0946.

**2b**: a pale yellow solid; mp 155.8-157.8 °C;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  3.67 (s, 3H), 6.76-6.79 (m, 2H), 7.20-7.74 (m, 12H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  55.0, 114.2, 124.1 (d,  $J_{\text{PC}} = 9.6$  Hz), 124.9 (d,  $J_{\text{PC}} = 8.6$  Hz), 127.8 (d,  $J_{\text{PC}} = 6.6$  Hz), 128.3 (d,  $J_{\text{PC}} = 10.7$  Hz), 128.6 (d,  $J_{\text{PC}} = 12.4$  Hz), 128.7, 130.0 (d,  $J_{\text{PC}} = 97.5$  Hz), 130.4 (d,  $J_{\text{PC}} = 10.7$  Hz), 131.9 (d,  $J_{\text{PC}} = 2.8$  Hz), 132.1 (d,  $J_{\text{PC}} = 108.3$  Hz), 133.0 (d,  $J_{\text{PC}} = 1.6$  Hz), 137.9 (d,  $J_{\text{PC}} = 94.1$  Hz), 134.0 (d,  $J_{\text{PC}} = 20.2$  Hz), 141.7 (d,  $J_{\text{PC}} = 28.3$  Hz), 159.9;  $^{31}\text{P}\{^1\text{H}\}$  NMR (162 MHz,  $\text{CDCl}_3$ )  $\delta$  39.7; HRMS Calcd. for  $\text{C}_{21}\text{H}_{17}\text{O}_2\text{P}$  332.0966, Found: 332.0965.

**2c**: a white solid; mp 131.2-132.3 °C;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.36-7.81 (m, 14H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  123.9 (quart,  $J_{\text{FC}} = 270.5$  Hz), 125.1 (d,  $J_{\text{PC}} = 9.7$  Hz), 125.8 (quart,  $J_{\text{FC}} = 3.8$  Hz), 126.7 (d,  $J_{\text{PC}} = 6.2$  Hz), 128.6, 129.0 (d,  $J_{\text{PC}} = 12.2$  Hz), 129.2, 129.6 (quart,  $J_{\text{FC}} = 87.2$  Hz), 129.7 (d,  $J_{\text{PC}} = 10.7$  Hz), 130.7 (d,  $J_{\text{PC}} = 10.8$  Hz), 130.9 (d,  $J_{\text{PC}} = 152.8$  Hz), 132.5 (d,  $J_{\text{PC}} = 2.9$  Hz), 133.4 (d,  $J_{\text{PC}} = 2.0$  Hz), 136.0 (d,  $J_{\text{PC}} = 10.8$  Hz), 137.5 (d,  $J_{\text{PC}} = 94.4$  Hz), 138.6 (d,  $J_{\text{PC}} = 19.6$  Hz), 141.1 (d,  $J_{\text{PC}} = 27.5$  Hz);  $^{31}\text{P}\{^1\text{H}\}$  NMR (62 MHz,  $\text{CDCl}_3$ )  $\delta$  39.3; HRMS (FAB, matrix: 2-nitorobenzyl alcohol) Calcd. for  $\text{C}_{21}\text{H}_{15}\text{F}_3\text{OP}$  [ $\text{M} + \text{H}^+$ ] 371.0813, Found: 371.0820.

**2d**: a pale yellow solid; mp 142.5-143.9 °C;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  6.86-6.89 (m, 1H), 7.17-7.43 (m, 9H), 7.52-7.58 (m, 1H), 7.71-7.77 (m, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  124.3 (d,  $J_{\text{PC}} = 9.6$  Hz), 126.2, 127.4 (d,  $J_{\text{PC}} = 3.8$  Hz), 128.0, 128.7, 128.7 (d,  $J_{\text{PC}} = 29.5$  Hz), 128.7 (d,  $J_{\text{PC}} = 12.3$  Hz), 128.9 (d,  $J_{\text{PC}} = 99.4$  Hz), 130.6 (d,  $J_{\text{PC}} = 10.8$  Hz), 131.6 (d,  $J_{\text{PC}} = 108.5$  Hz), 132.2 (d,  $J_{\text{PC}}$

= 2.8 Hz), 133.2 (d,  $J_{PC}$  = 1.9 Hz), 133.3 (d,  $J_{PC}$  = 94.0 Hz), 134.0 (d,  $J_{PC}$  = 19.1 Hz), 135.8 (d,  $J_{PC}$  = 14.6 Hz), 141.7 (d,  $J_{PC}$  = 27.5 Hz);  $^{31}\text{P}\{^1\text{H}\}$  NMR (162 MHz,  $\text{CDCl}_3$ )  $\delta$  38.7; Anal. Calcd. for  $\text{C}_{18}\text{H}_{13}\text{OPS}$ : C, 70.12; H, 4.25. Found: C, 69.96; H, 4.42.

**Synthesis of 3.** A typical example is as follows.

A mixture of **2a** (30 mg, 0.10 mmol) and trichlorosilane (27 mg, 0.2 mmol) in toluene (3 mL) was stirred at 110 °C for 1 h. To the mixture was added 2 N NaOH aq. (0.5 mL) at 0 °C. The product was extracted with toluene and the organic layer was dried over anhydrous  $\text{Na}_2\text{SO}_4$ . After filtration and removal of the solvent, the residue was chromatographed over silica gel ( $\text{CH}_2\text{Cl}_2/\text{hexane}$  = 1/2) to give **3a** (24 mg, 0.08 mmol, 83% yield).

**3a**: a white solid; mp 165.5-166.7 °C;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.22-7.26 (m, 6H), 7.30-7.41 (m, 4H), 7.59-7.67 (m, 5H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  124.0, 126.0 (d,  $J_{PC}$  = 8.0 Hz), 127.0 (d,  $J_{PC}$  = 9.2 Hz), 127.7, 128.5 (d,  $J_{PC}$  = 23.6 Hz), 128.7, 128.7, 128.1 (d,  $J_{PC}$  = 20.7 Hz), 129.4, 132.9 (d,  $J_{PC}$  = 6.4 Hz), 133.3 (d,  $J_{PC}$  = 20.0 Hz), 136.4 (d,  $J_{PC}$  = 16.7 Hz), 143.6, 146.0 (d,  $J_{PC}$  = 4.9 Hz), 149.6 (d,  $J_{PC}$  = 6.4 Hz);  $^{31}\text{P}\{^1\text{H}\}$  NMR (162 MHz,  $\text{CDCl}_3$ )  $\delta$  0.6; HRMS Calcd. for  $\text{C}_{20}\text{H}_{15}\text{P}$ : 286.0911, Found: 286.0913.

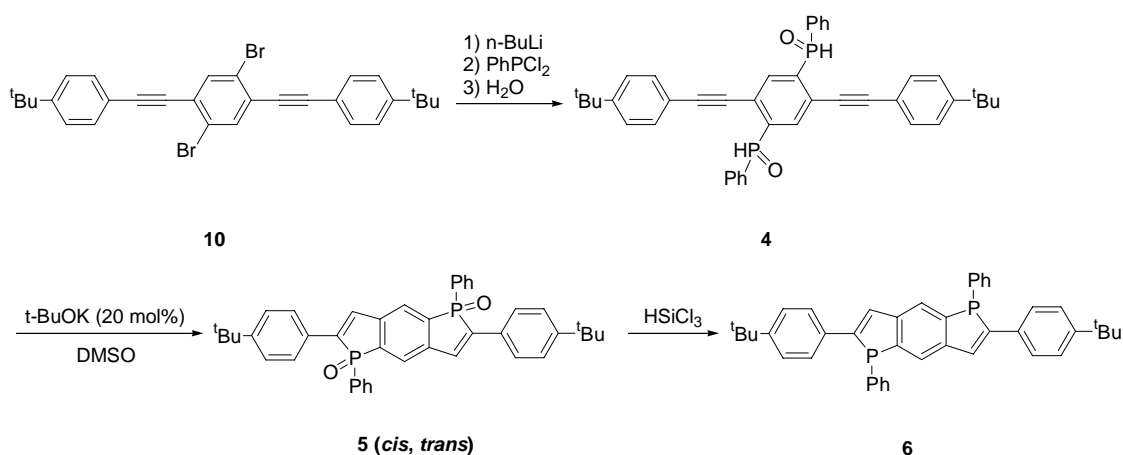
**3b**: a white solid; mp 159.2-159.7 °C;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  3.79 (s, 3H), 6.86-6.89 (m, 2H), 7.13-7.28 (m, 4H), 7.35-7.43 (m, 3H), 7.49-7.52 (m, 1H), 7.57-7.65 (m, 4H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  55.2, 114.1, 123.6, 125.6 (d,  $J_{PC}$  = 8.1 Hz), 128.1, 128.2 (d,  $J_{PC}$  = 3.8 Hz), 128.7 (d,  $J_{PC}$  = 8.0 Hz), 129.0 (d,  $J_{PC}$  = 20.6 Hz), 129.1 (d,  $J_{PC}$  = 17.3 Hz), 129.4, 131.1 (d,  $J_{PC}$  = 6.3 Hz), 133.2 (d,  $J_{PC}$  = 19.7 Hz), 133.7 (d,  $J_{PC}$  = 14.7 Hz), 143.2, 146.3 (d,  $J_{PC}$  = 4.7 Hz), 149.1 (d,  $J_{PC}$  = 6.2 Hz), 159.3;  $^{31}\text{P}\{^1\text{H}\}$  NMR (162 MHz,  $\text{CDCl}_3$ )  $\delta$  -0.6; HRMS Calcd. for  $\text{C}_{21}\text{H}_{17}\text{OP}$  316.1017, Found: 316.1015; Anal. Calcd. for  $\text{C}_{21}\text{H}_{17}\text{OP}$ : C, 79.73; H, 5.42. Found: C, 79.80; H, 5.24.

**3c**: a white solid; mp 131.7-132.9 °C;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.21-7.43 (m, 7H), 7.55-7.74 (m, 7H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  124.2 (quart,  $J_{FC}$  = 270.5 Hz), 124.5, 125.6 (quart,  $J_{FC}$  = 3.8 Hz), 126.6 (d,  $J_{PC}$  = 8.2 Hz), 127.1 (d,  $J_{PC}$  = 9.2 Hz), 128.5, 128.9 (d,  $J_{PC}$  = 8.1 Hz), 129.1 (quart,  $J_{FC}$  = 63.8 Hz), 129.2 (d,  $J_{PC}$  = 20.9 Hz), 129.8, 132.5 (d,  $J_{PC}$  = 14.9 Hz), 133.4 (d,  $J_{PC}$  = 20.1 Hz), 134.9 (d,  $J_{PC}$  = 6.5 Hz), 139.9 (d,  $J_{PC}$  = 16.6 Hz), 143.9, 145.6 (d,  $J_{PC}$  = 4.4 Hz), 148.3 (d,  $J_{PC}$  = 6.7 Hz);  $^{31}\text{P}\{^1\text{H}\}$  NMR (162 MHz,  $\text{CDCl}_3$ )  $\delta$  -0.2; HRMS Calcd. for  $\text{C}_{21}\text{H}_{14}\text{F}_3\text{P}$  354.0785, Found: 354.0795.

**3d**: a white solid; mp 129.8-130.5 °C;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  6.90-6.97 (m, 1H), 7.11-7.12 (m, 1H), 7.19-7.44 (m, 9H), 7.53-7.55 (m, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  123.7, 125.0, 125.7 (d,  $J_{PC}$  = 7.4 Hz), 126.1 (d,  $J_{PC}$  = 8.0 Hz), 127.8, 128.5, 128.8 (d,  $J_{PC}$  = 8.3 Hz), 129.1 (d,  $J_{PC}$  = 20.9 Hz), 129.7, 132.1 (d,  $J_{PC}$  = 5.9 Hz), 133.4 (d,  $J_{PC}$  = 15.8 Hz), 133.5 (d,  $J_{PC}$  = 20.3 Hz), 140.4 (d,  $J_{PC}$  = 21.9 Hz), 142.9 (d,  $J_{PC}$  = 21.9 Hz), 143.1, 146.0 (d,  $J_{PC}$  = 4.3 Hz);  $^{31}\text{P}\{^1\text{H}\}$  NMR (162 MHz,  $\text{CDCl}_3$ )  $\delta$  2.0; HRMS Calcd. for  $\text{C}_{18}\text{H}_{13}\text{PS}$  292.0476, Found: 292.0482. Anal. Calcd. for  $\text{C}_{18}\text{H}_{13}\text{PS}$ : C, 73.95; H, 4.48. Found: C, 73.55; H, 4.59.

**Synthesis of 5.**

**5** and **6** were synthesized as following scheme.



### Synthesis of 4.

To a solution of 1,4-di(4-*t*-butylphenylethynyl)-2,5-dibromobenzene **10** (1.52 g, 2.60 mmol) in Et<sub>2</sub>O (10 mL) was added a solution of 1.55 M *n*-butyllithium in hexane (3.4 mL, 5.2 mmol) at  $-50\text{ }^\circ\text{C}$ . After stirring at room temperature for 4 h, the mixture was added to a solution of dichlorophenylphosphine (0.93 g, 5.2 mmol) in Et<sub>2</sub>O (10 mL) at  $-78\text{ }^\circ\text{C}$ . The reaction mixture was allowed to room temperature and stirred for 19 h. After addition of H<sub>2</sub>O (10 mL), the product was extracted with CH<sub>2</sub>Cl<sub>2</sub> and the organic layer was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. After filtration and removal of the solvent, the residue was chromatographed over silica gel (CH<sub>2</sub>Cl<sub>2</sub>/acetone = 20/1) to give **4** (0.49 g, 0.77 mmol, 30% yield).

**4**: a pale yellow powder; mp  $106.0\text{--}109.3\text{ }^\circ\text{C}$ ; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  1.27 (s, 18H), 7.26–7.36 (m, 8H), 7.38–7.53 (m, 6H), 7.74–7.81 (m, 4H), 8.26–8.32 (m, 2H), 8.44 (d, 2H,  $J_{\text{PH}} = 504.6\text{ Hz}$ ); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  84.8 (d,  $J_{\text{PC}} = 8.5\text{ Hz}$ ), 100.6, 118.4, 124.6 (dd,  $J_{\text{PC}} = 19.3\text{ Hz}$ ,  $J_{\text{PC}} = 11.0\text{ Hz}$ ), 125.6, 128.9 (d,  $J_{\text{PC}} = 13.6\text{ Hz}$ ), 130.7 (d,  $J_{\text{PC}} = 103.7\text{ Hz}$ ), 130.8 (d,  $J_{\text{PC}} = 12.1\text{ Hz}$ ), 131.3, 132.8, 135.9 (dd,  $J_{\text{PC}} = 96.8\text{ Hz}$ ,  $J_{\text{PC}} = 2.1\text{ Hz}$ ), 136.4 (dd,  $J_{\text{PC}} = 8.3\text{ Hz}$ ,  $J_{\text{PC}} = 8.3\text{ Hz}$ ); <sup>31</sup>P{<sup>1</sup>H} NMR (162 MHz, CDCl<sub>3</sub>)  $\delta$  16.0; HRMS (FAB, matrix; 2-nitrobenzyl alcohol) Calcd. for C<sub>42</sub>H<sub>41</sub>O<sub>2</sub>P<sub>2</sub> (value for [M + H]<sup>+</sup>); 639.2582, Found; 639.2601.

### Synthesis of 5.

A mixture of **4** (120 mg, 0.19 mmol) and *t*-BuOK (9 mg, 0.075 mmol) in DMSO (5 mL) was stirred at  $70\text{ }^\circ\text{C}$  for 22 h. After addition of H<sub>2</sub>O (10 mL), the product was extracted with CH<sub>2</sub>Cl<sub>2</sub> and dried over Na<sub>2</sub>SO<sub>4</sub>. After removal of the solvent, preparative TLC (CH<sub>2</sub>Cl<sub>2</sub>/acetone = 10/1) gave **5-cis** ( $R_f = 0.40$ , 38 mg, 0.06 mmol, 32% yield) and **5-trans** ( $R_f = 0.64$ , 10 mg, 0.015 mmol, 8% yield).

**5-cis**: a pale yellow powder; mp  $> 300\text{ }^\circ\text{C}$ ; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  1.26 (s, 18H), 7.33 (d, 4H,  $J = 8.0\text{ Hz}$ ), 7.41–7.60 (M, 14H), 7.76–7.84 (m, 4H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) 31.1, 34.7, 124.5 (dd,  $J_{\text{PC}} = 10.6\text{ Hz}$ ,  $J_{\text{PC}} = 10.6\text{ Hz}$ ), 126.0, 126.5 (d,  $J_{\text{PC}} = 6.8\text{ Hz}$ ), 129.1, 129.3, (d,  $J_{\text{PC}} = 97.9\text{ Hz}$ ), 130.0 (d,  $J_{\text{PC}} = 109.1\text{ Hz}$ ), 130.7 (d,  $J_{\text{PC}} = 11.2\text{ Hz}$ ), 132.5, 134.7 (d,  $J_{\text{PC}} = 20.2\text{ Hz}$ ), 138.2 (d,  $J_{\text{PC}} = 101.1\text{ Hz}$ ), 139.2 (d,  $J_{\text{PC}} = 94.4\text{ Hz}$ ), 142.2 (dd,  $J_{\text{PC}} = 10.5\text{ Hz}$ ,  $J_{\text{PC}} = 29.3\text{ Hz}$ ); <sup>31</sup>P{<sup>1</sup>H} NMR (162 MHz, CDCl<sub>3</sub>)  $\delta$  38.2; HRMS (FAB, 2-nitobenzyl alcohol) Calcd. For C<sub>42</sub>H<sub>41</sub>O<sub>2</sub>P<sub>2</sub>: 639.2582, Found: 639.2592.

**5-trans**: a pale yellow powder; mp  $> 300\text{ }^\circ\text{C}$ ; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  1.26 (s, 18H), 7.33 (d,

4H,  $J = 8.2$  Hz) 7.45-7.61 (m, 14H), 7.76-7.83 (m, 4H);  $^{31}\text{P}\{^1\text{H}\}$  NMR (162 MHz,  $\text{CDCl}_3$ )  $\delta$  38.6; HRMS (FAB, 2-nitrobenzyl alcohol) Calcd. For  $\text{C}_{42}\text{H}_{41}\text{O}_2\text{P}_2$ : 639.2582, Found: 639.2581.

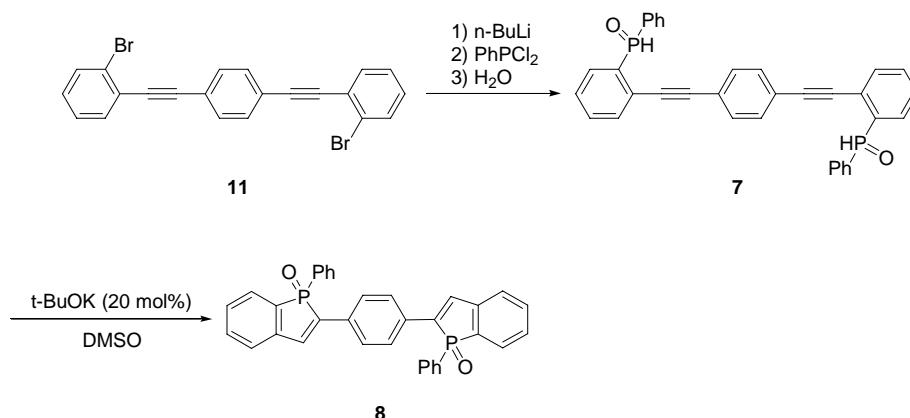
### Synthesis of 6.

A mixture of **5-cis** (70 mg, 0.11 mmol) and trichlorosilane (54 mg, 0.4 mmol) in toluene (10 mL) was stirred at 110 °C for 1 h. To the mixture was added 2 N NaOH aq. (1 mL) at 0 °C. The product was extracted with toluene and the organic layer was dried over anhydrous  $\text{Na}_2\text{SO}_4$ . After filtration and removal of the solvent, the residue was chromatographed over silica gel ( $\text{CH}_2\text{Cl}_2/\text{hexane} = 1/3$ ) to give **6** (35 mg, 60  $\mu\text{mol}$ , 52% yield).

**6**: a pale yellow powder; mp > 300 °C;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  1.28 (s, 18H), 7.26-7.32 (m, 10H), 7.39-7.44 (m, 4H), 7.52-7.55 (m, 6H), 7.75-7.78 (m, 2H);  $^{31}\text{P}\{^1\text{H}\}$  NMR (162 MHz,  $\text{CDCl}_3$ )  $\delta$  -1.6, -1.0; HRMS (FAB, matrix: 2-nitrobenzyl alcohol) Calcd. For  $\text{C}_{42}\text{H}_{40}\text{P}_2$ : 606.2605, Found: 606.2609.

### Synthesis of 8.

**8** was synthesized as following scheme.



To a solution of 1,4-di(2-bromophenylethynyl)benzene **11** (0.68 g, 1.57 mmol) in  $\text{Et}_2\text{O}$  (5 mL) was added a solution of 1.55 M  $n$ -butyllithium in hexane (2.0 mL, 3.1 mmol) at -50 °C. After stirring at room temperature for 4 h, the mixture was added to a solution of dichlorophenylphosphine (0.61 g, 3.41 mmol) in  $\text{Et}_2\text{O}$  (5 mL) at -78 °C. The reaction mixture was allowed to room temperature and stirred for 15 h. After addition of  $\text{H}_2\text{O}$  (10 mL), the product was extracted with  $\text{CH}_2\text{Cl}_2$  and the organic layer was dried over anhydrous  $\text{Na}_2\text{SO}_4$ . After filtration and removal of the solvent, the residue was chromatographed over silica gel ( $\text{CH}_2\text{Cl}_2/\text{acetone} = 2/1$ ) to give **7** (0.37 g, 0.71 mmol, 45% yield).

**7**: a white solid; mp 62.0-65.5 °C;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.10-7.14 (m, 4H), 7.24-7.40 (m, 13H), 7.52-7.59 (m, 4H), 7.77-7.83 (m, 2H), 9.05 (s, 1H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  87.7 (d,  $J_{\text{PC}} = 8.3$  Hz), 95.5, 122.0, 124.0 (d,  $J_{\text{PC}} = 9.2$  Hz), 128.2, (d,  $J_{\text{PC}} = 12.8$  Hz), 128.4, 130.1, 130.1 (d,  $J_{\text{PC}} = 11.4$  Hz), 130.6, 130.9, 131.3 (d,  $J_{\text{PC}} = 61.4$  Hz), 131.4 (d,  $J_{\text{PC}} = 57.6$  Hz), 131.8, 132.4 (d,  $J_{\text{PC}} = 8.3$  Hz);  $^{31}\text{P}\{^1\text{H}\}$  NMR (162 MHz,  $\text{CDCl}_3$ )  $\delta$  17.5; HRMS (FAB, 2-nitrobenzyl alcohol) Calcd. for  $\text{C}_{34}\text{H}_{25}\text{O}_2\text{P}_2$ : 527.1330, Found; 527.1338.

**Synthesis of 8.** A mixture of **7** (81 mg, 0.154 mmol) and  $\text{KO}^t\text{Bu}$  (7 mg, 0.062 mmol) in  $\text{DMSO}$  (5 mL) was stirred at 70 °C for 23 h. After addition of  $\text{H}_2\text{O}$  (10 mL), the product was extracted with

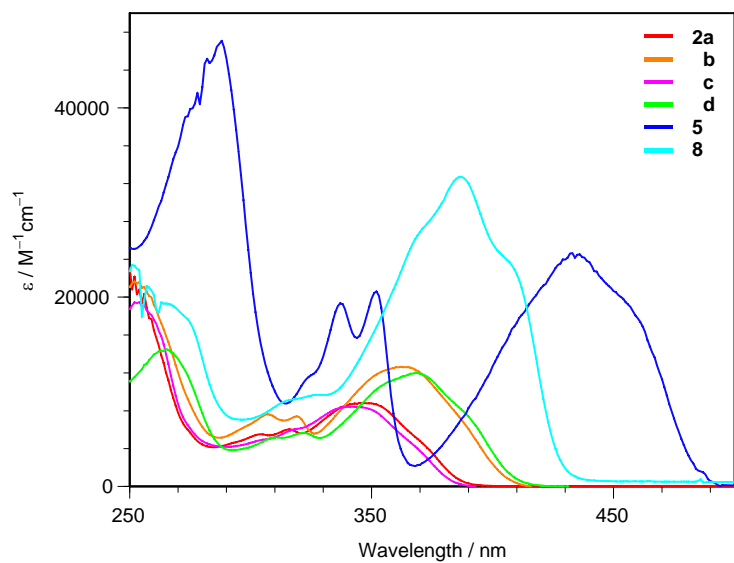
CH<sub>2</sub>Cl<sub>2</sub> and dried over Na<sub>2</sub>SO<sub>4</sub>. After removal of the solvent, the residue was chromatographed over neutral alumina (CH<sub>2</sub>Cl<sub>2</sub>/acetone = 2/1 or 1/2) to **8** (34 mg, 0.064 mmol, 42 %).

**8**: a pale yellow solid; mp > 300 °C; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ. 7.25-7.71 (m, 24H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 124.7 (d, *J*<sub>PC</sub> = 9.5 Hz), 127.1 (d, *J*<sub>PC</sub> = 6.5 Hz), 129.0 (d, *J*<sub>PC</sub> = 12.3 Hz), 129.1, 129.2 (d, *J*<sub>PC</sub> = 21.2 Hz), 129.6 (d, *J*<sub>PC</sub> = 97.7 Hz), 133.1 (d, *J*<sub>PC</sub> = 1.4 Hz), 136.6 (d, *J*<sub>PC</sub> = 19.8 Hz), 138.0 (d, *J*<sub>PC</sub> = 94.1 Hz), 141.5 (d, *J*<sub>PC</sub> = 27.9 Hz); <sup>31</sup>P{<sup>1</sup>H} NMR (162 MHz, CDCl<sub>3</sub>) δ 39.5; MS (FAB) *m/z* 527 [M + H<sup>+</sup>]; HRMS (FAB, matrix: 2-nitrobenzyl alcohol) Calcd. For C<sub>34</sub>H<sub>25</sub>O<sub>2</sub>P<sub>2</sub>: 527.1330, Found; 527.1342.

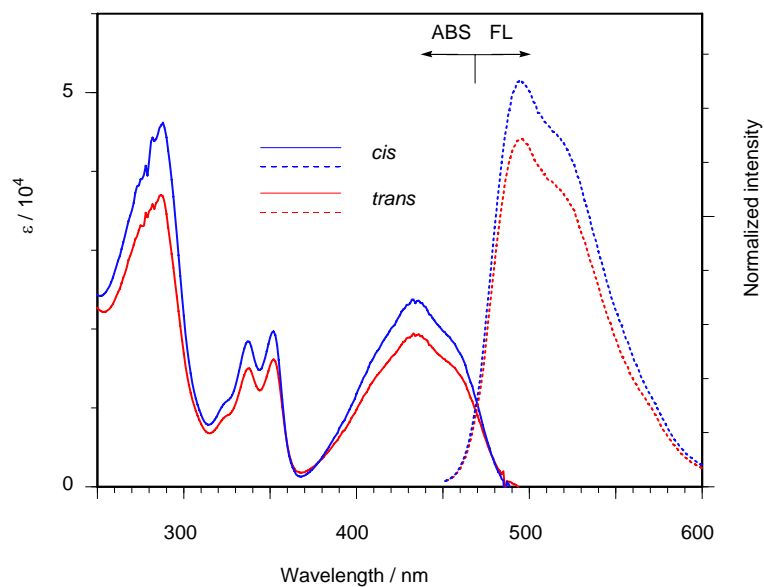
### **X-Ray Crystallography Analysis.**

A single crystal of **5-cis** for X-ray analysis was obtained from CHCl<sub>3</sub>/hexane solution. Data for **5-cis** were collected at -160 °C on a Rigaku Saturn CCD diffractometer equipped with monochromated Mo K $\alpha$  radiation ( $\lambda$  = 0.71073 Å). Calculations were carried out using the program package Crystal Structure, version 3.7, for Windows. A full-matrix least-squares refinement was used for the non-hydrogen atoms with anisotropic thermal parameters. Hydrogen atoms were located by assuming the ideal geometry and were included in the structure calculations without further refinement of the parameters.

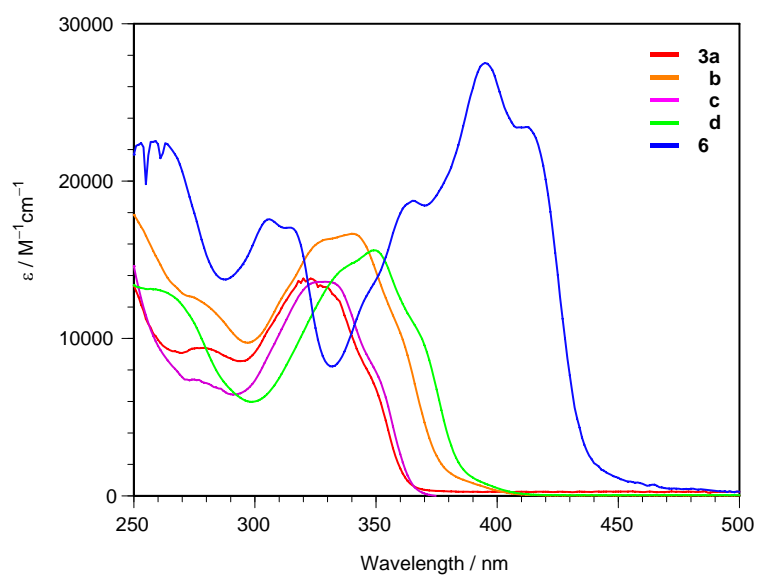
The crystal contained two solvent molecules (CHCl<sub>3</sub>) per solute molecule. The crystallographic data and the details of refinement are summarized in Appendix-1.



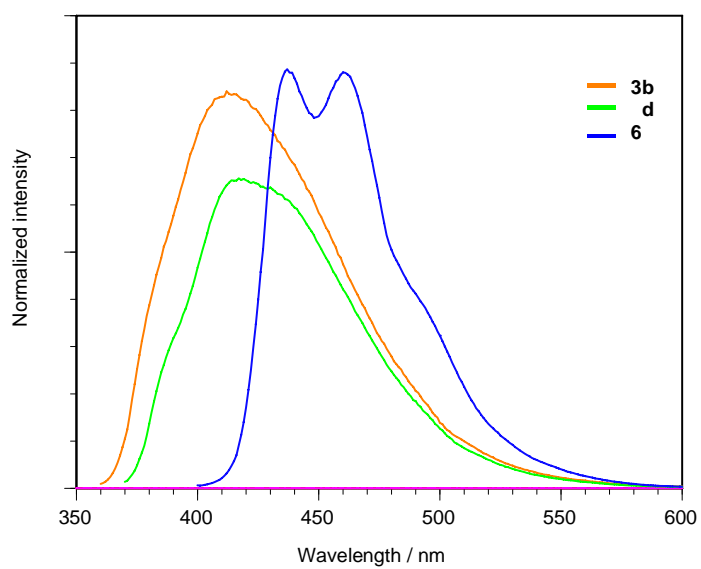
**Figure S1.** Absorption spectra of **2a-d**, **5-cis**, and **8** (THF).



**Figure S2.** Absorption and fluorescence spectra of **5-cis** and **trans** (THF).

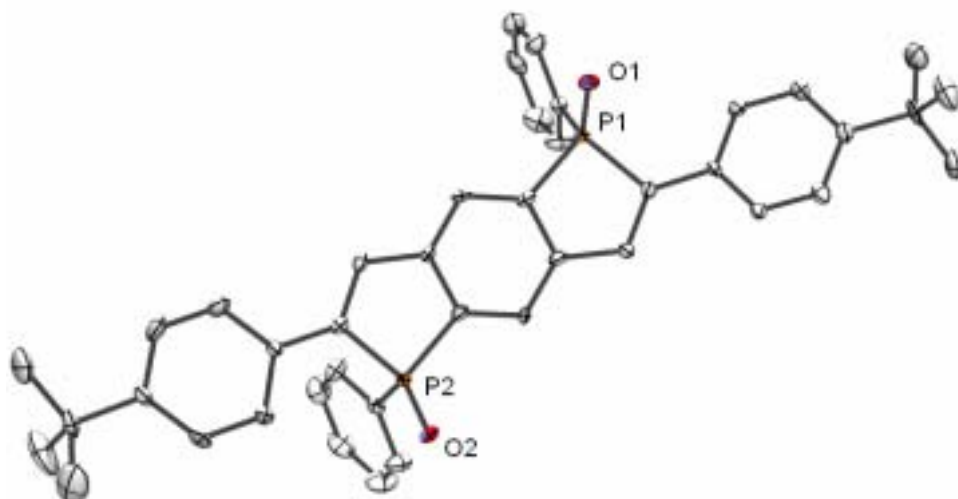


**Figure S3.** Absorption spectra of **3a-d** and **6** (THF).

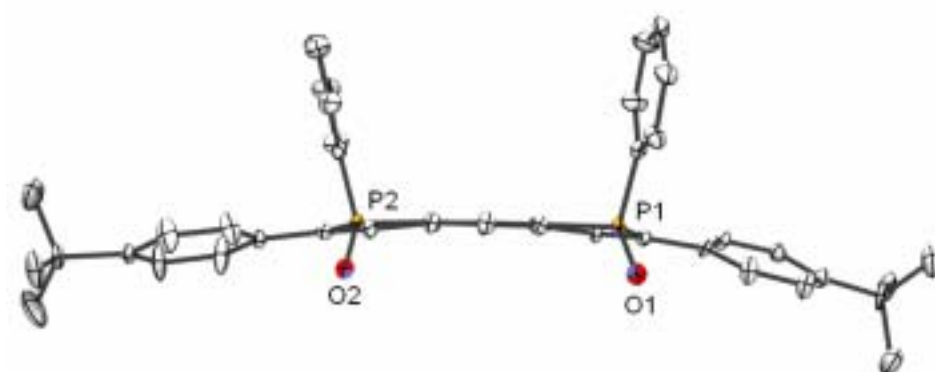


**Figure S4.** Fluorescence spectra of **3b**, **3d**, and **6** (THF).

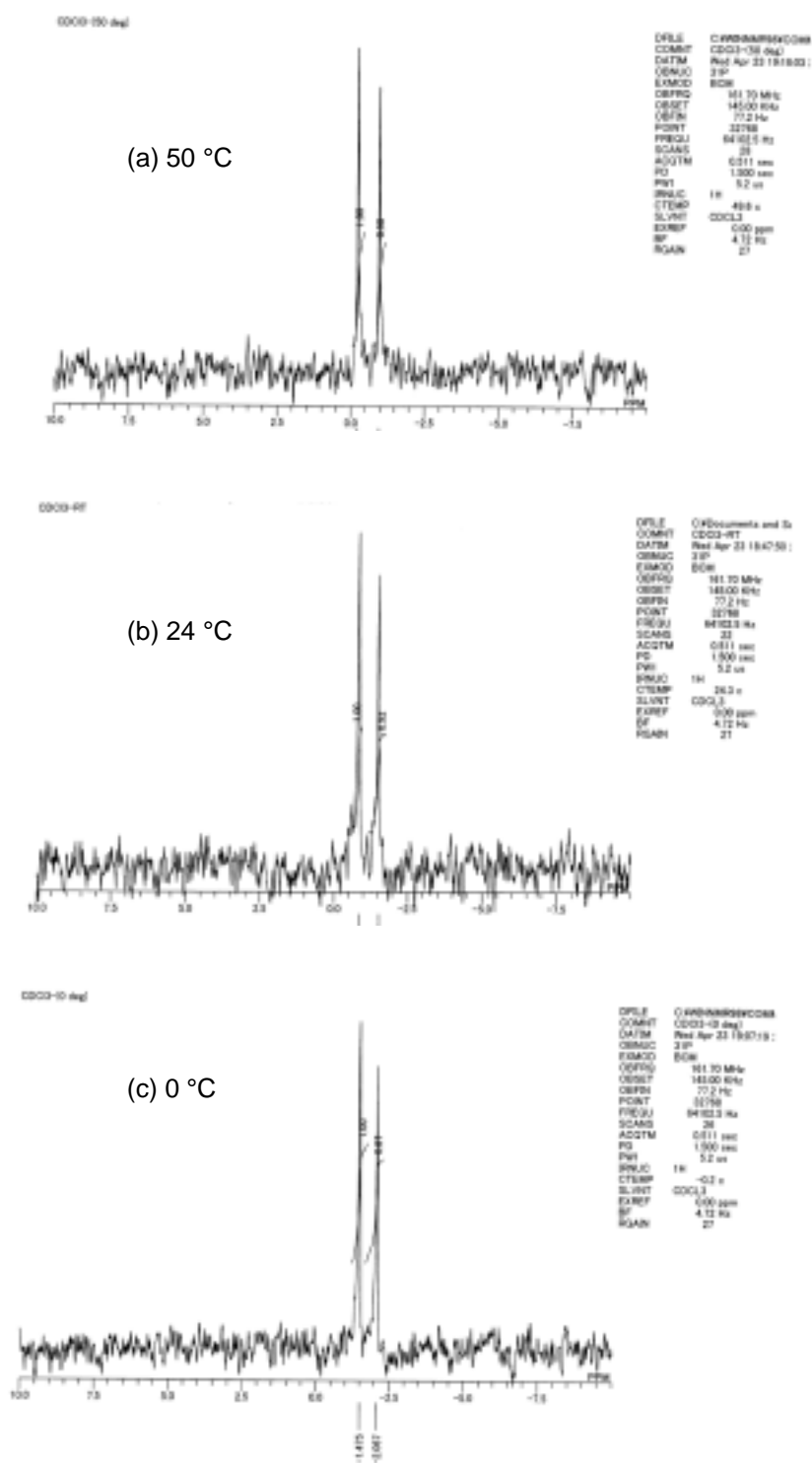
(a)



(b)



**Figure S5.** ORTEP drawing of *5-cis*. (a) Top and (b) side view.



**Figure S6.** Variable-temperature  $^{31}\text{P}$  NMR spectra of **6** at (a) 50 °C, (b) 24 °C, and (c) 0 °C ( $\text{CDCl}_3$ ).

Appendix-1

## X-ray Structure Report

March 22, 2008

## *Experimental*

### Data Collection

A yellow needle crystal of  $C_{44}H_{42}O_2P_2Cl_6$  having approximate dimensions of 1.20 x 0.25 x 0.30 mm was mounted on a glass fiber. All measurements were made on a Rigaku Saturn CCD area detector with graphite monochromated Mo-K $\alpha$  radiation.

Indexing was performed from 0 images that were exposed for 0 seconds. The crystal-to-detector distance was 45.09 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive triclinic cell with dimensions:

$$\begin{array}{ll} a = 12.273(4) \text{ \AA} & \alpha = 106.891(4)^\circ \\ b = 13.405(5) \text{ \AA} & \beta = 96.650(4)^\circ \\ c = 14.376(5) \text{ \AA} & \gamma = 95.930(4)^\circ \\ V = 2224.4(13) \text{ \AA}^3 & \end{array}$$

For  $Z = 2$  and F.W. = 877.48, the calculated density is 1.31 g/cm<sup>3</sup>. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

P-1 (#2)

The data were collected at a temperature of  $-160 \pm 1^\circ\text{C}$  to a maximum  $2\theta$  value of  $55.0^\circ$ . A total of 720 oscillation images were collected. A sweep of data was done using  $\omega$  scans from  $-110.0$  to  $70.0^\circ$  in  $0.5^\circ$  step, at  $\chi=45.0^\circ$  and  $\phi = 0.0^\circ$ . The exposure rate was 18.0 [sec./ $^\circ$ ]. The detector swing angle was  $-20.24^\circ$ . A second sweep was performed using  $\omega$  scans from  $-110.0$  to  $70.0^\circ$  in  $0.5^\circ$  step, at  $\chi=45.0^\circ$  and  $\phi = 90.0^\circ$ . The exposure rate was 18.0 [sec./ $^\circ$ ]. The detector swing angle was  $-20.24^\circ$ . The crystal-to-detector distance was 45.09 mm. Readout was performed in the 0.547 mm pixel mode.

### Data Reduction

Of the 16896 reflections that were collected, 9535 were unique ( $R_{int} = 0.030$ ); equivalent reflections were merged. Data were collected and processed using CrystalClear (Rigaku). Net intensities and sigmas were derived as follows:

$$F^2 = [\Sigma(P_i - mB_{ave})] \cdot L_p^{-1}$$

where  $P_i$  is the value in counts of the  $i^{th}$  pixel

$m$  is the number of pixels in the integration area

$B_{ave}$  is the background average

$L_p$  is the Lorentz and polarization factor

$$B_{ave} = \Sigma(B_j)/n$$

where  $n$  is the number of pixels in the background area

$B_j$  is the value of the  $j^{th}$  pixel in counts

$$\sigma^2(F^2_{hkl}) = [(\Sigma P_i) + m((\Sigma(B_{ave} - B_j)^2)/(n-1))] \cdot L_p \cdot \text{errmul} + (\text{erradd} \cdot F^2)^2$$

where  $\text{erradd} = 0.00$

$\text{errmul} = 1.00$

The linear absorption coefficient,  $\mu$ , for Mo-K $\alpha$  radiation is 4.9 cm<sup>-1</sup>. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.86 to 1.00. The data were corrected for Lorentz and polarization effects.

### Structure Solution and Refinement

The structure was solved by direct methods<sup>2</sup> and expanded using Fourier techniques<sup>3</sup>. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement<sup>4</sup> on  $F$  was based on 5599 observed reflections ( $I > 3.00\sigma(I)$ ) and 529 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma ||F_o| - |F_c|| / \Sigma |F_o| = 0.094$$

$$R_w = [\sum w (|F_o| - |F_c|)^2 / \sum w F_o^2]^{1/2} = 0.148$$

The standard deviation of an observation of unit weight<sup>5</sup> was 0.93. A Sheldrick weighting scheme was used. Plots of  $\sum w (|F_o| - |F_c|)^2$  versus  $|F_o|$ , reflection order in data collection,  $\sin \theta/\lambda$  and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 3.88 and -1.07 e<sup>-</sup>/Å<sup>3</sup>, respectively.

Neutral atom scattering factors were taken from Cromer and Waber<sup>6</sup>. Anomalous dispersion effects were included in  $F_{calc}$ <sup>7</sup>; the values for  $\Delta f'$  and  $\Delta f''$  were those of Creagh and McAuley<sup>8</sup>. The values for the mass attenuation coefficients are those of Creagh and Hubbell<sup>9</sup>. All calculations were performed using the CrystalStructure<sup>10,11</sup> crystallographic software package.

### *References*

- (1) CrystalClear: Rigaku Corporation, 1999. CrystalClear Software User's Guide, Molecular Structure Corporation, (c) 2000. J.W.Pflugrath (1999) Acta Cryst. D55, 1718-1725.
- (2) SIR97: Altomare, A., Burla, M., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A., Polidori, G., and Spagna, R. (1999). J. Appl. Cryst., 32, 115-119.
- (3) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.
- (4) Least Squares function minimized:

$$\sum w (|F_o| - |F_c|)^2 \quad \text{where } w = \text{Least Squares weights.}$$

- (5) Standard deviation of an observation of unit weight:

$$[\sum w (|F_o| - |F_c|)^2 / (N_o - N_v)]^{1/2}$$

where:  $N_O$  = number of observations  
 $N_V$  = number of variables

(6) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(7) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

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(10) CrystalStructure 3.6.0: Crystal Structure Analysis Package, Rigaku and Rigaku/MSK (2000-2004). 9009 New Trails Dr. The Woodlands TX 77381 USA.

(11) CRYSTALS Issue 10: Watkin, D.J., Prout, C.K. Carruthers, J.R. & Betteridge, P.W. Chemical Crystallography Laboratory, Oxford, UK. (1996)

## EXPERIMENTAL DETAILS

### A. Crystal Data

Empirical Formula	$C_{44}H_{42}O_2P_2Cl_6$
Formula Weight	877.48
Crystal Color, Habit	yellow, needle
Crystal Dimensions	1.20 X 0.25 X 0.30 mm
Crystal System	triclinic
Lattice Type	Primitive
Detector Position	45.09 mm
Pixel Size	0.137 mm
Lattice Parameters	$a = 12.273(4) \text{ \AA}$ $b = 13.405(5) \text{ \AA}$ $c = 14.376(5) \text{ \AA}$ $\alpha = 106.891(4)^\circ$ $\beta = 96.650(4)^\circ$ $\gamma = 95.930(4)^\circ$ $V = 2224.4(13) \text{ \AA}^3$
Space Group	P-1 (#2)
Z value	2
D <sub>calc</sub>	1.310 g/cm <sup>3</sup>
F <sub>000</sub>	908.00
$\mu(\text{MoK}\alpha)$	4.93 cm <sup>-1</sup>

## B. Intensity Measurements

Detector	Rigaku Saturn
Goniometer	Rigaku AFC10
Radiation	MoK $\alpha$ ( $\lambda = 0.71070 \text{ \AA}$ ) graphite monochromated
Detector Aperture	70 mm x 70 mm
Data Images	720 exposures
$\omega$ oscillation Range ( $\chi=45.0, \phi=0.0$ )	-110.0 - 70.0 $^{\circ}$
Exposure Rate	18.0 sec./ $^{\circ}$
Detector Swing Angle	-20.24 $^{\circ}$
$\omega$ oscillation Range ( $\chi=45.0, \phi=90.0$ )	-110.0 - 70.0 $^{\circ}$
Exposure Rate	18.0 sec./ $^{\circ}$
Detector Swing Angle	-20.24 $^{\circ}$
Detector Position	45.09 mm
Pixel Size	0.137 mm
$2\theta_{\max}$	55.0 $^{\circ}$
No. of Reflections Measured	Total: 16896 Unique: 9535 ( $R_{\text{int}} = 0.030$ )
Corrections	Lorentz-polarization Absorption (trans. factors: 0.8611 - 1.0000)

### C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR97)
Refinement	Full-matrix least-squares on F
Function Minimized	$\sum w ( F_o  -  F_c )^2$
Least Squares Weights	$1/[0.0235F_o^2+1.0000\sigma(F_o^2)]$
$2\theta_{\max}$ cutoff	55.0°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ( $I > 3.00\sigma(I)$ )	5599
No. Variables	529
Reflection/Parameter Ratio	10.58
Residuals: R ( $I > 3.00\sigma(I)$ )	0.094
Residuals: $R_w$ ( $I > 3.00\sigma(I)$ )	0.148
Goodness of Fit Indicator	0.929
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	$3.88 \text{ e}^-/\text{\AA}^3$
Minimum peak in Final Diff. Map	$-1.07 \text{ e}^-/\text{\AA}^3$

Table 1. Atomic coordinates and  $B_{iso}/B_{eq}$ 

atom	x	y	z	$B_{eq}$
C11	-0.3844(2)	-0.1579(2)	0.5498(2)	4.97(6)
C12	-0.3868(2)	0.0673(2)	0.6026(2)	5.40(7)
C13	-0.1996(3)	-0.0103(3)	0.6762(2)	7.15(9)
C14	-0.0775(3)	0.6004(3)	0.8015(3)	8.06(10)
C15	0.1109(2)	0.7609(2)	0.8445(2)	4.12(5)
C16	-0.0697(2)	0.7477(2)	0.6898(2)	5.51(7)
P1	0.14436(12)	0.16988(11)	0.66278(10)	0.99(3)
P2	-0.21270(12)	0.35370(11)	0.43273(11)	0.97(3)
O1	0.1934(3)	0.0771(3)	0.6078(3)	1.44(8)
O2	-0.1710(3)	0.4203(3)	0.3735(3)	1.36(8)
C1	0.2282(5)	0.2997(5)	0.7028(4)	1.17(11)
C2	0.1688(5)	0.3670(5)	0.6725(4)	1.12(10)
C3	0.0597(5)	0.3207(5)	0.6117(4)	1.23(11)
C4	0.0359(5)	0.2117(4)	0.5947(4)	1.13(11)
C5	-0.0589(5)	0.1510(5)	0.5317(4)	1.29(11)
C6	-0.0124(5)	0.3725(5)	0.5674(4)	1.23(11)
C7	-0.1068(5)	0.3124(5)	0.5059(4)	1.26(11)
C8	-0.1293(5)	0.2031(4)	0.4859(4)	1.12(11)
C9	-0.2256(5)	0.1531(5)	0.4078(4)	1.17(11)
C10	-0.2776(5)	0.2196(4)	0.3692(4)	1.06(10)
C11	0.0896(5)	0.1491(5)	0.7672(4)	1.25(11)
C12	0.0550(6)	0.2292(5)	0.8372(5)	2.1(1)
C13	0.0053(6)	0.2100(6)	0.9129(5)	2.7(2)
C14	-0.0110(5)	0.1096(6)	0.9193(5)	2.3(2)
C15	0.0221(6)	0.0278(6)	0.8512(5)	2.3(2)
C16	0.0737(5)	0.0461(5)	0.7738(5)	1.87(13)
C17	-0.3061(5)	0.4145(5)	0.5122(4)	1.26(11)
C18	-0.3019(6)	0.5234(5)	0.5377(5)	2.1(1)
C19	-0.3681(7)	0.5741(6)	0.6035(6)	2.7(2)
C20	-0.4420(6)	0.5134(6)	0.6385(5)	2.6(2)
C21	-0.4467(6)	0.4055(6)	0.6137(5)	2.6(2)
C22	-0.3789(6)	0.3568(5)	0.5507(5)	2.03(13)
C23	0.3398(5)	0.3221(5)	0.7586(4)	1.10(10)
C24	0.4056(5)	0.2431(5)	0.7593(5)	1.81(12)
C25	0.5131(5)	0.2654(5)	0.8051(5)	2.1(1)

C26	0.5641(5)	0.3688(5)	0.8544(5)	1.60(12)
C27	0.4969(5)	0.4508(5)	0.8588(4)	1.61(12)

Table 1. Atomic coordinates and  $B_{\text{iso}}/B_{\text{eq}}$  (continued)

atom	x	y	z	$B_{\text{eq}}$
C28	0.3893(5)	0.4265(5)	0.8098(4)	1.34(11)
C29	0.6860(5)	0.3924(6)	0.9015(5)	2.4(2)
C30	0.7265(6)	0.5085(6)	0.9477(6)	2.9(2)
C31	0.7562(6)	0.3476(8)	0.8216(6)	3.5(2)
C32	0.7036(6)	0.3380(7)	0.9807(6)	3.2(2)
C33	-0.3724(5)	0.1903(4)	0.2902(4)	1.08(10)
C34	-0.4395(6)	0.0938(5)	0.2616(5)	2.4(1)
C35	-0.5282(6)	0.0676(6)	0.1882(6)	2.7(2)
C36	-0.5577(5)	0.1369(5)	0.1362(4)	1.41(12)
C37	-0.4918(6)	0.2342(6)	0.1652(6)	2.7(2)
C38	-0.4012(6)	0.2622(6)	0.2414(6)	2.9(2)
C39	-0.6613(5)	0.1118(6)	0.0594(5)	1.97(13)
C40	-0.6419(8)	0.1608(9)	-0.0229(6)	4.8(2)
C41	-0.7561(7)	0.1610(9)	0.1120(7)	4.6(2)
C42	-0.6939(7)	-0.0086(7)	0.0122(6)	3.6(2)
C43	-0.3044(7)	-0.0348(7)	0.5758(6)	3.5(2)
C44	0.0108(9)	0.6759(9)	0.7540(7)	5.3(3)

$$B_{\text{eq}} = 8/3 \pi^2 (U_{11}(\text{aa}^*)^2 + U_{22}(\text{bb}^*)^2 + U_{33}(\text{cc}^*)^2 + 2U_{12}(\text{aa}^*\text{bb}^*)\cos \gamma + 2U_{13}(\text{aa}^*\text{cc}^*)\cos \beta + 2U_{23}(\text{bb}^*\text{cc}^*)\cos \alpha)$$

Table 2. Atomic coordinates and  $B_{iso}$  involving hydrogens/ $B_{eq}$ 

atom	x	y	z	$B_{eq}$
H1	-0.2708	-0.0332	0.5199	3.978
H2	0.0472	0.6298	0.7082	5.993
H3	0.7541	0.5364	0.8999	3.202
H4	0.7841	0.5185	1.0011	3.197
H5	0.6671	0.5438	0.9709	3.191
H6	0.7644	0.2771	0.8187	4.016
H7	0.8273	0.3891	0.8363	4.013
H8	0.7200	0.3487	0.7599	4.024
H9	0.6936	0.3843	1.0421	3.823
H10	0.7763	0.3197	0.9855	3.821
H11	0.6509	0.2761	0.9636	3.833
H12	-0.6618	0.2297	-0.0067	5.676
H13	-0.6866	0.1177	-0.0823	5.683
H14	-0.5661	0.1641	-0.0314	5.682
H15	-0.7945	0.1101	0.1353	5.254
H16	-0.8063	0.1810	0.0676	5.244
H17	-0.7251	0.2211	0.1661	5.253
H18	-0.6580	-0.0330	-0.0435	3.856
H19	-0.7719	-0.0232	-0.0075	3.858
H20	-0.6733	-0.0436	0.0590	3.855
H21	0.1957	0.4399	0.6884	1.279
H22	-0.0732	0.0770	0.5200	1.597
H23	0.0019	0.4464	0.5791	1.370
H24	-0.2489	0.0791	0.3868	1.301
H25	0.0669	0.2988	0.8330	2.847
H26	-0.0176	0.2660	0.9605	3.479
H27	-0.0453	0.0965	0.9713	3.004
H28	0.0095	-0.0418	0.8553	2.992
H29	0.0982	-0.0096	0.7269	2.214
H30	-0.2526	0.5627	0.5108	2.736
H31	-0.3641	0.6486	0.6222	3.418
H32	-0.4894	0.5476	0.6814	3.183
H33	-0.4962	0.3654	0.6399	3.172
H34	-0.3811	0.2825	0.5324	2.352
H35	0.3753	0.1719	0.7252	2.043

H36	0.5548	0.2094	0.8047	2.568
H37	0.5265	0.5218	0.8944	1.810

Table 2. Atomic coordinates and  $B_{\text{ISO}}$  involving hydrogens/ $B_{\text{Eq}}$  (continued)

atom	x	y	z	$B_{\text{Eq}}$
H38	0.3472	0.4823	0.8105	1.556
H39	-0.4230	0.0439	0.2945	2.790
H40	-0.5715	0.0001	0.1711	3.124
H41	-0.5098	0.2840	0.1326	3.221
H42	-0.3583	0.3299	0.2587	3.439

$$B_{\text{Eq}} = 8/3 \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos \gamma + 2U_{13}(aa^*cc^*)\cos \beta + 2U_{23}(bb^*cc^*)\cos \alpha)$$

Table 3. Anisotropic Displacement Parameters

atom	U <sub>11</sub>	U <sub>22</sub>	U <sub>33</sub>	U <sub>12</sub>	U <sub>13</sub>	U <sub>23</sub>
C11	0.062(2)	0.0343(12)	0.086(2)	-0.0066(11)	0.0082(13)	0.0144(12)
C12	0.074(2)	0.051(2)	0.084(2)	0.017(1)	0.023(2)	0.019(1)
C13	0.069(2)	0.130(3)	0.070(2)	-0.031(2)	-0.028(2)	0.057(2)
C14	0.102(3)	0.070(2)	0.140(3)	-0.005(2)	0.078(3)	0.025(2)
C15	0.064(2)	0.0452(13)	0.0418(12)	0.0121(11)	0.0060(10)	0.0043(10)
C16	0.064(2)	0.075(2)	0.059(2)	0.004(1)	0.0107(13)	0.005(1)
P1	0.0127(7)	0.0092(7)	0.0152(7)	0.0009(6)	-0.0014(5)	0.0045(6)
P2	0.0108(7)	0.0095(7)	0.0168(7)	0.0013(6)	-0.0006(5)	0.0054(6)
O1	0.022(2)	0.010(2)	0.020(2)	0.004(2)	0.001(2)	0.002(2)
O2	0.018(2)	0.010(2)	0.022(2)	-0.001(2)	0.001(2)	0.005(2)
C1	0.012(3)	0.016(3)	0.016(3)	-0.001(2)	0.001(2)	0.006(2)
C2	0.014(3)	0.011(3)	0.016(3)	0.002(2)	-0.002(2)	0.002(2)
C3	0.019(3)	0.010(3)	0.016(3)	-0.000(2)	-0.000(2)	0.004(2)
C4	0.019(3)	0.009(3)	0.015(3)	0.000(2)	0.003(2)	0.004(2)
C5	0.019(3)	0.009(3)	0.023(3)	0.002(2)	0.001(2)	0.008(2)
C6	0.012(3)	0.011(3)	0.021(3)	-0.003(2)	-0.006(2)	0.004(2)
C7	0.019(3)	0.011(3)	0.016(3)	-0.001(2)	-0.001(2)	0.004(2)
C8	0.012(3)	0.009(3)	0.020(3)	-0.002(2)	-0.001(2)	0.004(2)
C9	0.015(3)	0.012(3)	0.015(3)	-0.002(2)	-0.001(2)	0.002(2)
C10	0.015(3)	0.011(3)	0.013(3)	-0.000(2)	0.003(2)	0.003(2)
C11	0.014(3)	0.017(3)	0.017(3)	0.002(2)	0.001(2)	0.005(2)
C12	0.039(4)	0.022(3)	0.029(3)	0.012(3)	0.015(3)	0.014(3)
C13	0.043(4)	0.041(5)	0.026(4)	0.013(4)	0.012(3)	0.014(3)
C14	0.020(3)	0.049(5)	0.026(3)	0.007(3)	0.001(3)	0.026(3)
C15	0.027(4)	0.037(4)	0.031(4)	-0.001(3)	0.003(3)	0.022(3)
C16	0.026(3)	0.022(3)	0.022(3)	-0.003(3)	-0.001(3)	0.010(3)
C17	0.016(3)	0.016(3)	0.018(3)	0.004(2)	0.001(2)	0.008(2)
C18	0.033(4)	0.019(3)	0.035(4)	0.009(3)	0.013(3)	0.012(3)
C19	0.045(4)	0.025(4)	0.038(4)	0.016(3)	0.017(4)	0.008(3)
C20	0.031(4)	0.041(4)	0.029(4)	0.020(3)	0.011(3)	0.008(3)
C21	0.026(4)	0.042(5)	0.032(4)	0.006(3)	0.011(3)	0.012(3)
C22	0.026(3)	0.020(3)	0.028(3)	-0.004(3)	0.004(3)	0.005(3)
C23	0.009(3)	0.014(3)	0.019(3)	0.004(2)	0.001(2)	0.005(2)
C24	0.020(3)	0.012(3)	0.033(4)	-0.001(3)	-0.007(3)	0.006(3)
C25	0.018(3)	0.023(4)	0.040(4)	0.002(3)	-0.003(3)	0.014(3)

C26	0.015(3)	0.024(3)	0.022(3)	0.001(3)	-0.000(2)	0.007(3)
C27	0.019(3)	0.021(3)	0.017(3)	0.004(3)	-0.001(2)	0.001(3)

Table 3. Anisotropic Displacement Parameters (continued)

atom	U <sub>11</sub>	U <sub>22</sub>	U <sub>33</sub>	U <sub>12</sub>	U <sub>13</sub>	U <sub>23</sub>
C28	0.013(3)	0.014(3)	0.023(3)	0.003(2)	0.002(2)	0.003(2)
C29	0.010(3)	0.038(4)	0.042(4)	0.004(3)	-0.009(3)	0.014(3)
C30	0.017(3)	0.038(4)	0.045(4)	-0.003(3)	-0.009(3)	0.008(4)
C31	0.024(4)	0.058(6)	0.045(5)	0.013(4)	0.002(3)	0.008(4)
C32	0.030(4)	0.048(5)	0.043(5)	-0.001(4)	-0.012(3)	0.024(4)
C33	0.013(3)	0.012(3)	0.015(3)	0.003(2)	-0.001(2)	0.003(2)
C34	0.037(4)	0.021(4)	0.030(4)	-0.009(3)	-0.012(3)	0.016(3)
C35	0.036(4)	0.024(4)	0.039(4)	-0.015(3)	-0.014(3)	0.015(3)
C36	0.014(3)	0.024(3)	0.017(3)	0.005(2)	-0.001(2)	0.010(2)
C37	0.032(4)	0.029(4)	0.041(4)	-0.001(3)	-0.020(3)	0.023(3)
C38	0.028(4)	0.023(4)	0.058(5)	-0.007(3)	-0.021(3)	0.024(4)
C39	0.016(3)	0.033(4)	0.019(3)	-0.000(3)	-0.007(2)	0.005(3)
C40	0.066(6)	0.081(7)	0.033(4)	-0.020(6)	-0.017(4)	0.034(5)
C41	0.028(4)	0.089(8)	0.048(5)	0.025(5)	-0.004(4)	0.007(5)
C42	0.038(4)	0.046(5)	0.037(4)	-0.005(4)	-0.022(4)	0.005(4)
C43	0.036(4)	0.052(5)	0.038(4)	-0.008(4)	-0.004(3)	0.014(4)
C44	0.071(7)	0.068(7)	0.051(6)	-0.009(6)	0.036(5)	-0.006(5)

The general temperature factor expression:  $\exp(-2\pi^2(a^2U_{11}h^2 + b^2U_{22}k^2 + c^2U_{33}l^2 + 2a*b*U_{12}hk + 2a*c*U_{13}hl + 2b*c*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Cl(1)	C(43)	1.746(9)	Cl(2)	C(43)	1.76(1)
Cl(3)	C(43)	1.747(9)	Cl(4)	C(44)	1.74(1)
Cl(5)	C(44)	1.727(9)	Cl(6)	C(44)	1.80(1)
P(1)	O(1)	1.493(4)	P(1)	C(1)	1.825(6)
P(1)	C(4)	1.793(6)	P(1)	C(11)	1.794(7)
P(2)	O(2)	1.494(5)	P(2)	C(7)	1.809(7)
P(2)	C(10)	1.811(5)	P(2)	C(17)	1.800(6)
C(1)	C(2)	1.351(9)	C(1)	C(23)	1.460(7)
C(2)	C(3)	1.478(7)	C(3)	C(4)	1.402(8)
C(3)	C(6)	1.387(9)	C(4)	C(5)	1.403(7)
C(5)	C(8)	1.389(9)	C(6)	C(7)	1.388(7)
C(7)	C(8)	1.400(8)	C(8)	C(9)	1.483(7)
C(9)	C(10)	1.355(9)	C(10)	C(33)	1.462(7)
C(11)	C(12)	1.383(9)	C(11)	C(16)	1.41(1)
C(12)	C(13)	1.38(1)	C(13)	C(14)	1.37(1)
C(14)	C(15)	1.37(1)	C(15)	C(16)	1.41(1)
C(17)	C(18)	1.391(9)	C(17)	C(22)	1.39(1)
C(18)	C(19)	1.39(1)	C(19)	C(20)	1.39(1)
C(20)	C(21)	1.38(1)	C(21)	C(22)	1.37(1)
C(23)	C(24)	1.398(9)	C(23)	C(28)	1.411(7)
C(24)	C(25)	1.366(9)	C(25)	C(26)	1.401(8)
C(26)	C(27)	1.43(1)	C(26)	C(29)	1.530(8)
C(27)	C(28)	1.381(8)	C(29)	C(30)	1.51(1)
C(29)	C(31)	1.54(1)	C(29)	C(32)	1.53(1)
C(33)	C(34)	1.383(8)	C(33)	C(38)	1.40(1)
C(34)	C(35)	1.37(1)	C(35)	C(36)	1.40(1)
C(36)	C(37)	1.385(9)	C(36)	C(39)	1.523(8)
C(37)	C(38)	1.40(1)	C(39)	C(40)	1.54(1)
C(39)	C(41)	1.56(1)	C(39)	C(42)	1.55(1)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(2)	H(21)	0.9500	C(5)	H(22)	0.9500
C(6)	H(23)	0.9500	C(9)	H(24)	0.9500
C(12)	H(25)	0.9500	C(13)	H(26)	0.9500
C(14)	H(27)	0.9498	C(15)	H(28)	0.9500
C(16)	H(29)	0.9500	C(18)	H(30)	0.9500
C(19)	H(31)	0.9500	C(20)	H(32)	0.9499
C(21)	H(33)	0.9501	C(22)	H(34)	0.9501
C(24)	H(35)	0.9500	C(25)	H(36)	0.9500
C(27)	H(37)	0.9499	C(28)	H(38)	0.9500
C(30)	H(3)	0.9500	C(30)	H(4)	0.9500
C(30)	H(5)	0.9499	C(31)	H(6)	0.9500
C(31)	H(7)	0.9500	C(31)	H(8)	0.9500
C(32)	H(9)	0.9501	C(32)	H(10)	0.9500
C(32)	H(11)	0.9500	C(34)	H(39)	0.9500
C(35)	H(40)	0.9500	C(37)	H(41)	0.9499
C(38)	H(42)	0.9499	C(40)	H(12)	0.9500
C(40)	H(13)	0.9500	C(40)	H(14)	0.9500
C(41)	H(15)	0.9500	C(41)	H(16)	0.9500
C(41)	H(17)	0.9500	C(42)	H(18)	0.9499
C(42)	H(19)	0.9500	C(42)	H(20)	0.9499
C(43)	H(1)	0.9500	C(44)	H(2)	0.9500
H(23)	C(6)	0.9500	H(40)	C(35)	0.9500

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
Cl(1)	C(43)	Cl(2)	111.1(5)	Cl(1)	C(43)	Cl(3)	112.0(6)
Cl(2)	C(43)	Cl(3)	107.8(4)	Cl(4)	C(44)	Cl(5)	111.8(6)
Cl(4)	C(44)	Cl(6)	109.2(6)	Cl(5)	C(44)	Cl(6)	110.7(6)
C(1)	P(1)	O(1)	118.9(3)	C(4)	P(1)	O(1)	116.8(2)
C(11)	P(1)	O(1)	112.0(3)	C(4)	P(1)	C(1)	93.0(3)
C(11)	P(1)	C(1)	107.9(3)	P(1)	C(1)	C(2)	108.3(4)
P(1)	C(1)	C(23)	123.6(5)	C(11)	P(1)	C(4)	106.2(3)
P(1)	C(4)	C(3)	109.1(4)	P(1)	C(4)	C(5)	128.8(5)
P(1)	C(11)	C(12)	122.3(5)	P(1)	C(11)	C(16)	118.6(4)
C(7)	P(2)	O(2)	115.4(3)	C(10)	P(2)	O(2)	118.7(3)
C(17)	P(2)	O(2)	112.8(3)	C(10)	P(2)	C(7)	93.0(3)
C(17)	P(2)	C(7)	108.1(3)	P(2)	C(7)	C(6)	128.7(5)
P(2)	C(7)	C(8)	108.7(4)	C(17)	P(2)	C(10)	106.8(3)
P(2)	C(10)	C(9)	109.1(4)	P(2)	C(10)	C(33)	124.5(5)
P(2)	C(17)	C(18)	118.3(5)	P(2)	C(17)	C(22)	122.1(5)
C(1)	C(2)	C(3)	116.3(5)	C(23)	C(1)	C(2)	128.2(5)
C(1)	C(23)	C(24)	122.3(5)	C(1)	C(23)	C(28)	121.0(6)
C(2)	C(3)	C(4)	112.9(5)	C(2)	C(3)	C(6)	126.6(5)
C(3)	C(4)	C(5)	122.0(6)	C(6)	C(3)	C(4)	120.3(5)
C(3)	C(6)	C(7)	117.6(6)	C(4)	C(5)	C(8)	117.3(5)
C(5)	C(8)	C(7)	120.3(5)	C(5)	C(8)	C(9)	126.1(5)
C(6)	C(7)	C(8)	122.5(6)	C(7)	C(8)	C(9)	113.4(5)
C(8)	C(9)	C(10)	115.6(5)	C(9)	C(10)	C(33)	126.4(5)
C(10)	C(33)	C(34)	123.2(6)	C(10)	C(33)	C(38)	120.2(5)
C(11)	C(12)	C(13)	121.4(7)	C(16)	C(11)	C(12)	118.9(6)
C(11)	C(16)	C(15)	118.9(6)	C(12)	C(13)	C(14)	119.9(7)
C(13)	C(14)	C(15)	120.6(7)	C(14)	C(15)	C(16)	120.4(7)
C(17)	C(18)	C(19)	120.1(7)	C(22)	C(17)	C(18)	119.5(6)
C(17)	C(22)	C(21)	121.0(7)	C(18)	C(19)	C(20)	118.6(7)
C(19)	C(20)	C(21)	121.6(7)	C(20)	C(21)	C(22)	119.1(7)
C(28)	C(23)	C(24)	116.5(5)	C(23)	C(24)	C(25)	122.0(5)
C(23)	C(28)	C(27)	122.5(6)	C(24)	C(25)	C(26)	122.1(7)
C(25)	C(26)	C(27)	117.0(5)	C(25)	C(26)	C(29)	121.2(6)
C(26)	C(27)	C(28)	119.8(5)	C(29)	C(26)	C(27)	121.8(5)
C(26)	C(29)	C(30)	113.1(6)	C(26)	C(29)	C(31)	108.4(5)

C(26)	C(29)	C(32)	109.9(6)	C(31)	C(29)	C(30)	108.7(6)
C(32)	C(29)	C(30)	108.3(6)	C(32)	C(29)	C(31)	108.3(7)

Table 6. Bond angles (°) -- continued

atom	atom	atom	angle	atom	atom	atom	angle
C(38)	C(33)	C(34)	116.5(5)	C(33)	C(34)	C(35)	122.7(7)
C(33)	C(38)	C(37)	120.4(6)	C(34)	C(35)	C(36)	122.2(6)
C(35)	C(36)	C(37)	115.5(6)	C(35)	C(36)	C(39)	122.6(6)
C(36)	C(37)	C(38)	122.6(8)	C(39)	C(36)	C(37)	121.6(7)
C(36)	C(39)	C(40)	110.6(6)	C(36)	C(39)	C(41)	107.5(5)
C(36)	C(39)	C(42)	110.9(6)	C(41)	C(39)	C(40)	108.8(8)
C(42)	C(39)	C(40)	108.3(6)	C(42)	C(39)	C(41)	110.7(6)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
Cl(1)	C(43)	H(1)	108.4	Cl(2)	C(43)	H(1)	109.1
Cl(3)	C(43)	H(1)	108.3	Cl(4)	C(44)	H(2)	108.4
Cl(5)	C(44)	H(2)	108.1	Cl(6)	C(44)	H(2)	108.6
C(1)	C(2)	H(21)	122.1	H(21)	C(2)	C(3)	121.6
C(3)	C(6)	H(23)	121.2	C(4)	C(5)	H(22)	121.1
H(22)	C(5)	C(8)	121.6	H(23)	C(6)	C(7)	121.2
C(8)	C(9)	H(24)	121.7	H(24)	C(9)	C(10)	122.7
C(11)	C(12)	H(25)	118.7	C(11)	C(16)	H(29)	119.9
H(25)	C(12)	C(13)	119.9	C(12)	C(13)	H(26)	120.3
H(26)	C(13)	C(14)	119.9	C(13)	C(14)	H(27)	119.8
H(27)	C(14)	C(15)	119.7	C(14)	C(15)	H(28)	120.3
C(15)	C(16)	H(29)	121.2	H(28)	C(15)	C(16)	119.3
C(17)	C(18)	H(30)	119.5	C(17)	C(22)	H(34)	118.6
H(30)	C(18)	C(19)	120.4	C(18)	C(19)	H(31)	120.3
H(31)	C(19)	C(20)	121.1	C(19)	C(20)	H(32)	118.8
H(32)	C(20)	C(21)	119.6	C(20)	C(21)	H(33)	120.5
C(21)	C(22)	H(34)	120.4	H(33)	C(21)	C(22)	120.5
C(23)	C(24)	H(35)	119.0	C(23)	C(28)	H(38)	118.8
H(35)	C(24)	C(25)	119.0	C(24)	C(25)	H(36)	119.5
H(36)	C(25)	C(26)	118.4	C(26)	C(27)	H(37)	120.1
C(27)	C(28)	H(38)	118.7	H(37)	C(27)	C(28)	120.1
C(29)	C(30)	H(5)	109.7	C(29)	C(30)	H(3)	109.2
C(29)	C(30)	H(4)	109.5	C(29)	C(31)	H(8)	109.1
C(29)	C(31)	H(6)	109.4	C(29)	C(31)	H(7)	109.9
C(29)	C(32)	H(11)	108.9	C(29)	C(32)	H(9)	109.3
C(29)	C(32)	H(10)	110.2	H(5)	C(30)	H(3)	109.5
H(4)	C(30)	H(3)	109.5	H(5)	C(30)	H(4)	109.5
H(8)	C(31)	H(6)	109.5	H(7)	C(31)	H(6)	109.5
H(8)	C(31)	H(7)	109.5	H(11)	C(32)	H(9)	109.5
H(10)	C(32)	H(9)	109.5	H(11)	C(32)	H(10)	109.5
C(33)	C(34)	H(39)	118.5	C(33)	C(38)	H(42)	119.9
H(39)	C(34)	C(35)	118.8	C(34)	C(35)	H(40)	119.3
H(40)	C(35)	C(36)	118.5	C(36)	C(37)	H(41)	118.2
C(37)	C(38)	H(42)	119.7	H(41)	C(37)	C(38)	119.2
C(39)	C(40)	H(14)	110.0	C(39)	C(40)	H(12)	110.0

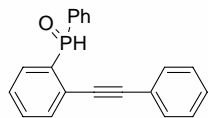
C(39)	C(40)	H(13)	108.5	C(39)	C(41)	H(17)	109.4
C(39)	C(41)	H(15)	108.6	C(39)	C(41)	H(16)	110.4

Table 7. Bond angles involving hydrogens ( $^{\circ}$ ) -- continued

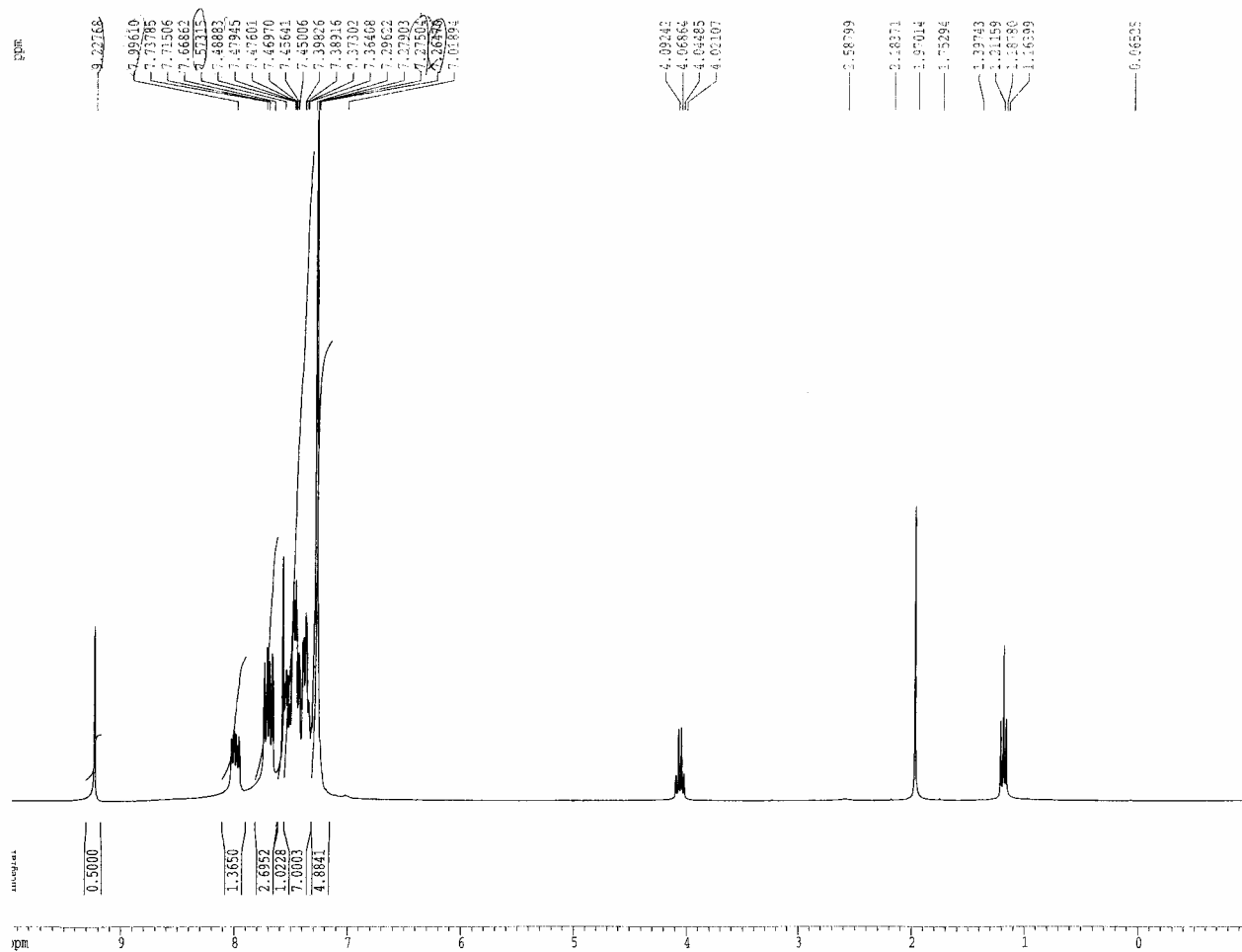
atom	atom	atom	angle	atom	atom	atom	angle
C(39)	C(42)	H(20)	109.7	C(39)	C(42)	H(18)	110.4
C(39)	C(42)	H(19)	108.3	H(14)	C(40)	H(12)	109.5
H(13)	C(40)	H(12)	109.5	H(14)	C(40)	H(13)	109.5
H(17)	C(41)	H(15)	109.5	H(16)	C(41)	H(15)	109.5
H(17)	C(41)	H(16)	109.5	H(20)	C(42)	H(18)	109.5
H(19)	C(42)	H(18)	109.5	H(20)	C(42)	H(19)	109.5

Appendix-2

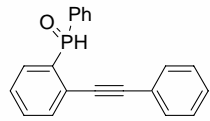
NMR Data



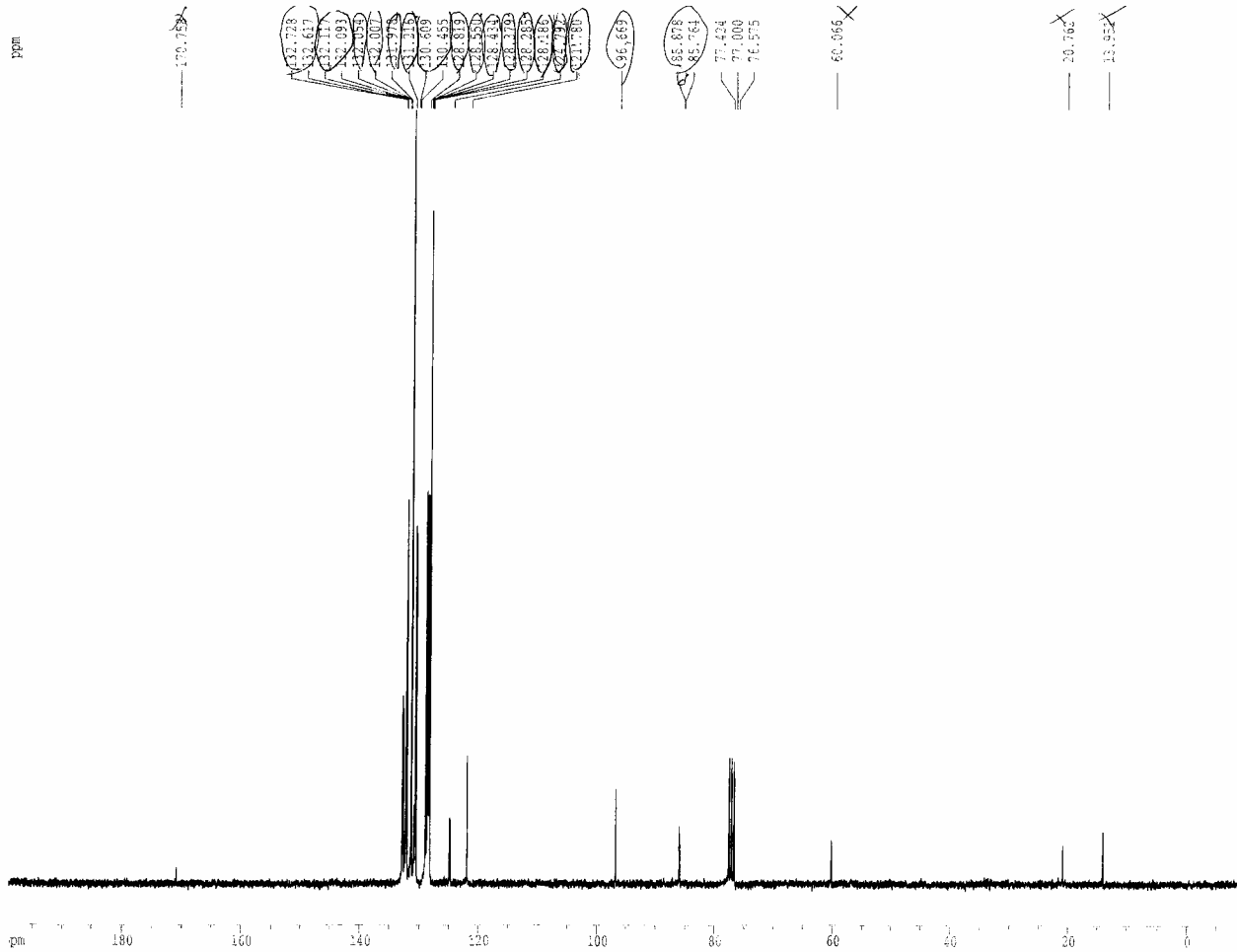
**1a**



$^1\text{H}$  NMR spectrum of **1a** ( $\text{CDCl}_3$ )



**1a**



Current Data Parameters  
 NAME shiraishi  
 EXPNO 705143  
 PROCNO 1

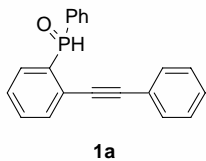
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 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 353  
 DS 2  
 SMH 23809.523 Hz  
 FIDRES 0.363304 Hz  
 AQ 1.3763061 sec  
 RG 8192  
 DW 21.000 usec  
 DE 4.50 usec  
 TE 300.0 K  
 D12 0.00002000 sec  
 PL13 16.00 dB  
 D1 1.00000000 sec  
 CPDPRG2 waltz16  
 PCPD2 100.00 usec  
 SFO2 300.1312005 MHz  
 NUC2 1H  
 PL2 -6.00 dB  
 PL12 16.00 dB  
 P1 5.70 usec  
 DE 4.50 usec  
 SFO1 75.4772501 MHz  
 NUC1 13C  
 PL1 -6.00 dB  
 D11 0.03000000 sec

F2 - Processing parameters  
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 SF 75.4677675 MHz  
 VDF EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

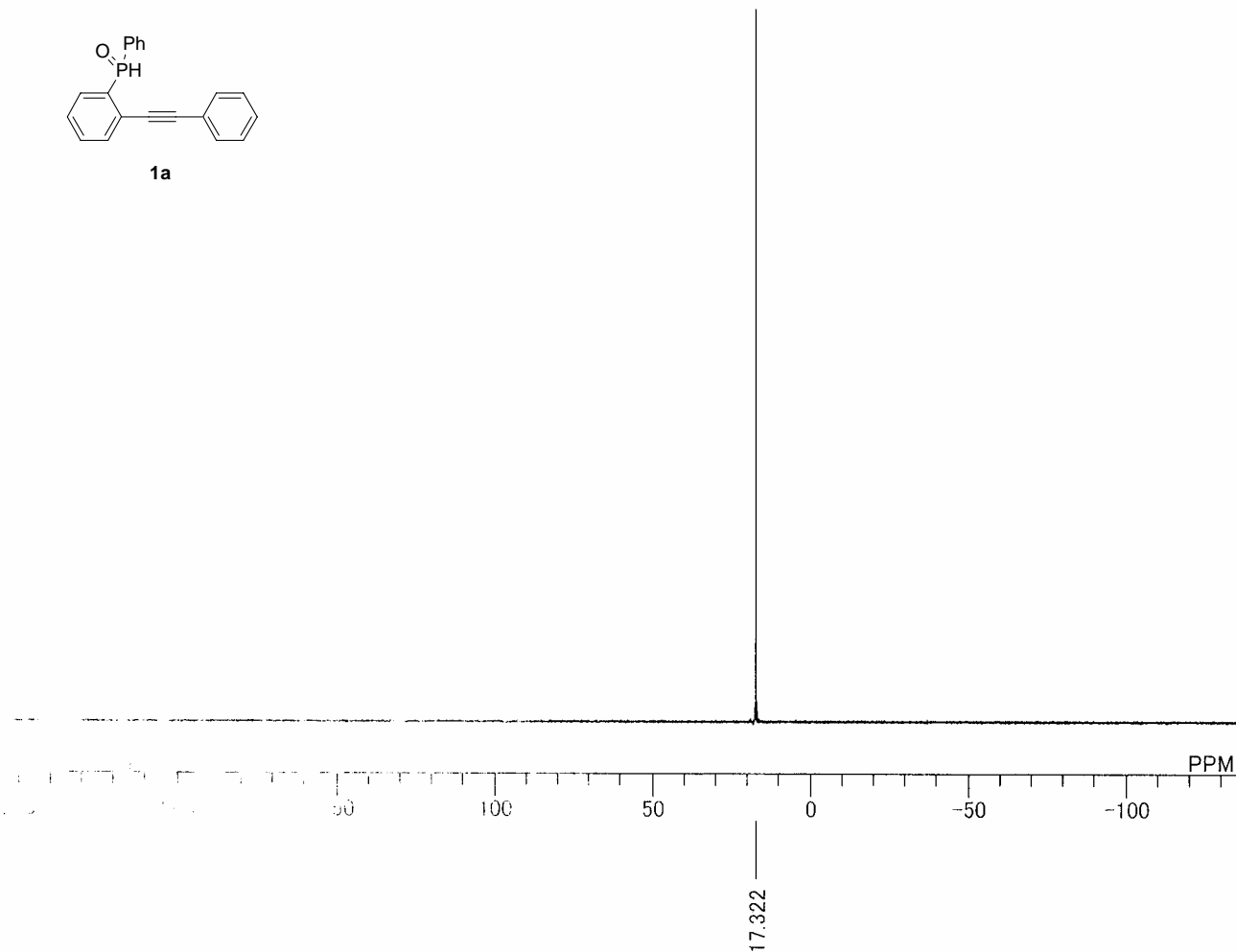
1D NMR plot parameters  
 CX 30.00 cm  
 F1P 200.000 ppm  
 F1 15093.55 Hz  
 F2P -10.000 ppm  
 F2 -754.66 Hz  
 PPMXN 7.00000 ppm/cm  
 HZCM 528.27429 Hz/cm

<sup>13</sup>C NMR spectrum of **1a** (CDCl<sub>3</sub>)

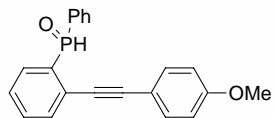
PHO-Ph-705141  
17.322



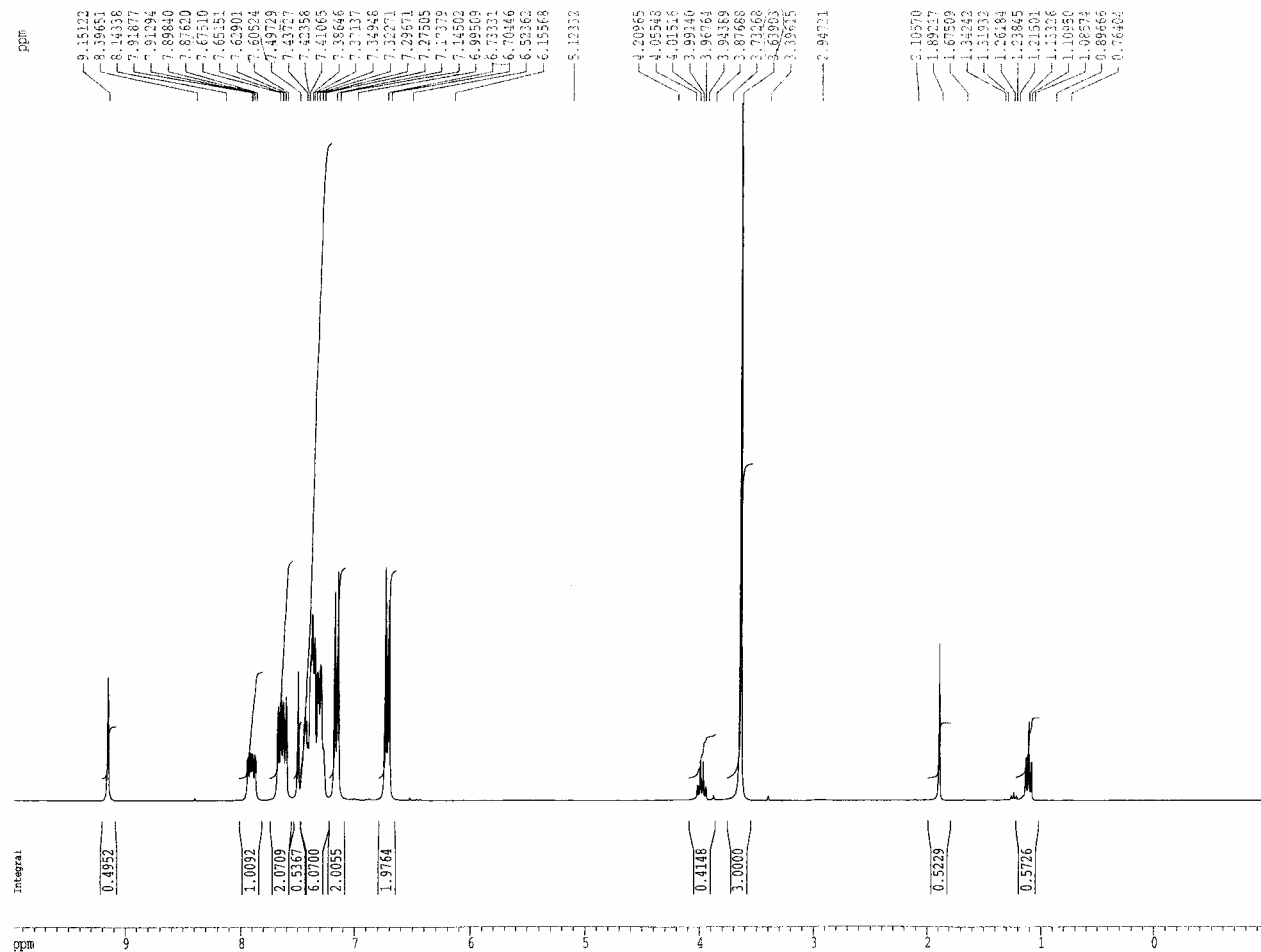
DFILE C:\WINNMR98\COM  
COMNT PHO-Ph-705141  
DATIM Mon May 14 19:32:21  
OBNUC 31P  
EXMOD BCM  
OBFRQ 161.70 MHz  
OBSET 145.00 KHz  
OBFIN 77.2 Hz  
POINT 32768  
FREQU 64102.5 Hz  
SCANS 10  
AQTM 0.511 sec  
PD 2.000 sec  
PW1 5.2 us  
IRNUC 1H  
CTEMP 21.1 c  
SLVNT CDCL3  
EXREF 0.00 ppm  
BF 1.20 Hz  
RGAIN 22



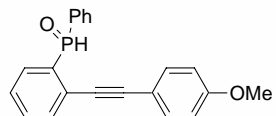
$^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of **1a** ( $\text{CDCl}_3$ )



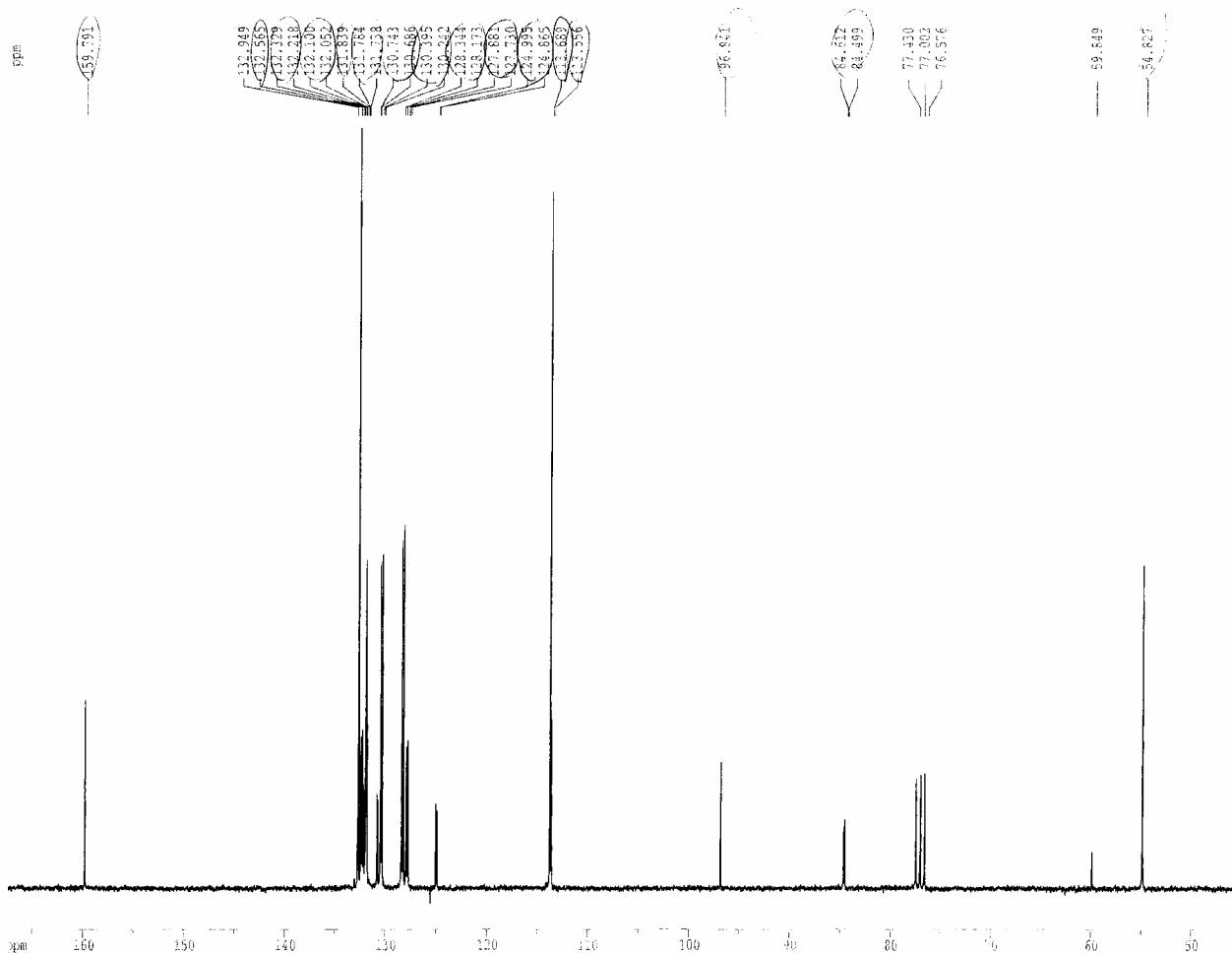
**1b**



<sup>1</sup>H NMR spectrum of **1b** (CDCl<sub>3</sub>)



**1b**



Current Data Parameters  
 NAME shiraishi  
 EXPNO 707282  
 PROCNO 1

F2 - Acquisition Parameters  
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 Time 17.52  
 INSTRUM qcx300  
 PROBHD 5 mm QNP 1H  
 PULPROG zgpg30  
 PD 65536  
 SOLVENT CDC13  
 NS 189  
 DS 2  
 SSB 23809.523 Hz  
 FIDRES 0.363304 Hz  
 AQ 1.3763061 sec  
 RG 4096  
 DM 21.000 usec  
 DE 4.50 usec  
 TE 300.0 K  
 D12 0.00002000 sec  
 PL13 16.00 dB  
 D1 1.00000000 sec  
 CPDPRG2 waltz16  
 PCPD2 100.00 usec  
 SFO2 300.1312005 MHz  
 NUC2 1H  
 PL2 -6.00 dB  
 PL12 16.00 dB  
 P1 5.70 usec  
 DE 4.50 usec  
 SFO1 75.4772501 MHz  
 NUC1 13C  
 PL1 -6.00 dB  
 D11 0.03000000 sec

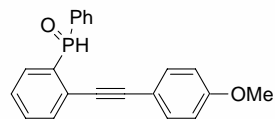
F2 - Processing parameters  
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 SF 75.4677784 MHz  
 MDL EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

ID NMR plot parameters  
 CX 30.00 cm  
 FIP 167.686 ppm  
 F1 12654.91 Hz  
 F2P 44.918 ppm  
 F2 3389.90 Hz  
 PPMCK 4.09126 ppm/cm  
 HZCK 508.63371 Hz/cm

<sup>13</sup>C NMR spectrum of **1b** (CDCl<sub>3</sub>)

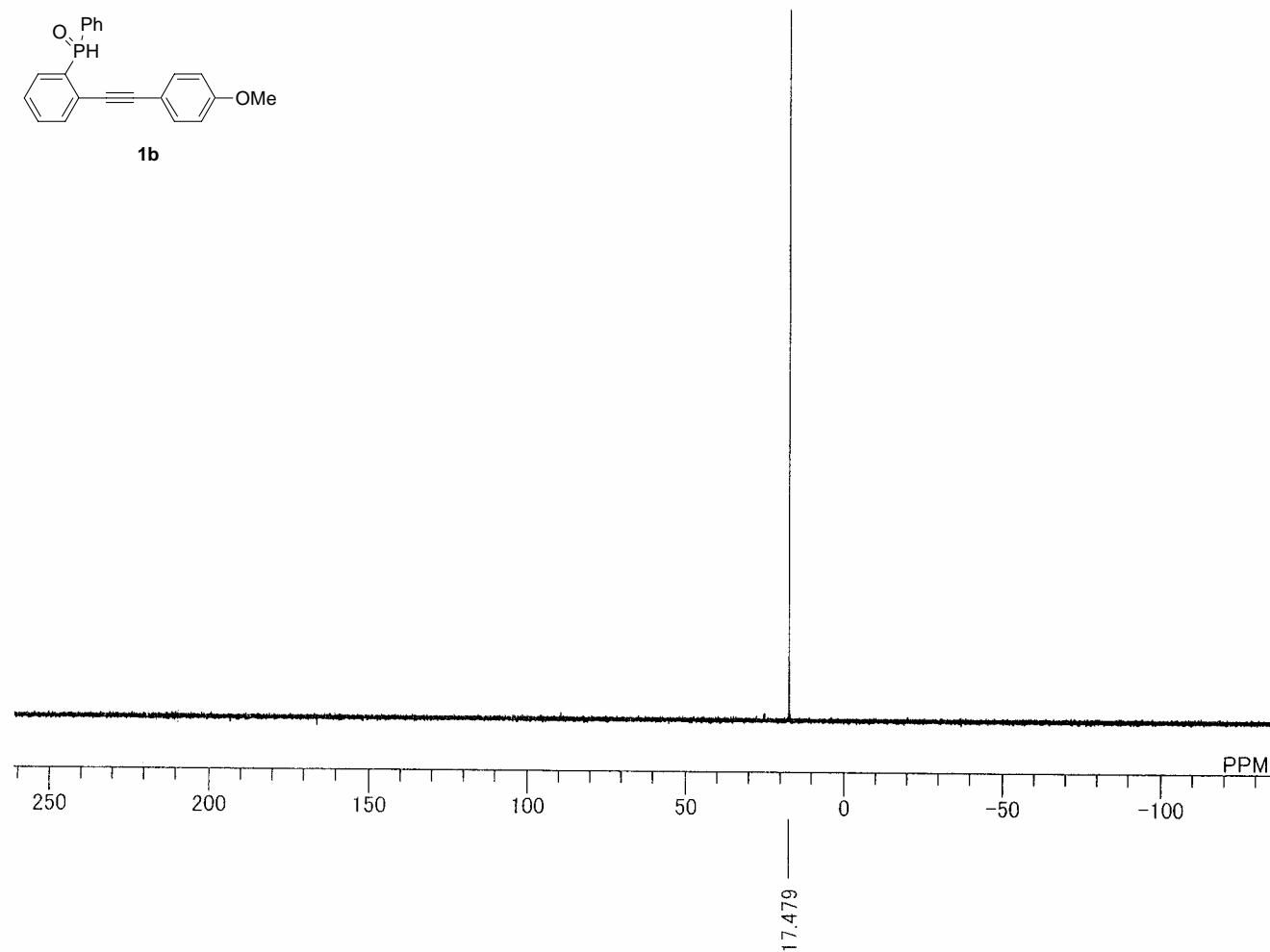
C:\WINNMR98\COMMON\DEFAULT.ALS

1b

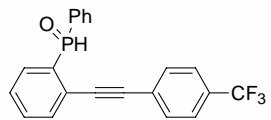


1b

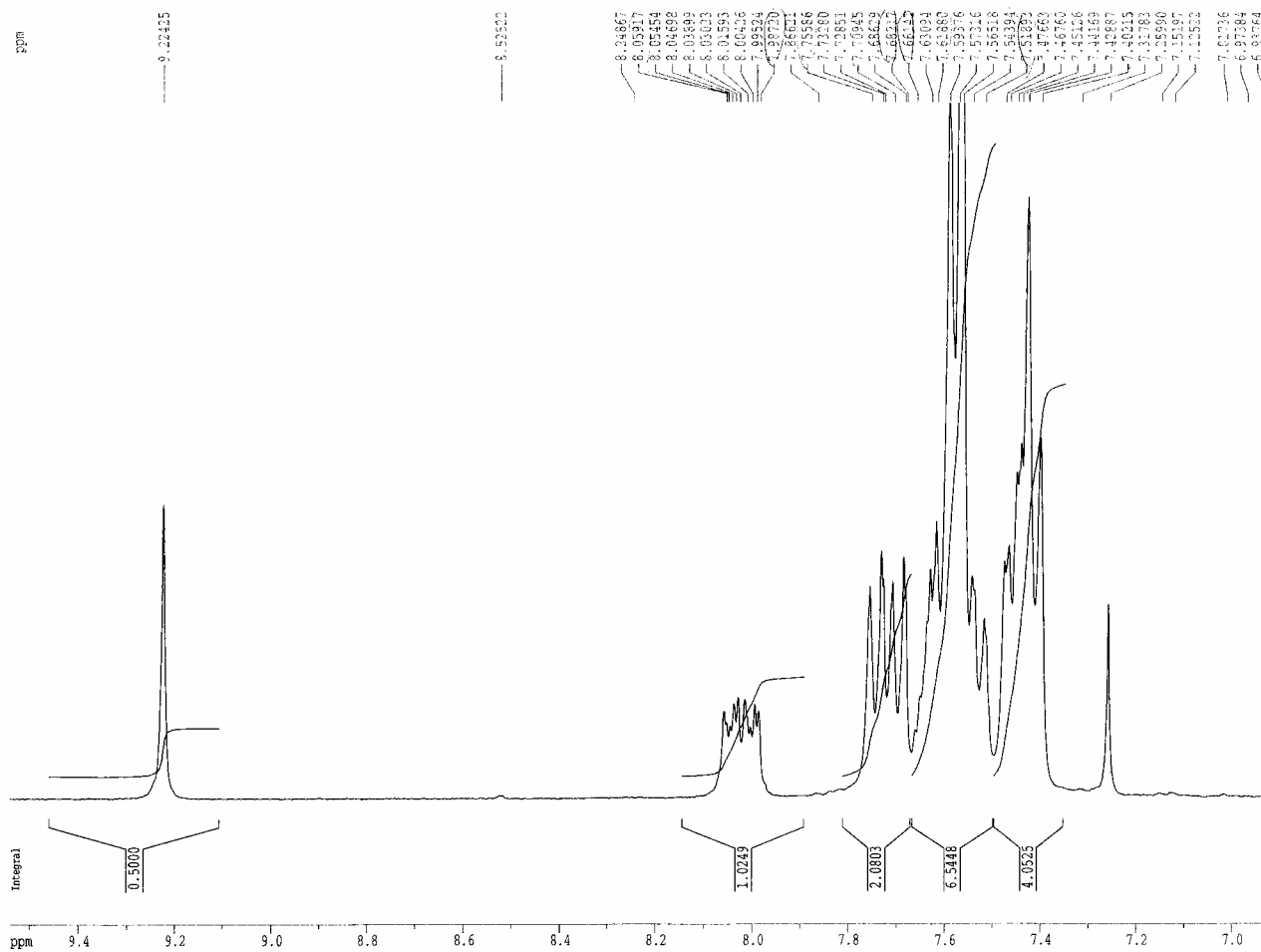
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COMNT PHO-PhOMe-70730  
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OBNUC 31P  
EXMOD BCM  
OBFRQ 161.70 MHz  
OBSET 145.00 KHz  
OBFIN 77.2 Hz  
POINT 32768  
FREQU 64102.5 Hz  
SCANS 4  
ACQTM 0.511 sec  
PD 1.500 sec  
PW1 5.2 us  
IRNUC 1H  
CTEMP 22.4 c  
SLVNT CDCL3  
EXREF 0.00 ppm  
BF 1.20 Hz  
RGAIN 25



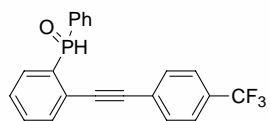
$^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of **1b** ( $\text{CDCl}_3$ )



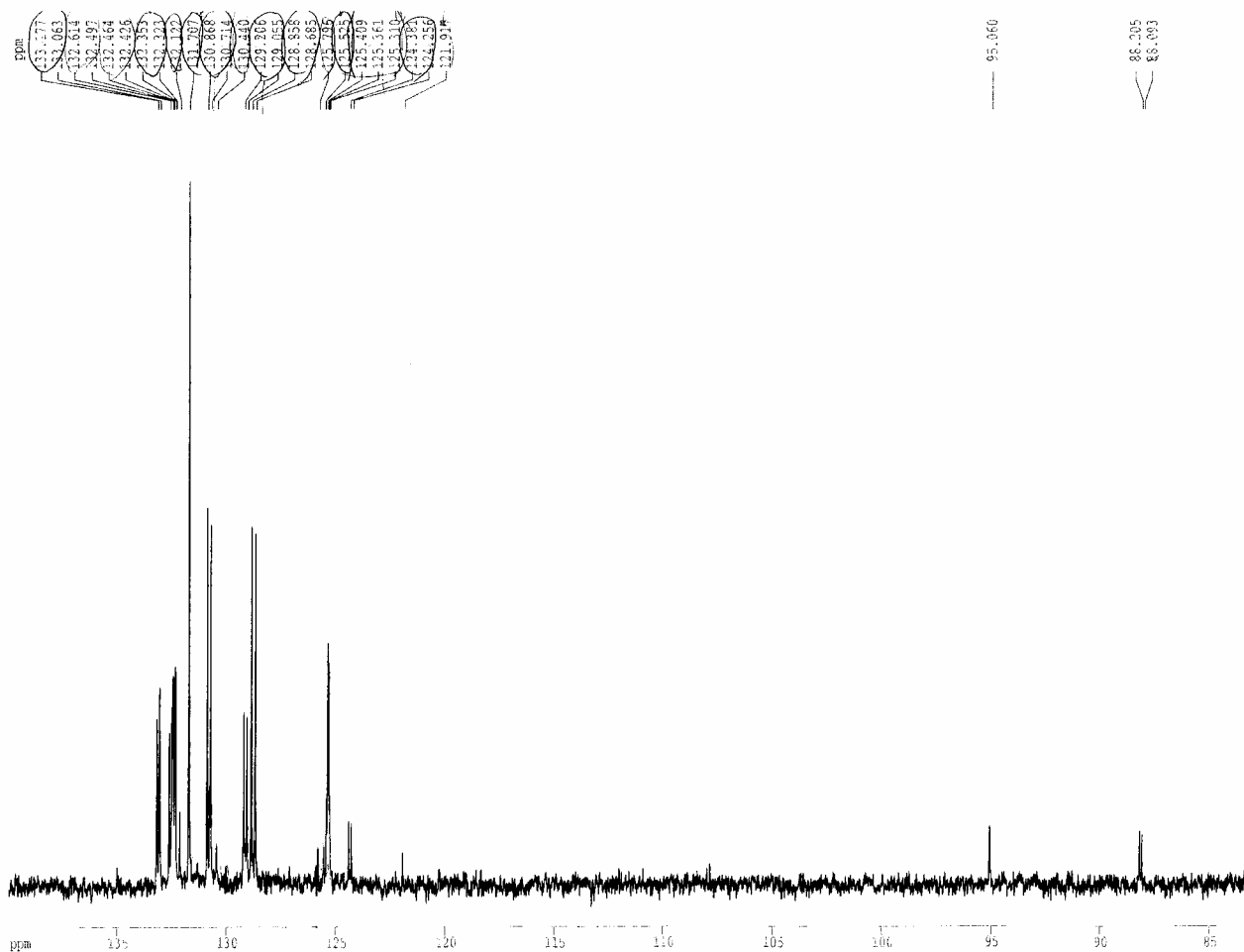
**1c**



$^1\text{H}$  NMR spectrum of **1c** ( $\text{CDCl}_3$ )



**1c**



Current Data Parameters  
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 EXPNO 708022  
 PROCNO 1

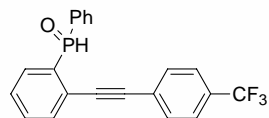
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 PULPROG zgpg30  
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 SOLVENT cdcl3  
 NS 665  
 DS 2  
 SMR 23809.523 Hz  
 FIDRES 0.363304 Hz  
 AQ 1.3763061 sec  
 RG 2580.3  
 DW 21.000 usec  
 DE 4.50 usec  
 TE 300.0 K  
 D12 0.0002000 sec  
 PL13 16.00 dB  
 D1 1.0000000 sec  
 CPDPRG2 waltz16  
 PCPD2 100.00 usec  
 SFO2 300.1312005 MHz  
 NU02 1H  
 PL2 -6.00 dB  
 PL12 16.00 dB  
 P1 5.70 usec  
 DE 4.50 usec  
 SFO1 75.4772501 MHz  
 NU01 13C  
 PL1 -6.00 dB  
 D11 0.0300000 sec

F2 Processing parameters  
 SI 32768  
 SF 75.4677516 MHz  
 NDF EM  
 SSB 0  
 LB 1.00 Hz  
 GE 0  
 PC 1.40

1D NMR plot parameters  
 CZ 30.00 cm  
 FLP 139.922 ppm  
 F1 10559.63 Hz  
 F2P 82.997 ppm  
 F2 6263.57 Hz  
 PPMCM 1.89753 ppm/cm  
 HZCM 143.20221 Hz/cm

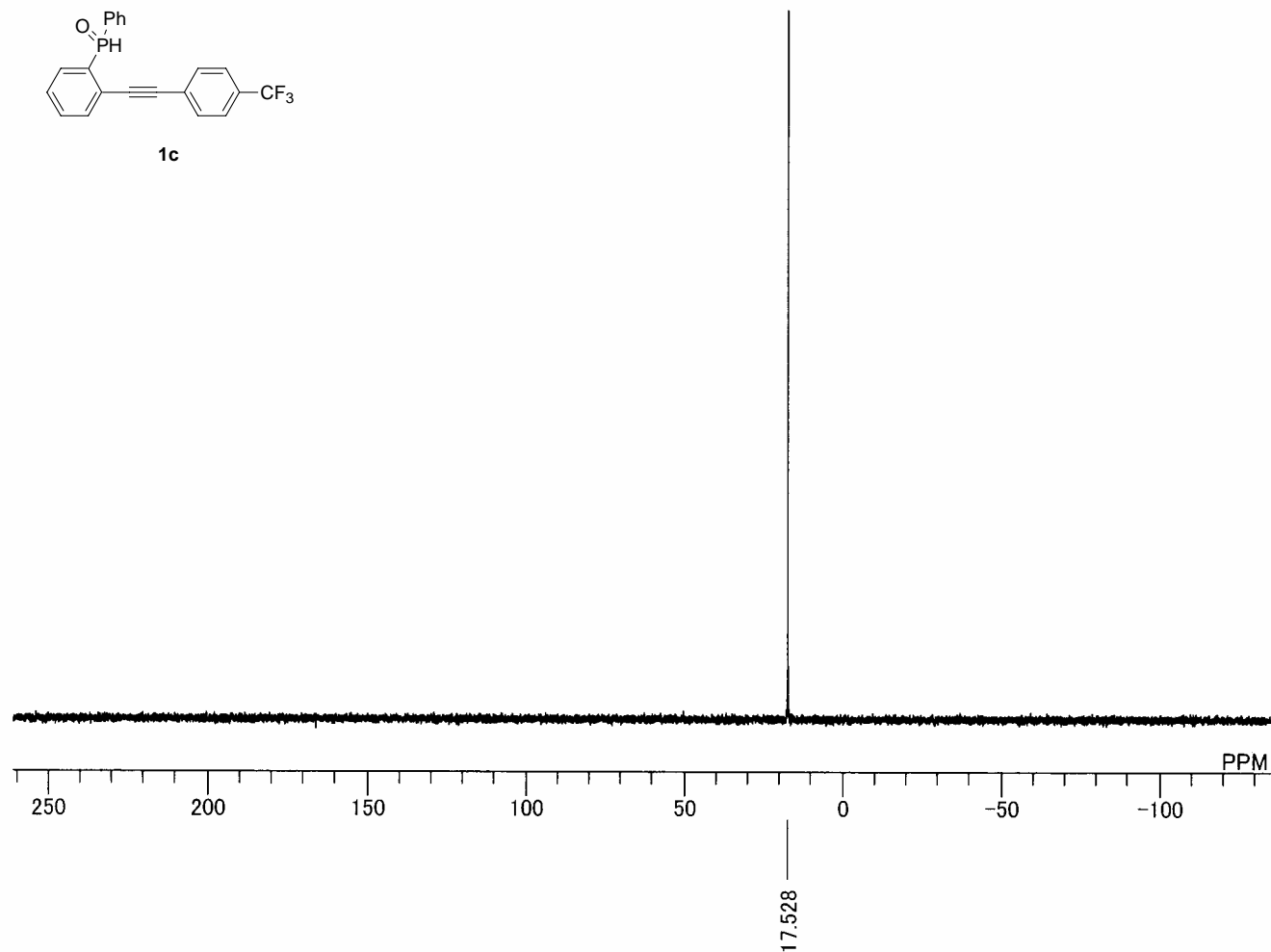
<sup>13</sup>C NMR spectrum of **1c** (CDCl<sub>3</sub>)

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PHO-PhTFM-708021

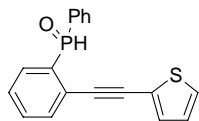


**1c**

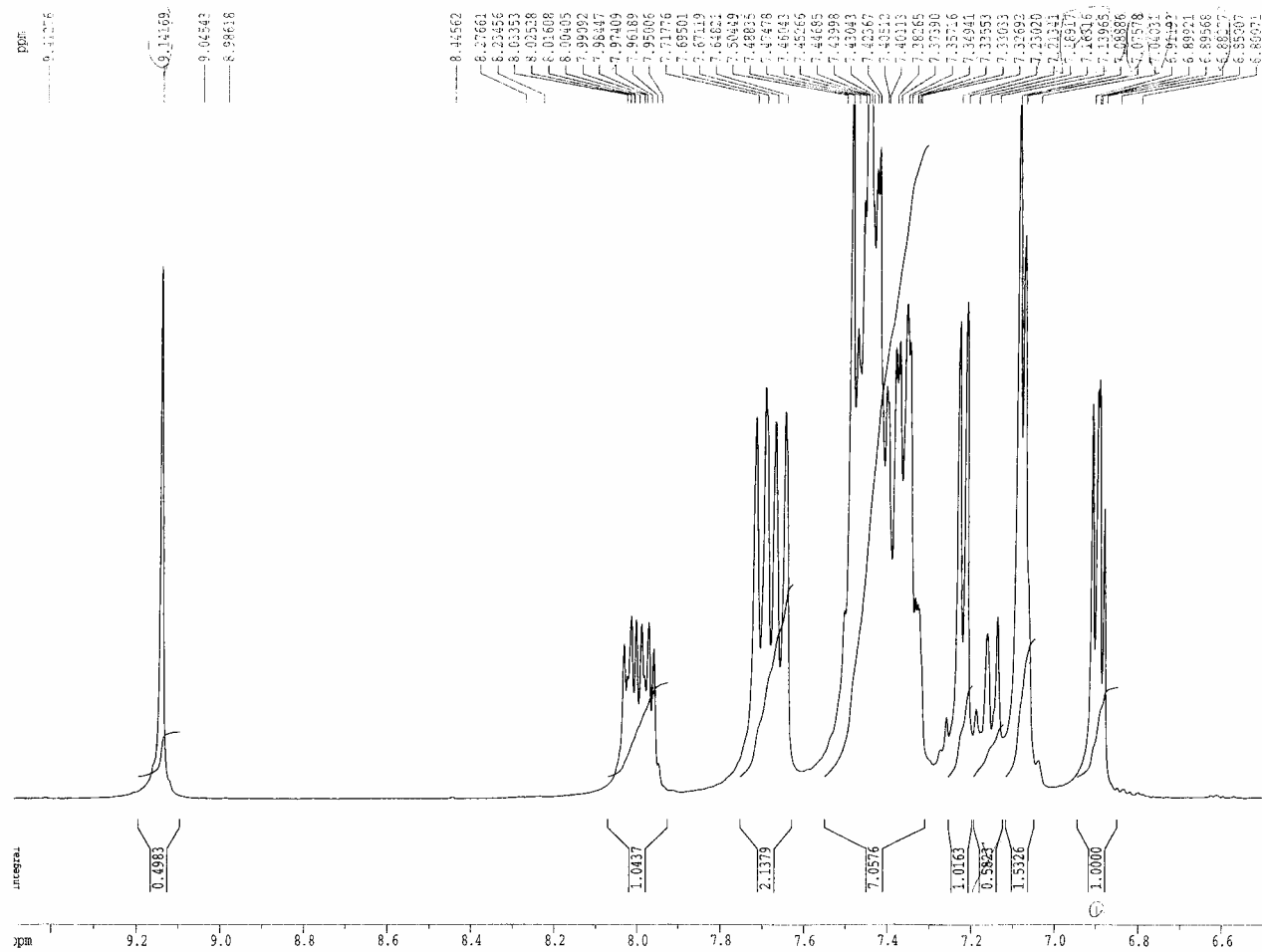
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COMNT PHO-PhTFM-708021  
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EXMOD BCM  
OBFRQ 161.70 MHz  
OBSET 145.00 KHz  
OBFIN 77.2 Hz  
POINT 32768  
FREQU 64102.5 Hz  
SCANS 4  
ACQTM 0.511 sec  
PD 2.000 sec  
PW1 5.2 us  
IRNUC 1H  
CTEMP 23.2 c  
SLVNT CDCL3  
EXREF 0.00 ppm  
BF 4.20 Hz  
RGAIN 26



$^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of **1c** ( $\text{CDCl}_3$ )



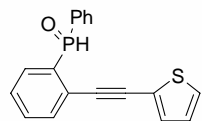
**1d**



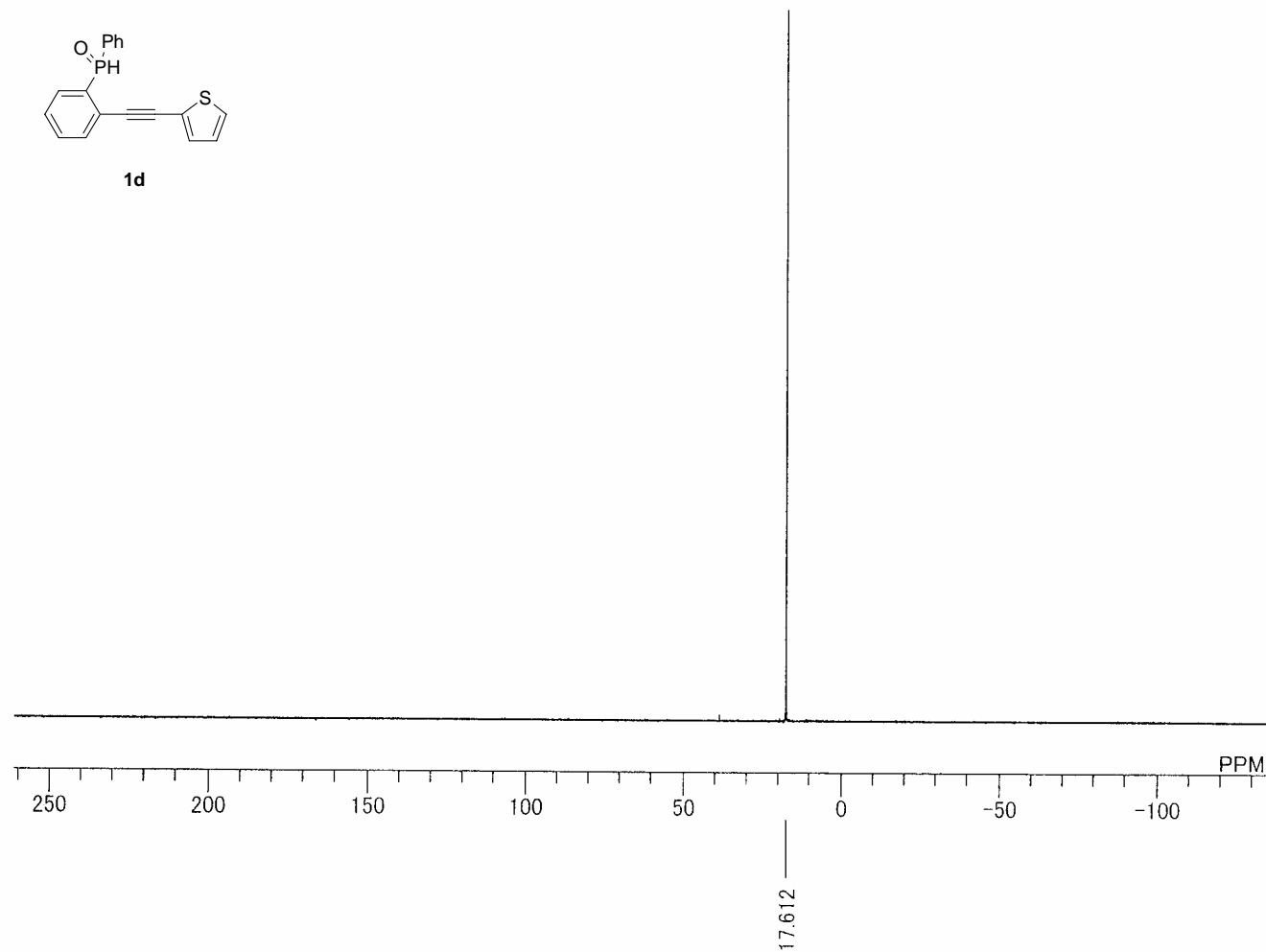
$^1\text{H}$  NMR spectrum of **1d** ( $\text{CDCl}_3$ )



C:\WINNMR98\COMMON\DEFAULT.ALS  
PHO-Thi-707261

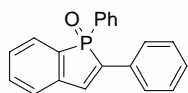


**1d**

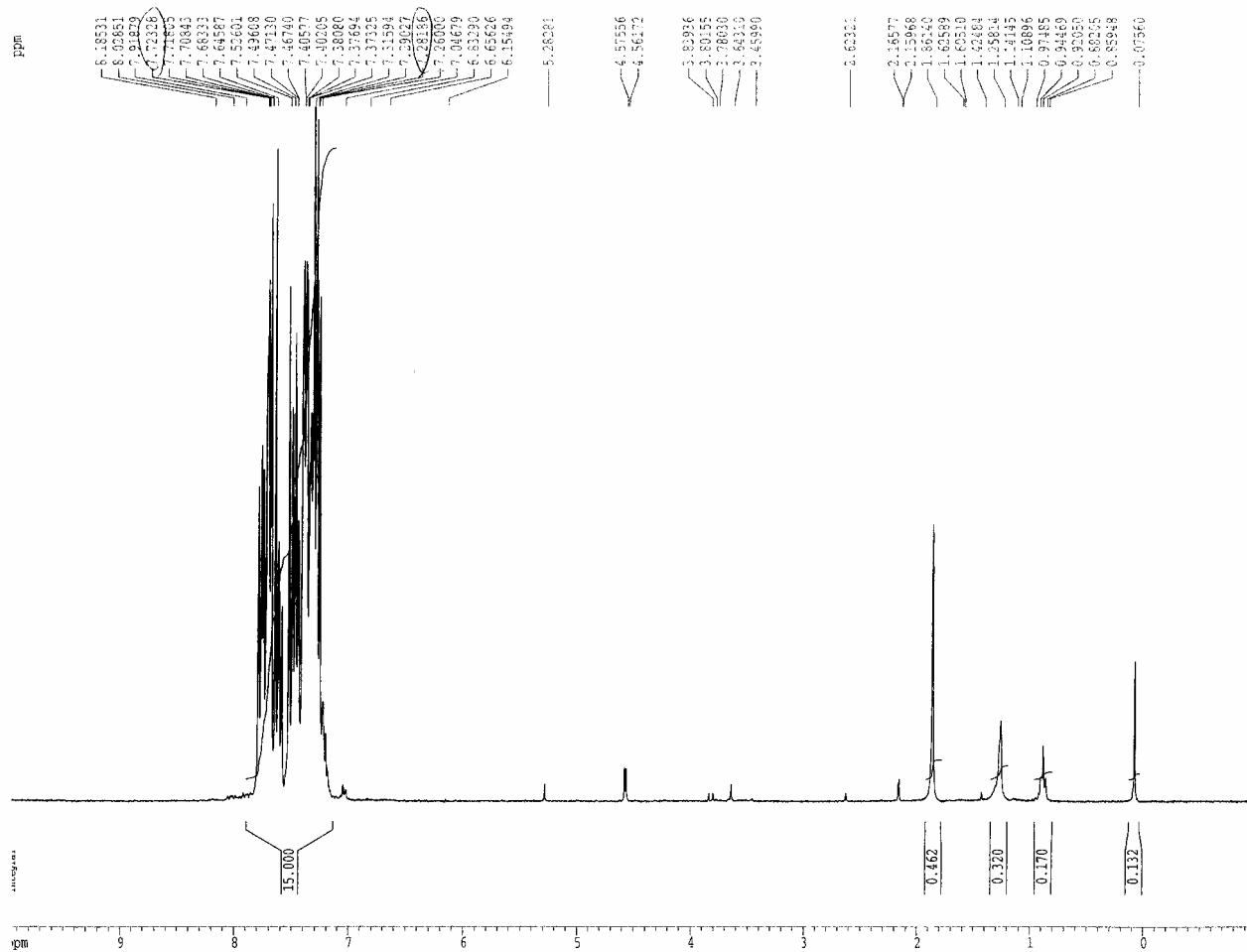


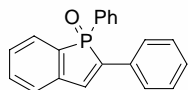
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COMNT	PHO-Thi-707261
DATIM	Thu Jul 26 15:15:46 2
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OBSET	145.00 KHz
OBFIN	77.2 Hz
POINT	32768
FREQU	64102.5 Hz
SCANS	4
ACQTM	0.511 sec
PD	2.000 sec
PW1	5.2 us
IRNUC	1H
CTEMP	22.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	1.20 Hz
RGAIN	21

$^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of **1d** ( $\text{CDCl}_3$ )

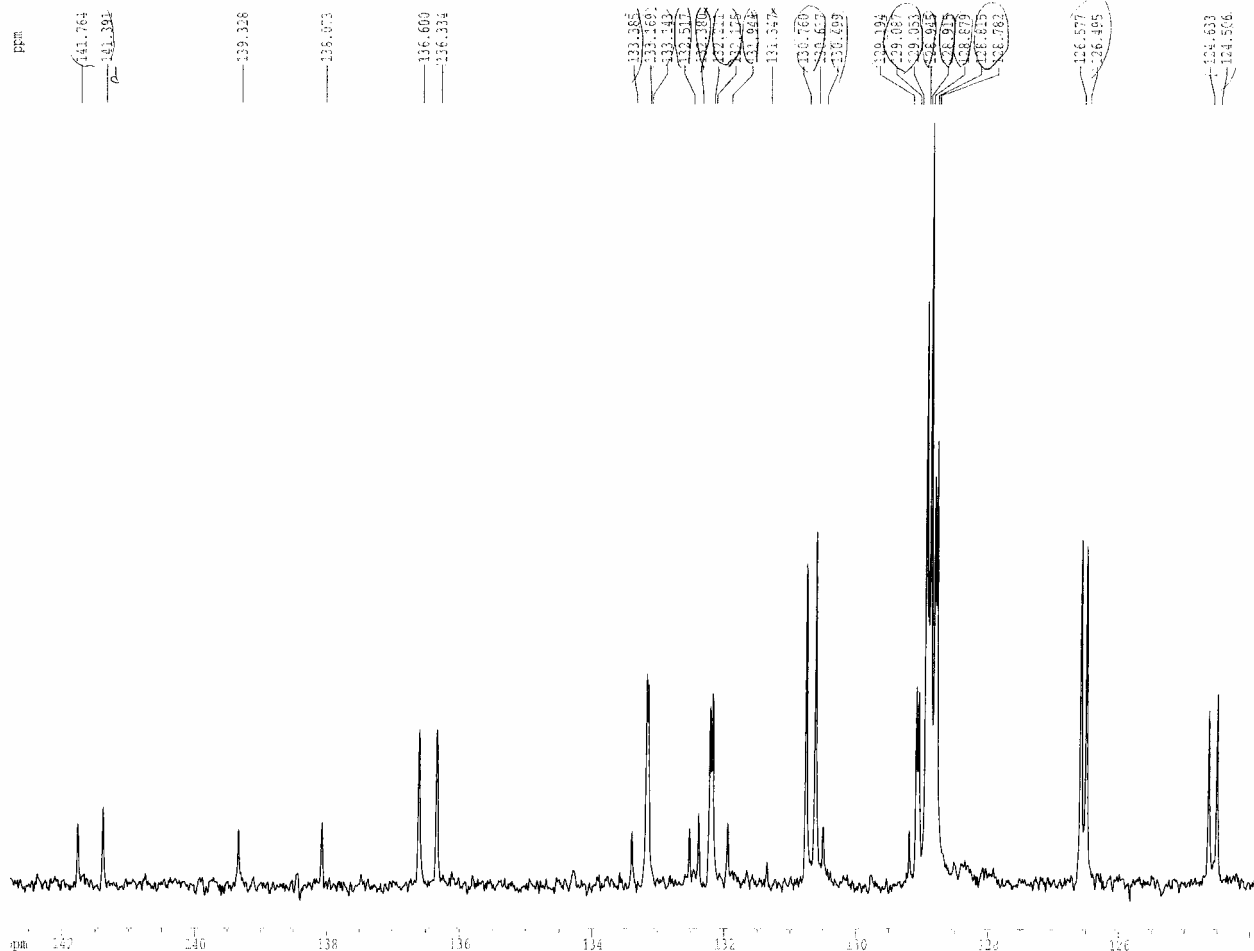


2a





**2a**



Current Data Parameters  
 NAME shiraiishi  
 EXPNO 705252  
 PROCNO 1

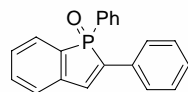
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 PROBHD 5 mm QNP 1H  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 498  
 DS 2  
 SSB 23809.523 Hz  
 FIDRES 0.363304 Hz  
 AQ 1.3763061 sec  
 RG 2048  
 LW 21.000 usec  
 DE 4.50 usec  
 TE 300.0 K  
 D12 0.00002000 sec  
 PL13 16.00 dB  
 D1 1.00000000 sec  
 CPDPRG2 waltz16  
 PCPD2 100.00 usec  
 SFO2 300.1312005 MHz  
 NUC2 1H  
 PL2 -6.00 dB  
 PL12 16.00 dB  
 P1 5.70 usec  
 DE 4.50 usec  
 SFO1 75.4772501 MHz  
 NUC1 13C  
 PL1 -6.00 dB  
 D11 0.03000000 sec

F2 - Processing parameters  
 SI 32768  
 SF 75.4677543 MHz  
 VDE EH  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 EC 1.40

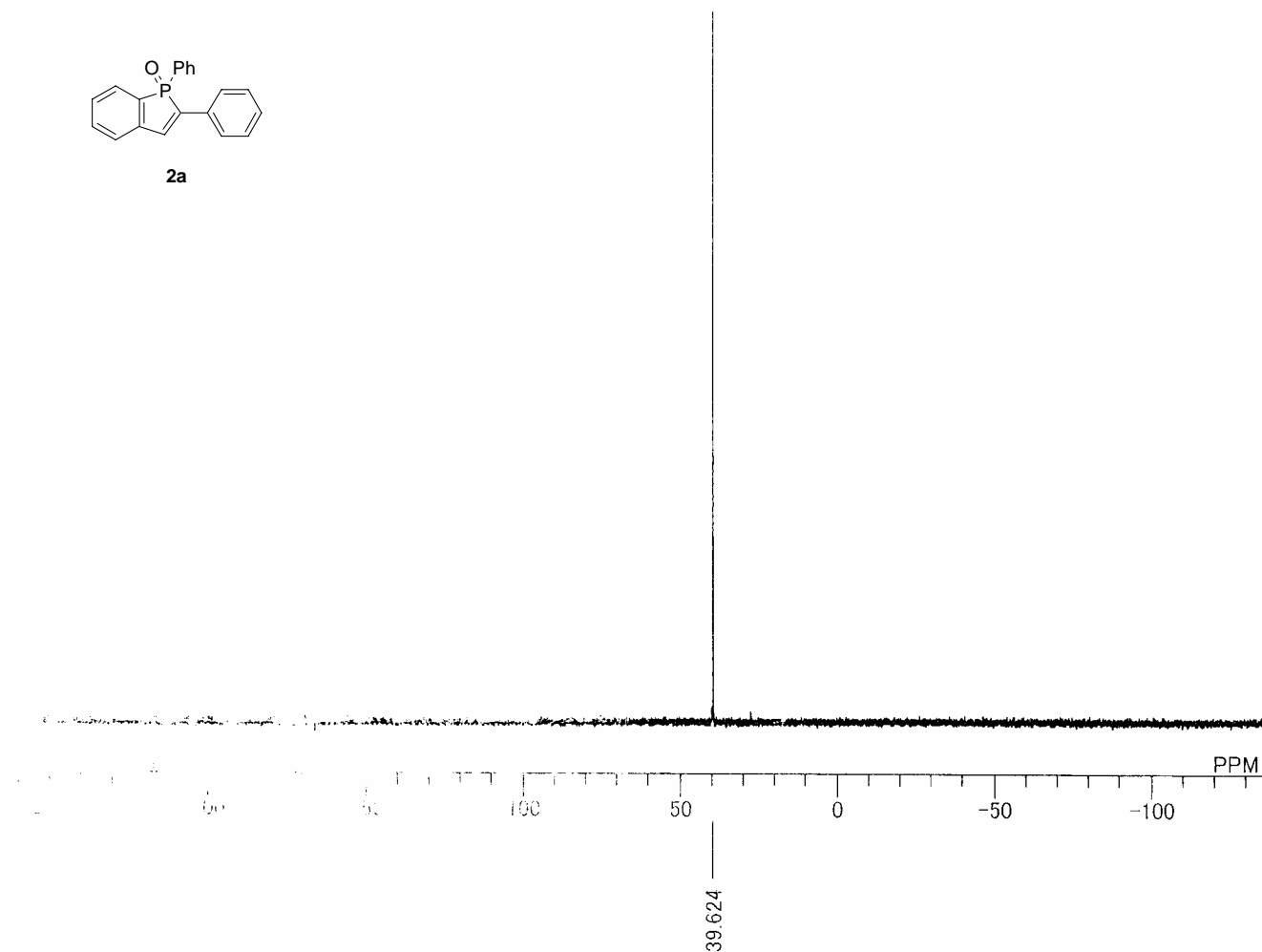
1D NMR plot parameters  
 CV 30.00 cm  
 F1P 142.866 ppm  
 F1 10781.79 Hz  
 F2P 123.797 ppm  
 F2 9322.65 Hz  
 PPGPH 0.63266 ppm/cm  
 HZ/CH 47.97147 Hz/cm

<sup>13</sup>C NMR spectrum of **2a** (CDCl<sub>3</sub>)

PO-Ph-705251  
17:09:21



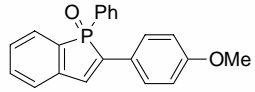
**2a**



201  
D:\WINNMR98\COM  
COMNT PO-Ph-705251  
DATIM Fri May 25 17:09:21 2  
OBNUC 31P  
EXMOD BCM  
OBFRQ 161.70 MHz  
OBSET 145.00 KHz  
OBFIN 77.2 Hz  
POINT 32768  
FREQU 64102.5 Hz  
SCANS 4  
ACQTM 0.511 sec  
PD 2.000 sec  
PW1 5.2 us  
IRNUC 1H  
CTEMP 22.3 c  
SLVNT CDCL3  
EXREF 0.00 ppm  
BF 4.20 Hz  
RGAIN 26

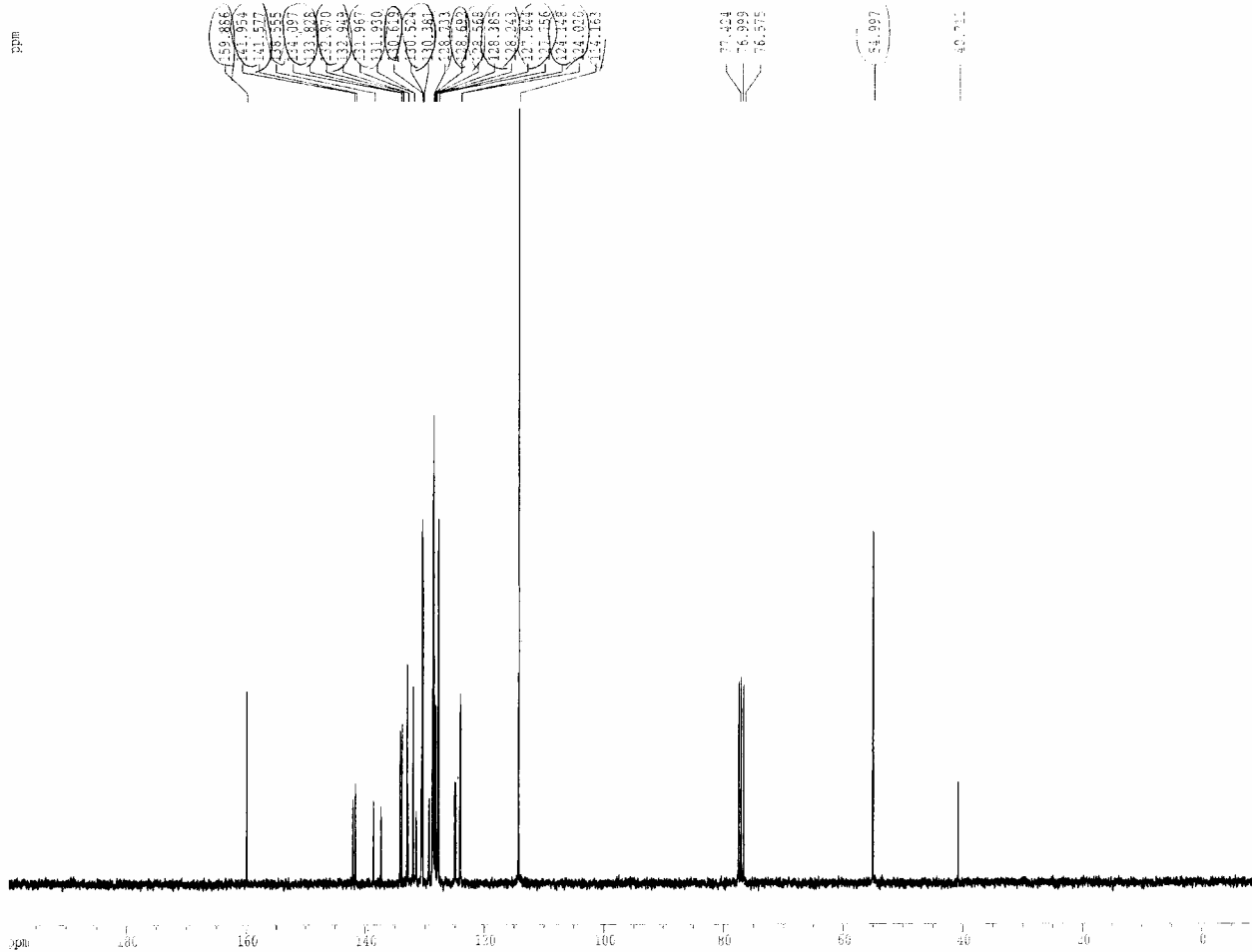
$^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of **2a** ( $\text{CDCl}_3$ )



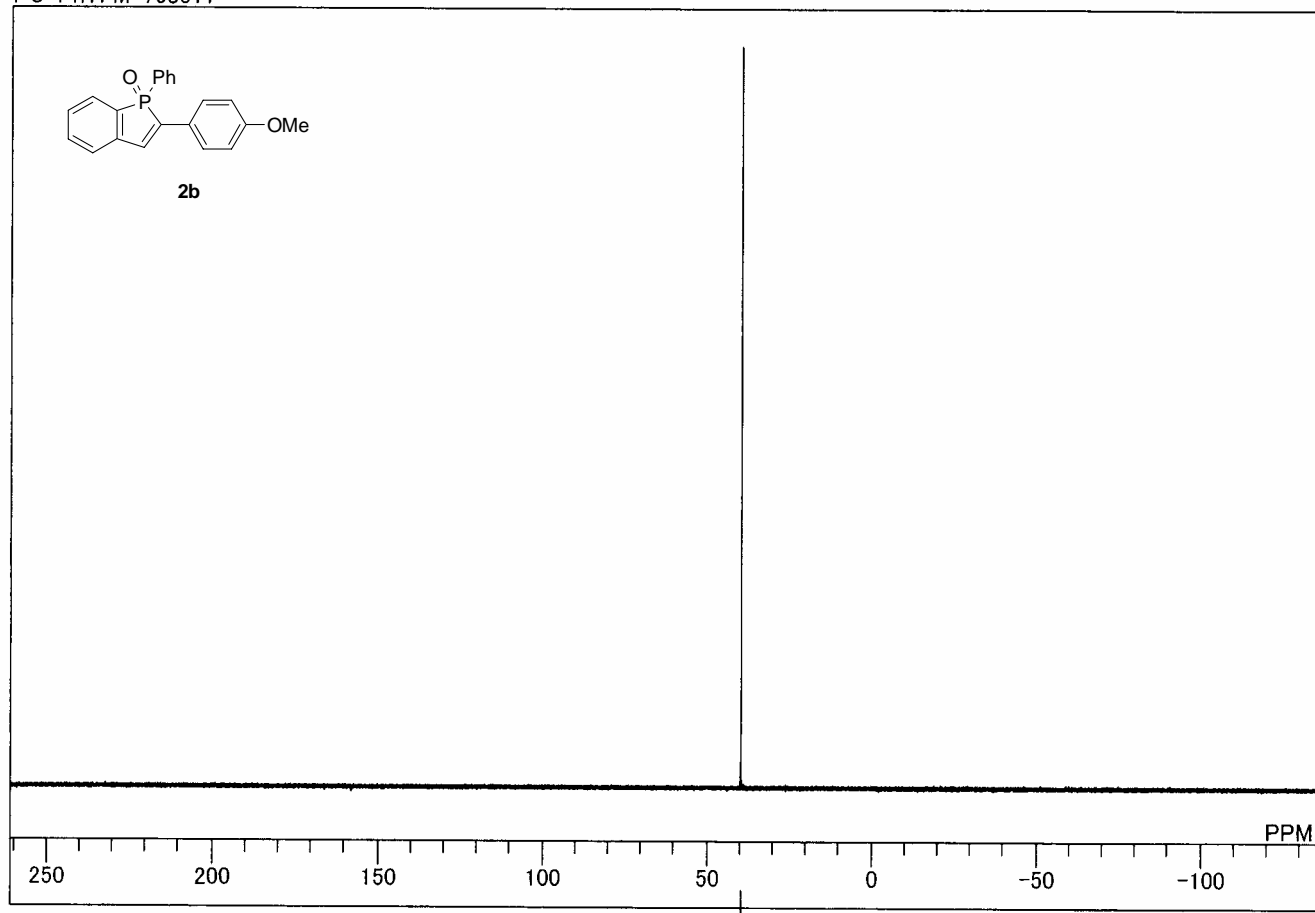


**2b**

Handwritten notes: C1=CC=C(C=C1)C=C2C=CC=C2P(=O)(C1=CC=C(C=C1))C3=CC=C(C=C3)OC  
26



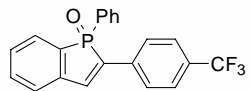
C:\WINNMR98\COMMON\DEFAULT.ALS  
PO-PhTFM-708011



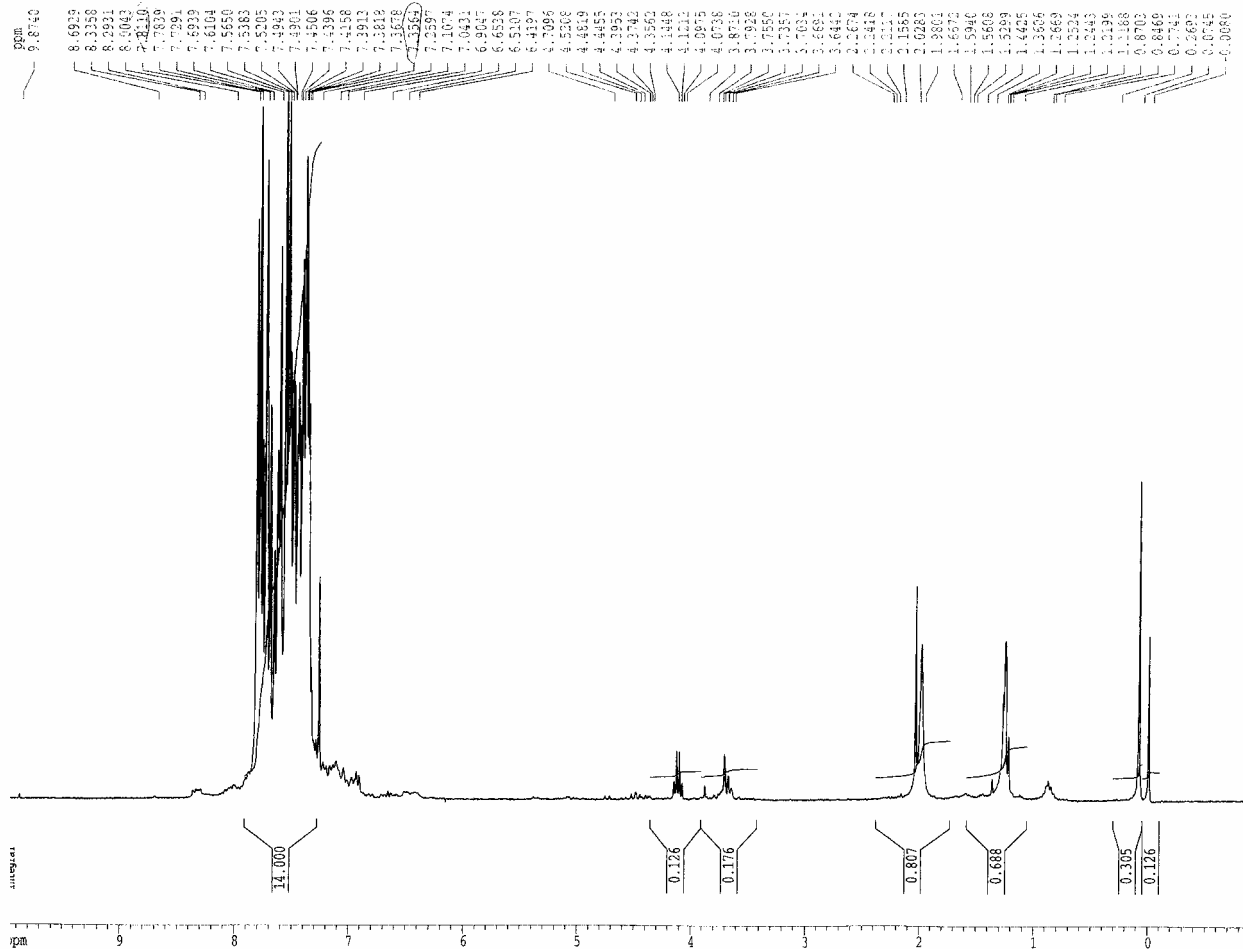
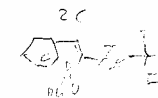
26  
OMe

DFILE	C:\WINNMR98\COMM
COMNT	PO-PhTFM-708011
DATIM	Wed Aug 01 17:50:07
OBNUC	31P
EXMOD	BCM
OBFRQ	161.70 MHz
OBSET	145.00 KHz
OBFIN	77.2 Hz
POINT	32768
FREQU	64102.5 Hz
SCANS	4
ACQTM	0.511 sec
PD	2.000 sec
PW1	5.2 us
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	23

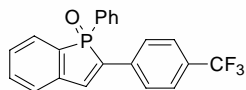
$^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of **2b** ( $\text{CDCl}_3$ )



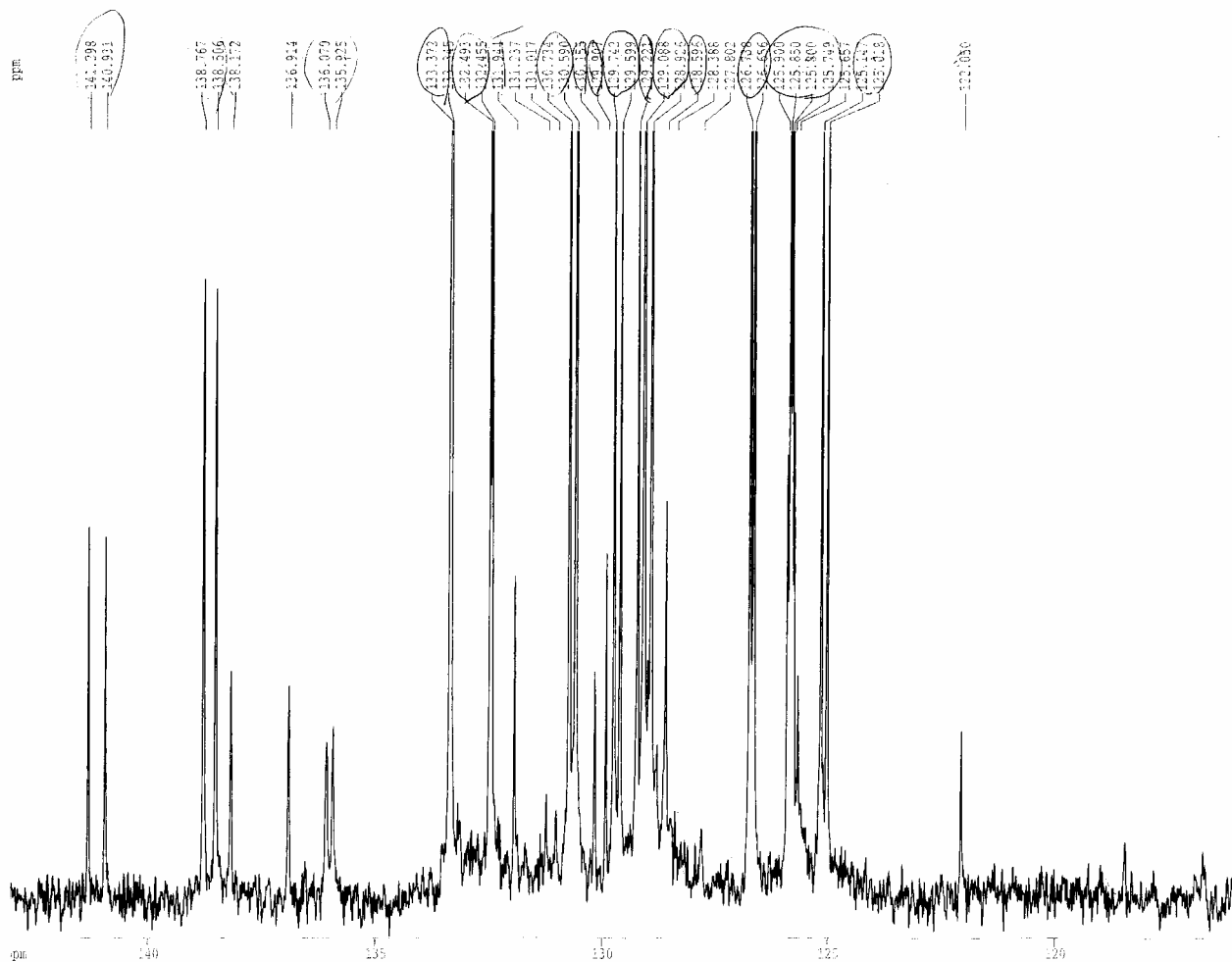
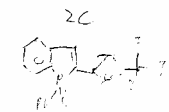
**2c**



$^1\text{H}$  NMR spectrum of **2c** ( $\text{CDCl}_3$ )



2c



```

Current Data Parameters
NAME      shiraishi
EXPNO    708042
PROCNO    1

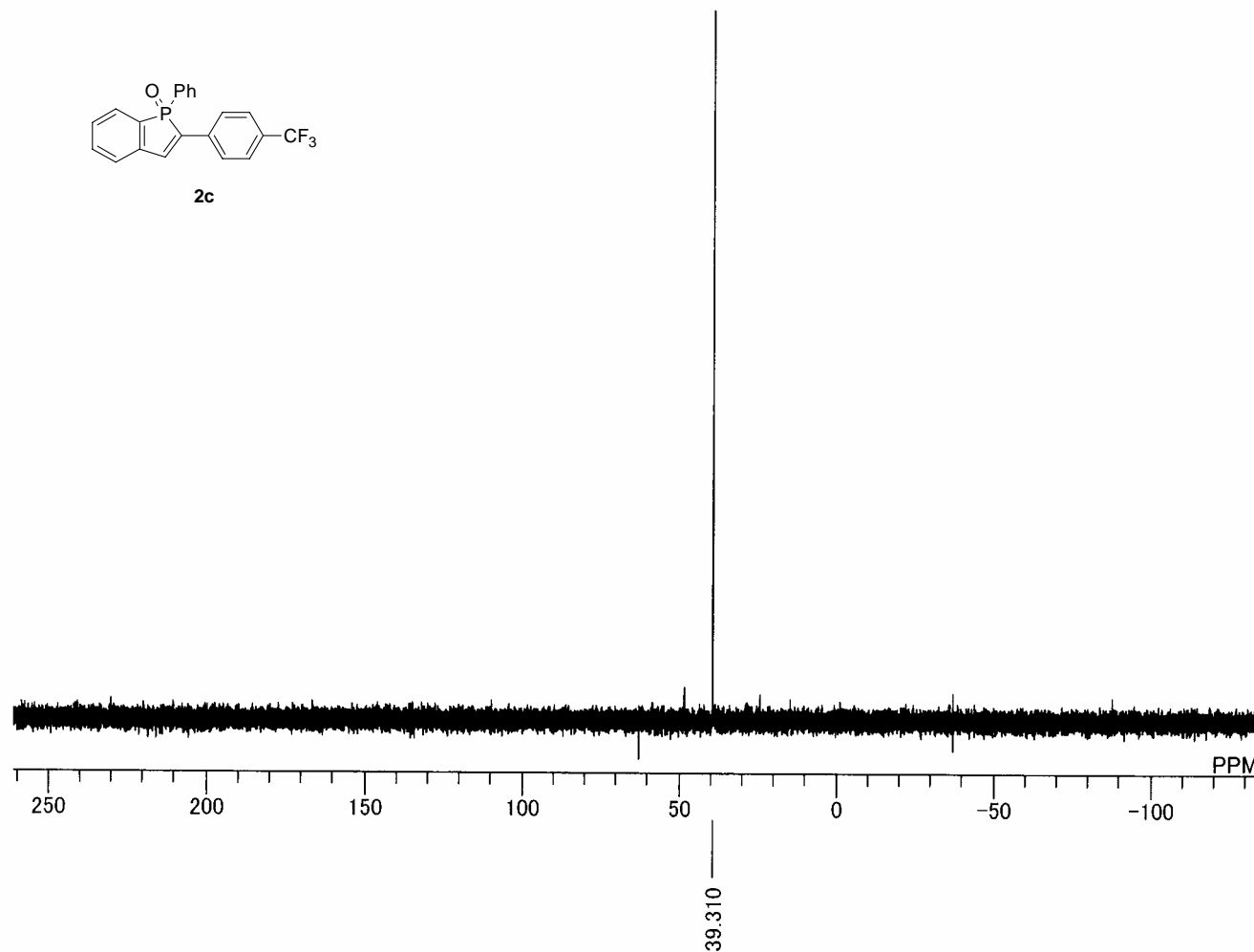
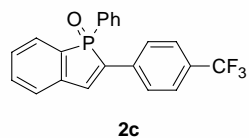
F2 - Acquisition Parameters
Date_     500000
Time      15.17
INSTRUM   dpx300
PROBHD    5 mm QNP 1H
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        1029
DS        2
SFO1      24809.524 Hz
FIDRES    0.363304 Hz
AQ        1.3763061 sec
RG        8192
EX        21.000 usec
DE        4.50 usec
TE        300.0 K
D12       0.00002000 sec
PL13      16.00 dB
D1        1.00000000 sec
CPDPRG2   waltz16
PCPD2     100.00 nsec
SFO2      300.1312005 MHz
NUC2      13C
PL2       -6.00 dB
PL12      16.00 dB
P1        5.70 usec
DE        4.50 usec
SFO1      75.4772501 MHz
NUC1      13C
PL1       -6.00 dB
D11       0.03000000 sec

F2 - Processing parameters
SI        32768
SF        75.4677530 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40

1D NMR plot parameters
CX        30.00 cm
FIP       143.117 ppm
F1        10800.71 Hz
P2P       215.951 ppm
F2        8750.54 Hz
PPM2CH    0.90554 ppm/cm
HZCH      68.53914 Hz/cm
  
```

<sup>13</sup>C NMR spectrum of 2c (CDCl<sub>3</sub>)

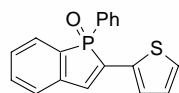
C:\WINNMR98\COMMON\DEFAULT.ALS  
PO-PhTFM-708041



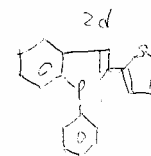
2 c

DFILE	C:\WINNMR98\COMM
COMNT	PO-PhTFM-708041
DATIM	Sat Aug 04 15:16:32 2
OBNUC	31P
EXMOD	BCM
OBFRQ	161.70 MHz
OBSET	145.00 KHz
OBFIN	77.2 Hz
POINT	32768
FREQU	64102.5 Hz
SCANS	11
ACQTM	0.511 sec
PD	2.000 sec
PW1	5.2 us
IRNUC	1H
CTEMP	22.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	26

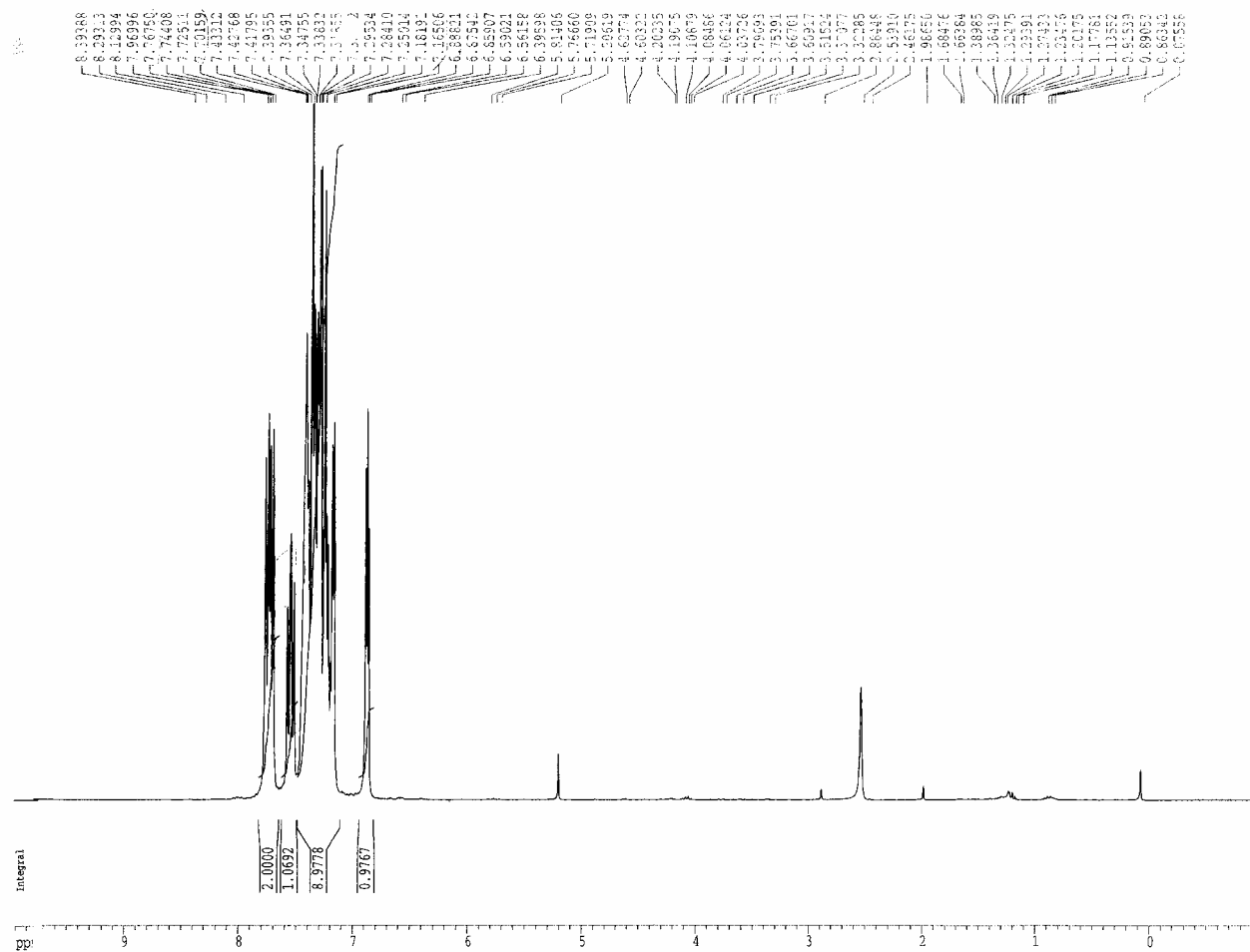
$^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of **2c** ( $\text{CDCl}_3$ )



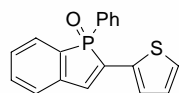
**2d**



2d



$^1\text{H}$  NMR spectrum of **2d** ( $\text{CDCl}_3$ )

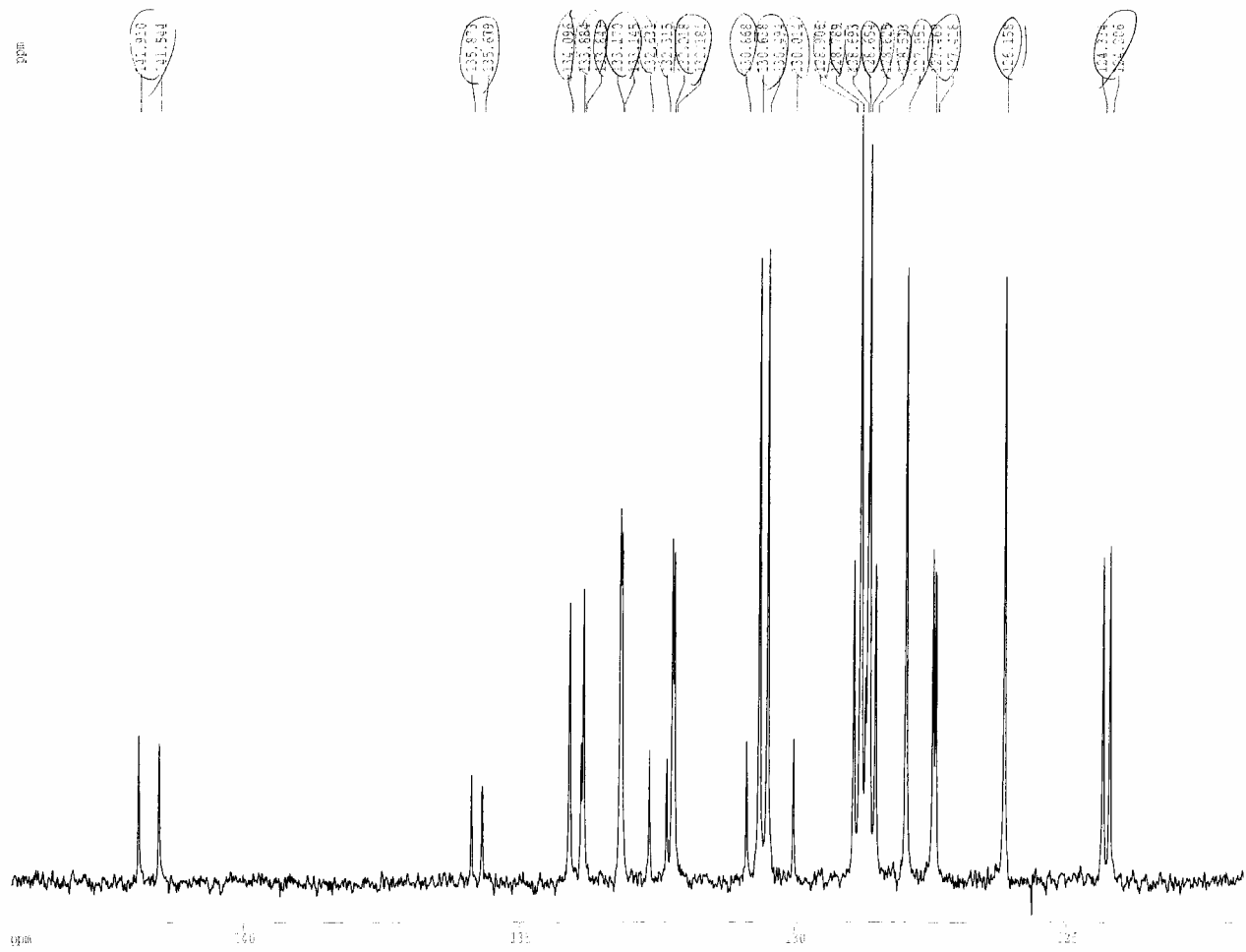


**2d**



2d

71



```

Current Data Parameters
NAME      shiraishi
EXPER    707103
PROCNO    1

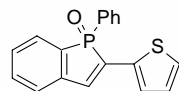
F2 Acquisition Parameters
Date_     500000
Time      16.16
INSTRUM   spect
PROBHD    5 mm QNP 1H
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        209
DS        2
SWH       23809.523 Hz
FIDRES    0.363304 Hz
AQ        1.3763061 sec
RG        8192
LR        23.000 uzer
DE        4.50 usec
TE        300.0 K
D12       0.00002000 sec
PL12      16.00 dB
D1        1.00000000 sec
CDEPRG2   waltz16
PCPD2     100.00 usec
SFO2      300.1312005 MHz
NUC2      13C
PL2       -6.00 dB
FL12      16.00 dB
P1        5.70 usec
DE        4.50 usec
SFO1      75.4772561 MHz
NUC1      13C
PL1       -6.00 dB
D11       0.03000000 sec

F2 - Processing parameters
SI         32768
SF         75.4677683 MHz
MEF        0
SSP        0
LE         1.00 Hz
GB         0
PC         1.40

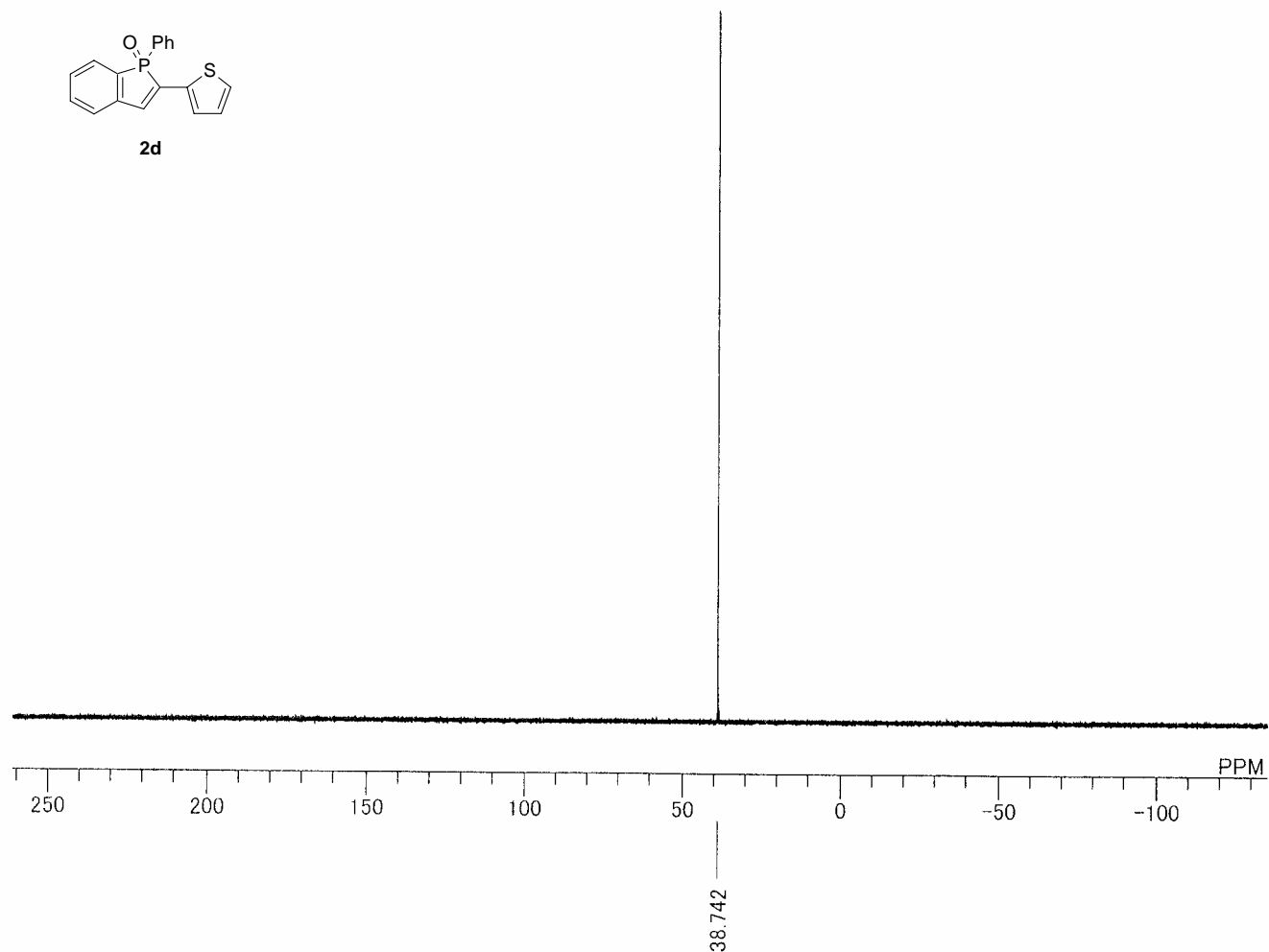
1D NMR plot parameters
CX         30.00 cm
F1P        144.265 ppm
F1         10887.35 Hz
F2P        121.750 ppm
F2         9189.74 Hz
FREQN      1.74981 ppm/cm
HZCN       50.58701 Hz/cm
  
```

<sup>13</sup>C NMR spectrum of **2d** (CDCl<sub>3</sub>)

C:\WINNMR98\COMMON\DEFAULT.ALS  
PO-Thi-707301



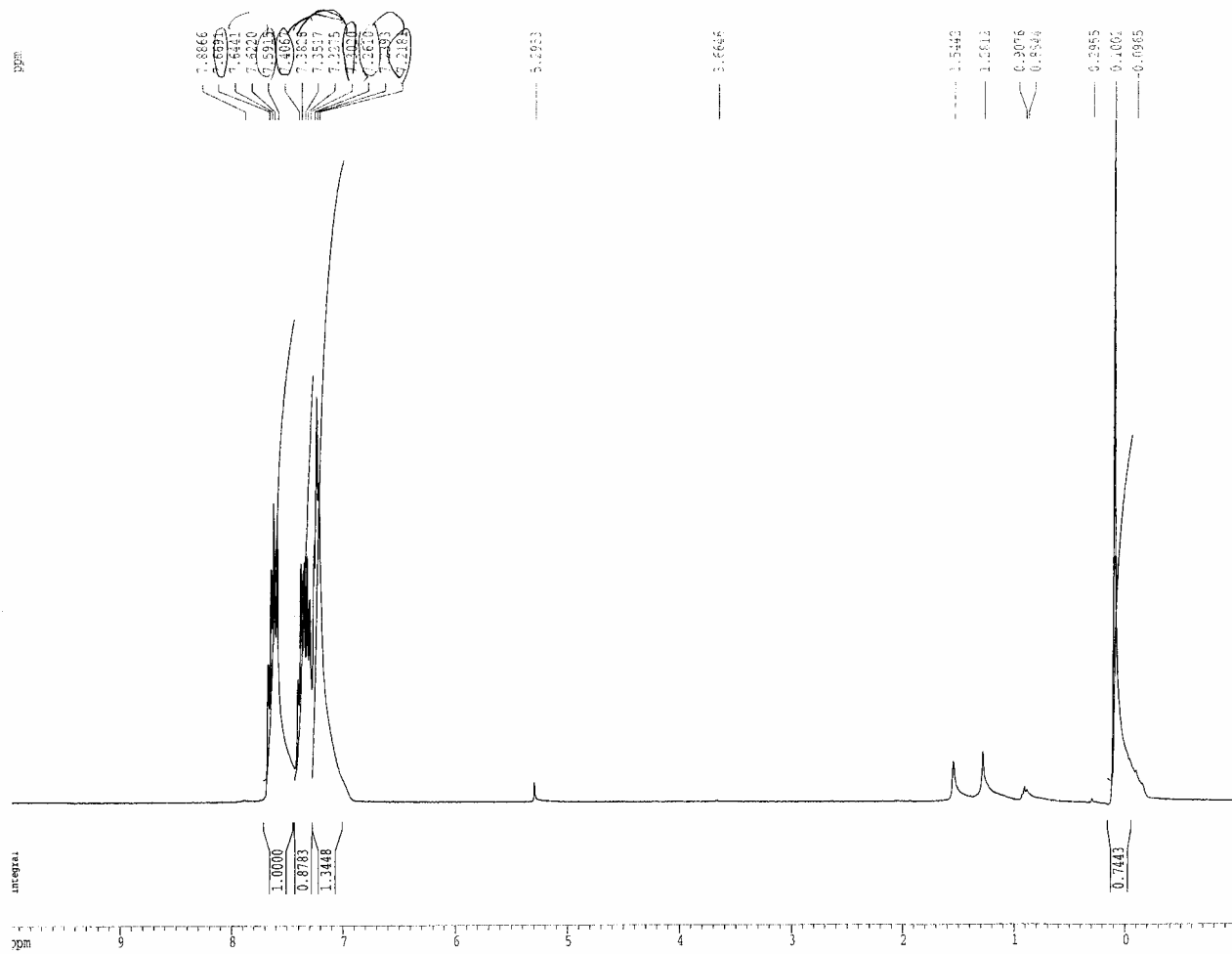
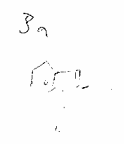
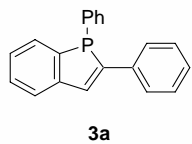
**2d**



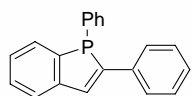
2d

DFILE	C:\WINNMR98\COMM
COMNT	PO-Thi-707301
DATIM	Mon Jul 30 17:48:21 2
OBNUC	31P
EXMOD	BCM
OBFRQ	161.70 MHz
OBSET	145.00 KHz
OBFIN	77.2 Hz
POINT	32768
FREQU	64102.5 Hz
SCANS	4
ACQTM	0.511 sec
PD	1.500 sec
PW1	5.2 us
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	23

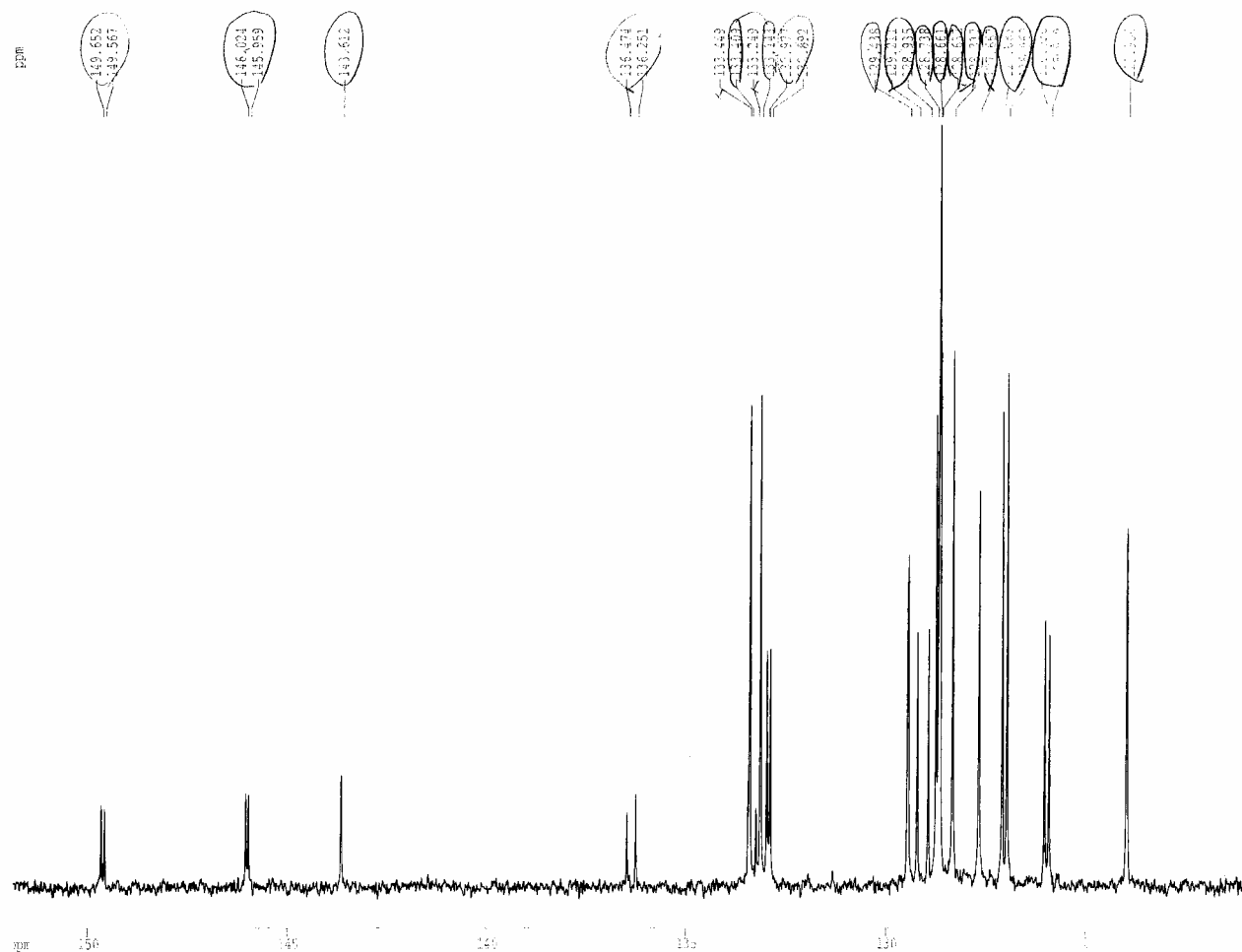
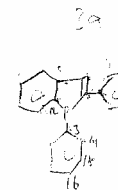
$^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of **2d** ( $\text{CDCl}_3$ )



$^1\text{H}$  NMR spectrum of **3a** ( $\text{CDCl}_3$ )



**3a**



```

Current Data Parameters
NAME      shiraishi
EXPNO    706051
PROCNO    1

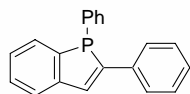
F2 - Acquisition Parameters
Date_     500000
Time      19.11
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PROBHD    5 mm QNP 1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
RG         848
DS         2
SFR        33809.531 Hz
FIDRES    0.363304 Hz
AQ         1.3763061 sec
RG         8192
SI         21.000 usec
DE         4.50 usec
TE        300.0 K
D1         0.00000000 sec
dL13      16.00 dB
d1         1.00000000 sec
CPDPRG2   waltz16
PCPD2     100.00 usec
RF02      300.1312005 MHz
REC2      4H
dL1       -6.00 dB
dL12      16.00 dB
d1         3.70 usec
DE         4.50 usec
SFO1      75.4772501 MHz
NUC1      13C
dL1       -6.00 dB
d1         0.00000000 sec

F2 - Processing parameters
SI         32768
SF         75.4677545 MHz
SFO1      75.4677545 MHz
WDW        EM
SSB        0
B          1.00 Hz
GB         0
PC         1.40

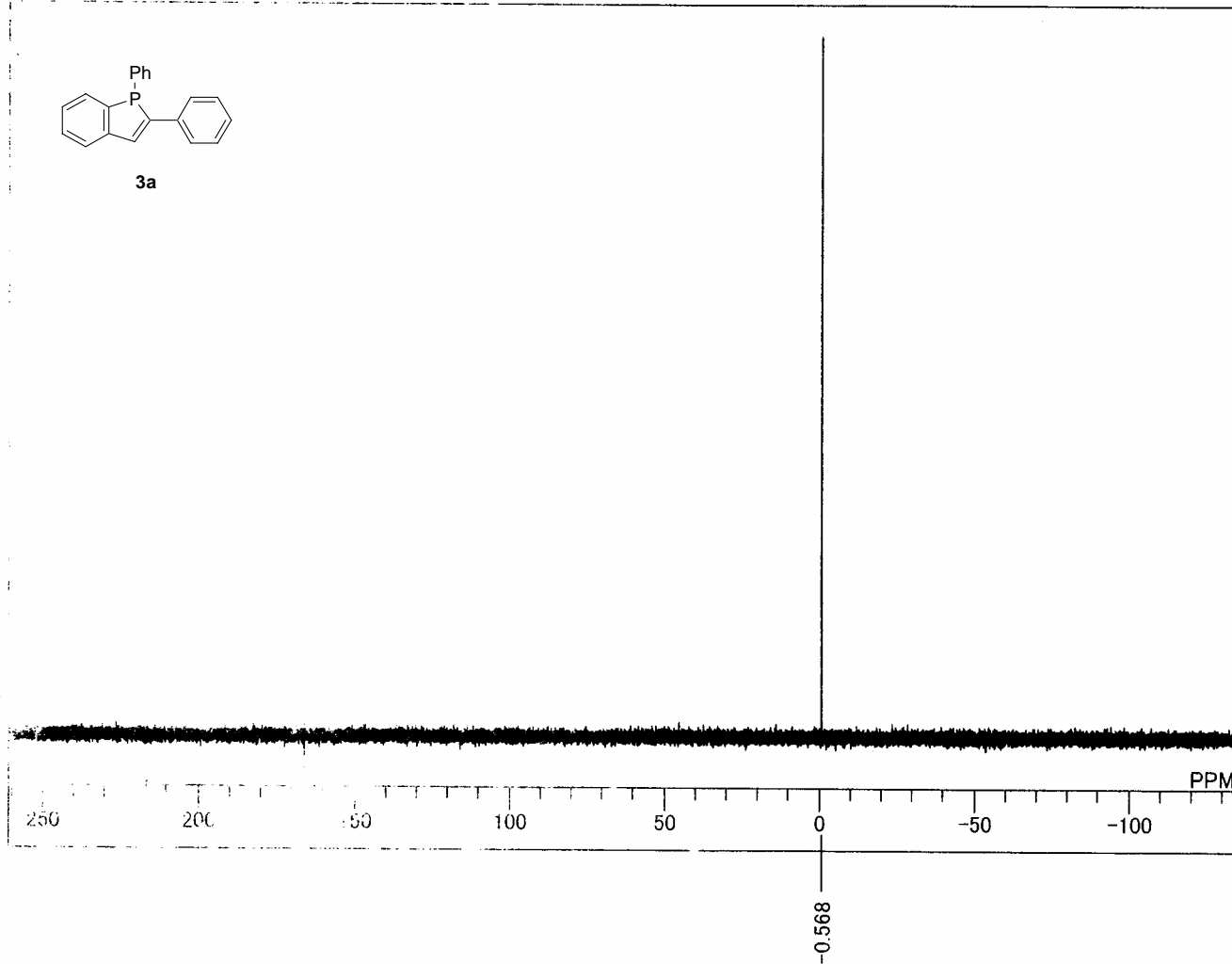
L F2 plot parameters
DE         30.00 cm
CFP        151.968 ppm
d1         12468.69 Hz
CFE        170.938 ppm
CF         9226.92 Hz
d1         1.03430 ppm cm
d1         78.05901 Hz cm
  
```

<sup>13</sup>C NMR spectrum of **3a** (CDCl<sub>3</sub>)

C:\WINNMR98\COM1\_DEFAULT.ALS  
P-Ph-705301



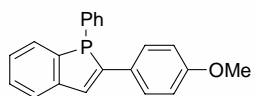
3a



3a

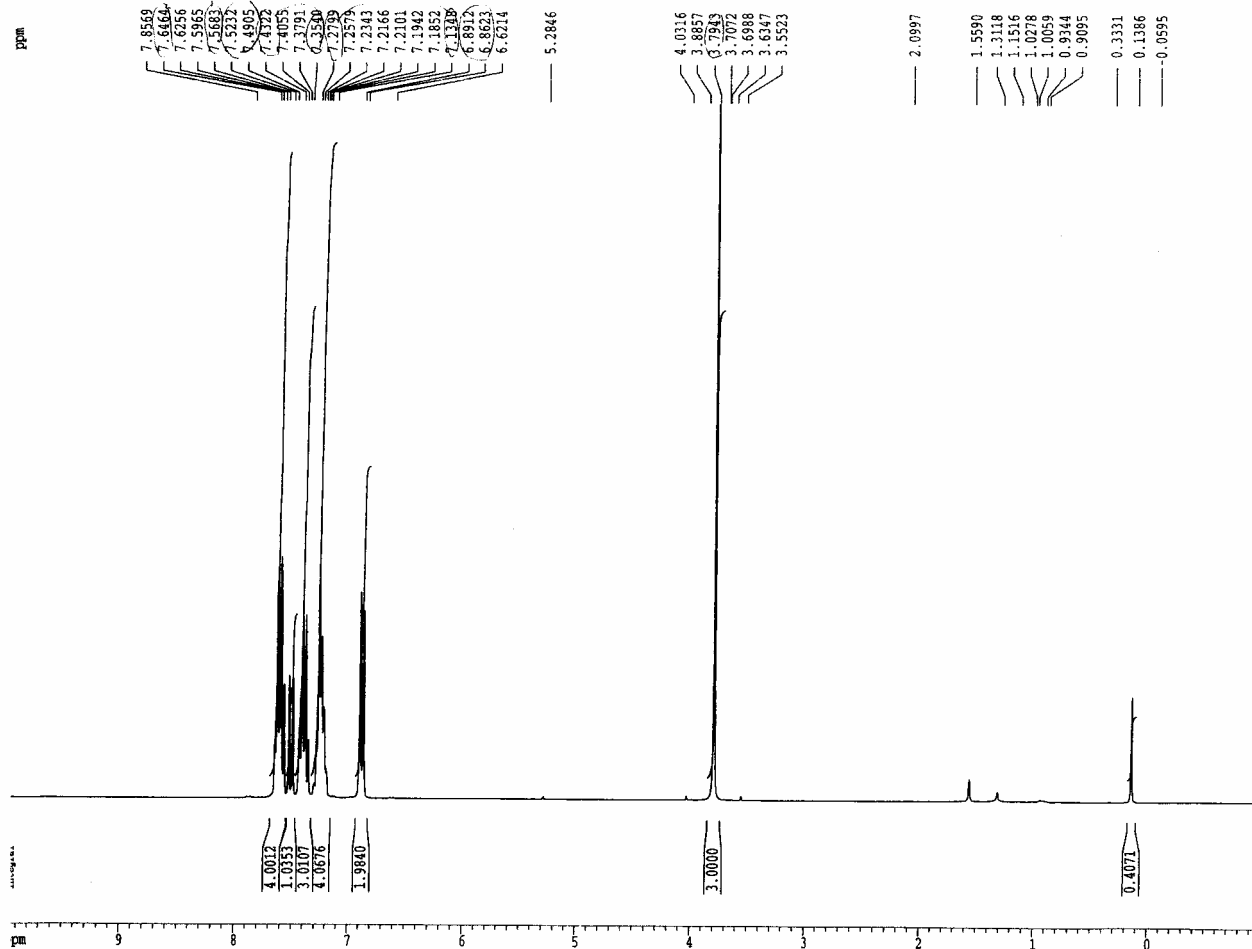
DFILE	C:\WINNMR98\COM1
COMNT	P-Ph-705301
DATIM	Wed May 30 13:06:46
OBNUC	31P
EXMOD	BCM
OBFRQ	161.70 MHz
OBSET	145.00 KHz
OBFIN	77.2 Hz
POINT	32768
FREQU	64102.5 Hz
SCANS	4
ACQTM	0.511 sec
PD	2.000 sec
PW1	5.2 us
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	26

$^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of **3a** ( $\text{CDCl}_3$ )



**3b**

56 P-Ph & Me



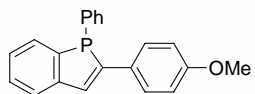
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=====

=====

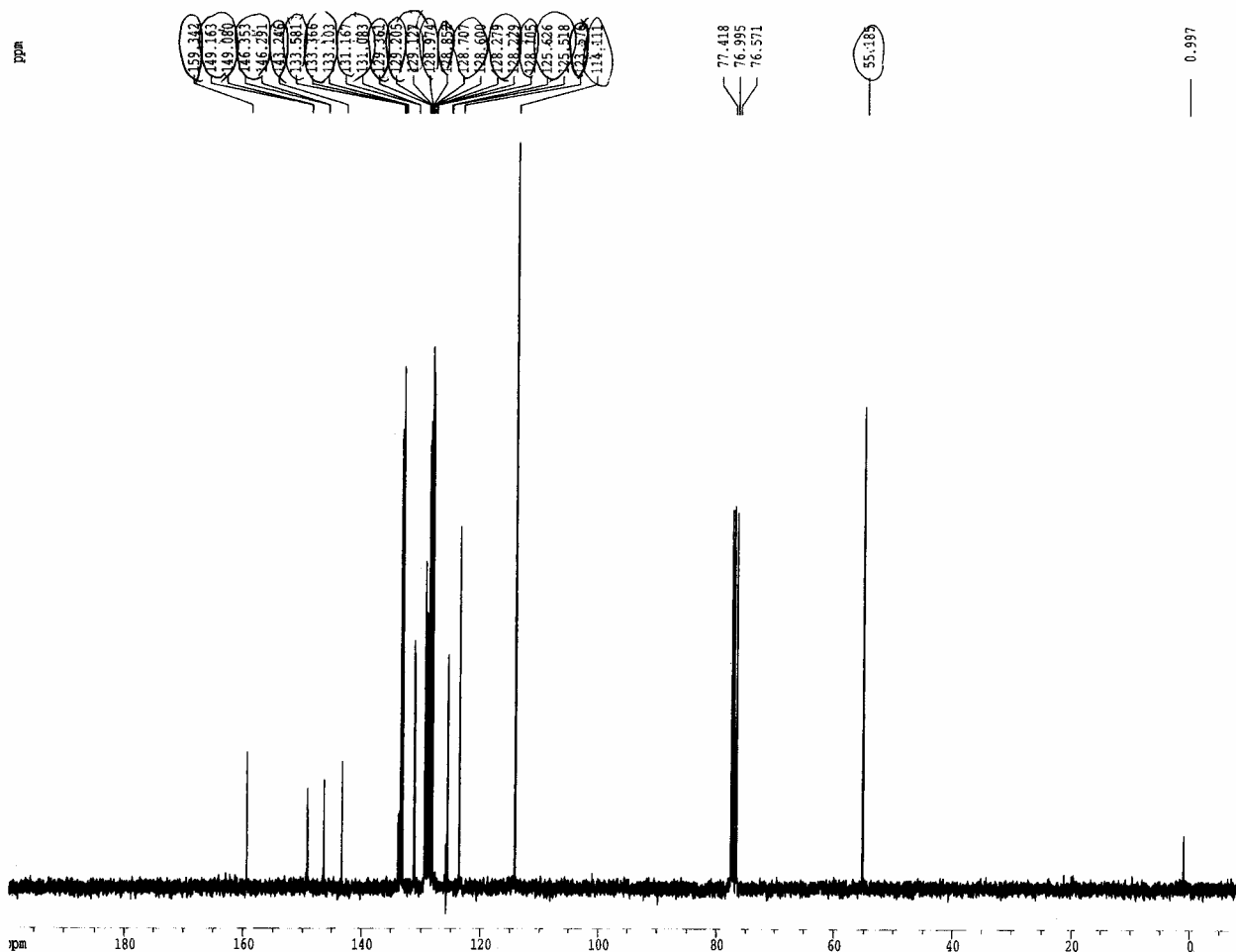
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<sup>1</sup>H NMR spectrum of **3b** (CDCl<sub>3</sub>)



**3b**

*sb*  
P-PhOMe



```

Current Data Parameters
NAME      shiraishi
EXPNO    708212
PROCNO   1

F2 - Acquisition Parameters
Date_    500000
Time     10.36
INSTRUM  dpx300
PROBHD   5 mm QNP 1H
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       310
DS       2
SWH      23809.523 Hz
FIDRES   0.363304 Hz
AQ       1.3763061 sec
RG       8192
DN       21.000 usec
DE       4.50 usec
TE       300.0 K
D12      0.00002000 sec
PL13     16.00 dB
D1       1.00000000 sec
CPDPRG2  waltzi6
PCPD2    100.00 usec
SFO2     300.1312005 MHz
NUC2     1H
PL2      -6.00 dB
PL12     16.00 dB
P1       5.70 usec
DE       4.50 usec
SFO1     75.4772501 MHz
NUC1     13C
PL1      -6.00 dB
D11      0.03000000 sec

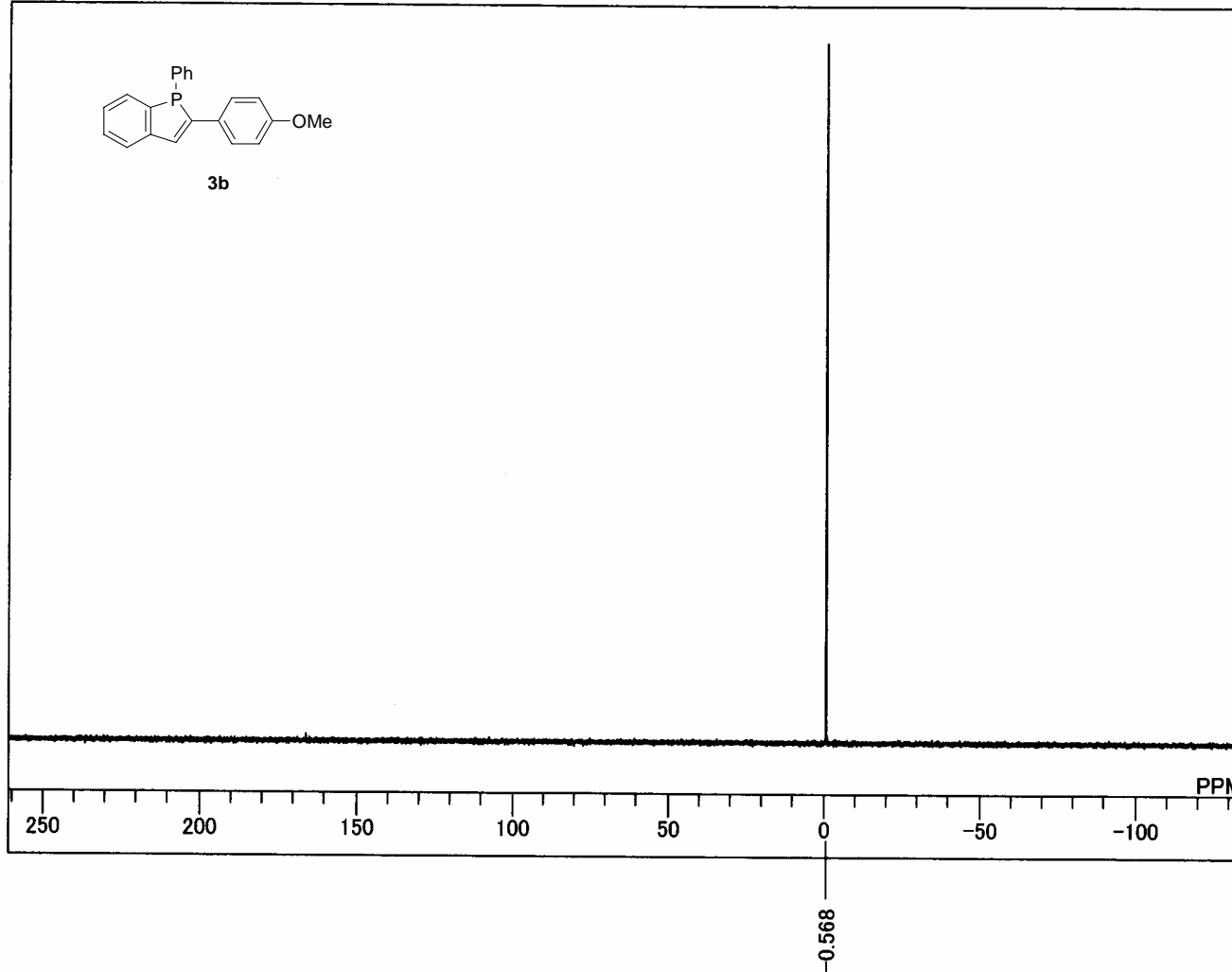
F2 Processing parameters
SI       32768
SF       75.4677566 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40

1D NMR plot parameters
CX       30.00 cm
FLP      200.000 ppm
F1       15093.55 Hz
F2P      -10.000 ppm
F2       -754.68 Hz
PPMCM    7.00000 ppm/cm
HZCM     528.27429 Hz/cm
  
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$^{13}\text{C}$  NMR spectrum of **3b** ( $\text{CDCl}_3$ )

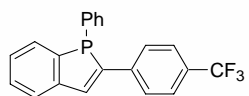
C:\WINNMR98\COMMON\DEFAULT.ALS  
P-PhOMe-708211

3b

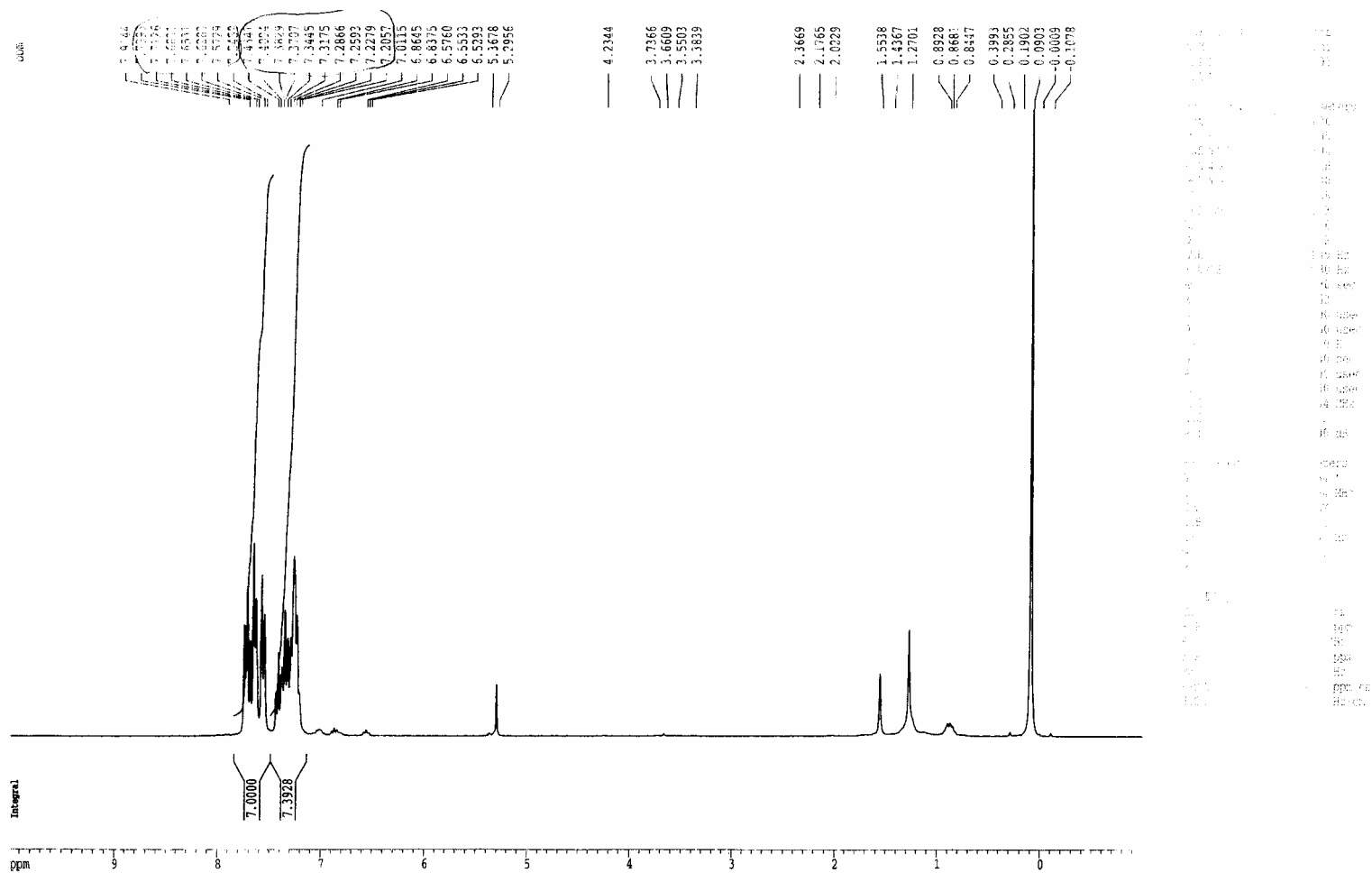
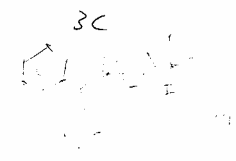


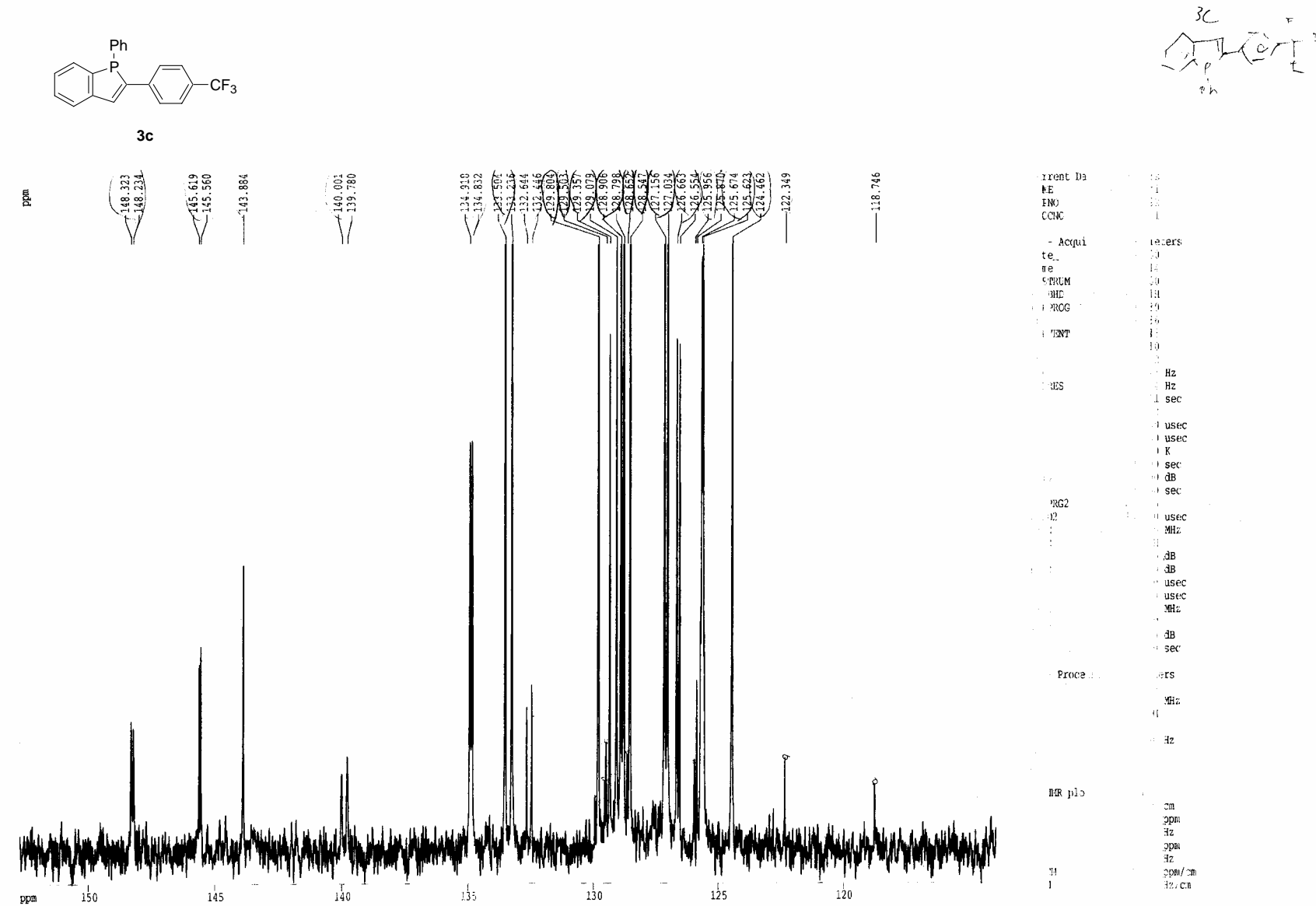
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COMNT P-PhOMe-708211  
DATIM Tue Aug 21 10:41:07  
OBNUC 31P  
EXMOD BCM  
OBFRQ 161.70 MHz  
OBSET 145.00 KHz  
OBFIN 77.2 Hz  
POINT 32768  
FREQU 64102.5 Hz  
SCANS 4  
ACQTM 0.511 sec  
PD 2.000 sec  
PW1 5.2 us  
IRNUC 1H  
CTEMP 23.7 c  
SLVNT CDCL3  
EXREF 0.00 ppm  
BF 4.20 Hz  
RGAIN 25

<sup>31</sup>P{<sup>1</sup>H} NMR spectrum of **3b** (CDCl<sub>3</sub>)



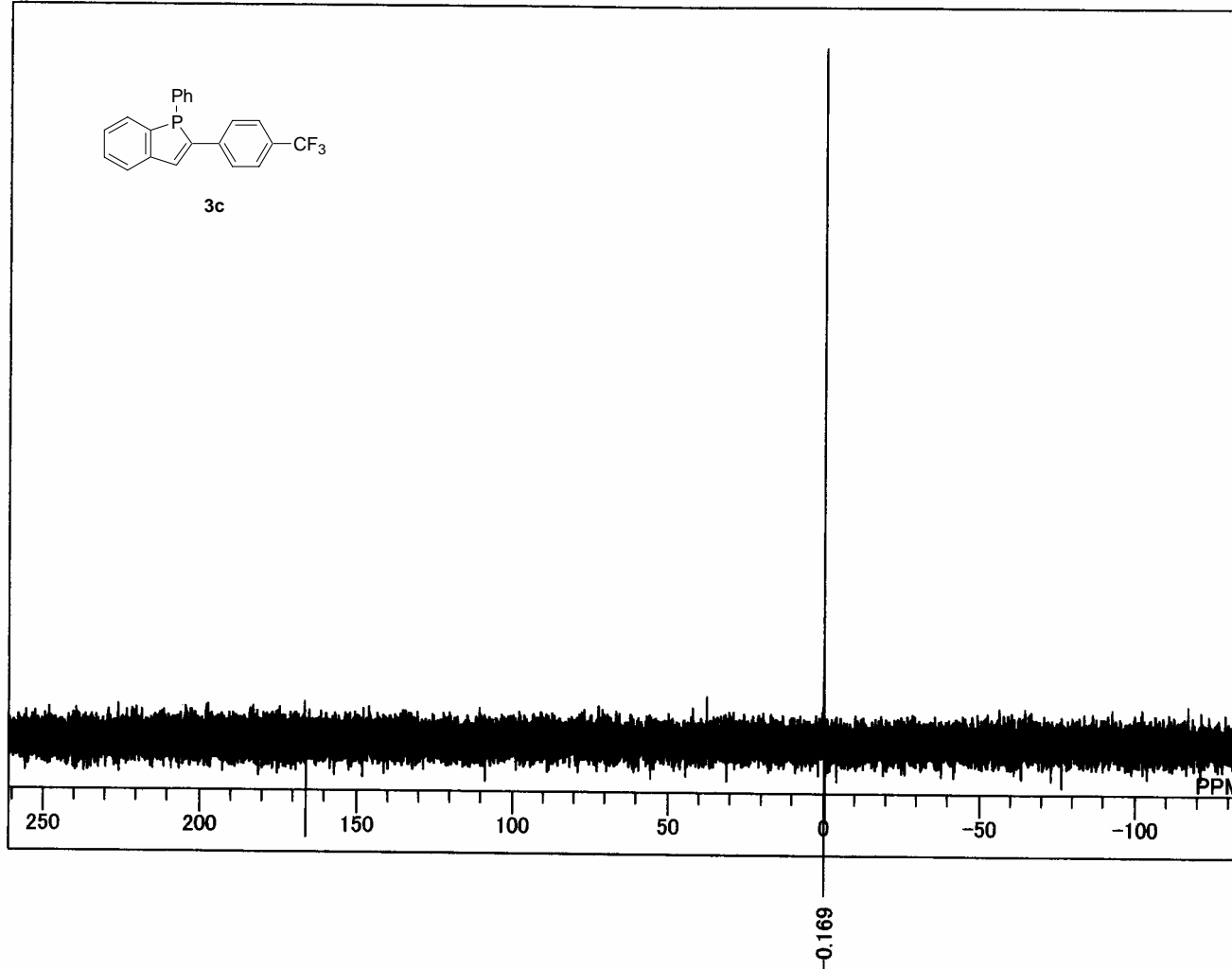
**3c**





<sup>13</sup>C NMR spectrum of **3c** (CDCl<sub>3</sub>)

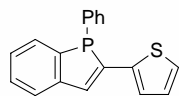
C:\WINNMR98\COMMON\DEFAULT.ALS  
P-TFM-70918



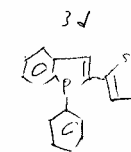
3c

DFILE	C:\WINNMR98\COM
COMNT	P-TFM-70918
DATIM	Tue Sep 18 20:50:40
OBNUC	31P
EXMOD	BCM
OBFRQ	161.70 MHz
OBSET	145.00 KHz
OBFIN	77.2 Hz
POINT	32768
FREQU	64102.5 Hz
SCANS	4
ACQTM	0.511 sec
PD	2.000 sec
PW1	5.2 us
IRNUC	1H
CTEMP	27.8 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	27

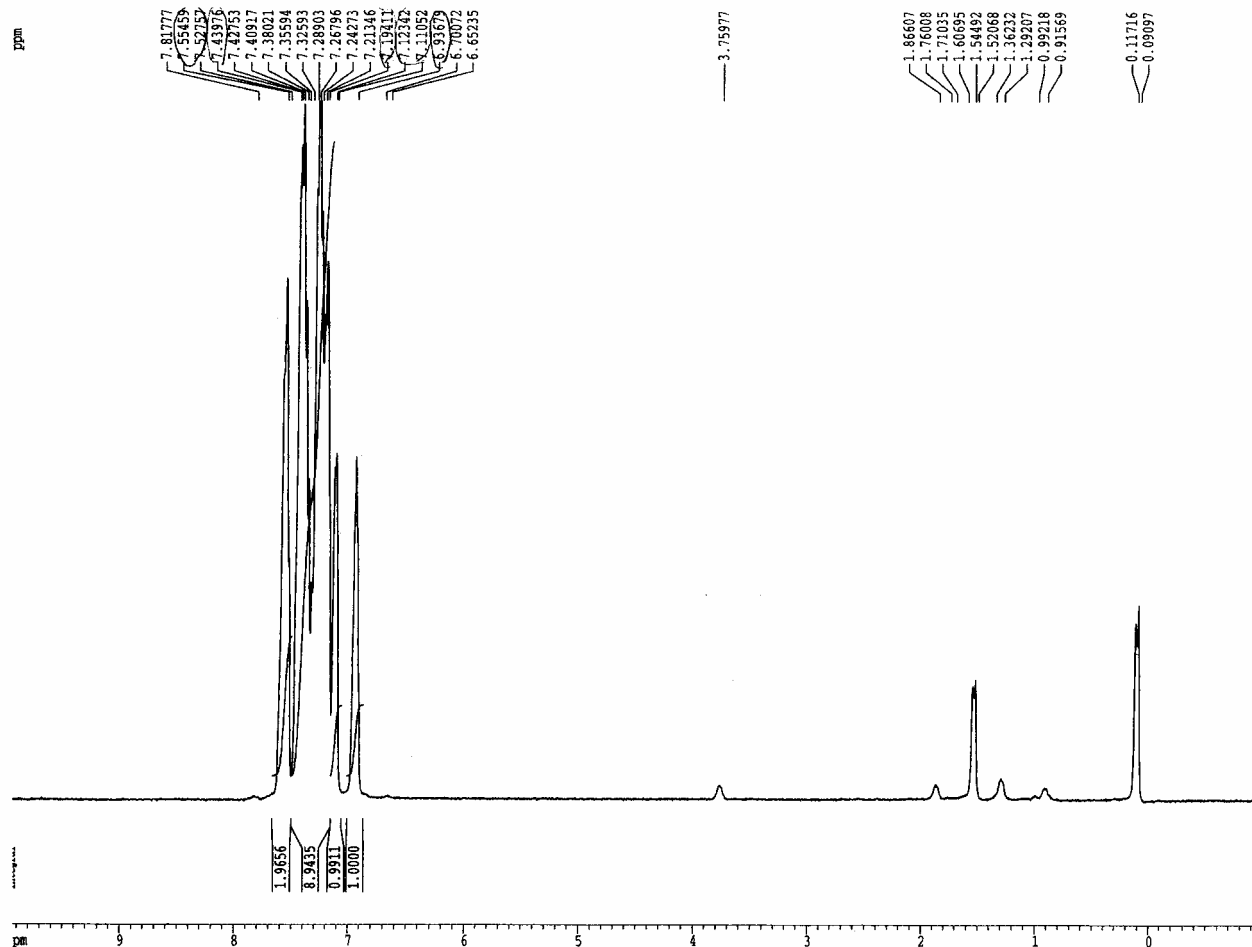
<sup>31</sup>P{<sup>1</sup>H} NMR spectrum of **3c** (CDCl<sub>3</sub>)



3d



131-1



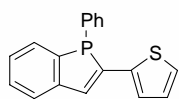
<sup>1</sup>H NMR spectrum of 3d (CDCl<sub>3</sub>)

Current Data Parameters  
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 PROCNO: 1

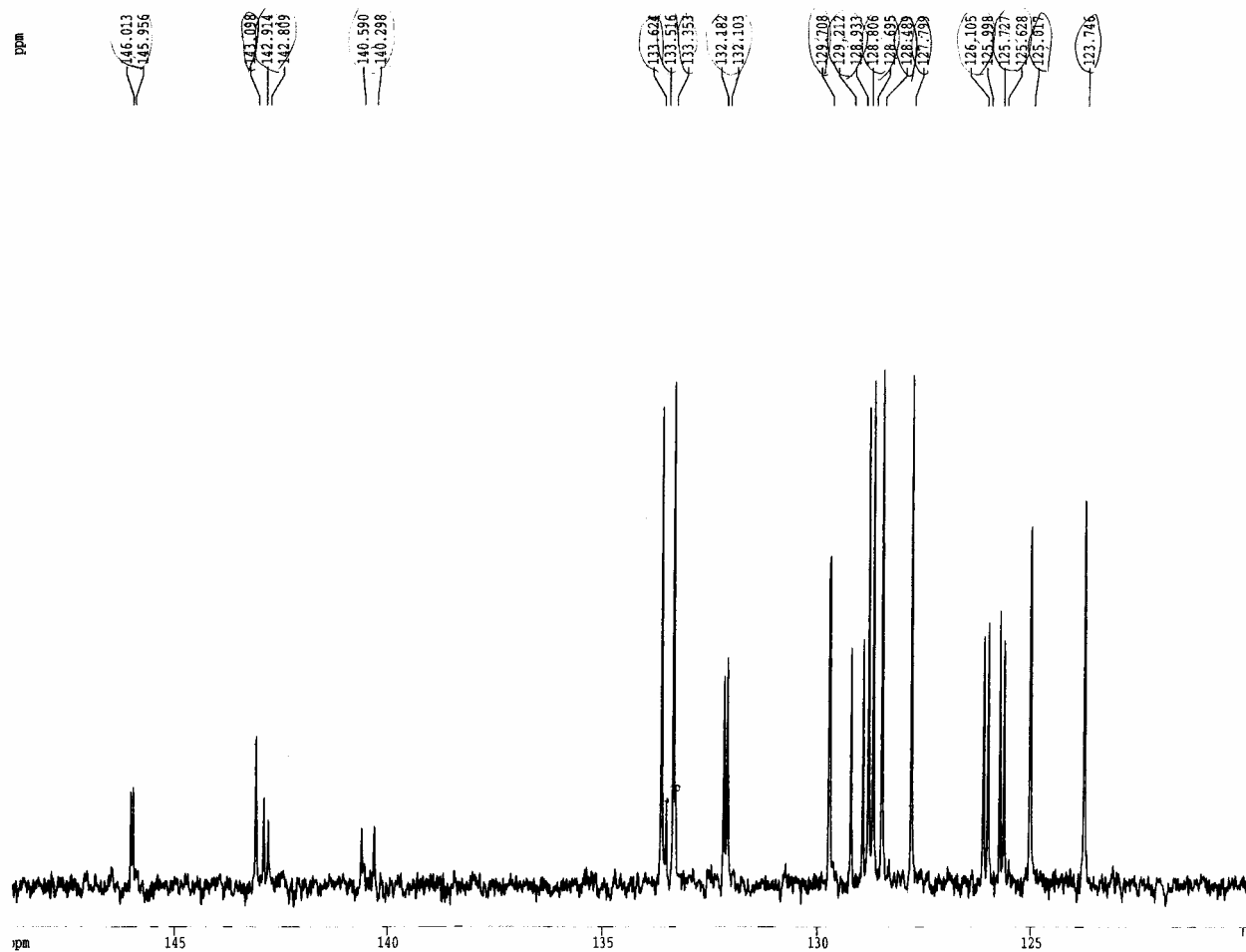
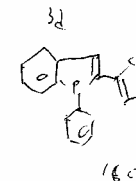
F2 - Acquisition Parameters  
 Date\_: 500000  
 Time: 17.35  
 INSTRUM: spect  
 PROGRAM: 5 wa QNP 1H  
 PULPROG: zgpg30  
 TD: 32768  
 SOLVENT: CDCl3  
 NS: 16  
 DS: 2  
 SSB: 0  
 FIDRES: 0.188386 Hz  
 AQ: 2.6542580 sec  
 RG: 456.1  
 RF: 01.000 MHz  
 DE: 4.50 usec  
 TE: 300.2 K  
 D: 0.00000000 sec  
 FI: 1.00 usec  
 DE: 4.50 usec  
 SF01: 100.1314534 MHz  
 NUC1: 1H  
 PUL1: 0.00 usec

F1 - Processing parameters  
 SI: 32768  
 SF: 100.1300664 MHz  
 NUC: 1H  
 SSB: 0  
 LB: 0.30 Hz  
 GB: 0  
 PC: 1.00

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 CW: 10.00 MHz  
 F1F2: 10.000 MHz  
 F1: 100.130 MHz  
 F2F1: 1.000 MHz  
 F2: 100.13 MHz  
 PRMCM: 0.36667 ppm/cm  
 NUCM: 116.0447 Hz/cm



3d



Current Data Parameters  
 NAME shiraishi  
 EXPNO 708215  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 500000  
 Time 17.40  
 INSTRUM dpx300  
 PROBHD 5 mm QNP 1H  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 346  
 DS 2  
 SWH 23809.523 Hz  
 FIDRES 0.363304 Hz  
 AQ 1.3763061 sec  
 RG 3251  
 DW 21.000 usec  
 DE 4.50 usec  
 TE 300.0 K  
 D12 0.00002000 sec  
 PL13 16.00 dB  
 D1 1.00000000 sec  
 CPDPRG2 waltz16  
 PCPD2 100.00 usec  
 SFO2 300.1312005 MHz  
 NUC2 1H  
 PL2 -6.00 dB  
 PL12 16.00 dB  
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 DE 4.50 usec  
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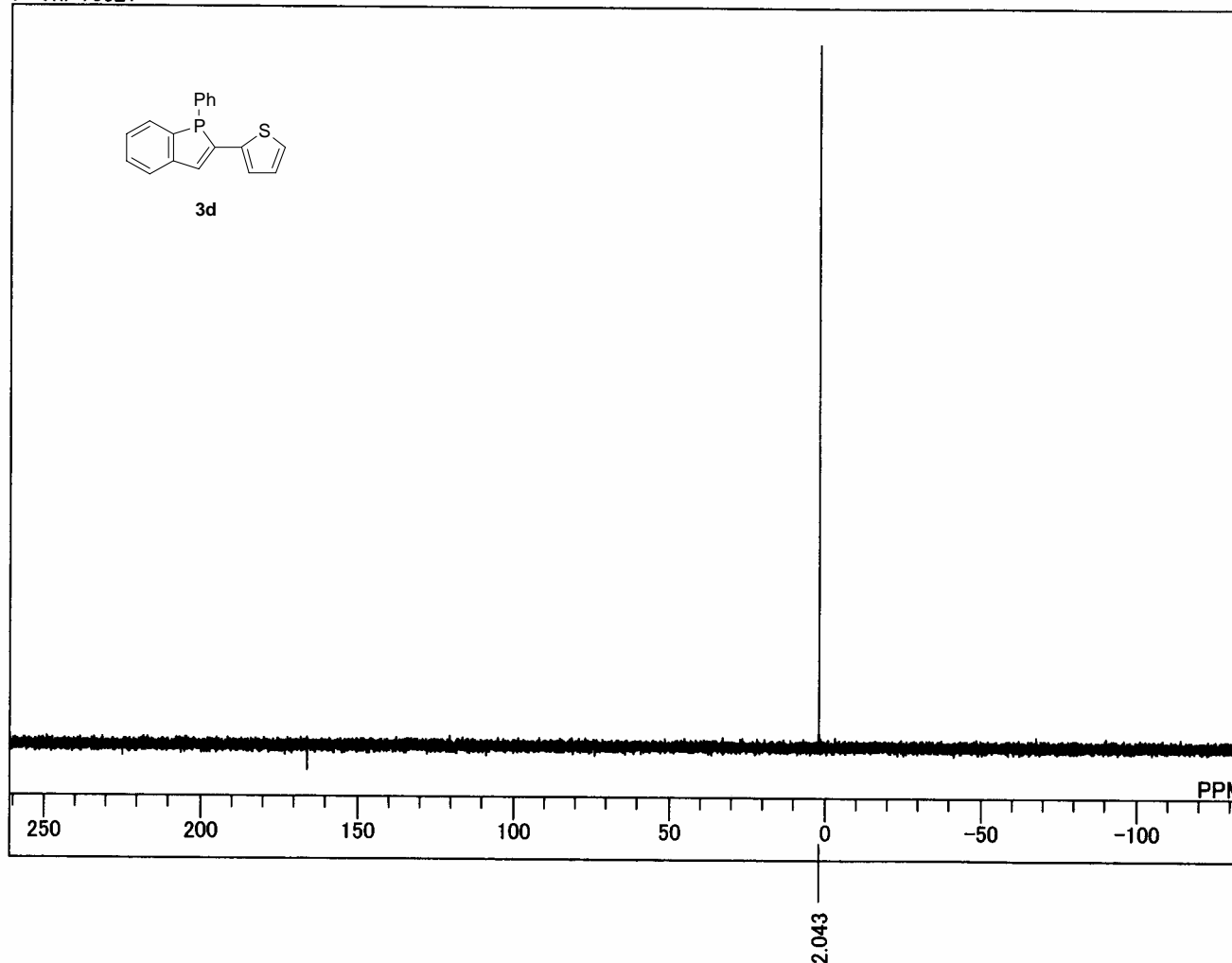
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1D NMR plot parameters  
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 F1 11236.78 Hz  
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 F2 9029.13 Hz  
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 HZCM 73.58835 Hz/cm

<sup>13</sup>C NMR spectrum of 3d (CDCl<sub>3</sub>)

3d

C:\WINNMR98\COMMON\DEFAULT.ALS  
P-Thi-70821

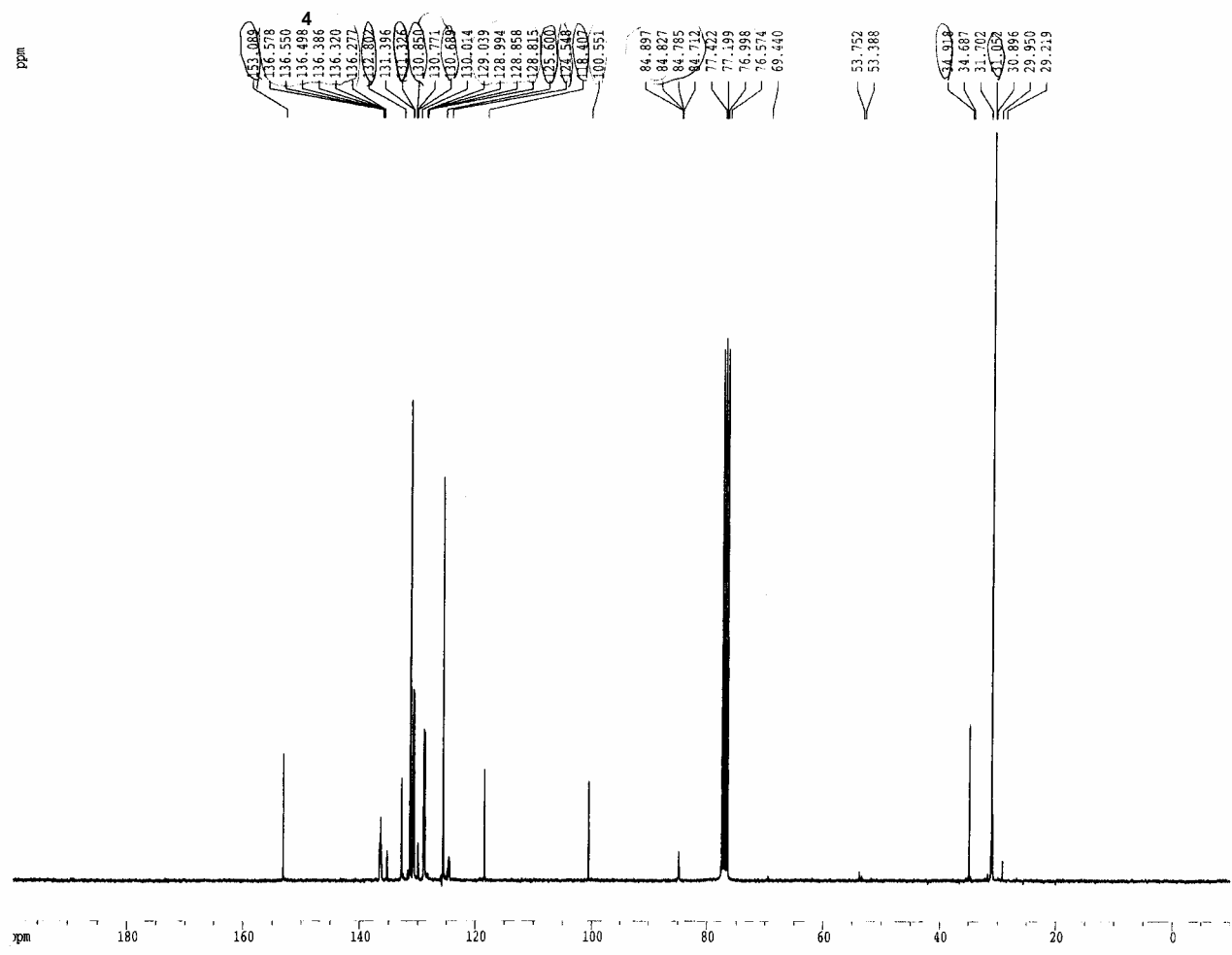
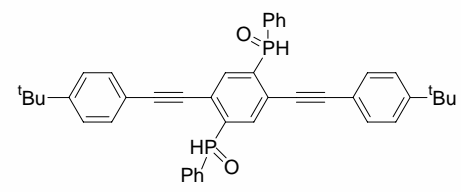


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COMNT P-Thi-70821  
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OBFIN 77.2 Hz  
POINT 32768  
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SCANS 4  
ACQTM 0.511 sec  
PD 2.000 sec  
PW1 5.2 us  
IRNUC 1H  
CTEMP 24.3 c  
SLVNT CDCL3  
EXREF 0.00 ppm  
BF 1.00 Hz  
RGAIN 26

$^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of **3d** ( $\text{CDCl}_3$ )



2 Ph-O-Ph<sub>2</sub> C<sub>6</sub>H<sub>4</sub>



Current Data Parameters  
NAME shiraishi  
EXPNO 802221  
PROCNO 1

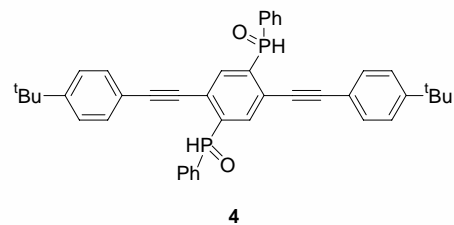
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DS 2  
SWH 23809.523 Hz  
FIDRES 0.363304 Hz  
AQ 1.3763061 sec  
RG 3649.1  
DW 21.000 usec  
DE 4.50 usec  
TE 300.0 K  
D12 0.0002000 sec  
PL13 16.00 dB  
D1 1.0000000 sec  
CPDPRG2 waltz16  
PCPD2 100.00 usec  
SFO2 300.1312005 MHz  
NUC2 1H  
PL2 -6.00 dB  
PL12 16.00 dB  
P1 5.70 usec  
DE 4.50 usec  
SFO1 75.4772501 MHz  
NUC1 13C  
PL1 -6.00 dB  
D11 0.03000000 sec

F2 - Processing parameters  
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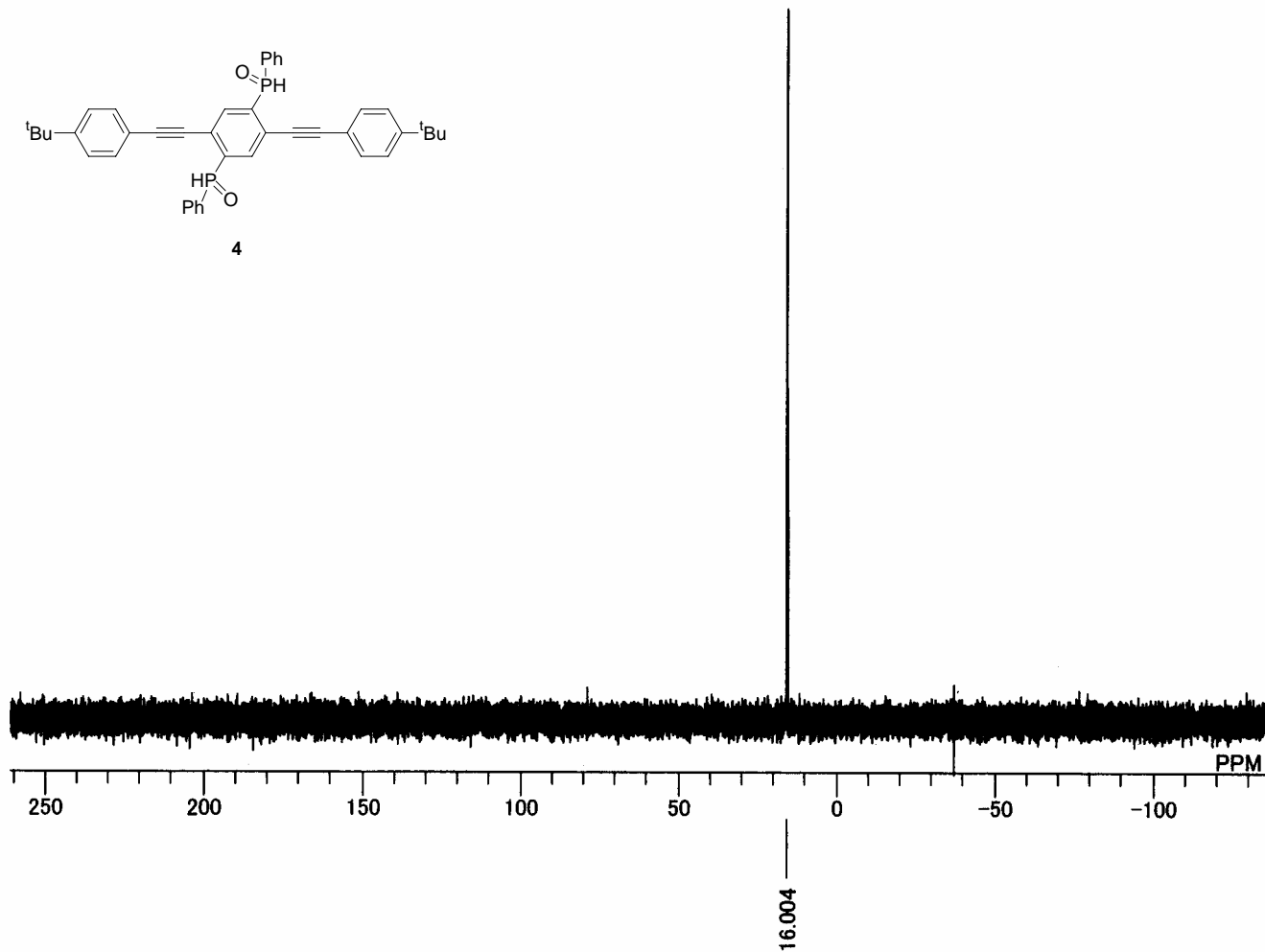
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F1 15093.55 Hz  
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F2 -754.68 Hz  
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HZCM 528.27423 Hz/cm

<sup>13</sup>C NMR Spectrum of 4

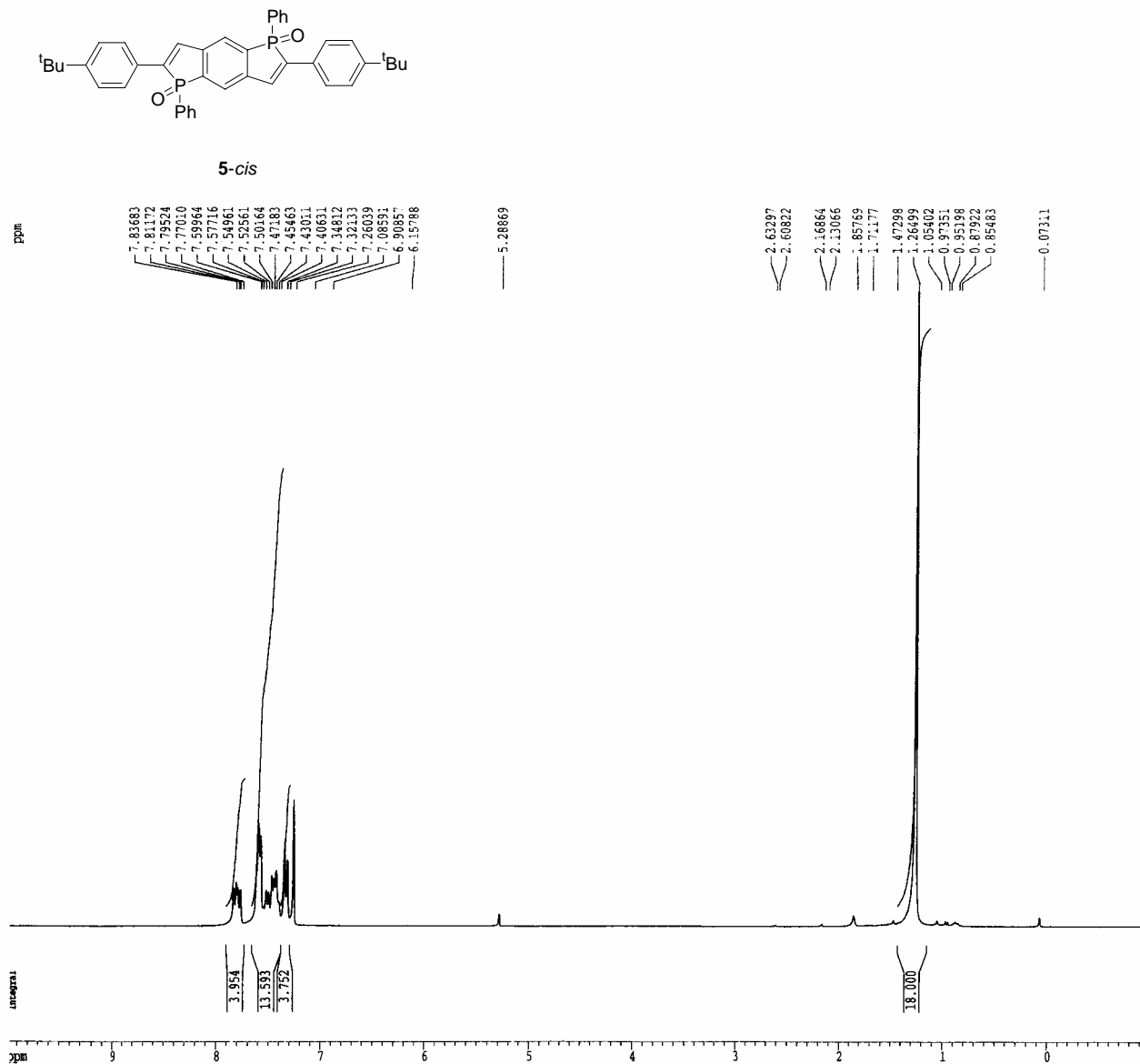
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2PHO-PhtBu-802221



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OBFIN 77.2 Hz  
POINT 32768  
FREQU 64102.5 Hz  
SCANS 4  
ACQTM 0.511 sec  
PD 1.500 sec  
PW1 5.2 us  
IRNUC 1H  
CTEMP 19.8 c  
SLVNT CDCL3  
EXREF 0.00 ppm  
BF 0.12 Hz  
RGAIN 26



<sup>31</sup>P NMR Spectrum of 4



200 MHz (Bu) @  
11 x 17 -

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1: 1.07298 (s, 1H)
2: 1.26499 (s, 1H)
3: 1.47298 (s, 1H)
4: 1.71177 (s, 1H)
5: 1.85789 (s, 1H)
6: 2.13066 (s, 1H)
7: 2.16864 (s, 1H)
8: 2.60822 (s, 1H)
9: 2.63297 (s, 1H)
10: 6.15788 (s, 1H)
11: 6.90857 (s, 1H)
12: 7.08591 (s, 1H)
13: 7.26039 (s, 1H)
14: 7.32133 (s, 1H)
15: 7.34812 (s, 1H)
16: 7.40631 (s, 1H)
17: 7.43011 (s, 1H)
18: 7.45463 (s, 1H)
19: 7.47783 (s, 1H)
20: 7.50164 (s, 1H)
21: 7.52561 (s, 1H)
22: 7.54961 (s, 1H)
23: 7.57716 (s, 1H)
24: 7.59964 (s, 1H)
25: 7.77010 (s, 1H)
26: 7.79524 (s, 1H)
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28: 7.83683 (s, 1H)

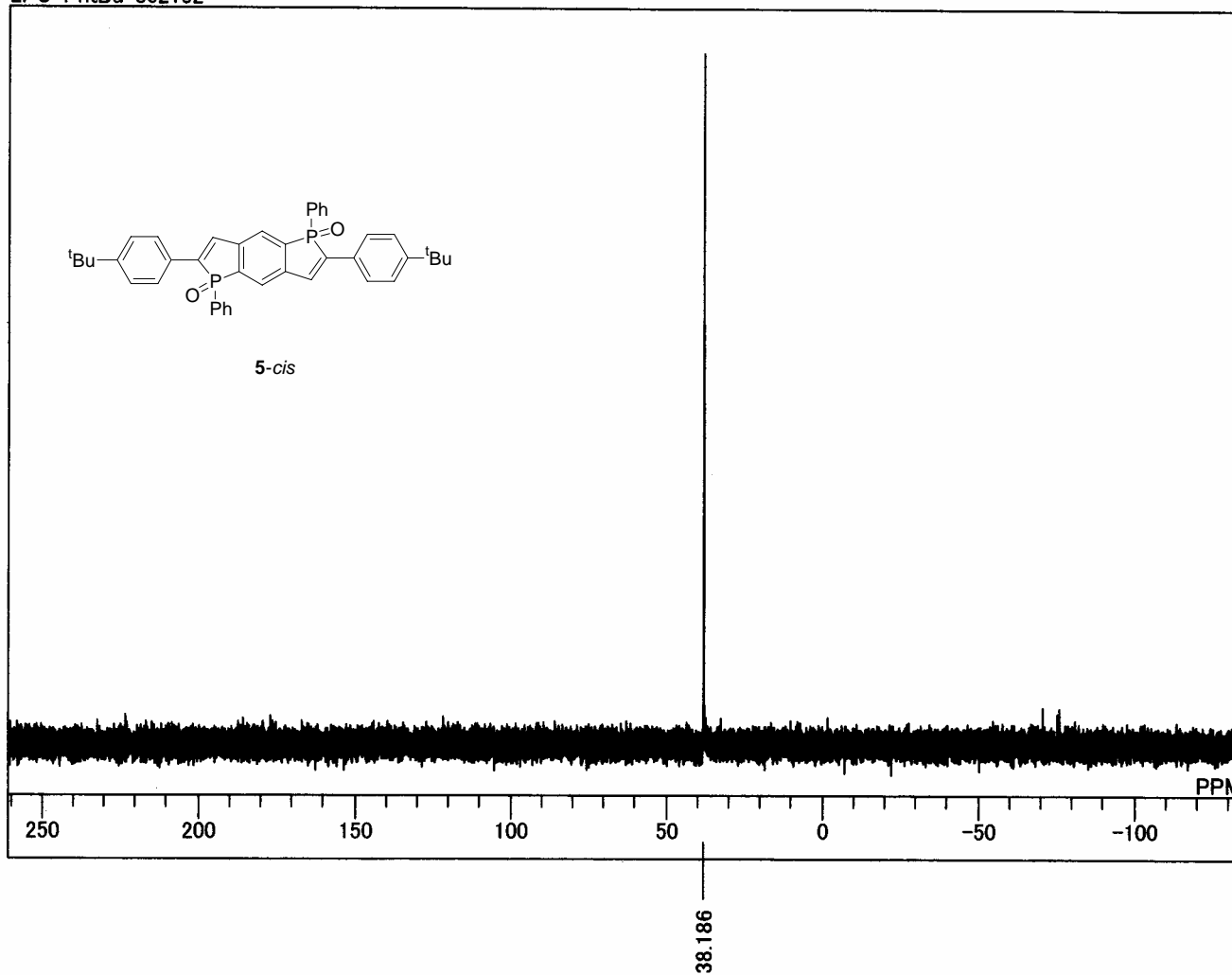
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<sup>1</sup>H NMR Spectrum of **5-cis**



C:\WINNMR98\COMMON\DEFAULT.ALS  
2PO-PhtBu-802132

x 2p-

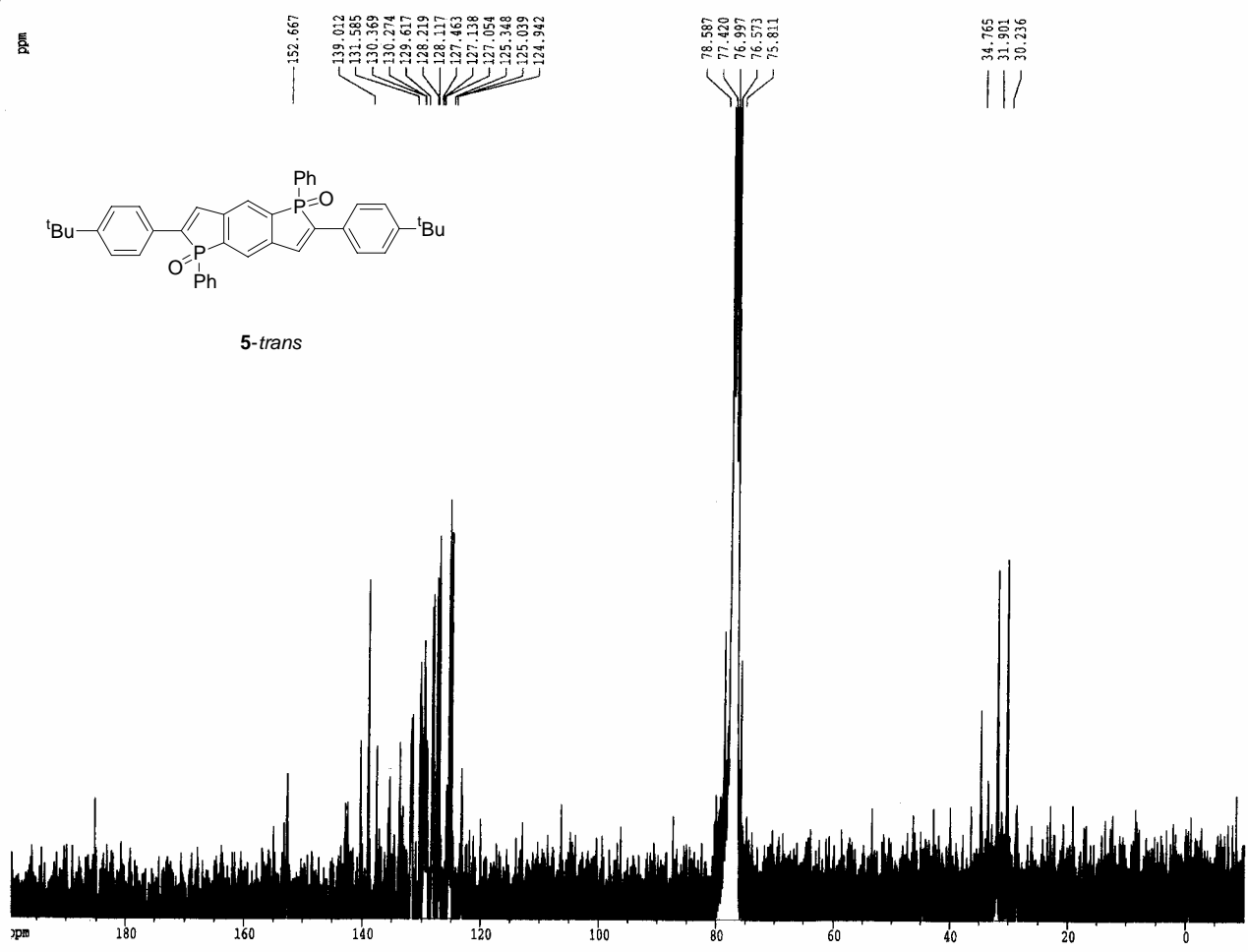


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PD 1.500 sec  
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CTEMP 21.5 c  
SLVNT CDCL3  
EXREF 0.00 ppm  
BF 0.12 Hz  
RGAIN 26

<sup>31</sup>P NMR Spectrum of 5-cis



217-  
711-06 x 06  
C



Current Data Parameters  
 NAME shiraishi  
 EXPNO 802144  
 PROCNO 1

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 Time 12.24  
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 PULPROG zgpg30  
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 NS 20874  
 DS 2  
 SMH 23809.523 Hz  
 FIDRES 0.363304 Hz  
 AQ 1.3763061 sec  
 RG 6502  
 DW 21.000 usec  
 DE 4.50 usec  
 TE 300.0 K  
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 PL13 16.00 dB  
 D1 1.00000000 sec  
 CPDPRG2 waltz16  
 PCPD2 100.00 usec  
 SFO2 300.1312005 MHz  
 NUC2 1H  
 PL2 -6.00 dB  
 PL12 16.00 dB  
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 DE 4.50 usec  
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 PL1 -6.00 dB  
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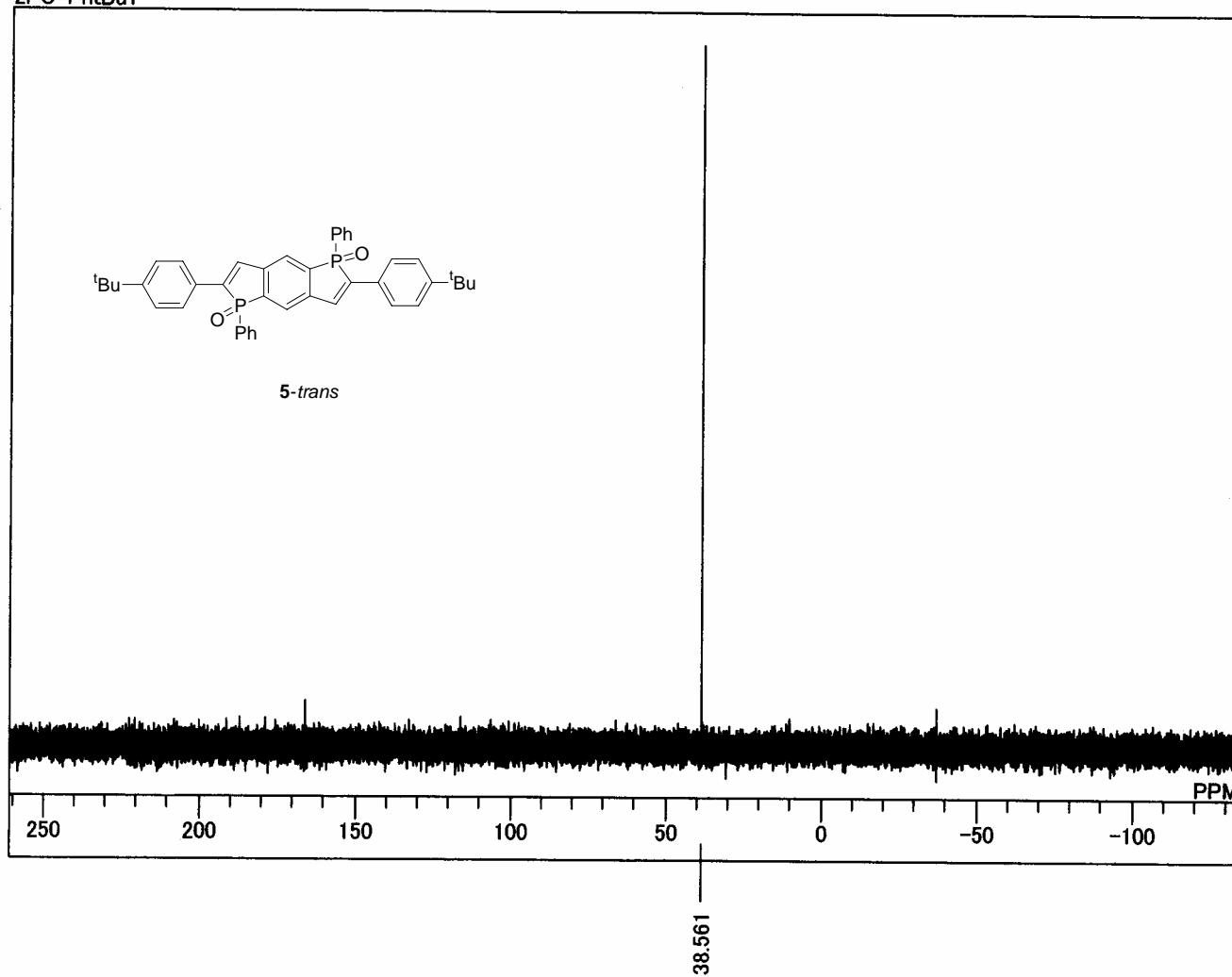
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 GB 0  
 PC 1.40

1D NMR plot parameters  
 CX 30.00 cm  
 FLP 200.000 ppm  
 F1 15093.55 Hz  
 F2P -10.000 ppm  
 F2 -754.68 Hz  
 PPMCM 7.00000 ppm/cm  
 HZCM 528.27423 Hz/cm

<sup>13</sup>C NMR Spectrum of **5-trans**

C:\WINNMR98\COMMON\DEFAULT.ALS  
2PO-PhtBu1

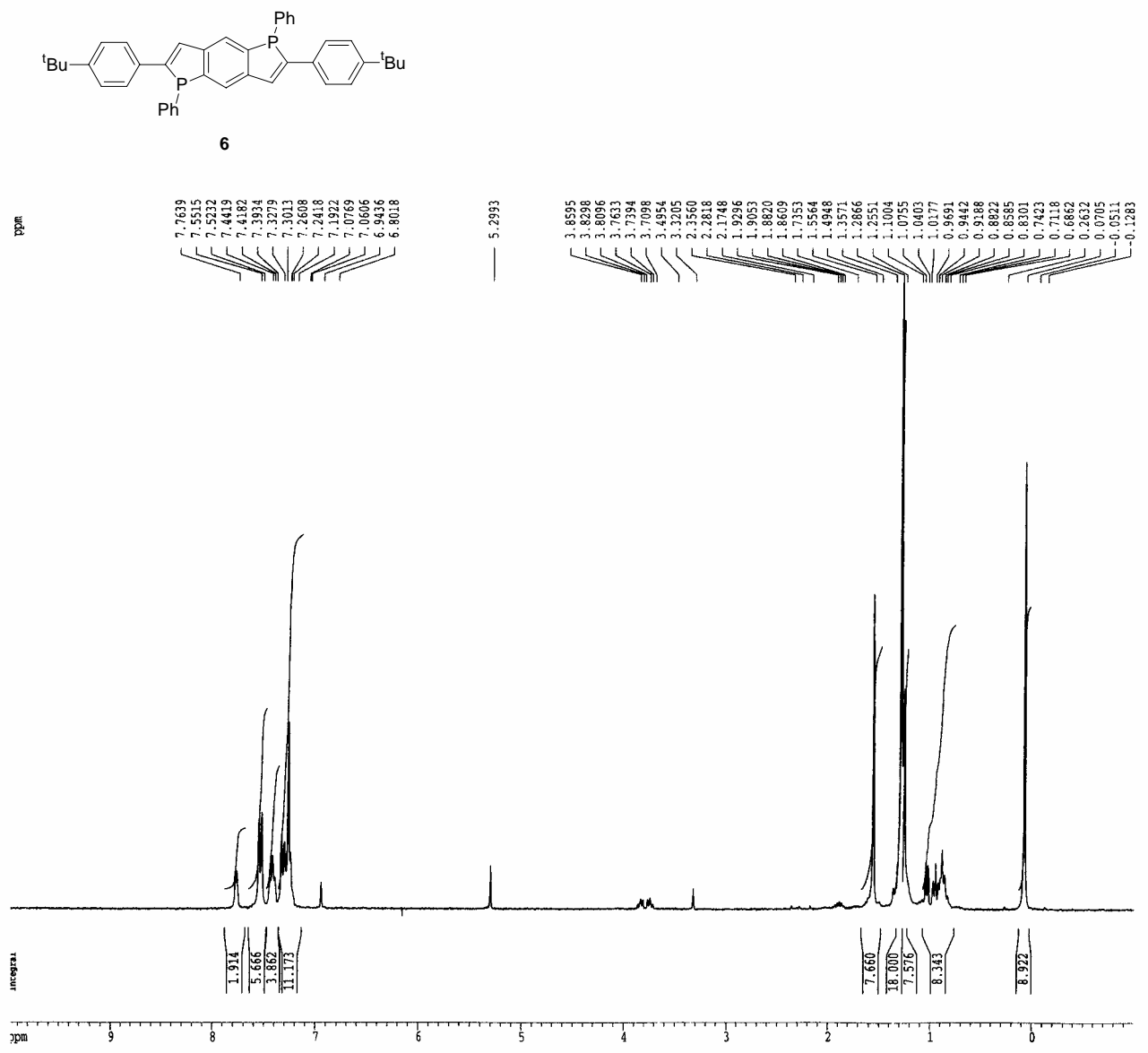
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OBSET 145.00 KHz  
OBFIN 77.2 Hz  
POINT 32768  
FREQU 64102.5 Hz  
SCANS 4  
ACQTM 0.511 sec  
PD 1.500 sec  
PW1 5.2 us  
IRNUC 1H  
CTEMP 20.7 c  
SLVNT CDCL3  
EXREF 0.00 ppm  
BF 0.12 Hz  
RGAIN 26

<sup>31</sup>P NMR Spectrum of 5-trans

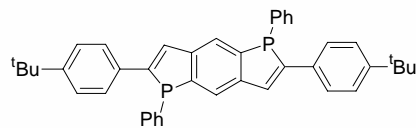
2 p-Ph-tBu



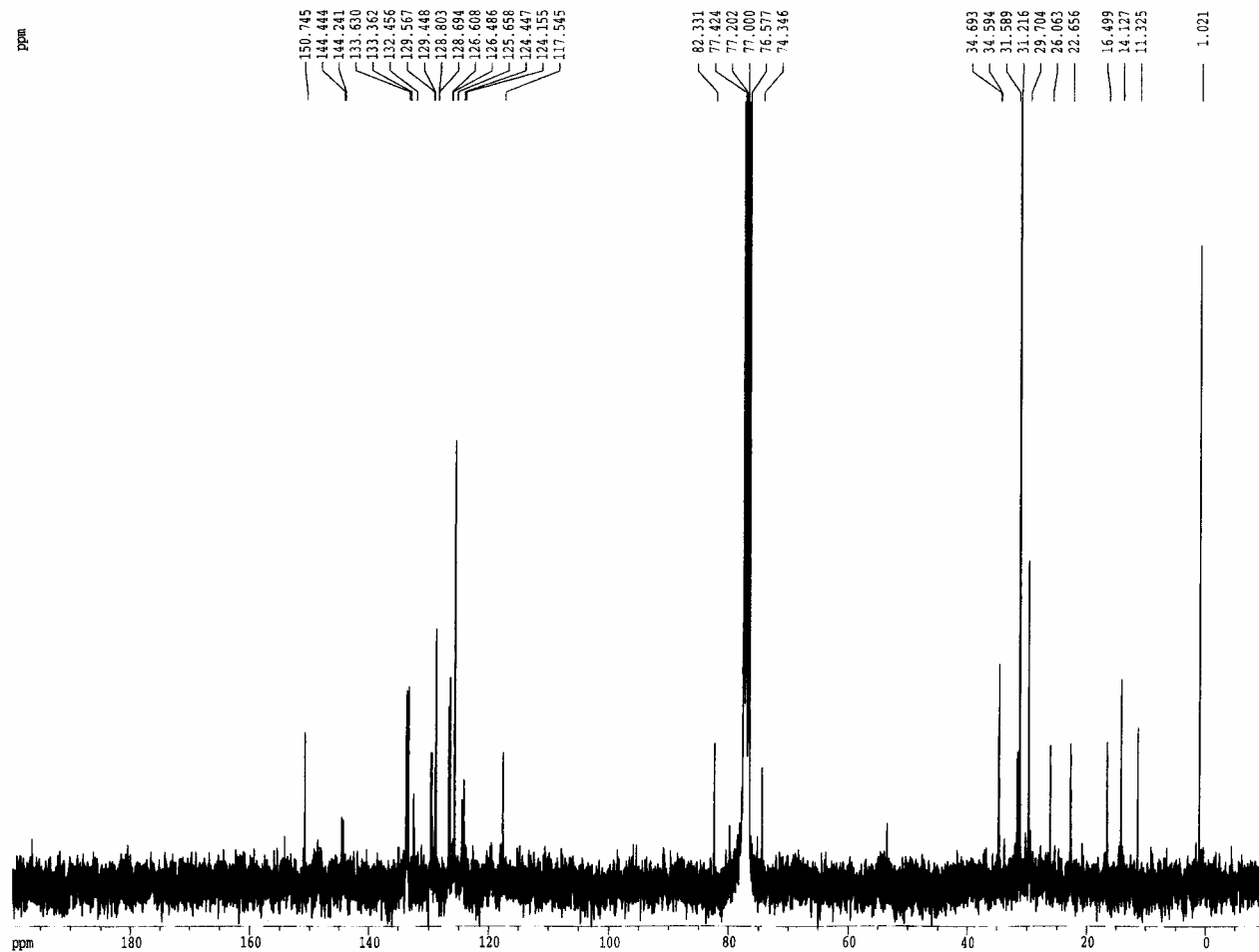
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<sup>1</sup>H NMR Spectrum of 6

2 p-Ph<sub>4</sub>



6



Current Data Parameters  
NAME shiraishi  
EXPNO 803112  
PROCNO 1

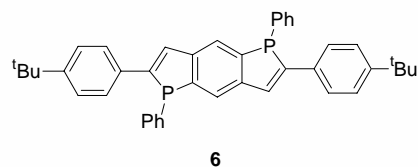
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PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 19110  
DS 2  
SWH 23809.523 Hz  
FIDRES 0.363304 Hz  
AQ 1.3763061 sec  
RG 1625.5  
DW 21.000 usec  
DE 4.50 usec  
TE 300.0 K  
D12 0.00002000 sec  
FL13 16.00 dB  
D1 1.00000000 sec  
CPDPRG2 waltz16  
PCPD2 100.00 usec  
SFO2 300.1312005 MHz  
NUC2 13C  
PL2 -6.00 dB  
PL12 16.00 dB  
P1 5.70 usec  
DE 4.50 usec  
SFO1 75.4772501 MHz  
NUC1 13C  
PL1 -6.00 dB  
D11 0.03000000 sec

F2 - Processing parameters  
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SF 75.4677494 MHz  
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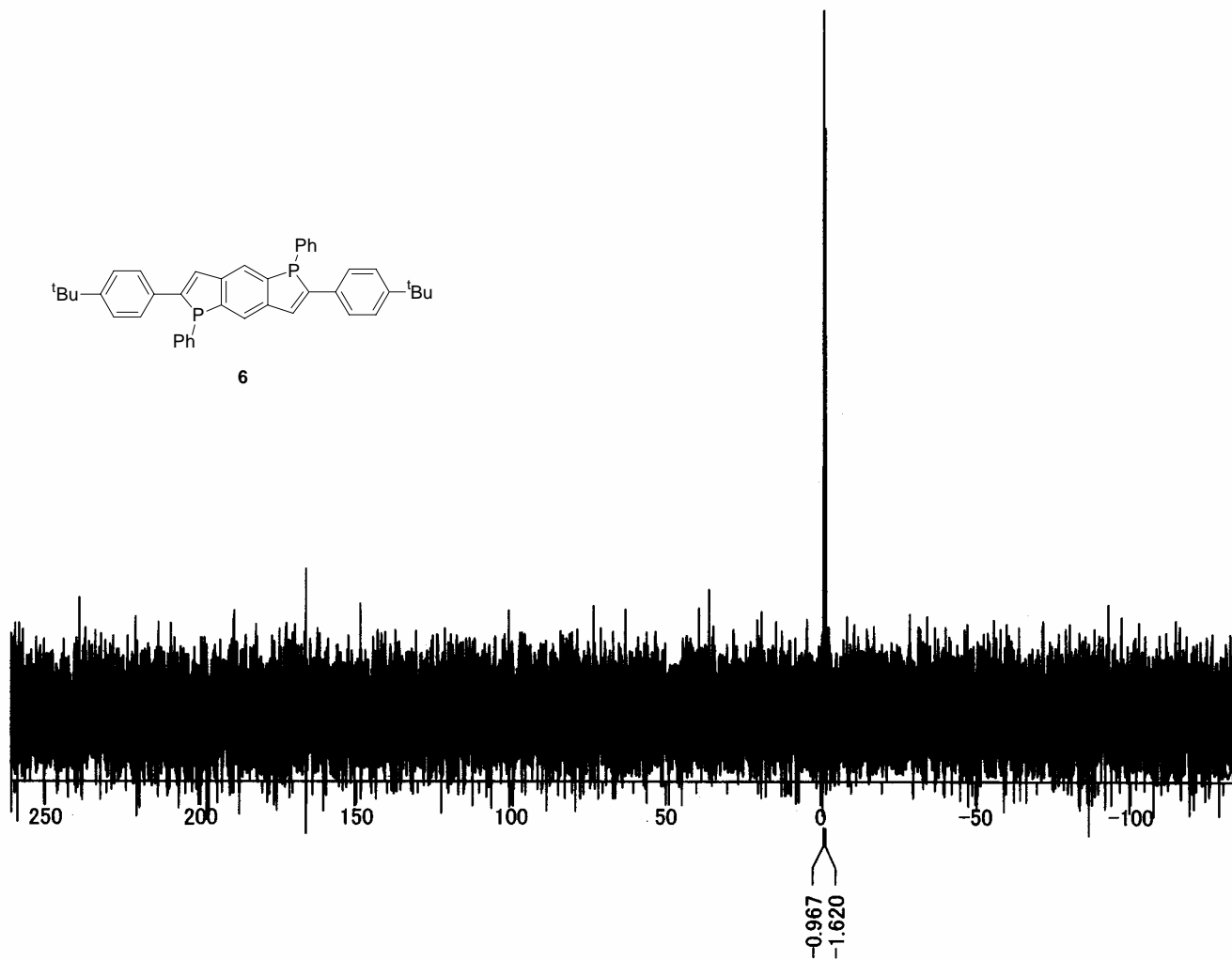
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F2P -10.000 ppm  
F2 -754.68 Hz  
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HZCM 528.27423 Hz/cm

<sup>13</sup>C NMR Spectrum of 6

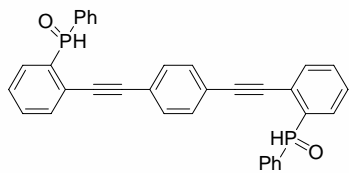
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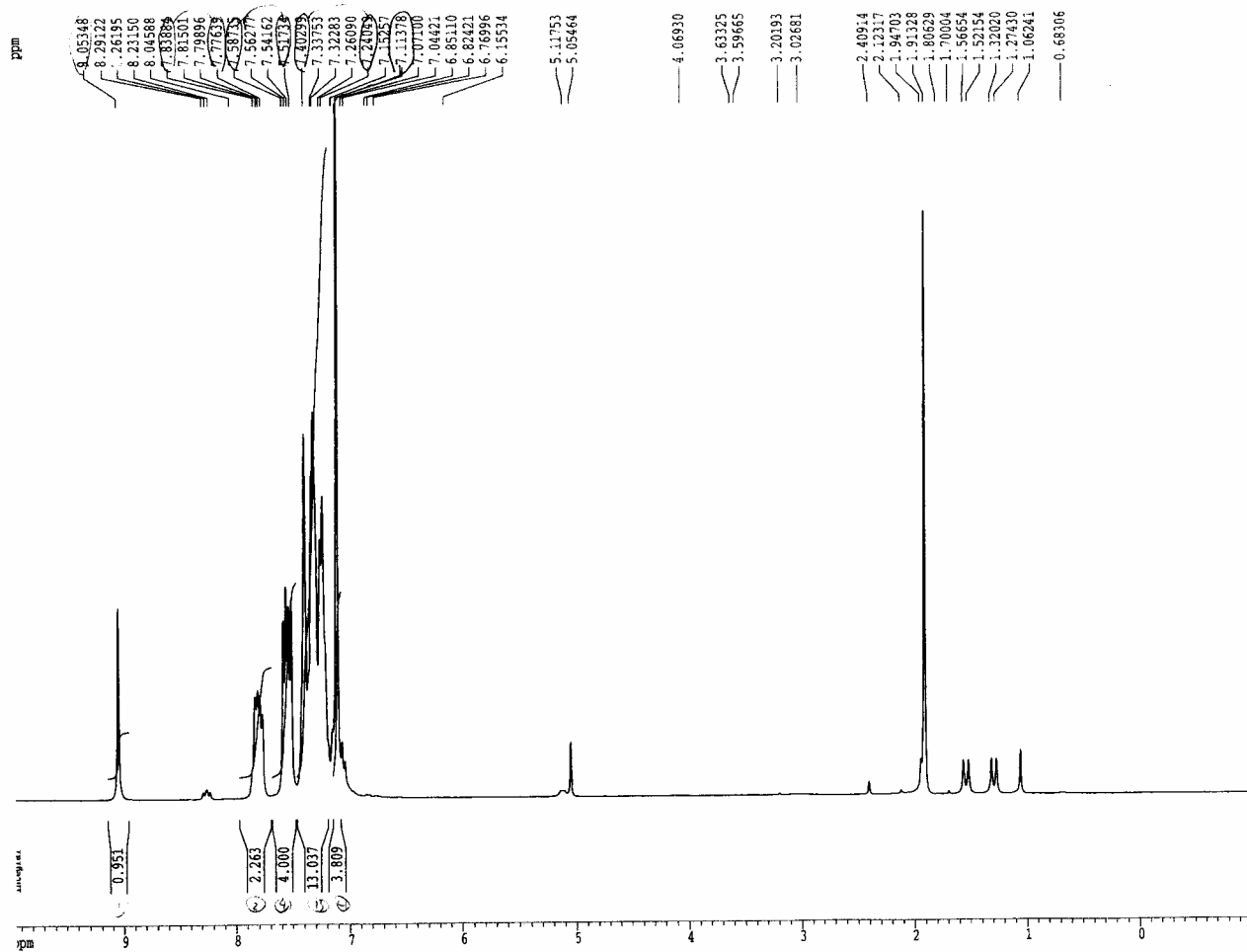
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EXREF	0.00 ppm
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RGAIN	27



<sup>31</sup>P NMR Spectrum of 6



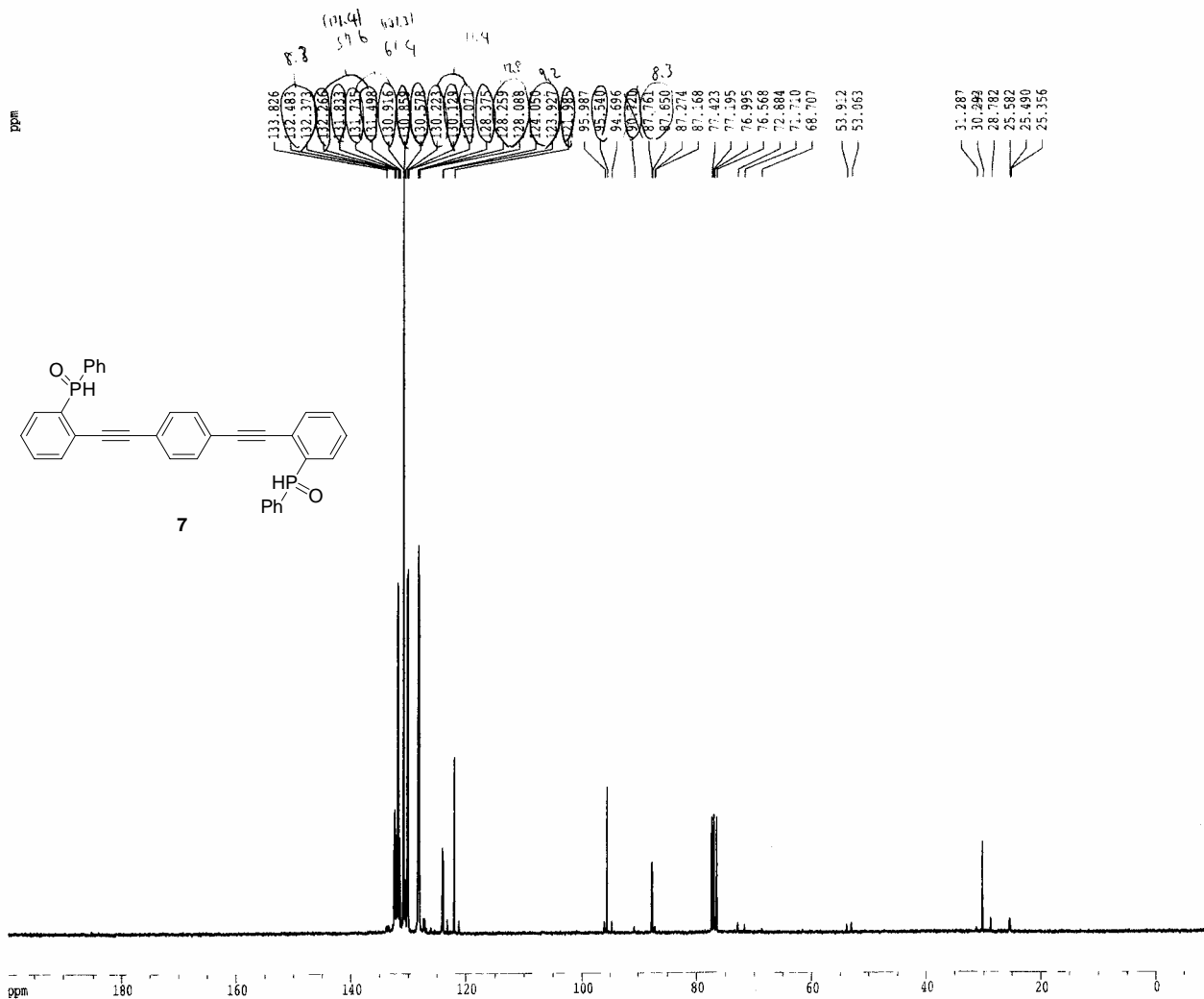
PhO-Ph-PhO



1H NMR (400 MHz, CDCl<sub>3</sub>) spectrum of compound 7. The spectrum shows a complex multiplet between 6.5 and 8.5 ppm, a sharp singlet at approximately 7.2 ppm, and a multiplet between 1.0 and 2.5 ppm. Integration values are shown below the peaks: 0.951, 2.265, 4.000, 11.037, and 3.809.

<sup>1</sup>H NMR Spectrum of 7

PhO-Ph-Ph-C



Current Data Parameters

NAME shiraishi  
 EXPNO 801252  
 PROCNO 1

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 SOLVENT CDCl3  
 NS 519  
 DS ?  
 SWH 23809.132  
 FIDRES 0.363304 Hz  
 AQ 1.3763061 sec  
 RG 3251  
 DW 21.000 usec  
 DE 4.50 usec  
 TE 300.0 K  
 D12 0.0002000 sec  
 EL13 16.00 dB  
 D1 1.00000000 sec  
 CPDPRG2 waltz16  
 PCPD2 100.00 usec  
 SFO2 300.1312005 MHz  
 NUC2 1H  
 PL2 -6.00 dB  
 PL12 16.00 dB  
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 DE 4.50 usec  
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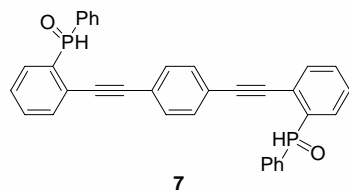
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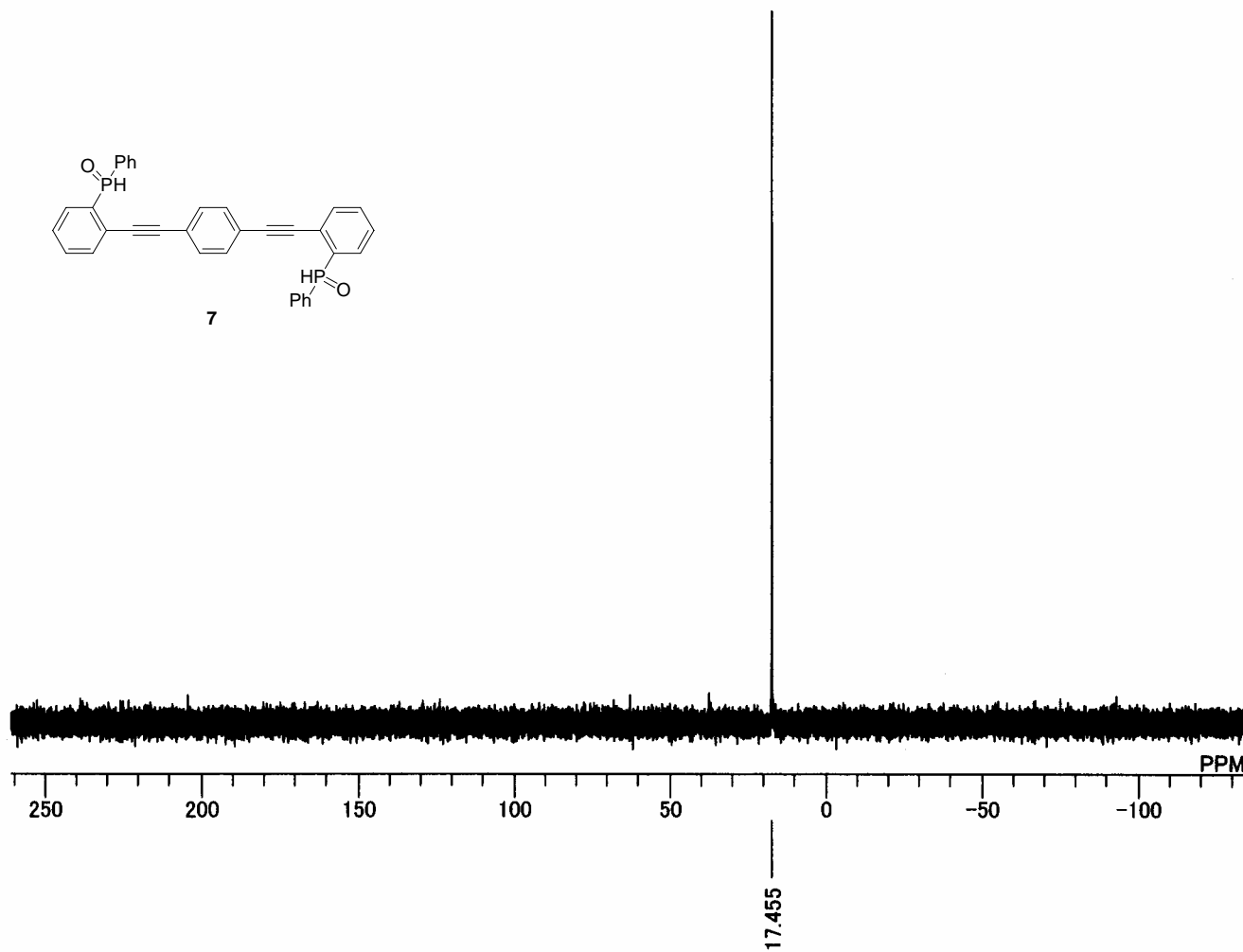
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 HZCM 528.27448 Hz/cm

<sup>13</sup>C NMR spectrum of 7

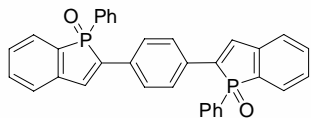


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POINT 32768  
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SCANS 2  
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PD 2.000 sec  
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CTEMP 21.1 c  
SLVNT CDCL3  
EXREF 0.00 ppm  
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RGAIN 25



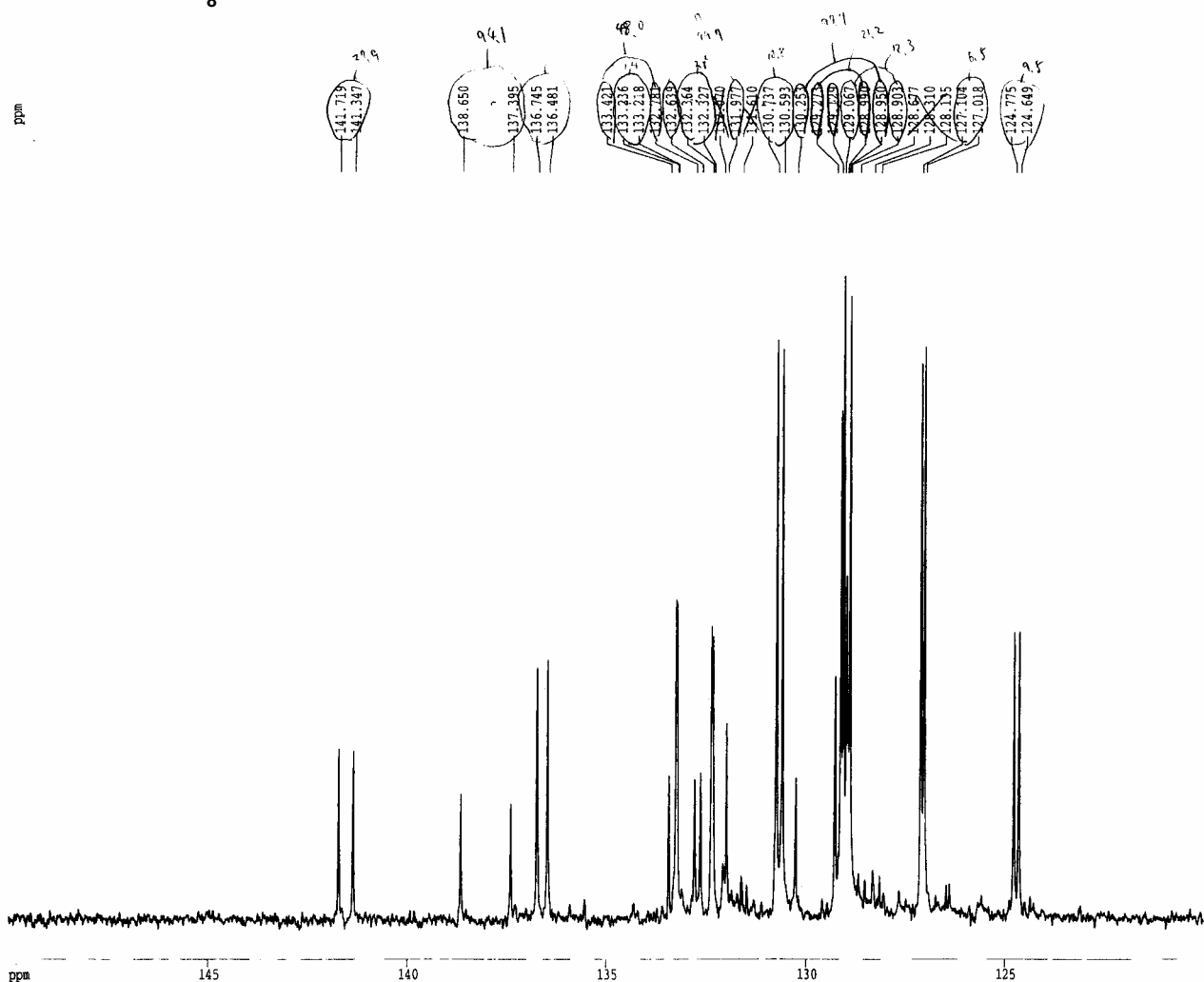
<sup>31</sup>P NMR Spectrum of 7





8

PD-P4-PD  
5



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Current Data Parameters
NAME      shiraiishi
EXPNO     803103
PROCNO    1

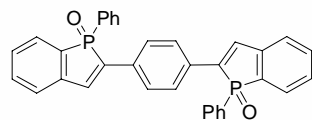
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SWH        23809.523 Hz
FIDRES     0.363304 Hz
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RG          8192
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TE          300.0 K
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PCPD2      100.00 usec
SFO2       300.1312005 MHz
NUC2        1H
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PL12        16.00 dB
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NUC1        13C
PL1         -6.00 dB
D11         0.0300000 sec

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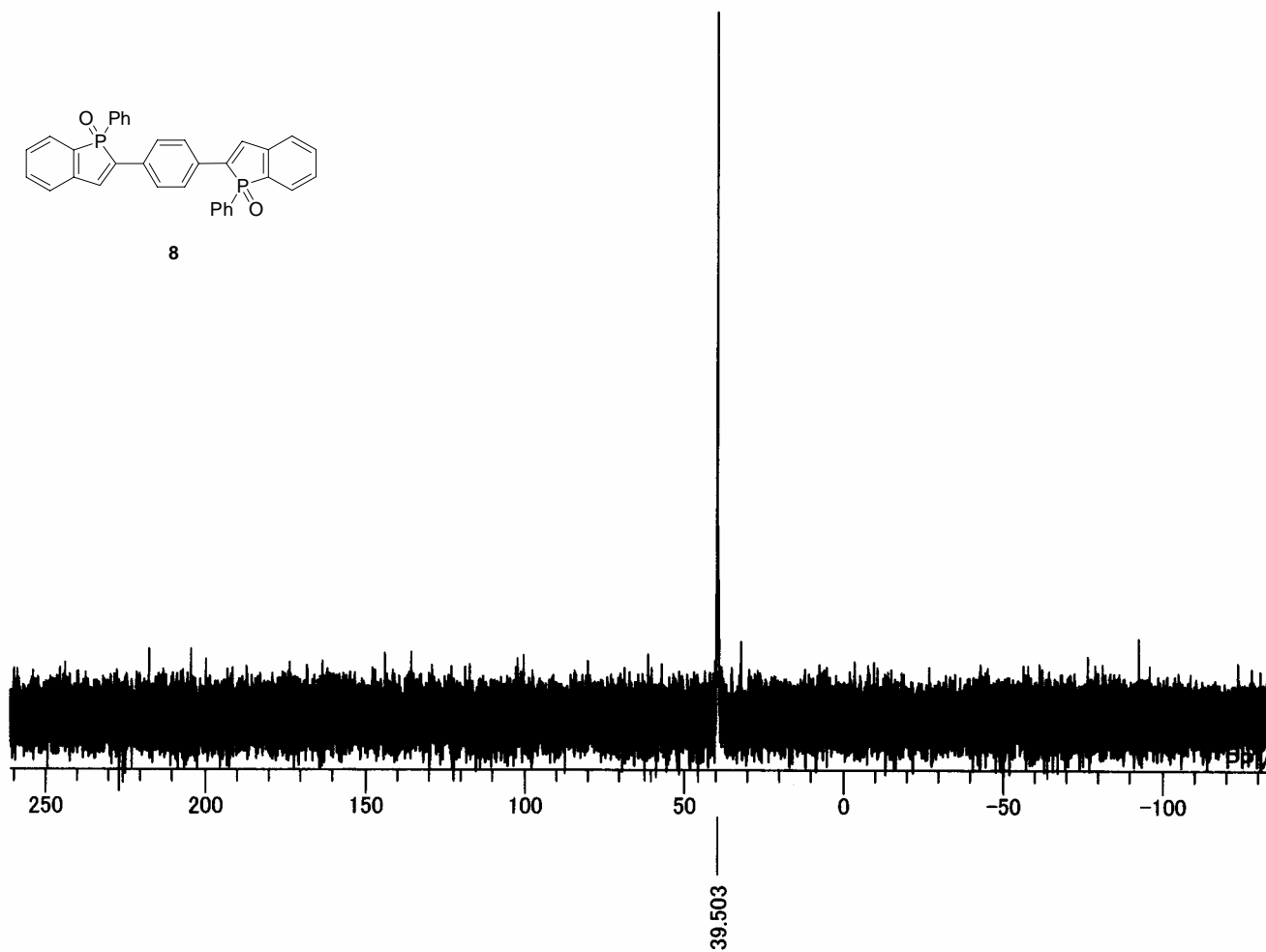
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<sup>13</sup>C NMR Spectrum of 8

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PO-Ph-PO-711131

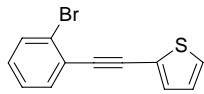


8

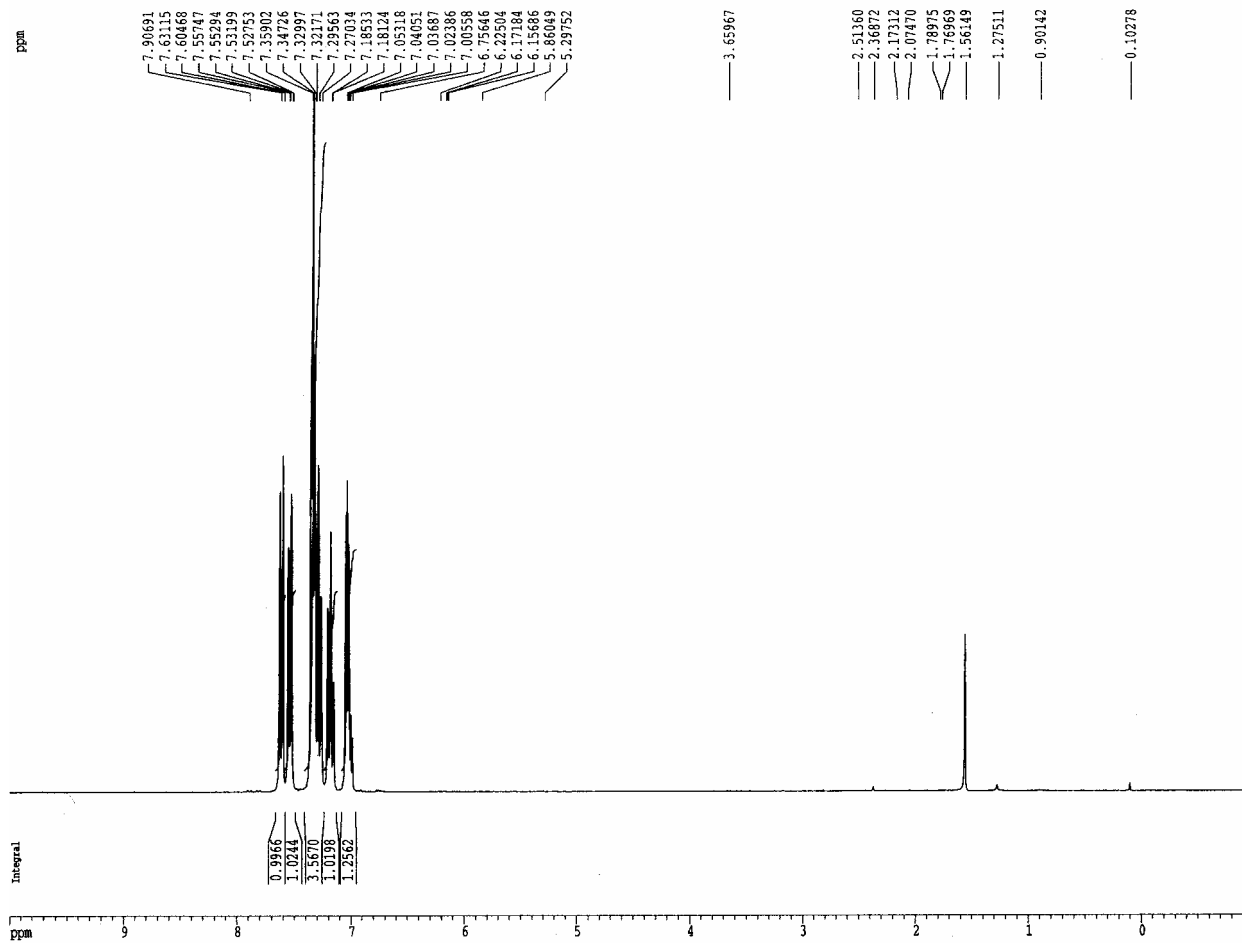


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OBFIN	77.2 Hz
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FREQU	64102.5 Hz
SCANS	86
ACQTM	0.511 sec
PD	2.000 sec
PW1	5.2 us
IRNUC	1H
CTEMP	20.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	26

<sup>31</sup>P NMR Spectrum of 8



**9d**

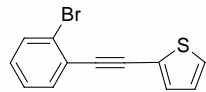


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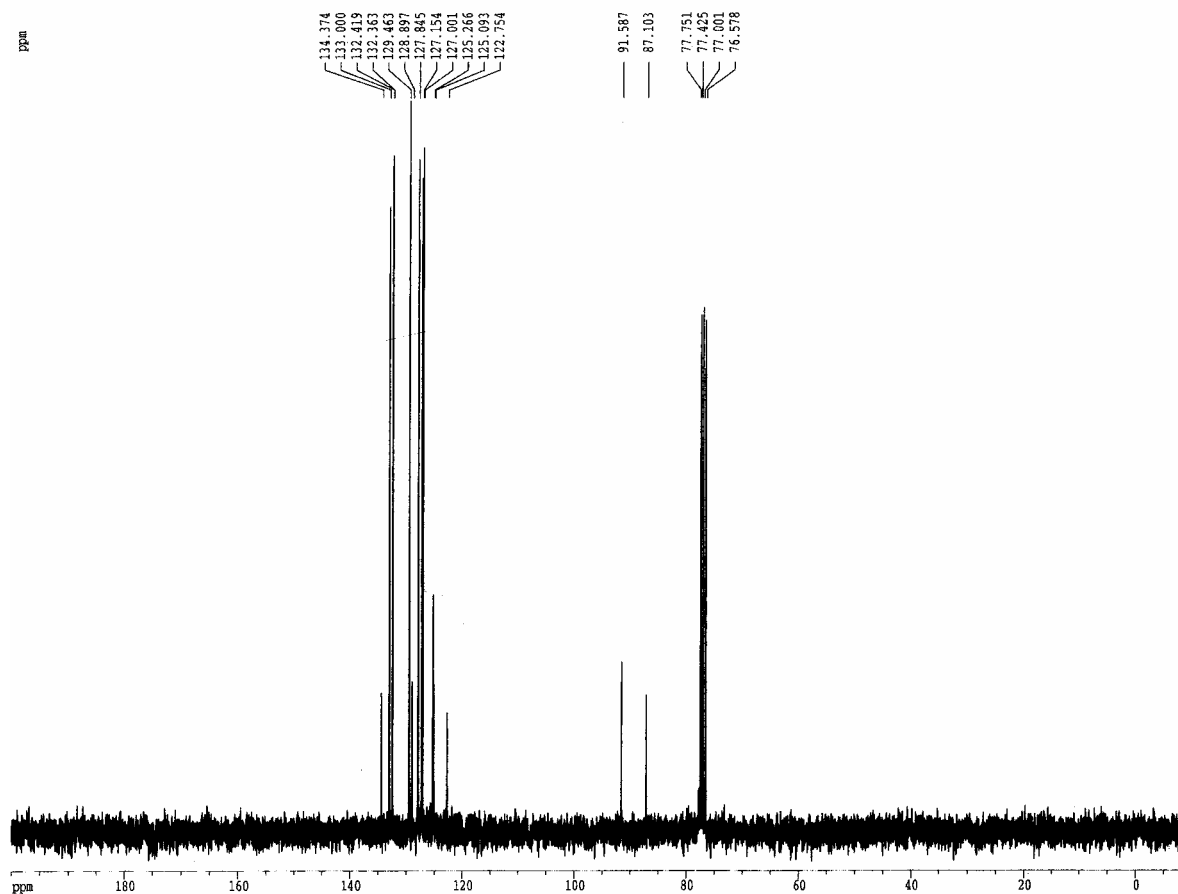
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PROBHD                5mm QNP 1H
PULPROG              zgpg30
TD                   65536
SOLVENT              CDCl3
AQ                   1.1766
RG                   327.13
AQ1                   0.0000
RG1                   1.0000
RG2                   1.0000
RG3                   1.0000
AQ2                   0.0000
RG4                   1.0000
RG5                   1.0000
RG6                   1.0000
RG7                   1.0000
RG8                   1.0000
RG9                   1.0000
RG10                  1.0000
RG11                  1.0000
RG12                  1.0000
RG13                  1.0000
RG14                  1.0000
RG15                  1.0000
RG16                  1.0000
RG17                  1.0000
RG18                  1.0000
RG19                  1.0000
RG20                  1.0000
RG21                  1.0000
RG22                  1.0000
RG23                  1.0000
RG24                  1.0000
RG25                  1.0000
RG26                  1.0000
RG27                  1.0000
RG28                  1.0000
RG29                  1.0000
RG30                  1.0000
RG31                  1.0000
RG32                  1.0000
RG33                  1.0000
RG34                  1.0000
RG35                  1.0000
RG36                  1.0000
RG37                  1.0000
RG38                  1.0000
RG39                  1.0000
RG40                  1.0000
RG41                  1.0000
RG42                  1.0000
RG43                  1.0000
RG44                  1.0000
RG45                  1.0000
RG46                  1.0000
RG47                  1.0000
RG48                  1.0000
RG49                  1.0000
RG50                  1.0000
RG51                  1.0000
RG52                  1.0000
RG53                  1.0000
RG54                  1.0000
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RG56                  1.0000
RG57                  1.0000
RG58                  1.0000
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RG60                  1.0000
RG61                  1.0000
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RG63                  1.0000
RG64                  1.0000
RG65                  1.0000
RG66                  1.0000
RG67                  1.0000
RG68                  1.0000
RG69                  1.0000
RG70                  1.0000
RG71                  1.0000
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RG73                  1.0000
RG74                  1.0000
RG75                  1.0000
RG76                  1.0000
RG77                  1.0000
RG78                  1.0000
RG79                  1.0000
RG80                  1.0000
RG81                  1.0000
RG82                  1.0000
RG83                  1.0000
RG84                  1.0000
RG85                  1.0000
RG86                  1.0000
RG87                  1.0000
RG88                  1.0000
RG89                  1.0000
RG90                  1.0000
RG91                  1.0000
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RG93                  1.0000
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RG95                  1.0000
RG96                  1.0000
RG97                  1.0000
RG98                  1.0000
RG99                  1.0000
RG100                 1.0000
=====
1D - Processing parameters
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SI                32768
SF                400.1460000 MHz
WDW                EM
SSB                0
LB                 1.30 Hz
GB                 0
PC                 1.00
RG                 327.13
AQ                 1.1766
RG1                1.0000
RG2                1.0000
RG3                1.0000
RG4                1.0000
RG5                1.0000
RG6                1.0000
RG7                1.0000
RG8                1.0000
RG9                1.0000
RG10               1.0000
RG11               1.0000
RG12               1.0000
RG13               1.0000
RG14               1.0000
RG15               1.0000
RG16               1.0000
RG17               1.0000
RG18               1.0000
RG19               1.0000
RG20               1.0000
RG21               1.0000
RG22               1.0000
RG23               1.0000
RG24               1.0000
RG25               1.0000
RG26               1.0000
RG27               1.0000
RG28               1.0000
RG29               1.0000
RG30               1.0000
RG31               1.0000
RG32               1.0000
RG33               1.0000
RG34               1.0000
RG35               1.0000
RG36               1.0000
RG37               1.0000
RG38               1.0000
RG39               1.0000
RG40               1.0000
RG41               1.0000
RG42               1.0000
RG43               1.0000
RG44               1.0000
RG45               1.0000
RG46               1.0000
RG47               1.0000
RG48               1.0000
RG49               1.0000
RG50               1.0000
RG51               1.0000
RG52               1.0000
RG53               1.0000
RG54               1.0000
RG55               1.0000
RG56               1.0000
RG57               1.0000
RG58               1.0000
RG59               1.0000
RG60               1.0000
RG61               1.0000
RG62               1.0000
RG63               1.0000
RG64               1.0000
RG65               1.0000
RG66               1.0000
RG67               1.0000
RG68               1.0000
RG69               1.0000
RG70               1.0000
RG71               1.0000
RG72               1.0000
RG73               1.0000
RG74               1.0000
RG75               1.0000
RG76               1.0000
RG77               1.0000
RG78               1.0000
RG79               1.0000
RG80               1.0000
RG81               1.0000
RG82               1.0000
RG83               1.0000
RG84               1.0000
RG85               1.0000
RG86               1.0000
RG87               1.0000
RG88               1.0000
RG89               1.0000
RG90               1.0000
RG91               1.0000
RG92               1.0000
RG93               1.0000
RG94               1.0000
RG95               1.0000
RG96               1.0000
RG97               1.0000
RG98               1.0000
RG99               1.0000
RG100              1.0000
=====
1D - NMR (1H) parameters
-----
SI                32768
SF                400.1460000 MHz
WDW                EM
SSB                0
LB                 1.30 Hz
GB                 0
PC                 1.00
RG                 327.13
AQ                 1.1766
RG1                1.0000
RG2                1.0000
RG3                1.0000
RG4                1.0000
RG5                1.0000
RG6                1.0000
RG7                1.0000
RG8                1.0000
RG9                1.0000
RG10               1.0000
RG11               1.0000
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RG13               1.0000
RG14               1.0000
RG15               1.0000
RG16               1.0000
RG17               1.0000
RG18               1.0000
RG19               1.0000
RG20               1.0000
RG21               1.0000
RG22               1.0000
RG23               1.0000
RG24               1.0000
RG25               1.0000
RG26               1.0000
RG27               1.0000
RG28               1.0000
RG29               1.0000
RG30               1.0000
RG31               1.0000
RG32               1.0000
RG33               1.0000
RG34               1.0000
RG35               1.0000
RG36               1.0000
RG37               1.0000
RG38               1.0000
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RG43               1.0000
RG44               1.0000
RG45               1.0000
RG46               1.0000
RG47               1.0000
RG48               1.0000
RG49               1.0000
RG50               1.0000
RG51               1.0000
RG52               1.0000
RG53               1.0000
RG54               1.0000
RG55               1.0000
RG56               1.0000
RG57               1.0000
RG58               1.0000
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RG60               1.0000
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RG63               1.0000
RG64               1.0000
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RG67               1.0000
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RG74               1.0000
RG75               1.0000
RG76               1.0000
RG77               1.0000
RG78               1.0000
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RG83               1.0000
RG84               1.0000
RG85               1.0000
RG86               1.0000
RG87               1.0000
RG88               1.0000
RG89               1.0000
RG90               1.0000
RG91               1.0000
RG92               1.0000
RG93               1.0000
RG94               1.0000
RG95               1.0000
RG96               1.0000
RG97               1.0000
RG98               1.0000
RG99               1.0000
RG100              1.0000
=====

```

<sup>1</sup>H NMR spectrum of **9d**



**9d**



```

Current Data Parameters
NAME      shiraishi
EXPNO     706084
PROCNO    1

F2 - Acquisition Parameters
Date_     500000
Time      20.42
INSTRUM   dpx300
PROBHD    5 mm QNP 1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         113
DS         2
SWH        23809.523 Hz
FIDRES     0.363304 Hz
AQ         1.3763061 sec
RG         3649.1
DW         21.000 usec
DE         4.50 usec
TE         300.0 K
D12        0.00002000 sec
PL13       16.00 dB
D1         1.00000000 sec
CPDPRG2    waltz16
PCPD2      100.00 usec
SFO2       300.1312005 MHz
NUC2       13C
PL2        -6.00 dB
PL12       16.00 dB
P1         5.70 usec
DE         4.50 usec
SFO1       75.4772501 MHz
NUC1       13C
PL1        -6.00 dB
D11        0.03000000 sec

F2 - Processing parameters
SI         32768
SF         75.4677552 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

1D NMR plot parameters
CX         30.00 cm
F1P        200.000 ppm
F1         15093.55 Hz
F2P        -10.000 ppm
F2         -754.68 Hz
PWCMM      7.00000 ppm/cm
HZCM       528.27429 Hz/cm
  
```

<sup>13</sup>C NMR spectrum of **9d**