

# Supporting Information:

## Palladium-Catalyzed Cross-Coupling Reactions of Benzyl Indium Reagents with Aryl Iodides

Louis S. Chupak,<sup>\*1</sup> Joanna P. Wolkowski,<sup>2</sup> and Yves A. Chantigny<sup>3</sup>

*Groton Laboratories, Pfizer Inc., Eastern Point Road, 8220/2255, Groton, CT 06475*  
*louis.Chupak@bms.com*

<b>Contents</b>	<b>Page</b>
<b>Materials, Methods, General Procedure</b>	<b>S2</b>
<b>Characterization Data for Entries 1 through 23 in Table 1</b>	<b>S2-S6</b>
<b><sup>1</sup>H NMR Spectra for Entries 1 through 23 in Table 1</b>	<b>S7-S26</b>
<b>X-ray Crystallography for Entry 14 in Table 1</b>	<b>S27-S35</b>

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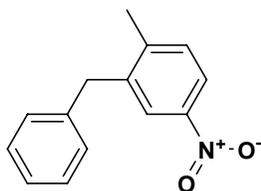
<sup>1</sup> Early Discovery Chemistry, Bristol-Myers Squibb Co., 5 Research Parkway, Wallingford, CT 06492.

<sup>2</sup> Sierra Nevada Brewing Company

<sup>3</sup> MethylGene Inc.

**Materials and Methods.** Reactions were conducted either in an ambient laboratory atmosphere or under N<sub>2</sub>, as noted. All solvents were reagent grade. Tetrakis(triphenylphosphine)palladium(0), Indium powder (-60 mesh, 99.999%), lithium chloride, iodobenzene, 3-iodoanisole, 1-iodo-3-nitrobenzene, 2-iodo-4-nitrotoluene, were purchased from commercial sources and used without purification. 2-Iodo-1-isopropyl-4-nitrobenzene was prepared according to literature procedures (Kraszkiwicz, L.; Sosnowski, M.; Skulski, L. *Tetrahedron*, **2004**, *60*, 9113.) The benzyl bromides used in this study were purchased from commercial sources and used without further purification. Chromatography was performed on an MPLC system using normal phase disposable silica gel columns. <sup>1</sup>H NMR spectra were recorded on either 400 MHz spectrometer or a 500 MHz spectrometer. Chemical shifts are reported relative to chloroform (δ 7.26). LC/MS data were obtained using a 3.74 minute run used a gradient of 95% A / 5% B to 100% B (A = 0.01% formic acid, 2% acetonitrile, 97.99% water; B = 0.005% formic acid in acetonitrile). The DAD scanned wavelengths 190-400 nm to provide a UV trace of the compound. Due to the nature of the products, there is limited potential for ionization. Thus, the retention time of the single product peak is reported for all compounds. In the instance of ionization, a mass corresponding to M±1 is also reported. FAB HRMS was carried out on a high field mass spectrometer. A cesium ion gun was used to generate protonated molecular ions for the acquired mass spectra.

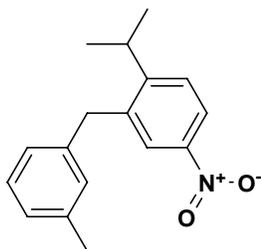
**General Procedure.** To a 1 mM solution of benzyl bromide (5 mmol bromide in 5 mL of DMF), were added 1.5 equivalents of indium. The reaction mixture was stirred at room temperature under an ambient laboratory atmosphere until TLC revealed disappearance of starting material. To a separate flask were added: 0.4 equiv (2 mmol) of aryl iodide, 0.8 equiv (4 mmol) of LiCl, and 2% (0.1 mmol) Pd(PPh<sub>3</sub>)<sub>4</sub>. The DMF solution containing the benzylindium was then added to the reaction mixture, which was subsequently diluted with DMF (5 mL) to a 0.5 mM solution. The resulting reaction mixture was stirred under N<sub>2</sub> at 100 °C until no starting aryl iodide was observed by TLC. After dilution with Et<sub>2</sub>O, the reaction mixture was washed with 3 N HCl (5x), water (3x), and brine (1x). The reaction mixture was dried over MgSO<sub>4</sub>, filtered, and solvents were removed by rotary evaporation. Chromatography on SiO<sub>2</sub> with an appropriate solvent system of ethyl acetate in hexanes yielded clean product.



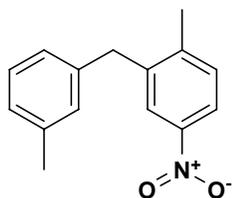
**Table 1 Entry 1. (2-Methyl-5-nitro-benzyl)-benzene.** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 2.33 (s, 3H), 4.04 (s, 2H), 7.10 (d, 7.5 Hz, 2H), 7.20-7.25 (m, 1H), 7.27-7.31 (m, 3H), 7.99 (m, 2H). <sup>13</sup>C NMR (500 MHz, CDCl<sub>3</sub>) δ 20.2, 36.9, 121.7, 124.1, 126.7, 127.2, 129.2, 131.1, 131.2, 136.7, 136.8, 140.6, 145.0, 146.9. LC/MS 3.0 min. FAB HRMS: Calc'd for C<sub>14</sub>H<sub>13</sub>NO<sub>2</sub> (M<sup>+</sup>): 228.1024, Found 228.1036.



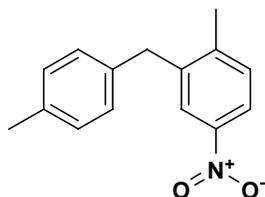
**Table 1 Entry 2. 1-methyl-2-(2-methylbenzyl)-4-nitrobenzene.** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 2.19 (3H), 2.39 (s, 3H), 3.97 (s, 2H), 6.87 (d, 7.0 Hz, 2H), 7.10-7.25 (m, 3H), 7.54 (d, 8.7 Hz, 1H), 7.75 (d, 2.5 Hz, 1H), 8.0 (dd, 8.31 Hz, 2.5 Hz, 1H). <sup>13</sup>C NMR (500 MHz, CDCl<sub>3</sub>) δ 19.9, 20.2, 36.9, 121.7, 124.1, 126.7, 127.2, 129.3, 131.1, 131.2, 136.7, 136.8, 140.6, 145.0, 146.9. LC/MS 3.1 min. Anal. Calc'd for C<sub>15</sub>H<sub>15</sub>NO<sub>2</sub>: C, 74.67; H, 6.27; N, 5.81; O, 11.88; Found C, 74.73; H, 6.21; N, 5.74.



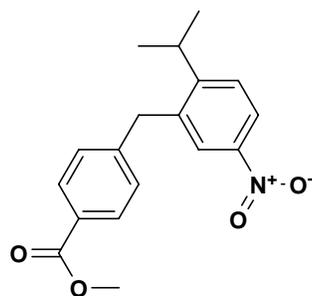
**Table 1 Entry 3. 3-(2-Isopropyl-5-nitro-benzyl)-toluene.** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 1.15 (d, 6.65 Hz, 6H), 2.29 (s, 3H), 3.21 (sept, 6.65 Hz, 1H), 4.07 (s, 2H), 6.87 (m, 2H), 7.02 (d, 7.9 Hz, 1H), 7.16 (t, 7.5 Hz, 1H), 7.43 (d, 8.7 Hz, 1H), 7.97 (d, 2.5 Hz, 1H), 8.07 (dd, 8.31 Hz, 2.5 Hz, 1H). <sup>13</sup>C NMR (500 MHz, CDCl<sub>3</sub>) δ 20.4, 21.7 28.7, 39.5, 121.7, 124.8, 125.9, 127.6, 128.9, 129.7, 131.2, 137.1, 141.4, 145.0, 146.8. FAB HRMS: Calc'd for C<sub>17</sub>H<sub>19</sub>NO<sub>2</sub> (M<sup>+</sup>): 270.1494, Found 270.1501.



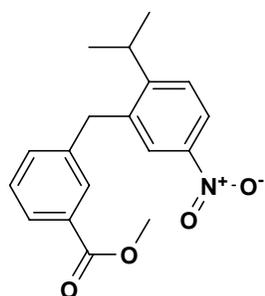
**Table 1 Entry 4. 1-methyl-2-(3-methylbenzyl)-4-nitrobenzene.**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  2.33 (3H), 2.36 (s, 3H), 4.03 (s, 2H), 6.99 (m, 2H), 7.06 (d, 5.2 Hz, 1H), 7.21 (t, 7.2 Hz, 1H), 7.32 (d, 6.3 Hz, 1H) 8.05 (m, 2H).  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  20.3, 21.7, 39.5, 121.8, 124.9, 125.9, 127.6, 128.9, 129.6, 131.3, 138.7, 141.0, 145.0, 146.8. LC/MS 2.2 min. FAB HRMS: Calc'd for  $\text{C}_{15}\text{H}_{15}\text{NO}_2$  ( $\text{M}^{+1}$ ): 242.1181, Found 242.1174.



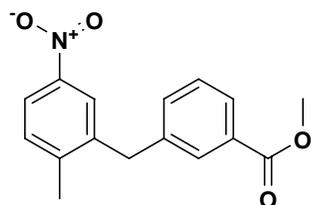
**Table 1 Entry 5. 1-methyl-2-(4-methylbenzyl)-4-nitrobenzene.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  2.32 (3H), 2.34 (s, 3H), 4.0 (s, 2H), 6.99 (d, 7.8 Hz, 2H), 7.16 (d, 7.8 Hz, 2H), 7.29 (d, 8.3 Hz, 1H), 7.99 (m, 2H).  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  20.3, 21.3, 39.2, 121.8, 124.8, 128.8, 129.7, 131.3, 135.7, 136.4 141.2, 145.0, 146.7. LC/MS 1.9 min. Anal. Calc'd for  $\text{C}_{15}\text{H}_{15}\text{NO}_2$ : C, 74.67; H, 6.27; N, 5.81; O, 11.88; Found C, 74.60; H, 6.21; N, 5.71.



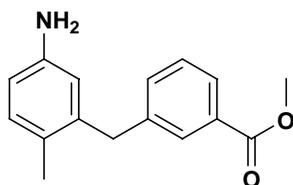
**Table 1 Entry 6. Methyl 4-(2-isopropyl-5-nitrobenzyl)benzoate.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  1.14 (d, 6.65 Hz, 6H), 3.16 (sept, 6.65 Hz, 1H), 3.91 (s, 3H), 4.21 (s, 2H), 7.19 (d, 8.0 Hz, 2H), 7.36 (s, 1H), 7.91 (d, 8.0 Hz, 2H) 8.02 (m, 2H).  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  19.2, 20.1, 36.7, 124.8, 128.7, 129.5, 132.2, 135.7, 136.4 140.9, 145.0, 146.7, 167.1. Anal. Calc'd for  $\text{C}_{18}\text{H}_{19}\text{NO}_4$ : C, 68.99; H, 6.11; N, 4.47; Found C, 69.05; H, 6.15; N, 4.39.



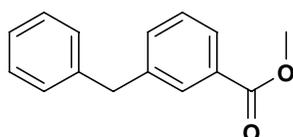
**Table 1 Entry 7. 3-(2-isopropyl-5-nitrobenzyl)-benzoic acid methyl ester.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  1.14 (d, 6.65 Hz, 6H), 3.16 (sept, 6.65 Hz, 1H), 3.88 (s, 3H), 4.16 (s, 2H), 7.25 (d, 5.8 Hz, 1H), 7.36 (t, 7.5 Hz, 1H), 7.44 (d, 8.3 Hz, 1H), 7.8 (s, 1H), 7.89 (d, 7.7 Hz, 1H), 7.97 (d, 2.5 Hz, 1H), 8.08 (dd, 8.3 Hz, 2.5 Hz, 1H). LC/MS 3.1 min.  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  20.1, 36.8, 121.7, 123.9, 126.6, 128.0, 129.2, 131.1, 131.2, 136.72, 136.79 140.8, 145.5, 146.9, 168.0. Anal. Calc'd for  $\text{C}_{18}\text{H}_{19}\text{NO}_4$ : C, 68.99; H, 6.11; N, 4.47; Found C, 69.11; H, 6.05; N, 4.49.



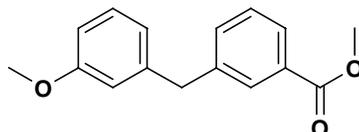
**Table 1 Entry 8. 3-(2-Methyl-5-nitro-benzyl)-benzoic acid methyl ester.**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  2.35 (s, 3H), 3.92 (s, 3H), 4.12 (s, 2H), 7.31 (d, 7.8 Hz, 1H), 7.34 (d, 8.3 Hz, 1H), 7.40 (t, 7.8 Hz, 1H), 7.84 (s, 1H), 7.94 (d, 7.8 Hz, 1H), 7.99 (d, 2.1 Hz, 1H), 8.05 (dd, 8.3 Hz, 2.1 Hz, 1H).  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  19.2, 20.1, 36.8, 121.7, 123.9, 126.6, 128.0, 129.2, 131.1, 131.2, 136.72, 136.79 140.8, 145.5, 146.9, 168.0 FAB HRMS:Calc'd for  $\text{C}_{16}\text{H}_{15}\text{NO}_4$  ( $\text{M}^+$ ) 286.1080, Found 286.1088.



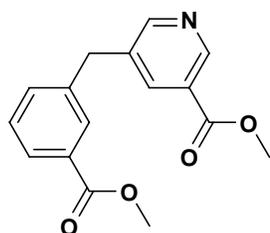
**Table 1 Entry 9. 3-(5-Amino-2-methyl-benzyl)-benzoic acid methyl ester.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  2.11 (s, 3H), 3.52 (bs, 2H), 3.88 (s, 3H), 3.92 (s, 2H), 6.41 (d, 2.49 Hz, 1H), 6.50 (dd, 7.89 Hz, 2.49 Hz, 1H), 6.94 (d, 7.89 Hz, 1H), 7.28-7.34 (m, 2H), 7.84-7.87 (m, 2H).  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  18.9, 39.5, 52.3, 113.7, 117.2, 126.6, 127.5, 128.6, 130.2, 130.4, 131.3, 133.6, 139.3, 141.0, 144.6, 167.4. LC/MS 1.7 min ( $\text{M}^+$ ) 256.4. FAB HRMS:Calc'd for  $\text{C}_{16}\text{H}_{17}\text{NO}_2$  ( $\text{M}^+$ ) 256.1338, Found 256.1350.



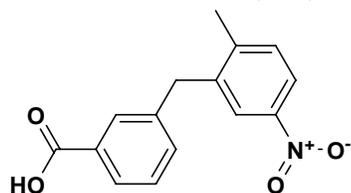
**Table 1 Entry 10. 3-Benzylbenzoic acid methyl ester.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  3.89 (s, 3H), 4.01 (s, 2H), 7.18 (m, 2H), 7.25 (m, 3H), 7.36 (m, 2H), 7.89 (m, 2H). LC/MS 2.8 min ( $\text{M}^+$ ) 227.4 FAB HRMS:Calc'd for  $\text{C}_{15}\text{H}_{14}\text{O}_2$  ( $\text{M}^+$ ) 227.1024, Found 227.1030. Chahen, L.; Doucet, H.; Santelli, M. *Synlett* **2003**, 1668.



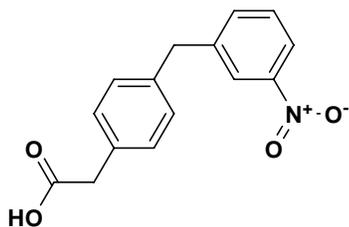
**Table 1 Entry 11. 3-(3-Methoxy-benzyl)-benzoic acid methyl ester.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  3.76 (s, 3H), 3.89 (s, 3H), 3.99 (s, 2H), 6.74 (m, 2H), 7.21 (t, 6.9 Hz, 1H), 7.36 (m, 2H), 7.90 (m, 2H).  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  39.6, 52.1, 52.3, 110.7, 112.7, 119.5, 128.6, 130.1, 130.5, 130.8, 131.0, 132.9, 141.0, 142.6, 160.7 167.4. Anal. Calc'd for  $\text{C}_{16}\text{H}_{16}\text{O}_3$ : C, 74.98; H, 6.29; Found C, 75.15; H, 6.31.



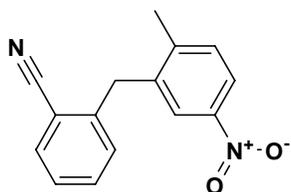
**Table 1 Entry 12. Methyl 5-(3-(methoxycarbonyl)benzyl)nicotinate.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  3.88 (s, 3H), 3.91 (s, 3H), 4.06 (s, 2H), 7.36 (m, 2H), 7.85 (s, 1H), 7.91(dt, 7.1 Hz, 1.6 Hz, 1H), 8.06 (t, 2.1 Hz, 1H), 8.63 (d, 2.5 Hz, 1H), 9.05 (d, 2.1 Hz, 1H).  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  38.8, 52.4, 52.6, 126.2, 128.3, 129.2, 130.1, 131.0, 133.5, 136.1, 139.6, 149.1, 153.9, 165.9, 165.0. LC/MS 2.08 min. ( $\text{M}^+$ ) 286.1. FAB HRMS:Calc'd for  $\text{C}_{16}\text{H}_{15}\text{NO}_4$  ( $\text{M}^+$ ) 286.1080, Found 286.1080.



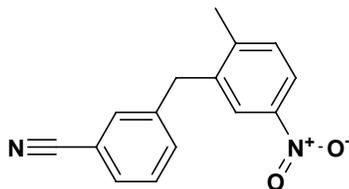
**Table 1 Entry 13. 3-(2-Methyl-5-nitro-benzyl)-benzoic acid.**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  2.36 (s, 3H), 4.14 (s, 2H), 7.36 (d, 8.3 Hz, 1H), 7.37 (d, 7.26 Hz, 1H), 7.44 (t, 7.78 Hz, 1H), 7.90 (s, 1H), 8.00-8.02 (m, 2H), 8.05-8.07 (m, 1H). LC/MS 2.6 min. ( $\text{M}^-$ ) 270.2. Lu, F.; Chi, S.; Kim, D.; Han, K.; Kuntz, I. D.; Guy, R. K. *J. Comb. Chem.* **2006**, 8, 315.



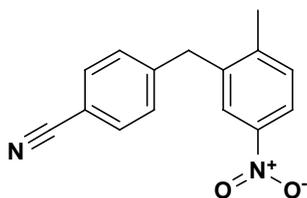
**Table 1 Entry 14. 2-(4-(3-Nitrobenzyl)phenyl)acetic acid.**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  3.65 (s, 2H), 4.08 (s, 2H), 7.17 (d, 8.3 Hz, 2H), 7.25 (d, 8.3 Hz, 2H), 7.47 (t, 7.8 Hz, 1H), 7.52 (d, 7.8 Hz, 1H), 8.08 (m, 2H).  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  40.7, 41.3, 121.6, 123.9, 129.4, 129.6, 130.0, 131.9, 135.3, 138.8, 143.1, 148.5, 177.5. LC/MS 2.3 min. ( $\text{M}^+$ ) 270.2. FAB HRMS: Calc'd for  $\text{C}_{16}\text{H}_{15}\text{NO}_4$  ( $\text{M}^+$ ) 272.0923, Found 272.0934.



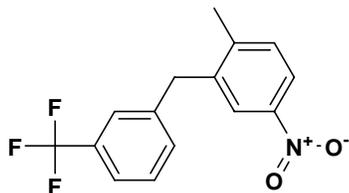
**Table 1 Entry 15. 2-(2-Methyl-5-nitro-benzyl)-benzonitrile.**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  2.36 (s, 3H), 4.26 (s, 2H), 7.06 (d, 7.9 Hz, 1H), 7.36 (m, 2H), 7.50 (dt, 1.6 Hz, 7.9 Hz, 1H), 7.70 (dd, 7.5 Hz, 1.25 Hz, 1H), 7.84 (d, 1.9 Hz, 1H), 8.04 (dd, 2.5 Hz, 8.3 Hz, 1H).  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  20.2, 37.5, 113.1, 117.8, 122.4, 124.5, 127.6, 129.7, 131.5, 133.4, 138.6, 142.5, 145.0. LC/MS 2.8 min. ( $\text{M}^+$ ) 251.1. FAB HRMS: Calc'd for  $\text{C}_{15}\text{H}_{13}\text{N}_2\text{O}_4$  ( $\text{M}^+$ ) 253.0978, Found 253.0976.



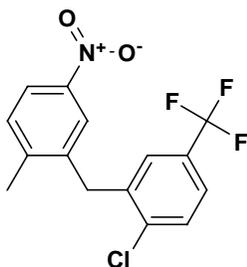
**Table 1 Entry 16. 3-(2-Methyl-5-nitro-benzyl)- benzonitrile.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  2.31 (s, 3H), 4.08 (s, 2H), 7.25-7.36 (m, 3H), 7.40-7.43 (m, 1H), 7.53 (d, 7.89 Hz, 1H), 7.96 (d, 2.08 Hz, 1H), 8.05 (dd, 8.31 Hz, 2.49 Hz, 1H).  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  19.7, 38.1, 112.8, 117.8, 122.4, 123.6, 127.9, 129.2, 132.1, 133.9, 135.4, 138.0, 142.3, 145.6. LC/MS 2.7 min. ( $\text{M}^+$ ) 251.3. FAB HRMS: Calc'd for  $\text{C}_{15}\text{H}_{13}\text{N}_2\text{O}_4$  ( $\text{M}^+$ ) 253.0978, Found 253.0989.



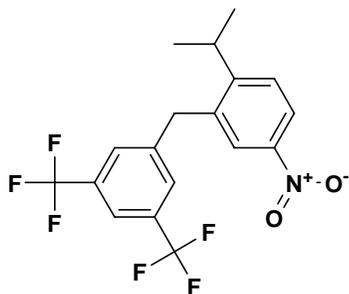
**Table 1 Entry 17. 4-(2-Methyl-5-nitro-benzyl)-benzonitrile.**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  2.33 (s, 3H), 4.14 (s, 2H), 7.24 (d, 8.30 Hz, 2H), 7.37 (d, 8.30 Hz, 1H), 7.62 (d, 8.30 Hz, 2H), 8.00 (s, 1H), 8.08 (d, 8.30 Hz, 1H).  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  20.2, 39.6, 110.9, 118.9, 122.4, 124.9, 124.9, 129.6, 131.6, 132.8, 139.1, 144.4, 144.9. LC/MS 2.8 min. ( $\text{M}^+$ ) 251.3. FAB HRMS: Calc'd for  $\text{C}_{15}\text{H}_{13}\text{N}_2\text{O}_4$  ( $\text{M}^+$ ) 253.0978, Found 253.0995.



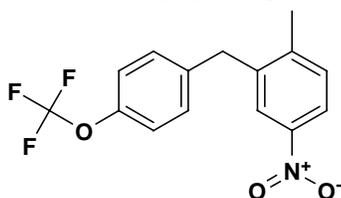
**Table 1 Entry 18. 1-Trifluoromethyl-3-(2-methyl-5-nitro-benzyl)-benzene.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  2.32 (s, 3H), 4.11 (s, 2H), 7.25 (s, 1H), 7.32-7.37 (m, 2H), 7.41 (t, 7.89 Hz, 1H), 7.50 (d, 7.89 Hz, 1H), 7.97 (d, 2.49 Hz, 1H), 8.04 (dd, 8.31 Hz, 2.49 Hz, 1H).  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  20.3, 39.5, 112.9, 119.9, 120.4, 122.5, 123.7, 125.5, 127.9, 129.8, 130.6, 135.4, 142.3, 145.3, 146.9, 147.9. FAB HRMS: Calc'd for  $\text{C}_{15}\text{H}_{13}\text{N}_2\text{O}_4$  ( $\text{M}^+$ ) 296.0898, Found 296.0904.



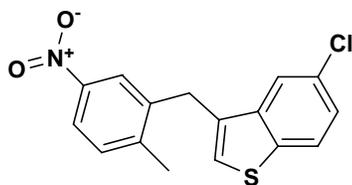
**Table 1 Entry 19. 1-Chloro-2-(2-Methyl-5-nitro-benzyl)-4-trifluoromethylbenzene.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  2.36 (s, 3H), 4.16 (s, 2H), 7.18 (d, 1.25 Hz, 1H), 7.37 (d, 8.31 Hz, 1H), 7.48-7.57 (m, 2H), 7.80 (d, 2.49 Hz, 1H), 8.06 (dd, 8.31 Hz, 2.49 Hz, 1H).  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  19.3, 40.3, 113.5, 119.1, 120.6, 122.5, 124.9, 126.9, 128.0, 130.1, 130.4, 130.5, 134.9, 138.9 140.4, 146.9, 147.7. Anal. Calc'd for  $\text{C}_{15}\text{H}_{11}\text{ClF}_3\text{NO}_2$ : C, 54.64; H, 3.36; N, 4.25; Found C, 54.77; H, 3.32; N, 4.28.



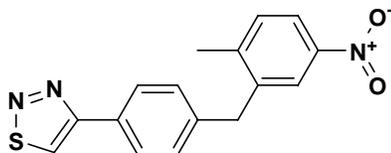
**Table 1 Entry 20. 1,3-Bistrifluoromethyl-5-(2-isopropyl-5-nitro-benzyl)-benzene.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  1.16 (d, 7.06 Hz, 6H), 3.05 (sept., 7.06 Hz, 1H), 4.25 (s, 2H), 7.49-7.52 (m, 3H), 7.75 (s, 1H), 7.99 (d, 2.49 Hz, 1H), 8.16 (dd, 8.72 Hz, 2.49 Hz, 1H).  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  19.2, 20.1, 40.8, 119.9, 121.7, 122.1, 124.3, 128.4, 129.0, 129.3, 131.9, 132.0, 132.2, 132.5, 136.2, 140.4.  $\text{C}_{18}\text{H}_{15}\text{F}_6\text{NO}_2$  Anal. Calc'd for C, 55.25; H, 3.86; N, 3.58; Found C, 55.20; H, 3.83; N, 3.60



**Table 1 Entry 21. 1-Trifluoromethoxy-4-(2-methyl-5-nitro-benzyl)-benzene.**  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  2.35 (s, 3H), 4.08 (s, 2H), 7.14 (d, 8.81 Hz, 2H), 7.17 (d, 8.81 Hz, 2H), 7.35 (d, 8.29 Hz, 1H), 8.01 (d, 2.59 Hz, 1H), 8.06 (dd, 8.29 Hz, 2.07 Hz, 1H).  $^{13}\text{C}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  22.3, 40.6, 117.4, 119.5, 121.2, 121.3, 121.5, 123.6, 124.6, 130.4, 130.5, 136.5, 142.1, 145.4, 149.1. LC/MS 3.1 min. (M-1) 310.1.  $\text{C}_{15}\text{H}_{12}\text{F}_3\text{NO}_3$  Anal. Calc'd for C, 57.88; H, 3.89; F, N, 4.50; Found C, C, 57.92; H, 3.87; N, 4.50.



**Table 1 Entry 22. 5-Chloro-3-(2-methyl-5-nitro-benzyl)-benzo[b]thiophene.**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  2.36 (s, 3H), 4.12 (s, 2H), 6.80 (s, 1H), 7.32 (d, 8.31 Hz, 1H), 7.35 (d, 8.31 Hz, 1H), 7.66 (s, 1H), 7.75 (d, 8.72 Hz, 1H), 7.94 (s, 1H), 8.03 (d, 8.31 Hz, 1H).  $^{13}\text{C}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  20.0, 32.5, 121.3, 122.2, 124.2, 124.5, 125.3, 125.4, 130.8, 131.4, 133.0, 138.9, 139.0, 139.8, 144.9, 146.8. LC/MS 3.3 min. FAB HRMS: Calc'd for  $\text{C}_{15}\text{H}_{13}\text{N}_2\text{O}_4$  ( $\text{M}^{+1}$ ) 317.0277, Found 317.0268



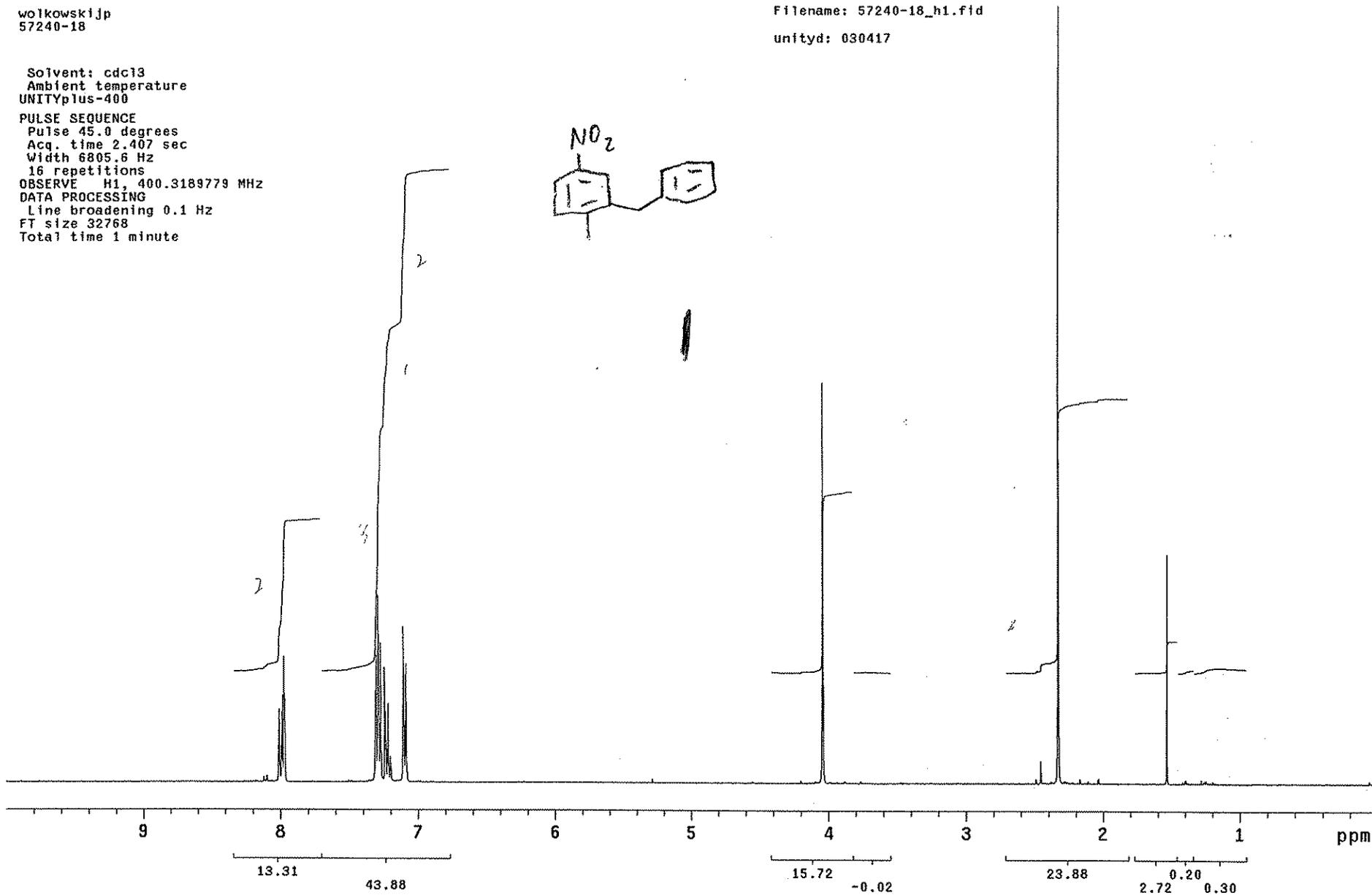
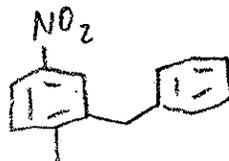
**Table 1 Entry 23. 4-[4-(2-Methyl-5-nitro-benzyl)-phenyl]-[1,2,3]thiadiazole.**  $^1\text{H}$  NMR (400 MHz, DMSO)  $\delta$  2.34 (s, 3H), 4.16 (s, 2H), 7.34 (d, 8.31 Hz, 2H), 7.46 (d, 7.48 Hz, 1H), 8.00-8.02 (m, 2H), 8.06 (d, 8.31 Hz, 2H), 9.56 (s, 1H).  $^{13}\text{C}$  NMR (500 MHz, DMSO)  $\delta$  20.2, 38.5, 122.1, 124.6, 128.0, 129.5, 130.3, 132.0, 133.6, 141.0, 141.6, 145.9, 146.6, 162.3. LC/MS 2.9 min (M+1) 312.3. FAB HRMS: Calc'd for  $\text{C}_{15}\text{H}_{13}\text{N}_2\text{O}_4$  ( $\text{M}^{+1}$ ) 311.0728, Found 311.0739.

wolkowski.jp  
57240-18

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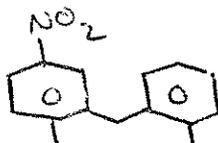


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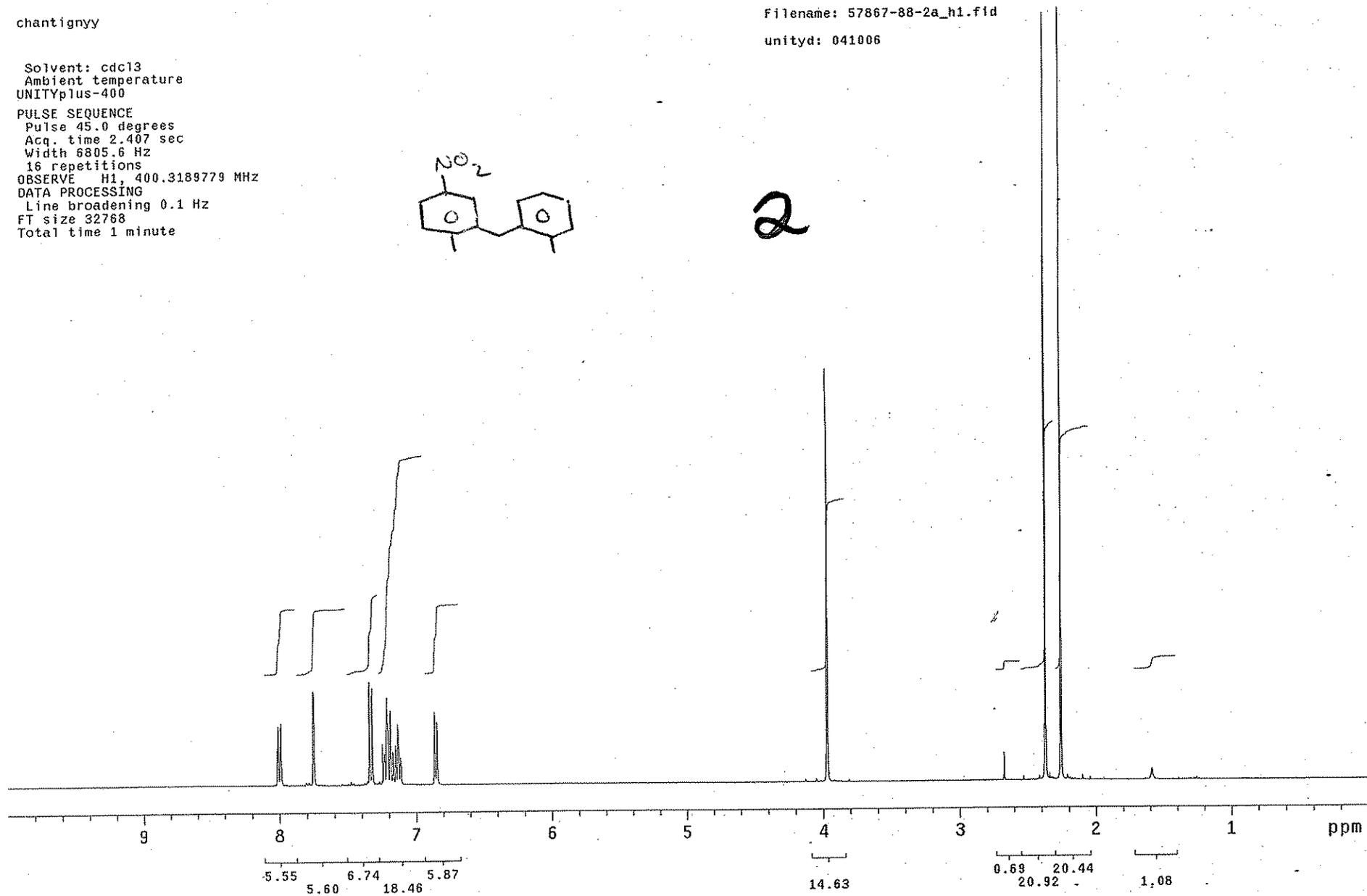
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2



57240-15

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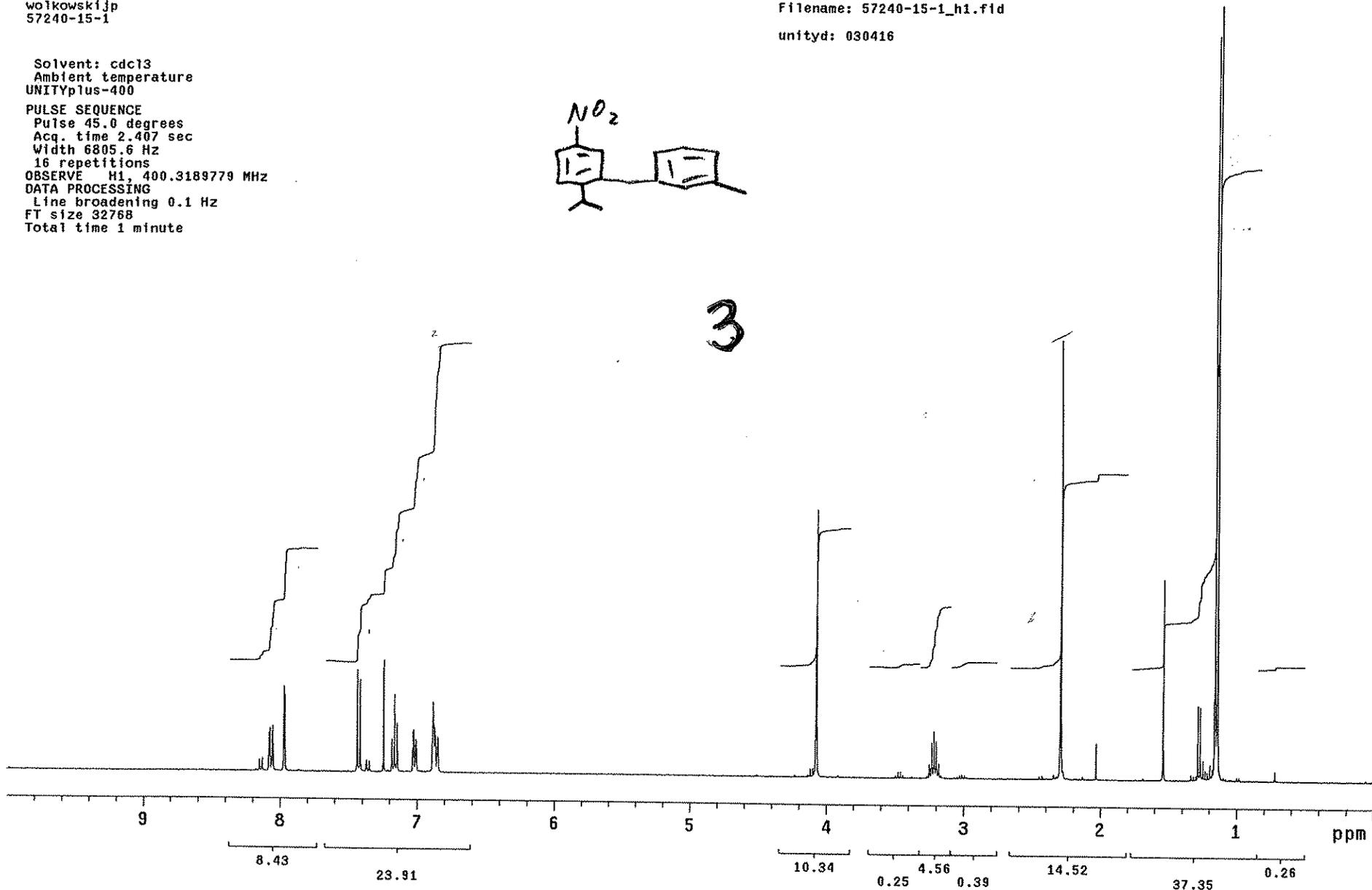
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3



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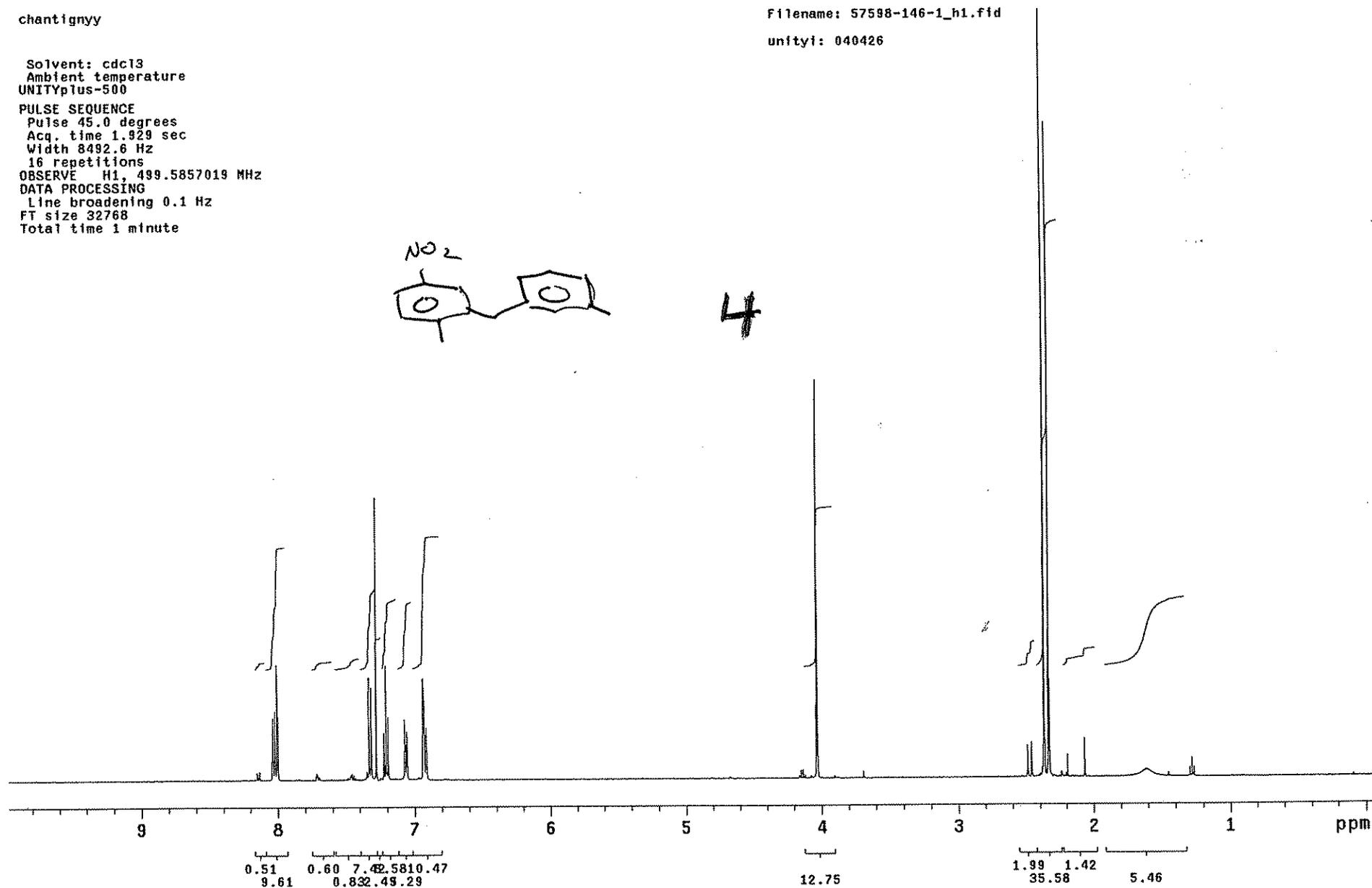
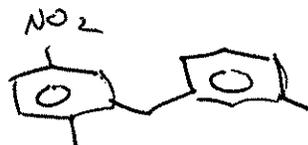
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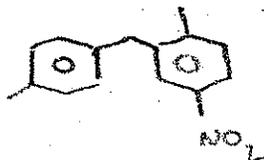
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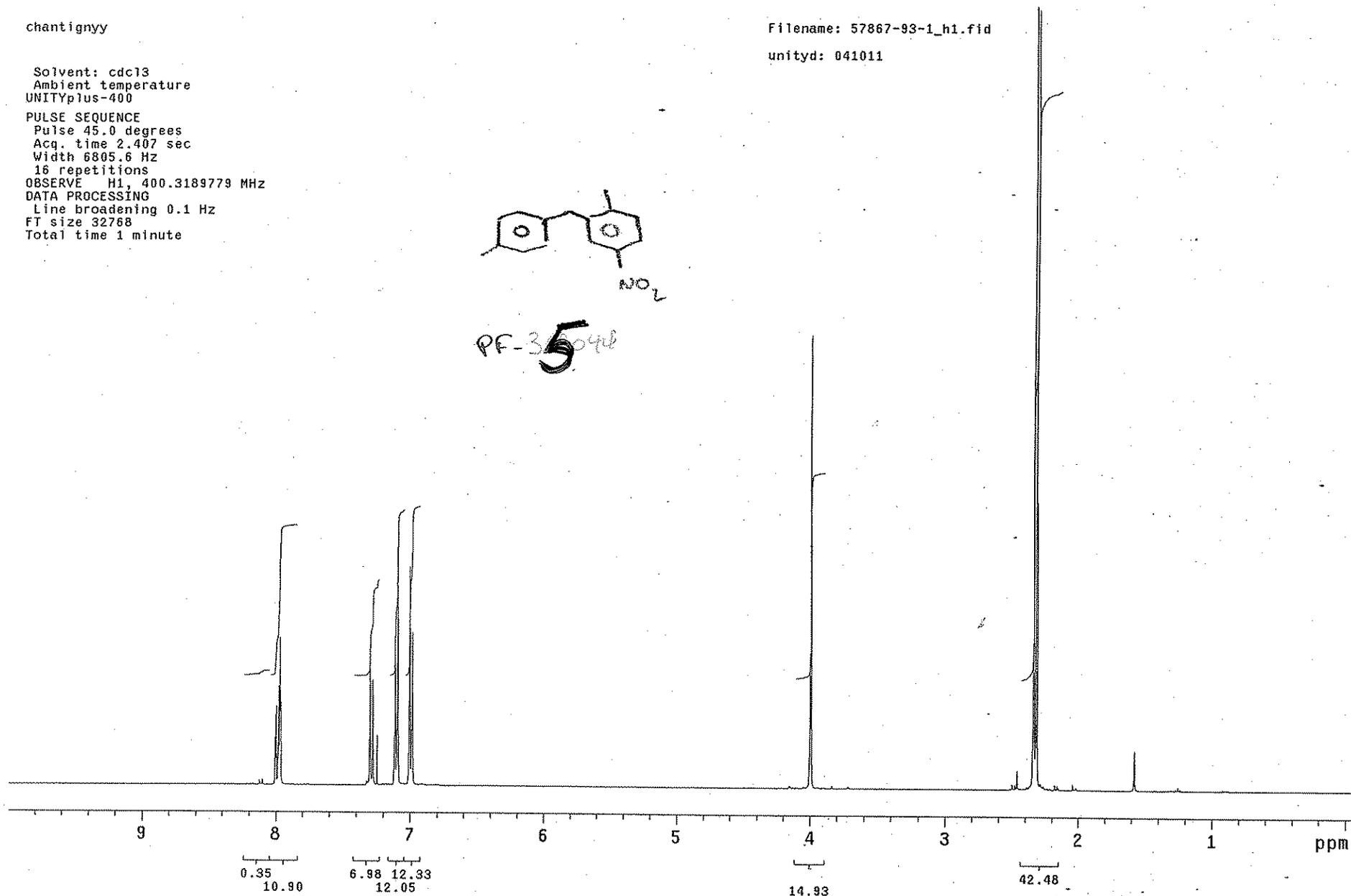
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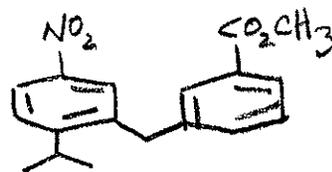
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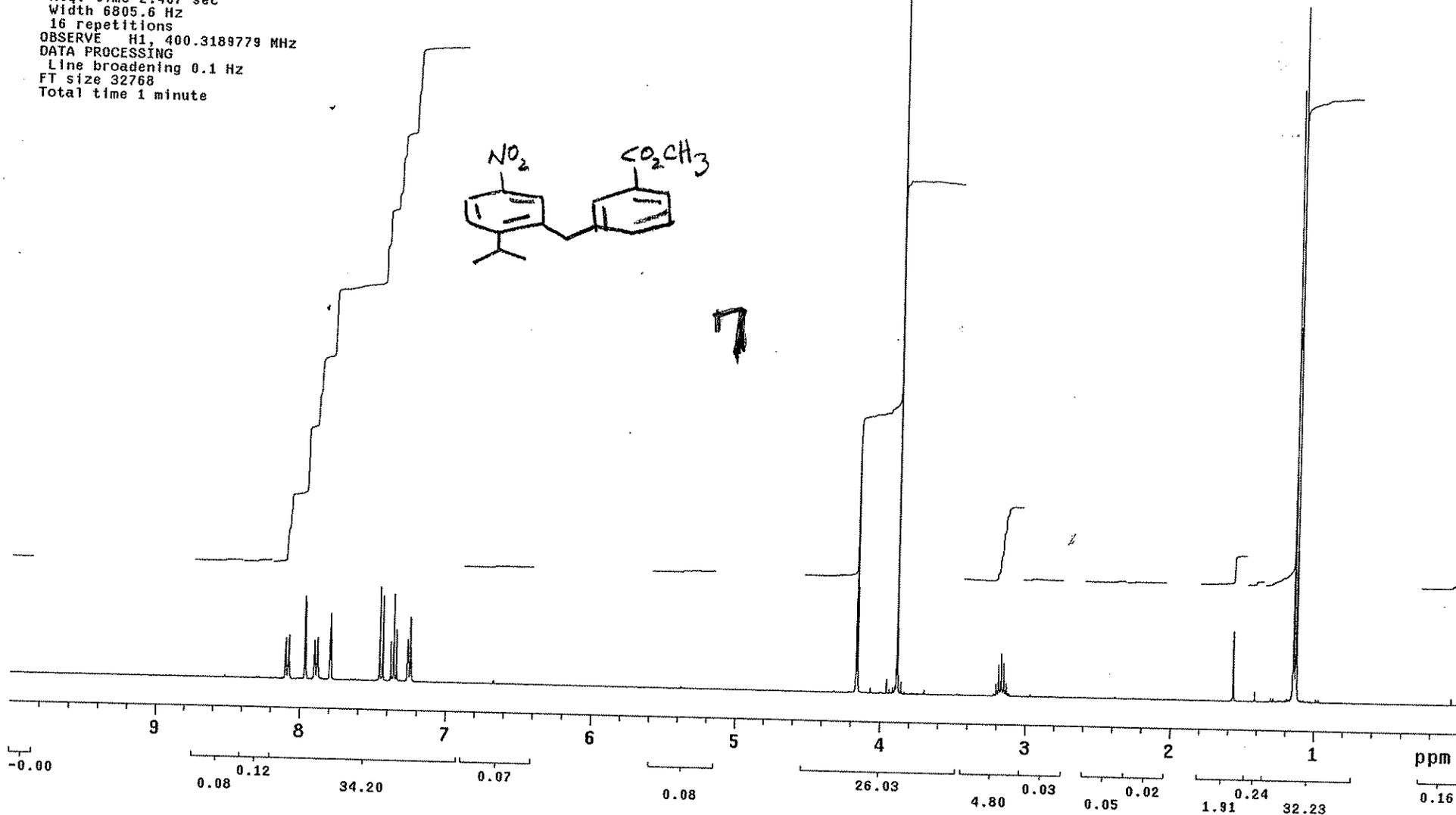
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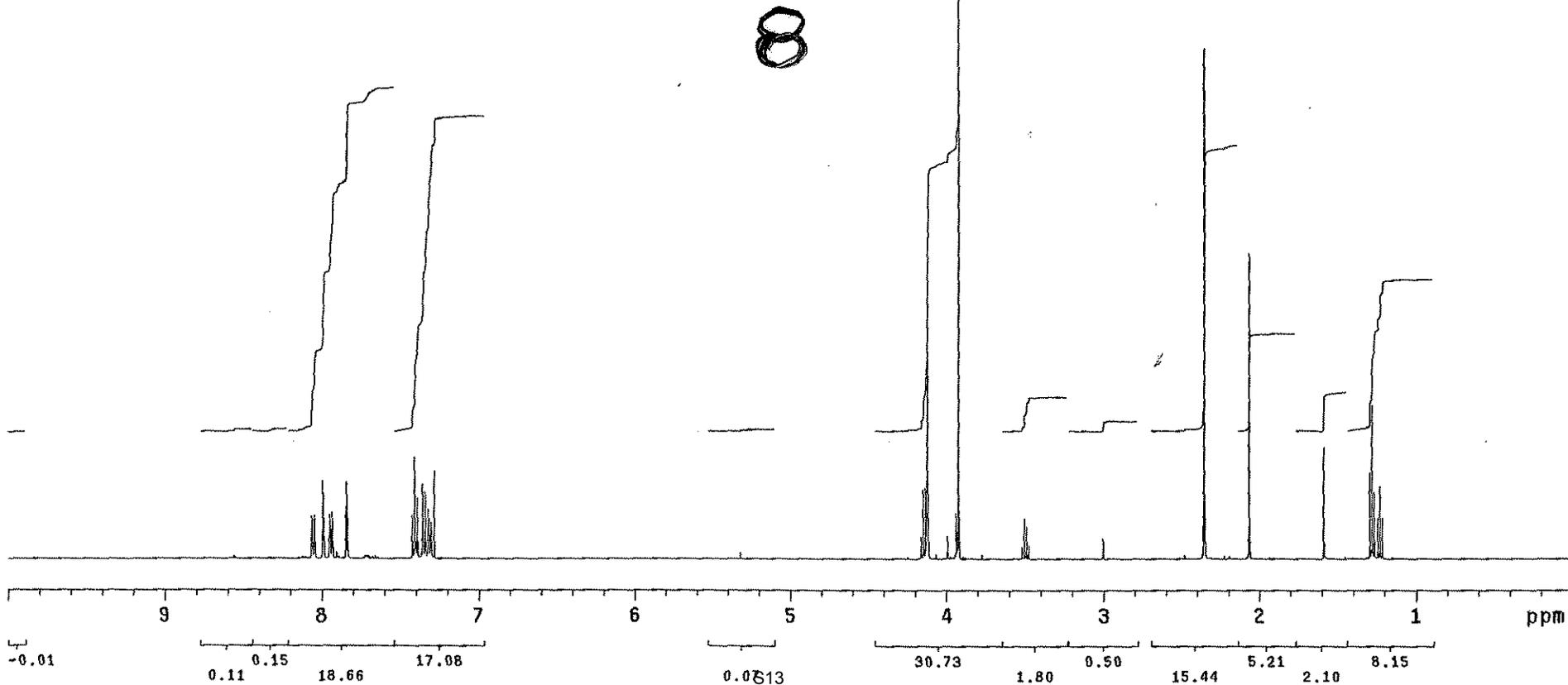
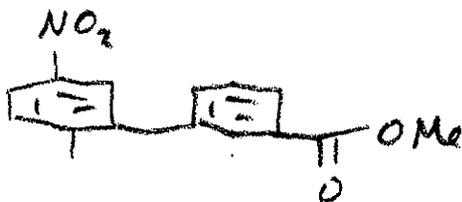


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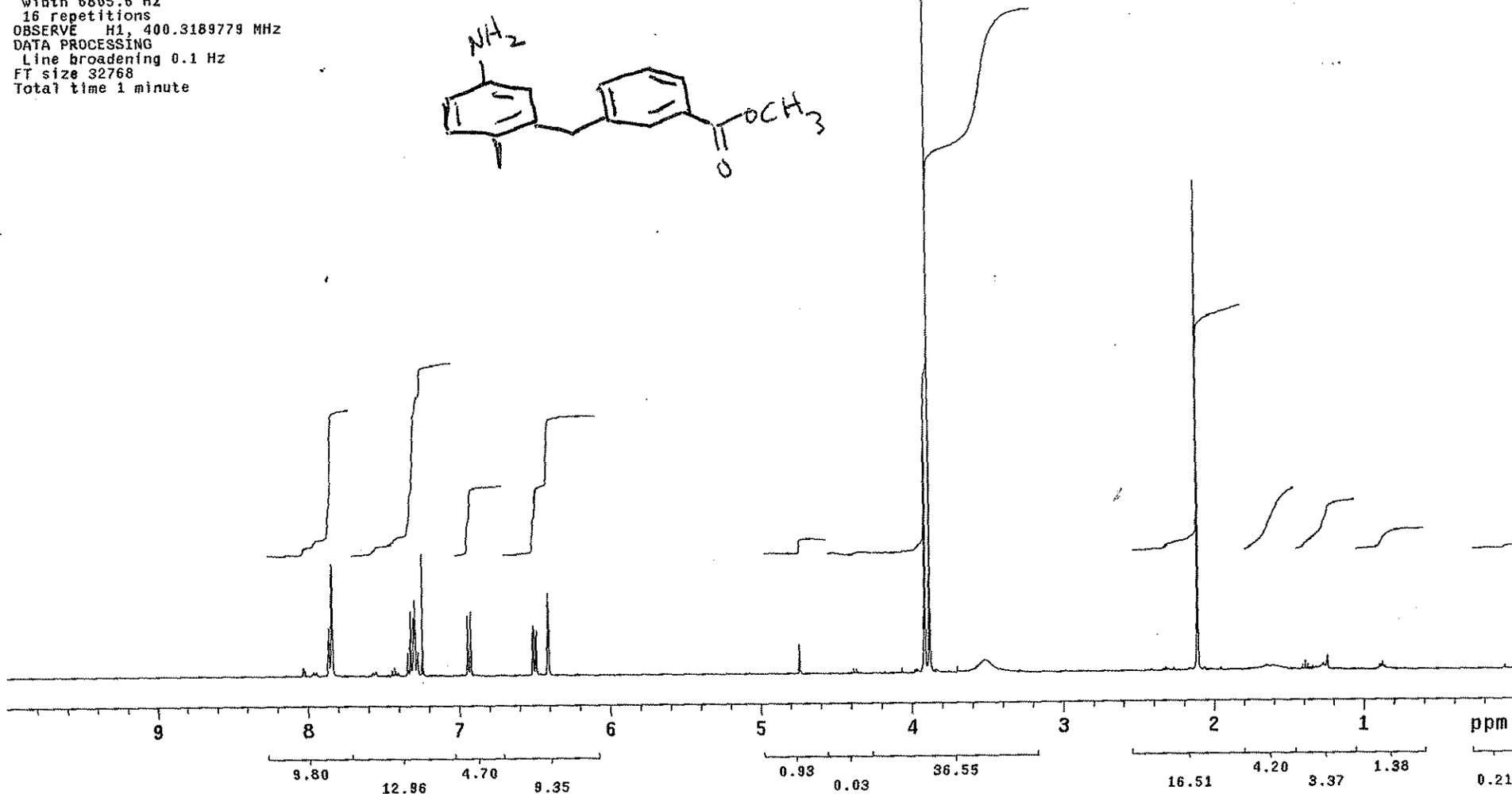
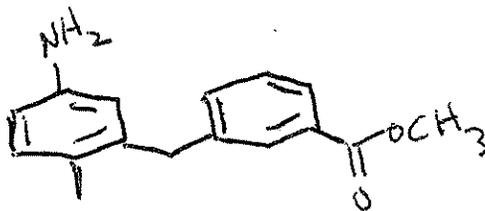


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9



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57240-108

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Ambient temperature  
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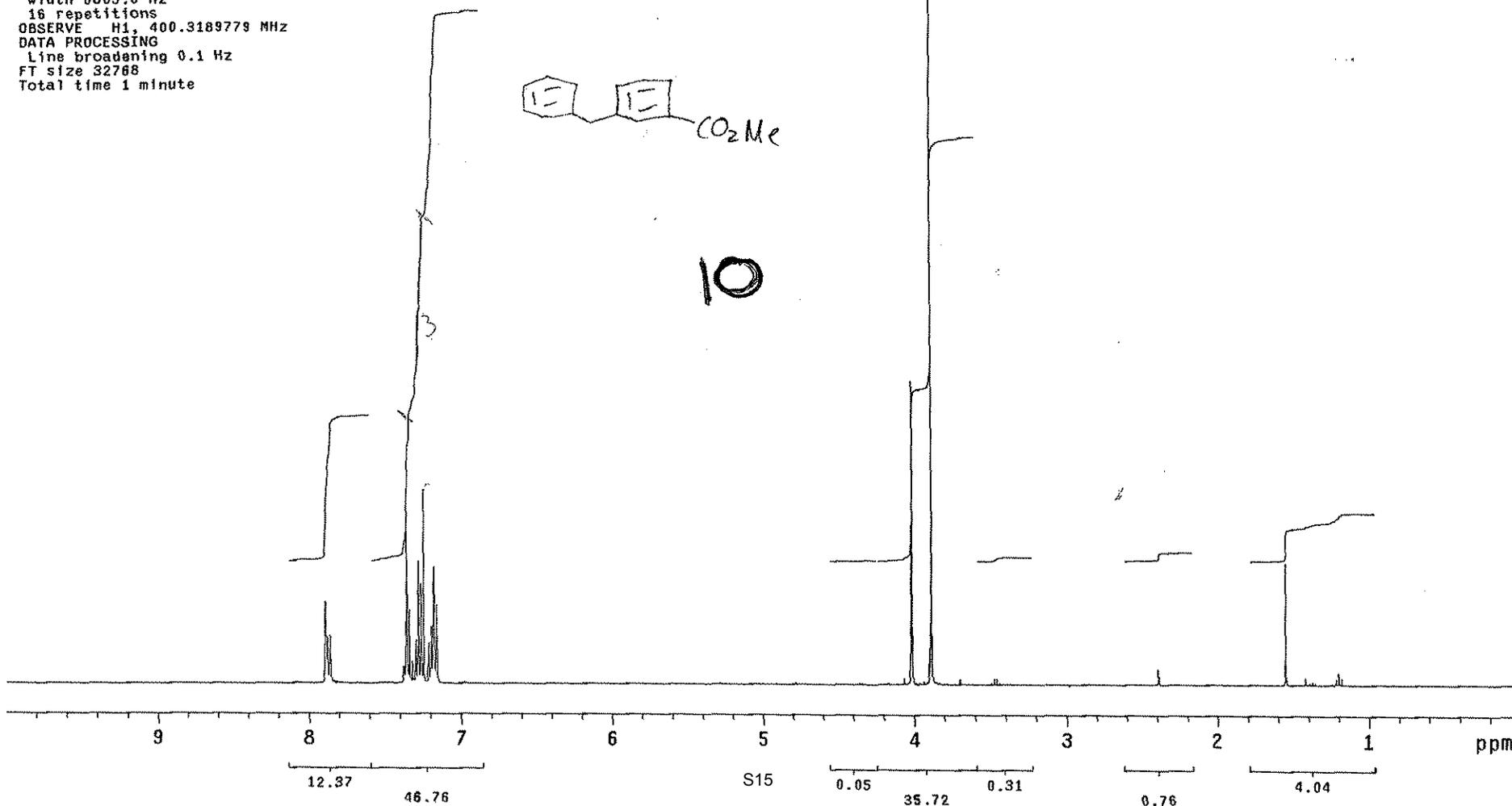
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10



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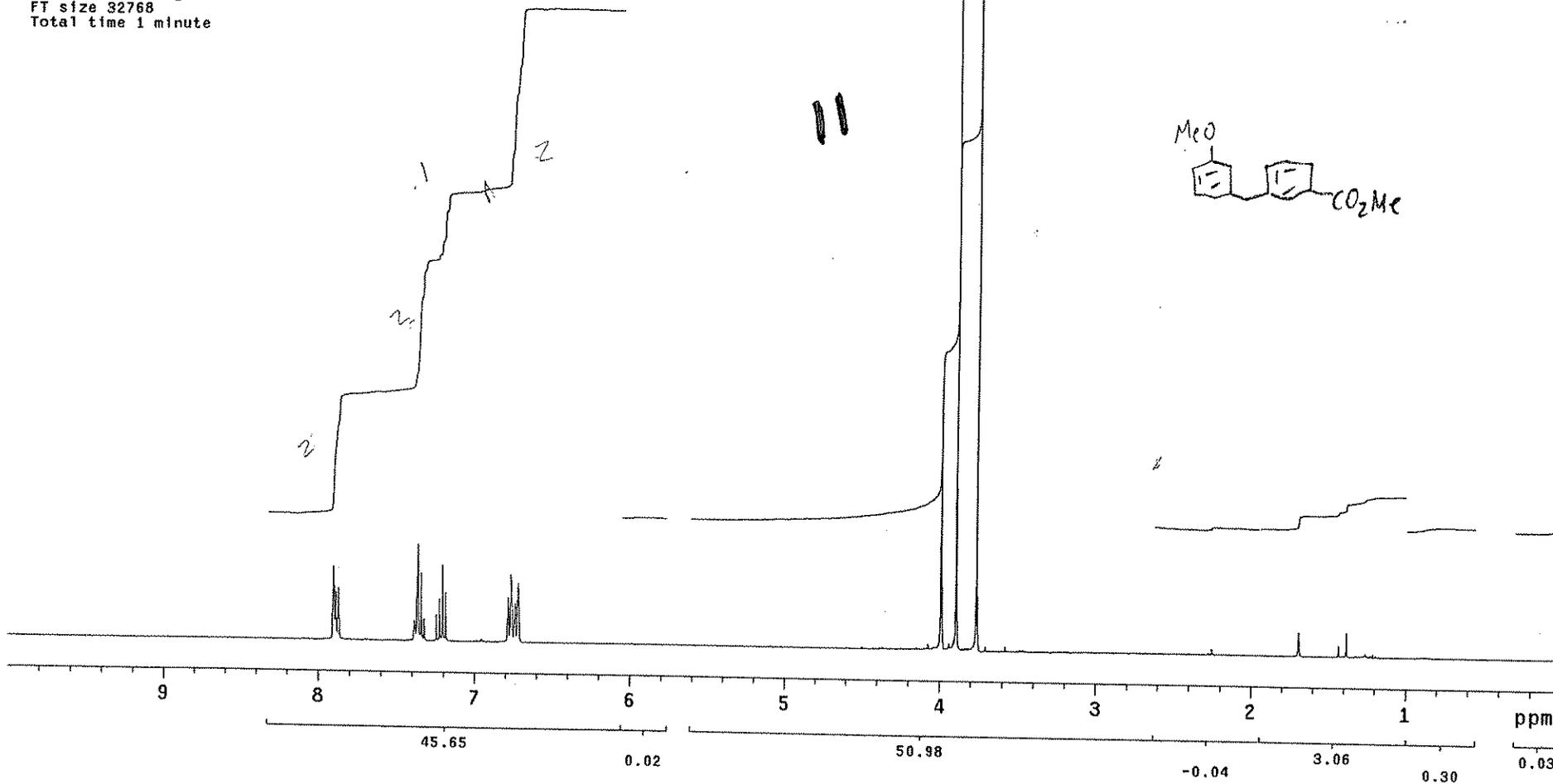
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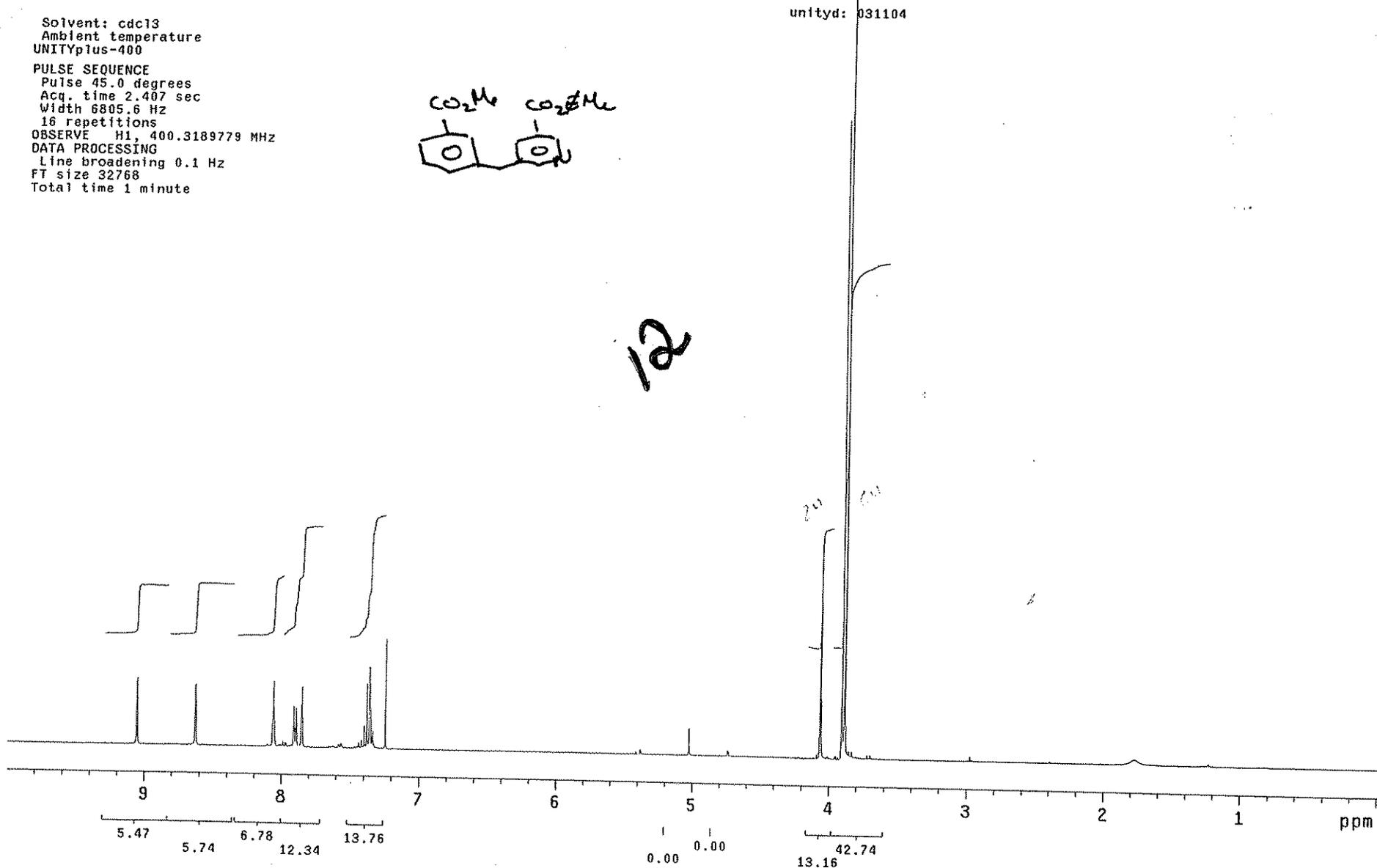
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12



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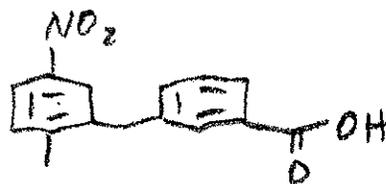
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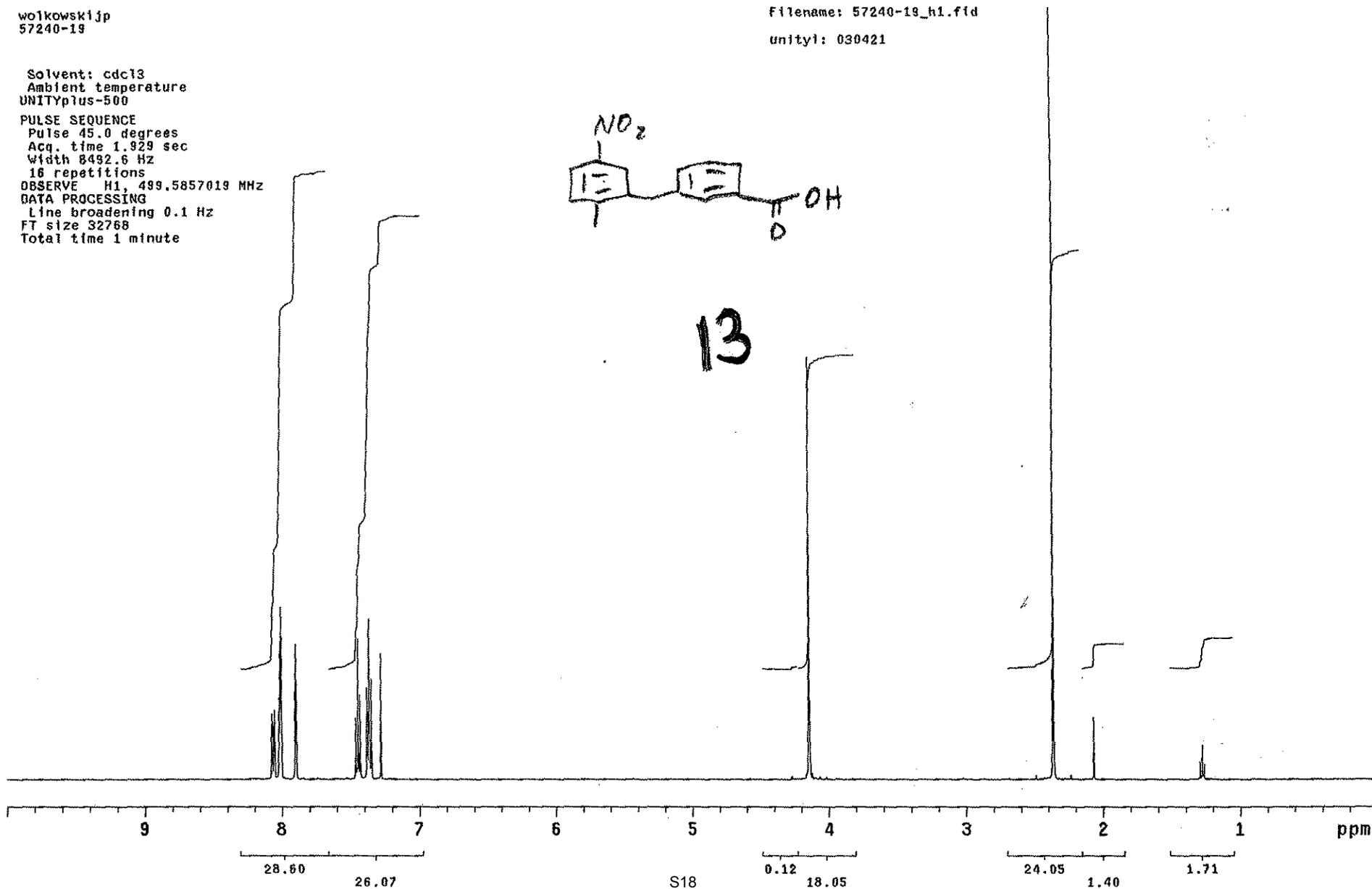
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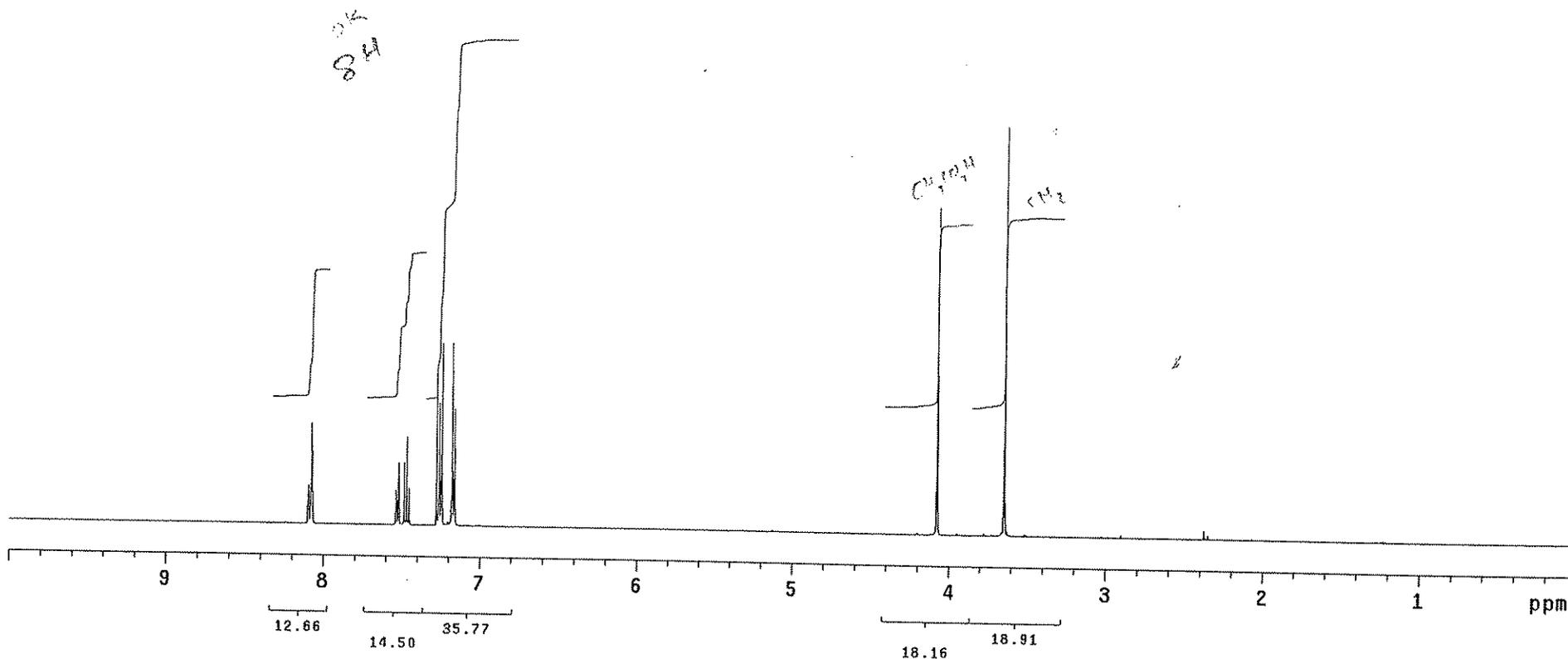
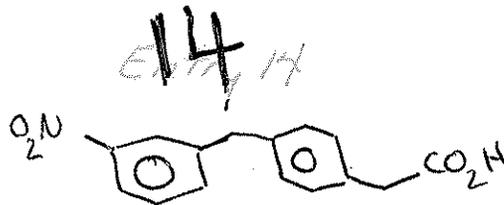
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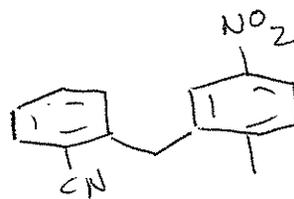


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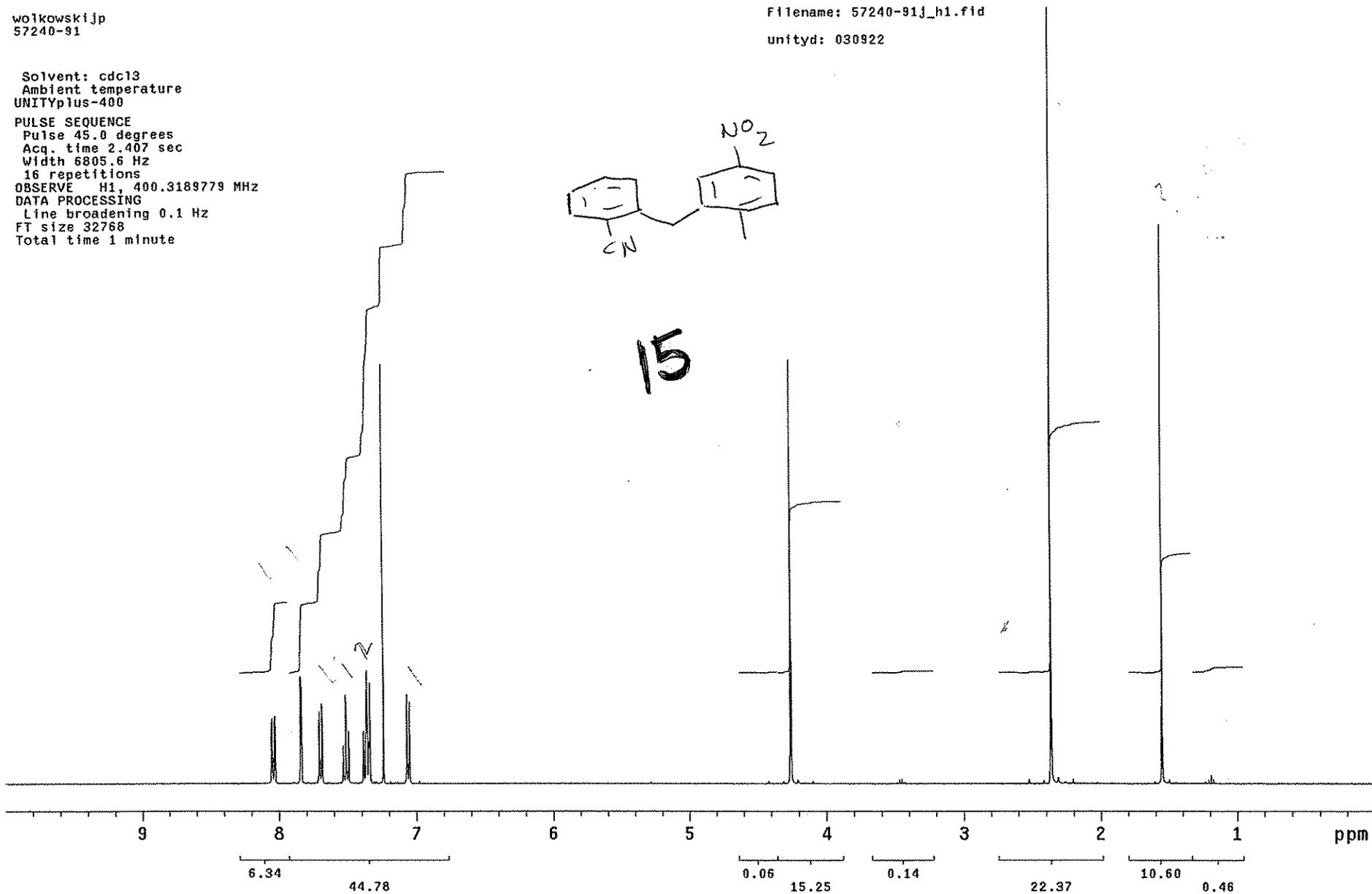
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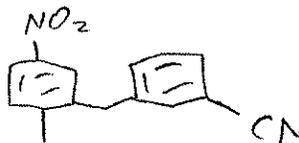
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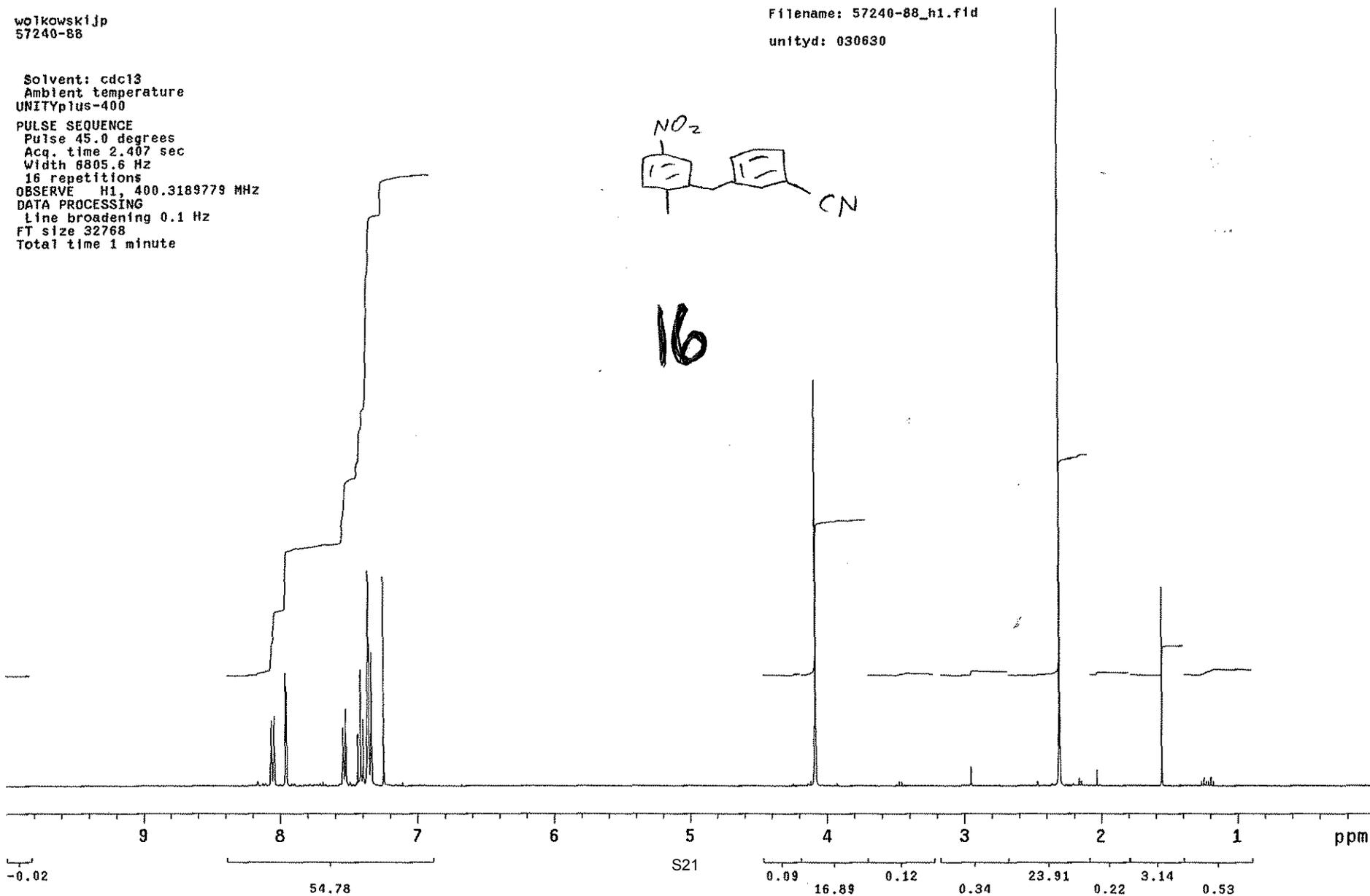
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16



wolkowski.jp  
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DATA PROCESSING

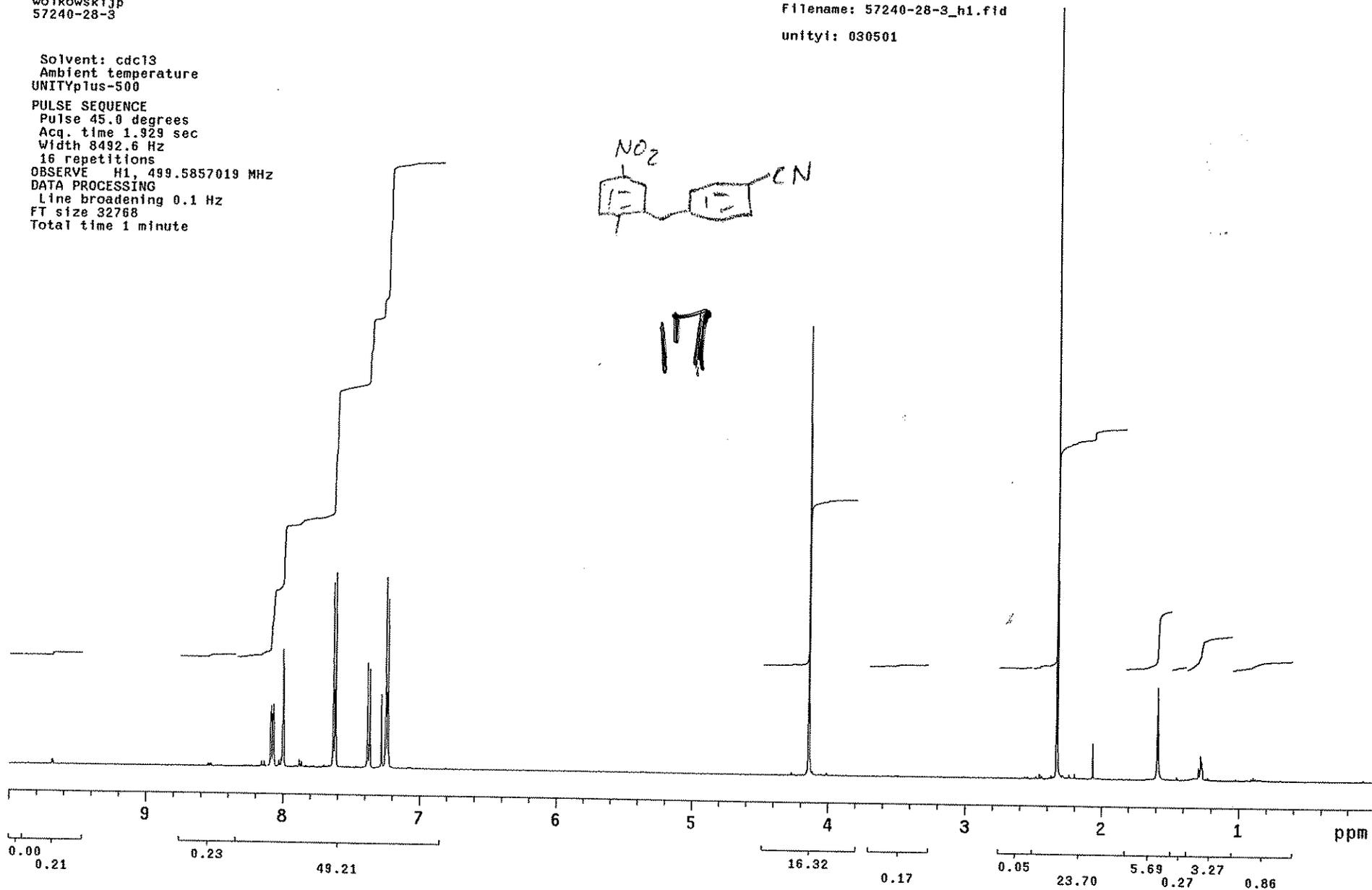
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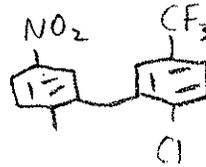
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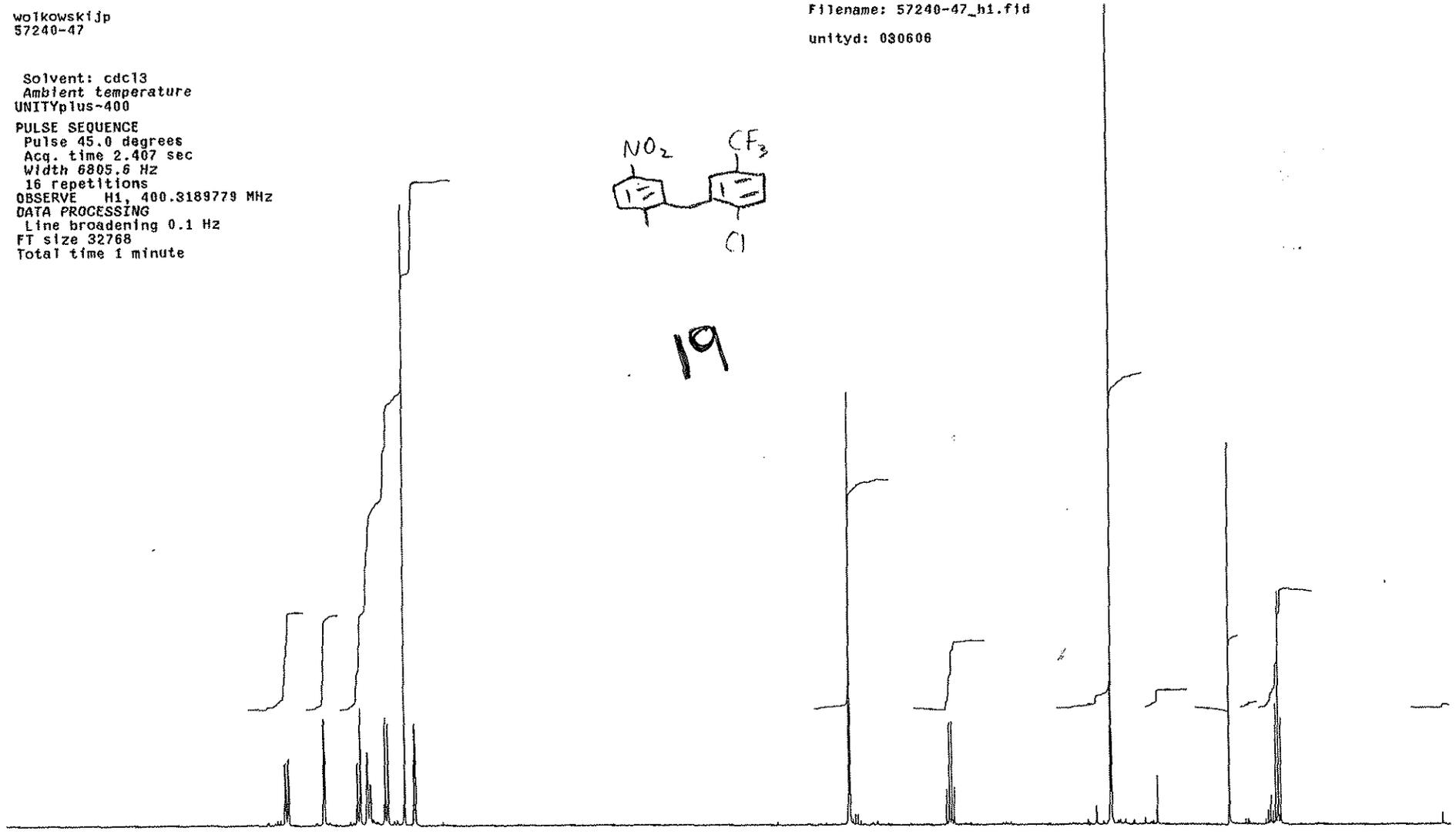
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19



9

8

7

6

5

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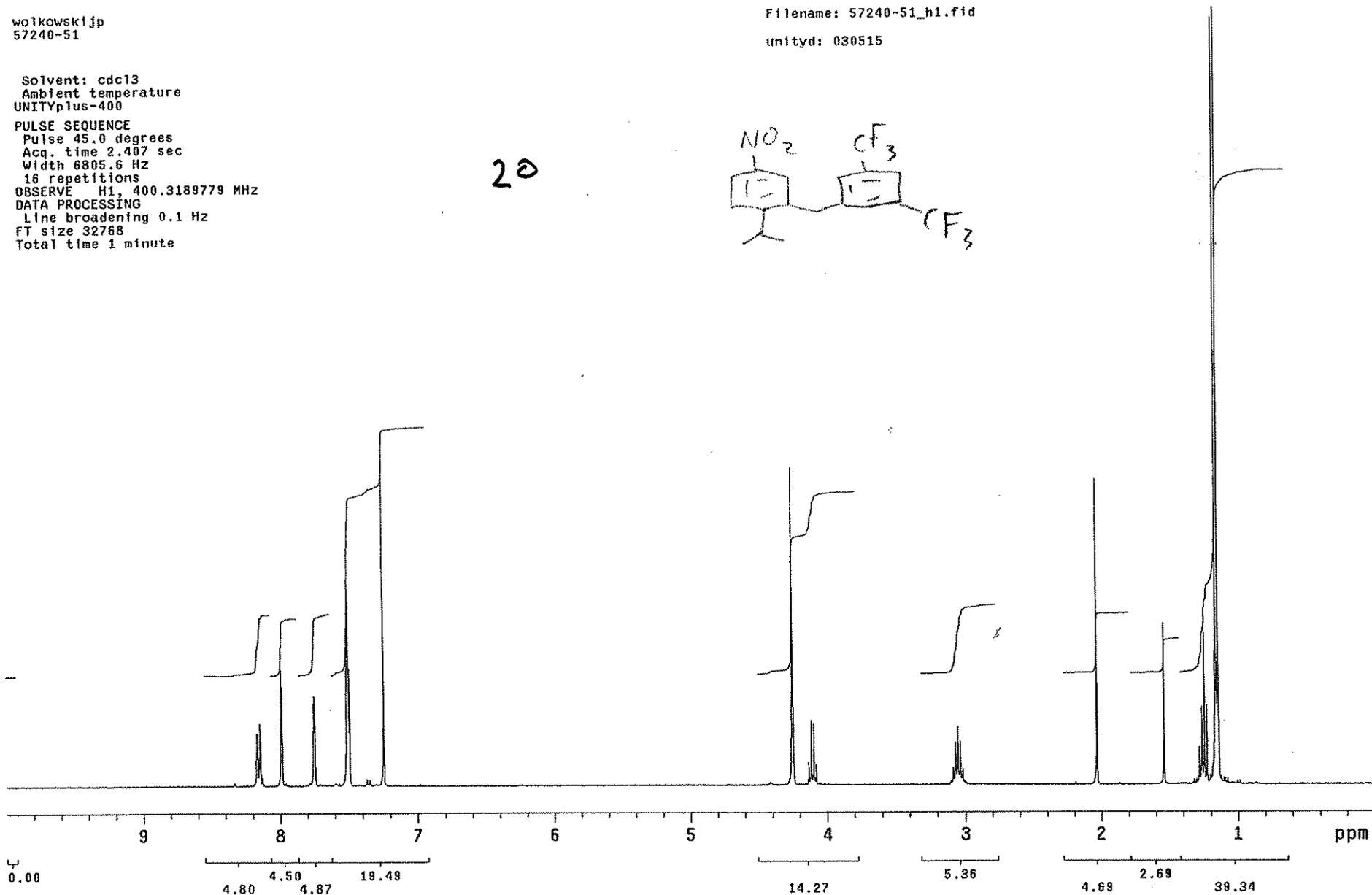
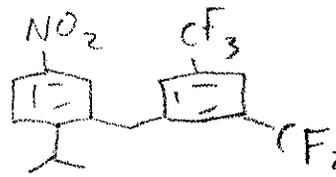
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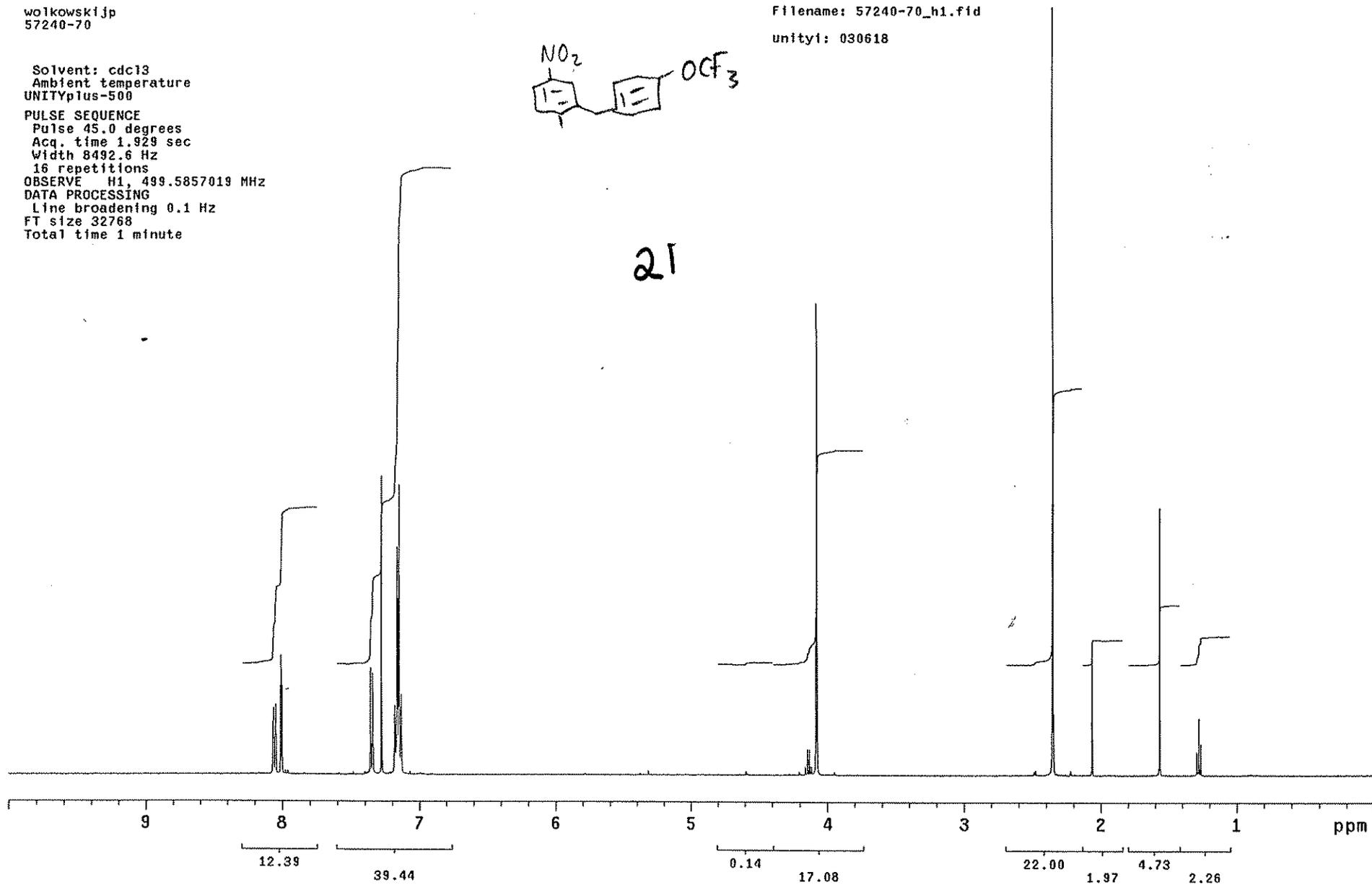
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21

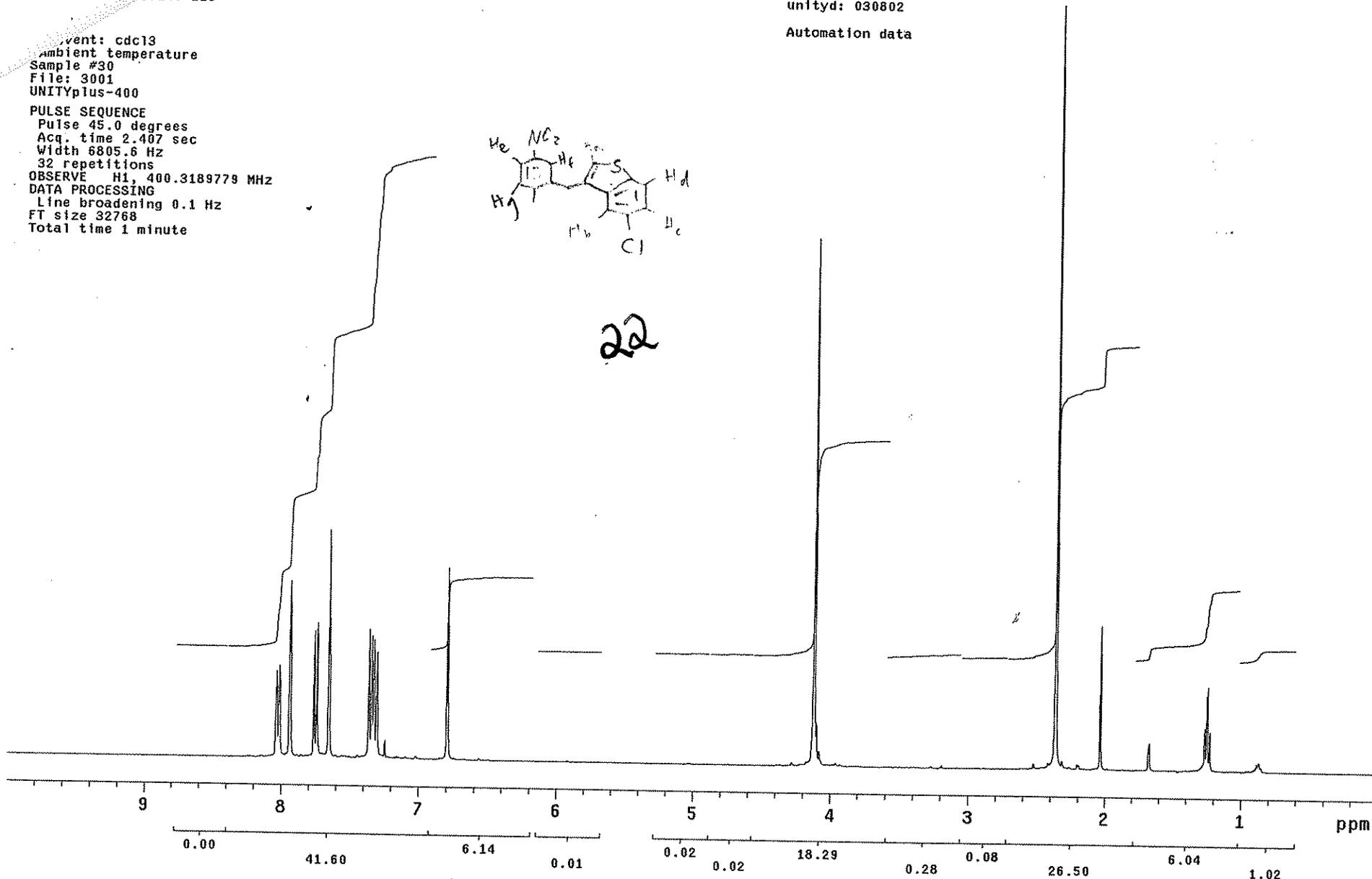
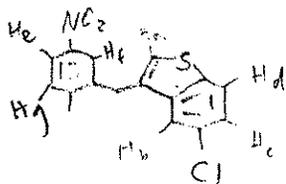


s III  
d III

87240-128

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File: 3001  
UNITYplus-400  
PULSE SEQUENCE  
Pulse 45.0 degrees  
Acq. time 2.407 sec  
Width 6805.6 Hz  
32 repetitions  
OBSERVE H1, 400.3189779 MHz  
DATA PROCESSING  
Line broadening 0.1 Hz  
FT size 32768  
Total time 1 minute





GLOBAL RESEARCH & DEVELOPMENT  
GROTON, CT 06340

**X-RAY CRYSTALLOGRAPHY**

JON BORDNER    IVAN SAMARDJIEV

860-441-4510

860-715-5846

**PFIZER ID NUMBER:**

PF-00733741

57598-35/57240-112-3

**X-RAY CODE:**

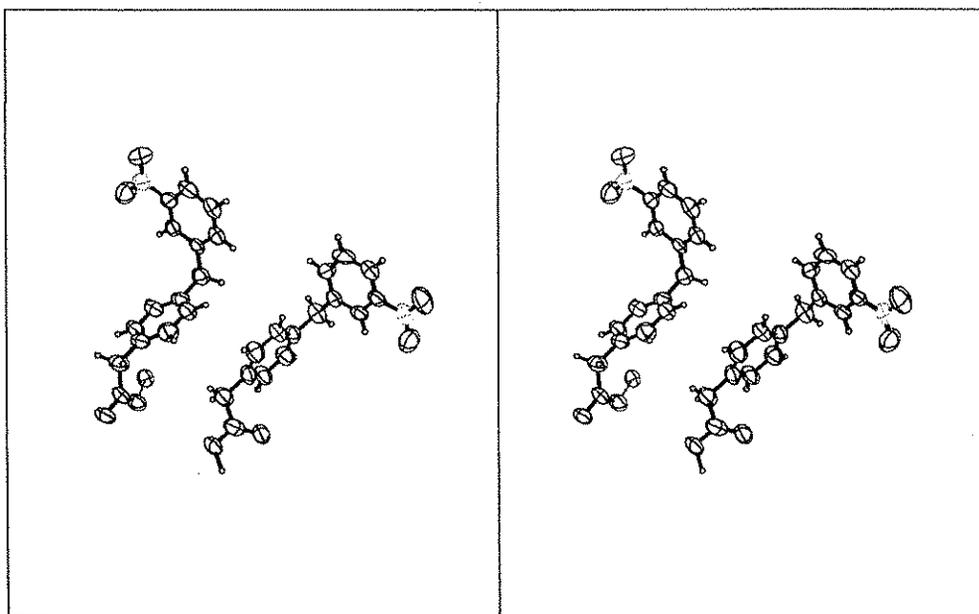
I212

**INVESTIGATOR(S):**

YVES A. CHANTIGNY

**DATE:**

January 13, 2004



POTASSIUM SALT AND FREE ACID

ADDITIONAL DRAWING(S) ON FOLLOWING PAGE(S)

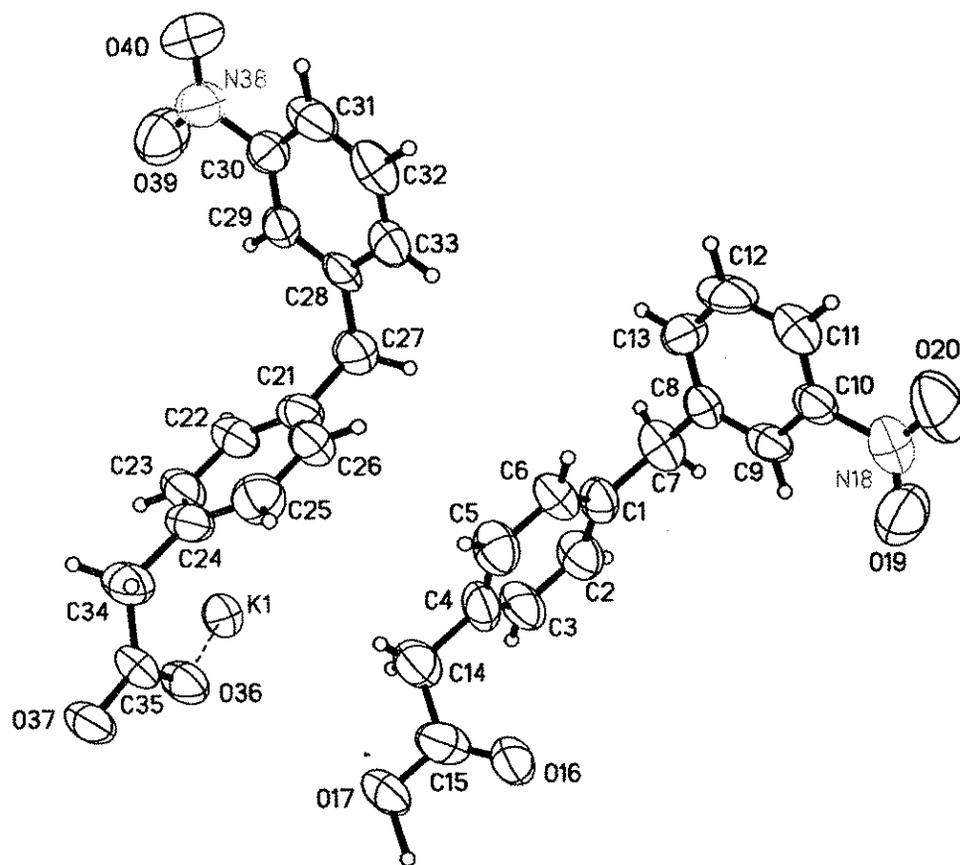


Table 1. Crystal data and structure refinement for PF-00733741.

Identification code	I212	
Empirical formula	$C_{15}H_{13}NO_4$ $C_{15}H_{12}NO_4^- K^+$	
Formula weight	580.62	
Temperature	298(2) K	
Wavelength	1.54178 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	$a = 7.2357(8)$ Å	$\alpha = 95.823(4)^\circ$ .
	$b = 8.3135(9)$ Å	$\beta = 97.965(4)^\circ$ .
	$c = 23.305(3)$ Å	$\gamma = 93.529(4)^\circ$ .
Volume	$1377.1(3)$ Å <sup>3</sup>	
Z	2	
Density (calculated)	1.400 Mg/m <sup>3</sup>	
Absorption coefficient	2.163 mm <sup>-1</sup>	
F(000)	604	
Crystal size	0.12 x 0.04 x 0.02 mm <sup>3</sup>	
Reflections collected	2942	
Independent reflections	2117 [R(int) = 0.2183]	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F <sup>2</sup>	
Data / restraints / parameters	2117 / 0 / 371	
Goodness-of-fit on F <sup>2</sup>	0.944	
Final R indices [I > 2σ(I)]	R1 = 0.0658, wR2 = 0.1569	
Extinction coefficient	0.0063(11)	
Largest diff. peak and hole	0.236 and -0.308 e.Å <sup>-3</sup>	



## Experimental

**Single Crystal X-Ray Analysis.** A representative crystal was surveyed and a 1 Å data set (maximum  $\sin \Theta/\lambda=0.5$ ) was collected on a Bruker 2k CCD/R diffractometer. Atomic scattering factors were taken from the International Tables for X-Ray Crystallography<sup>1</sup>. All crystallographic calculations were facilitated by the SHELXTL<sup>2</sup> system. All diffractometer data were collected at room temperature. Pertinent crystal, data collection, and refinement are summarized in Table I.

A trial structure was obtained by direct methods. Examination of the first E maps indicated the crystal contained two organic molecules. One of these molecules was a potassium salt and the other a free acid. This trial structure refined routinely. Hydrogen positions were calculated wherever possible. The hydrogen parameters were added to the structure factor calculations but were not refined. The shifts calculated in the final cycles of least squares refinement were all less than 0.1 of the corresponding standard deviations. The final R-index was 6.58%. A final difference Fourier revealed no missing or misplaced electron density.

The refined structure was plotted using the SHELXTL plotting package (Figure 1). Coordinates, anisotropic temperature factors, distances and angles are available as supplementary material (Tables 1-5).

## References

1. **International Tables for X-Ray Crystallography**, Vol. IV, pp. 55,99,149 Birmingham: Kynoch Press, 1974.
2. **SHELXTL**, Version 5.1, Bruker AXS, 1997.

Table 2. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for PF-00733741.  $U(\text{eq})$  is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

	x	y	z	$U(\text{eq})$
K(1)	8435(2)	11521(2)	-413(1)	67(1)
C(1)	2372(9)	5789(7)	-2246(3)	60(2)
C(2)	2170(10)	5974(9)	-1663(3)	77(2)
C(3)	3712(12)	6413(9)	-1239(3)	76(2)
C(4)	5459(10)	6661(8)	-1374(4)	71(2)
C(5)	5654(10)	6547(9)	-1963(3)	77(2)
C(6)	4123(10)	6088(8)	-2386(3)	69(2)
C(7)	681(9)	5317(9)	-2701(3)	78(2)
C(8)	1057(8)	4472(9)	-3278(3)	59(2)
C(9)	1106(8)	2797(9)	-3337(3)	58(2)
C(10)	1364(8)	2012(9)	-3876(3)	59(2)
C(11)	1567(9)	2799(11)	-4346(3)	71(2)
C(12)	1556(10)	4462(12)	-4286(3)	82(3)
C(13)	1273(9)	5278(9)	-3753(4)	72(2)
C(14)	7106(10)	7108(9)	-908(3)	78(2)
C(15)	7903(11)	5733(11)	-614(3)	75(2)
O(16)	7197(7)	4334(7)	-723(2)	90(2)
O(17)	9412(8)	6110(6)	-246(2)	113(2)
N(18)	1361(8)	222(9)	-3932(3)	79(2)
O(19)	1431(9)	-453(7)	-3496(3)	108(2)
O(20)	1451(8)	-461(7)	-4417(3)	117(2)
C(21)	7605(9)	10907(9)	-2343(3)	63(2)
C(22)	8130(9)	12149(9)	-1892(3)	64(2)
C(23)	9876(10)	12204(9)	-1576(3)	68(2)
C(24)	11182(9)	11104(10)	-1673(3)	61(2)
C(25)	10629(10)	9853(9)	-2121(3)	74(2)
C(26)	8883(10)	9793(9)	-2445(3)	69(2)
C(27)	5768(9)	10831(9)	-2741(3)	72(2)
C(28)	6011(8)	11391(10)	-3328(3)	57(2)
C(29)	6152(8)	13015(9)	-3384(3)	53(2)
C(30)	6413(9)	13491(9)	-3921(3)	63(2)
C(31)	6586(9)	12433(11)	-4400(3)	71(2)
C(32)	6465(10)	10824(11)	-4324(3)	75(2)
C(33)	6185(9)	10292(9)	-3806(4)	70(2)
C(34)	13081(9)	11216(9)	-1318(3)	75(2)
C(35)	13166(10)	10627(8)	-730(3)	56(2)
O(36)	11769(6)	10245(5)	-511(2)	66(1)
O(37)	14847(6)	10521(5)	-473(2)	68(1)
N(38)	6565(8)	15252(9)	-3975(3)	79(2)
O(39)	6560(8)	16196(7)	-3531(3)	98(2)
O(40)	6660(8)	15693(7)	-4448(3)	111(2)

Table 3. Bond lengths [Å] and angles [°] for PF-00733741.

C(1)-C(6)	1.366(9)	C(21)-C(26)	1.375(10)
C(1)-C(2)	1.381(8)	C(21)-C(22)	1.392(9)
C(1)-C(7)	1.508(8)	C(21)-C(27)	1.506(8)
C(2)-C(3)	1.388(9)	C(22)-C(23)	1.367(8)
C(3)-C(4)	1.354(9)	C(23)-C(24)	1.380(10)
C(4)-C(5)	1.392(9)	C(24)-C(25)	1.395(9)
C(4)-C(14)	1.498(9)	C(24)-C(34)	1.494(8)
C(5)-C(6)	1.383(9)	C(25)-C(26)	1.375(8)
C(7)-C(8)	1.519(9)	C(27)-C(28)	1.518(9)
C(8)-C(13)	1.374(9)	C(28)-C(29)	1.368(9)
C(8)-C(9)	1.388(9)	C(28)-C(33)	1.390(9)
C(9)-C(10)	1.396(8)	C(29)-C(30)	1.384(8)
C(10)-C(11)	1.352(9)	C(30)-C(31)	1.373(9)
C(10)-N(18)	1.480(9)	C(30)-N(38)	1.481(9)
C(11)-C(12)	1.376(10)	C(31)-C(32)	1.366(10)
C(12)-C(13)	1.399(10)	C(32)-C(33)	1.364(9)
C(14)-C(15)	1.497(10)	C(34)-C(35)	1.496(8)
C(15)-O(16)	1.229(8)	C(35)-O(36)	1.233(7)
C(15)-O(17)	1.292(8)	C(35)-O(37)	1.291(7)
N(18)-O(19)	1.205(7)	N(38)-O(40)	1.206(7)
N(18)-O(20)	1.225(7)	N(38)-O(39)	1.233(7)
C(6)-C(1)-C(2)	117.6(6)	C(26)-C(21)-C(22)	117.7(6)
C(6)-C(1)-C(7)	122.5(7)	C(26)-C(21)-C(27)	120.0(7)
C(2)-C(1)-C(7)	119.8(6)	C(22)-C(21)-C(27)	122.1(7)
C(1)-C(2)-C(3)	120.5(7)	C(23)-C(22)-C(21)	118.7(7)
C(4)-C(3)-C(2)	122.1(7)	C(22)-C(23)-C(24)	124.5(7)
C(3)-C(4)-C(5)	117.5(6)	C(23)-C(24)-C(25)	116.2(6)
C(3)-C(4)-C(14)	121.1(7)	C(23)-C(24)-C(34)	122.6(7)
C(5)-C(4)-C(14)	121.4(7)	C(25)-C(24)-C(34)	121.2(7)
C(6)-C(5)-C(4)	120.4(6)	C(26)-C(25)-C(24)	119.9(7)
C(1)-C(6)-C(5)	121.8(6)	C(25)-C(26)-C(21)	123.0(7)
C(1)-C(7)-C(8)	116.0(6)	C(21)-C(7)-C(28)	111.8(5)
C(13)-C(8)-C(9)	118.2(6)	C(29)-C(28)-C(33)	119.0(6)
C(13)-C(8)-C(7)	123.1(7)	C(29)-C(28)-C(27)	119.5(6)
C(9)-C(8)-C(7)	118.7(7)	C(33)-C(28)-C(27)	121.5(7)
C(8)-C(9)-C(10)	118.7(6)	C(28)-C(29)-C(30)	118.3(6)
C(11)-C(10)-C(9)	123.4(7)	C(31)-C(30)-C(29)	124.0(7)
C(11)-C(10)-N(18)	119.0(7)	C(31)-C(30)-N(38)	118.4(7)
C(9)-C(10)-N(18)	117.6(7)	C(29)-C(30)-N(38)	117.6(7)
C(10)-C(11)-C(12)	118.2(7)	C(32)-C(31)-C(30)	116.0(7)
C(11)-C(12)-C(13)	119.6(6)	C(31)-C(32)-C(33)	122.3(7)
C(8)-C(13)-C(12)	122.0(7)	C(32)-C(33)-C(28)	120.5(7)
C(4)-C(14)-C(15)	115.9(6)	C(24)-C(34)-C(35)	115.8(5)
O(16)-C(15)-O(17)	122.1(7)	O(36)-C(35)-O(37)	122.5(6)
O(16)-C(15)-C(14)	122.5(7)	O(36)-C(35)-C(34)	123.6(6)
O(17)-C(15)-C(14)	115.5(7)	O(37)-C(35)-C(34)	113.9(6)
O(19)-N(18)-O(20)	124.2(8)	O(40)-N(38)-O(39)	123.3(7)
O(19)-N(18)-C(10)	118.6(7)	O(40)-N(38)-C(30)	118.7(7)
O(20)-N(18)-C(10)	116.9(7)	O(39)-N(38)-C(30)	118.0(7)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for PF-00733741. The anisotropic displacement factor exponent takes the form:  $-2\pi^2 [ h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12} ]$

	$U_{11}$	$U_{22}$	$U_{33}$	$U_{23}$	$U_{13}$	$U_{12}$
K(1)	50(1)	74(1)	72(1)	13(1)	-3(1)	-8(1)
C(1)	42(5)	57(5)	74(6)	-1(4)	-4(4)	1(3)
C(2)	60(5)	102(6)	65(6)	7(4)	11(5)	-16(4)
C(3)	81(6)	94(6)	46(5)	-9(4)	2(5)	-8(5)
C(4)	52(5)	63(5)	87(6)	-3(4)	-13(5)	-6(4)
C(5)	61(5)	92(6)	74(6)	-2(4)	15(5)	-8(4)
C(6)	63(5)	81(5)	55(5)	-7(4)	-10(5)	1(4)
C(7)	65(5)	89(6)	72(5)	-6(4)	0(4)	-1(4)
C(8)	49(4)	65(6)	57(5)	1(4)	-8(4)	-8(4)
C(9)	45(4)	82(6)	43(5)	15(4)	-1(3)	-10(4)
C(10)	50(4)	74(6)	47(5)	10(5)	-9(4)	-8(4)
C(11)	62(5)	87(6)	57(6)	-6(5)	-4(4)	-3(4)
C(12)	76(5)	131(8)	43(5)	36(5)	10(4)	-15(5)
C(13)	70(5)	68(5)	76(6)	30(5)	-6(4)	-8(4)
C(14)	80(6)	79(6)	67(5)	-5(4)	-9(5)	7(4)
C(15)	81(6)	83(6)	58(5)	3(5)	-8(4)	24(5)
O(16)	98(4)	61(4)	95(4)	-6(3)	-35(3)	5(3)
O(17)	99(4)	86(4)	129(5)	18(3)	-71(4)	-3(3)
N(18)	69(4)	79(6)	80(5)	-14(4)	-1(4)	-3(3)
O(19)	146(5)	68(4)	111(5)	8(4)	23(4)	1(3)
O(20)	134(5)	106(5)	95(4)	-39(4)	1(4)	5(4)
C(21)	44(4)	86(6)	61(5)	29(4)	-4(4)	-1(4)
C(22)	58(5)	79(5)	51(5)	8(4)	-4(4)	6(4)
C(23)	59(5)	96(6)	43(4)	11(4)	-5(4)	-15(5)
C(24)	44(5)	89(6)	52(5)	28(4)	2(4)	-1(4)
C(25)	73(6)	71(6)	79(6)	8(5)	7(5)	14(4)
C(26)	60(5)	82(6)	60(5)	10(4)	-9(4)	-8(5)
C(27)	59(5)	85(6)	67(5)	16(4)	-3(4)	-11(4)
C(28)	43(4)	76(6)	44(5)	6(4)	-15(3)	-13(4)
C(29)	50(4)	58(5)	47(5)	1(4)	1(3)	-8(3)
C(30)	47(4)	69(6)	68(6)	20(5)	-9(4)	-4(4)
C(31)	69(5)	94(7)	43(5)	0(5)	-5(4)	1(4)
C(32)	74(5)	78(7)	61(6)	-19(5)	-6(4)	-1(4)
C(33)	64(5)	65(5)	71(6)	5(5)	-20(4)	-8(4)
C(34)	63(5)	111(6)	53(5)	30(4)	2(4)	-9(4)
C(35)	42(5)	74(5)	47(5)	0(4)	-9(4)	5(4)
O(36)	42(3)	98(4)	59(3)	20(2)	-1(2)	-3(2)
O(37)	46(3)	105(4)	51(3)	24(2)	-5(2)	-2(2)
N(38)	67(4)	94(6)	76(5)	29(4)	5(4)	-2(4)
O(39)	119(5)	75(4)	102(4)	8(3)	25(4)	4(3)
O(40)	143(5)	111(5)	84(4)	45(4)	11(4)	-6(4)

Table 5. Hydrogen coordinates ( $\times 10^4$ ) and isotropic displacement parameters ( $\text{\AA}^2 \times 10^{-3}$ ) for PF-00733741.

	x	y	z	U(eq)
H(2A)	991	5803	-1554	80
H(3A)	3539	6542	-849	80
H(5A)	6821	6781	-2072	80
H(6A)	4291	5979	-2776	80
H(7A)	57	6289	-2779	80
H(7B)	-180	4609	-2540	80
H(9A)	968	2210	-3025	80
H(11A)	1709	2232	-4700	80
H(12A)	1737	5041	-4597	80
H(13A)	1231	6399	-3721	80
H(14A)	6738	7884	-614	80
H(14B)	8088	7649	-1079	80
H(17A)	10000	5000	0	80
H(22A)	7308	12927	-1806	80
H(23A)	10206	13040	-1276	80
H(25A)	11441	9062	-2201	80
H(26A)	8551	8962	-2747	80
H(27A)	4922	11510	-2558	80
H(27B)	5208	9725	-2800	80
H(29A)	6074	13778	-3070	80
H(31A)	6774	12790	-4754	80
H(32A)	6579	10065	-4636	80
H(33A)	6109	9186	-3771	80
H(34A)	13577	12340	-1267	80
H(34B)	13895	10598	-1538	80
H(37A)	15000	10000	0	80