

Electronic Supplementary Information

Highly Selective Conversion of Nitrobenzenes Using a Simple Reducing System Combined with a Trivalent Indium Salt and a Hydrosilane

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Table of Contents

P.1-P.9	Spectral data for prepared compounds
P.10-P.41	NMR spectra for prepared compounds

General Methods

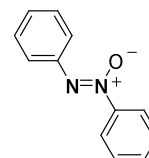
All reactions were carried out under a N₂ atmosphere, unless otherwise noted. Dichloromethane and chloroform were freshly distilled from P₂O₅ prior to use. Dimethylformamide (DMF) was distilled over CaH₂. THF was freshly distilled from sodium-benzophenone. All indium salts were commercially available and were used without further purification. Organosilanes were used without further purification. Reactions were monitored by TLC analysis of reaction aliquots. Thin-layer chromatography (TLC) was performed on silica gel 60 F₂₅₄, and the components were located by observation under UV light. Column chromatography was also performed using silica gel. ¹H NMR spectra were measured at 500 (or 300) MHz using tetramethylsilane as an internal standard. ¹³C NMR spectra were measured at 125 (or 75) MHz using the center peak of chloroform (77.0 ppm) as an internal standard. High resolution mass spectra were measured using NBA (3-nitrobenzylalcohol) as a matrix. Nitrobenzene (**1a**), 2-nitrotoluene (**1b**), 3-nitrotoluene (**1c**), 4-nitrotoluene (**1d**), 4-chloronitrobenzene (**1e**), 4-bromobenzene (**1f**), 4-nitrobenzonitrile (**1g**), 4-nitroacetophenone (**1h**), methyl 4-nitrobenzoate (**1i**), 2,2'-dinitrobiphenyl (**6**) were also purchased.

General procedure for indium-catalyzed reductive synthesis of azoxy compounds

To a 5 mL screw-capped vial containing a freshly distilled THF (0.6 mL) were successively added under N₂ aromatic nitro compound **1** (0.6 mmol), InBr₃ (0.03 mmol) and Et₃SiH (1.32 mmol). The resulting mixture was stirred at 60 °C (bath temperature), and monitored by TLC analysis until the starting material was completely consumed. The reaction was quenched with H₂O. The aqueous layer was extracted with EtOAc, the organic phases were dried over anhydrous Na₂SO₄, filtered, and evaporated under reduced pressure. The crude product was purified by recrystallization (hexane / AcOEt) or silica gel column chromatography to give the corresponding azoxybenzene compound **3**.

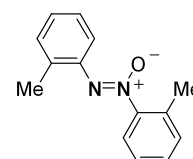
Azoxybenzene^[1] (**3a**):

96% yield; a yellow solid; mp 32.2-33.1 °C; ¹H NMR (300 MHz, CDCl₃) δ 7.36-7.41 (m, 1H), 7.46-7.56 (m, 5H), 8.15-8.29 (m, 2H), 8.30-8.32 (m, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 122.3, 125.5, 128.7, 128.8, 129.6, 131.6, 144.0, 148.3; MS (FAB): *m/z* 199 (M+H), 73 (100%).



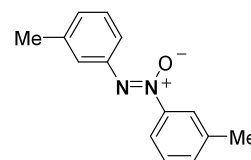
2,2'-Dimethylazoxybenzene^[1] (**3b**):

82% yield; a brown solid; mp 53.3-54.1 °C; ¹H NMR (500 MHz, CDCl₃) δ 2.37 (s, 3H), 2.51 (s, 3H), 7.25-7.31 (m, 5H), 7.36 (m, 1H), 7.67 (d, *J* = 8.0 Hz), 8.03 (d, *J* = 8.0 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 18.3, 18.4, 121.5, 123.5, 126.0, 126.5, 128.5, 130.0, 130.7, 131.2, 134.1, 142.8, 142.4; MS (FAB): *m/z* 227 (M+H), 73 (100%).



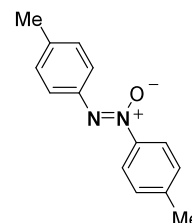
3,3'-Dimethylazoxybenzene^[2] (3c):

74% yield; a brown oil; ¹H NMR (500 MHz, CDCl₃) δ 2.42 (s, 3H), 2.45 (s, 3H), 7.19-7.20 (m, 1H), 7.34-7.38 (m, 3H), 7.96-7.98 (m, 2H), 8.07-8.10 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 18.3, 18.4, 121.5, 123.5, 126.0, 126.5, 128.5, 130.0, 130.7, 131.2, 134.1, 142.8, 142.4; MS (FAB): *m/z* 227 (M+H, 100%) .



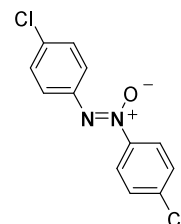
4,4'-Dimethylazoxybenzene^[1] (3d):

93% yield; a pale yellow solid; mp 61.1-62.0 °C; ¹H NMR (500 MHz, CDCl₃) δ 2.39 (s, 3H), 2.41 (s, 3H), 7.25-7.27 (m, 2H), 8.11 (d, *J* = 8.5 Hz, 2H), 8.16 (d, *J* = 8.5 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 21.2, 21.5, 122.1, 123.4, 125.6, 129.2, 129.7, 139.9, 141.8, 146.2; MS (FAB): *m/z* 227 (M+H, 100%).



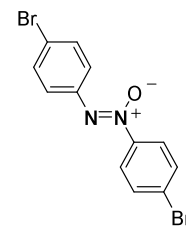
4,4'-Dichloroazoxybenzene^[1] (3e):

81% yield; a pale yellow powder; mp 153.0-154.0 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.44 (d, *J* = 8.5 Hz, 2H), 7.48 (d, *J* = 8.5 Hz, 2H), 8.16 (d, *J* = 8.5 Hz, 2H), 8.25 (d, *J* = 8.5 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 123.7, 127.1, 128.9, 129.0, 135.3, 138.1, 142.2, 146.6; MS (FAB): *m/z* 267 (M⁺), 154 (100%).



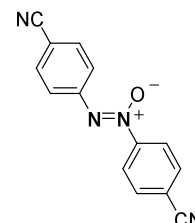
4,4'-Dibromoazoxybenzene^[1] (3f):

62% yield; a yellow needles; mp 159.9-161.3 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.60 (d, *J* = 9.0 Hz, 2H), 7.64 (d, *J* = 9.0 Hz, 2H), 8.08 (d, *J* = 9.0 Hz, 2H), 8.17 (d, *J* = 9.0 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 123.6, 123.9, 126.5, 127.2, 131.9, 132.0, 142.6, 147.1; MS (ESI): *m/z* 356 (M⁺, 100%).



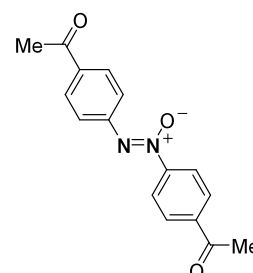
4,4'-Dicyanoazoxybenzene^[3] (3g):

88% yield; a colorless solid; mp 206.0-208.1 °C; ¹H NMR (300 MHz, CDCl₃) δ 7.80 (d, *J* = 8.5 Hz, 2H), 7.87 (d, *J* = 8.5 Hz, 2H), 8.23 (d, *J* = 8.5 Hz, 2H), 8.47 (d, *J* = 8.5 Hz, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 113.1, 116.1, 117.3, 118.2, 123.4, 126.0, 132.8, 133.1, 146.3, 150.2; MS (EI): *m/z* 248 (M⁺, 100%).



4,4'-Diacetylazoxybenzene^[4] (3h):

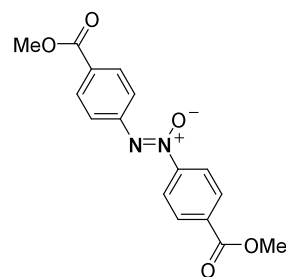
97% yield; a pale yellow solid; mp 190.8-191.5 °C; ¹H NMR (300 MHz, CDCl₃) δ 2.66 (s, 3H), 2.69 (s, 3H), 8.07-8.13 (m, 4H), 8.22 (d, *J* = 9.0 Hz, 2H), 8.42 (d, *J* = 9.0 Hz, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 26.7, 26.9, 122.9, 125.6, 129.0, 129.1, 137.4, 139.6,



147.0, 150.8; MS (FAB): m/z 283 (M+H), 154 (100%).

DimethylAzoxybenzene-4,4'-dicarboxylate^[5] (3i):

64% yield; a pale yellow powder; mp 209.5-210 °C; ¹H NMR (500 MHz, CDCl₃) δ 3.96 (s, 3H), 3.98 (s, 3H), 8.15-8.21(m, 6H), 8.38-8.40 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 52.3, 52.6, 122.6, 125.4, 130.2, 130.4, 130.8, 133.3, 147.0, 150.9, 165.8, 166.3; MS (FAB): m/z 315 (M+H), 154 (100%).

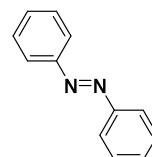


General procedure for indium-catalyzed reductive synthesis of azobenzene compounds

To a 5 mL screw-capped vial containing freshly distilled DMF (0.6 mL) were successively added under N₂ aromatic nitro compound **1** (0.6 mmol), In(OTf)₃ (0.03 mmol) and Et₃SiH (1.5 mmol). The resulting mixture was stirred at 60 °C (bath temperature), and monitored by TLC analysis. After completion of the reaction, the mixture was stirred under air for 15 h. The reaction was quenched with H₂O. The aqueous layer was extracted with AcOEt, and the organic phases were dried over anhydrous Na₂SO₄, filtered, and evaporated under reduced pressure. The crude product was purified by recrystallization (hexane / AcOEt) or silica gel column chromatography to give the corresponding azobenzene derivative **4**.

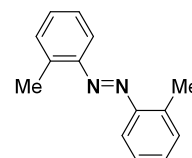
Azobenzene^[2] (4a):

99% yield; an orange solid; mp 65.0-66.0 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.46-7.54 (m, 3H), 7.92-7.93 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 122.8, 129.1, 131.0, 152.7; MS (FAB): m/z 183 (M+H), 154 (100%).



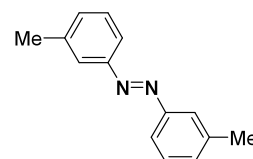
2,2'-Dimethylazobenzene^[2] (4b):

62% yield; a red solid; mp 53.0-54.0 °C; ¹H NMR (500 MHz, CDCl₃) δ 2.74 (s, 6H), 7.25 (m, 2H), 7.33 (m, 4H), 7.61-7.64 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 17.6, 115.8, 126.3, 130.7, 131.2, 138.0, 151.1; MS (EI): m/z 210 (M⁺, 100%).



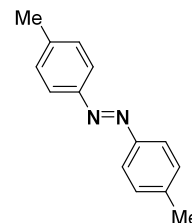
3,3'-Dimethylazobenzene^[2] (4c):

78% yield; a dark orange solid; mp 50.3-51.6 °C; ¹H NMR (500 MHz, CDCl₃) δ 2.46 (s, 6H), 7.28-7.29(m, 2H), 7.39-7.42 (m, 2H), 7.72 (m, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 21.4, 120.4, 122.9, 128.9, 131.7, 139.0, 152.8; MS (EI): m/z 210 (M⁺, 100%).



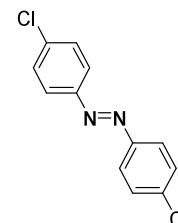
4,4'-Dimethylazobenzene^[2] (4d):

72% yield; a pale orange solid; mp 145.0-146.0 °C; ¹H NMR (500 MHz, CDCl₃) δ 2.43 (s, 6H), 7.30 (d, *J* = 8.0 Hz, 4H), 7.81 (d, *J* = 8.0 Hz, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 21.5, 122.7, 129.7, 141.2, 150.8; MS (EI): *m/z* 210 (M⁺, 100%).



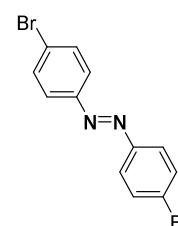
4,4'-Dichloroazobenzene^[1] (4e):

70% yield; an orange needles; mp 184.5-185.5 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.49 (d, *J* = 8.5 Hz, 4H), 7.86 (d, *J* = 8.5 Hz, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 124.2, 129.4, 137.2, 150.8; MS (ESI): *m/z* 250 (M⁺, 100%), 252 (M⁺+2).



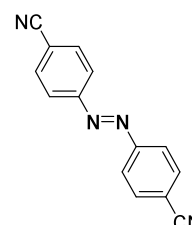
4,4'-Dibromoazobenzene^[2] (4f):

80% yield; an orange powder; mp 203.0-204.5 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.65 (d, *J* = 9.0 Hz, 4H), 7.79 (d, *J* = 9.0 Hz, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 124.4, 125.8, 132.4, 151.2; MS (ESI): *m/z* 340 (M⁺, 100%).



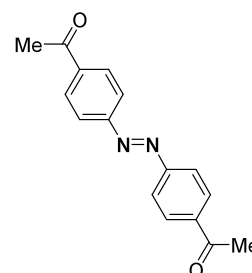
4,4'-Dicyanoazobenzene^[6] (4g):

64% yield; a red solid; mp 234.6-235.2 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.86 (d, *J* = 8.0 Hz, 4H), 8.04 (d, *J* = 8.0 Hz, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 115.2, 118.1, 123.7, 133.4, 154.0; MS (FAB): *m/z* 232 (M⁺, 100%).



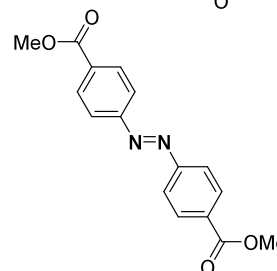
4,4'-Diacetylazobenzene^[4] (4h):

92% yield; a pale red powder; mp 208.5-209.8 °C; ¹H NMR (500 MHz, CDCl₃) δ 2.68 (s, 6H), 8.01 (d, *J* = 8.0 Hz, 4H), 8.13 (d, *J* = 8.0 Hz, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 26.8, 123.2, 129.4, 138.9, 154.8, 197.3; MS (FAB): *m/z* 267 (M+H), 73 (100%).



DimethylAzobenzene-4,4'-dicarboxylate^[6] (4i):

95% yield; an orange powder; mp 223.1-224.4 °C; ¹H NMR (500 MHz, CDCl₃) δ 3.97 (s, 6H), 7.99 (d, *J* = 8.0 Hz, 4H), 8.21 (d, *J* = 8.0 Hz, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 52.4, 122.9, 130.7, 132.4, 154.9, 166.4; MS (FAB): *m/z* 298 (M⁺, 100%).

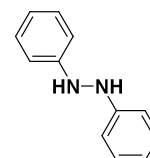


General procedure for indium-catalyzed reductive synthesis of hydrazobenzene derivatives

To a 5 mL screw-capped vial containing a freshly distilled DMF (0.6 mL) were successively added under N₂ aromatic nitro compound **1** (0.6 mmol), InBr₃ (0.03 mmol) and Et₃SiH (2.4 mmol). The resulting mixture was stirred at 60 °C (bath temperature), and monitored by TLC analysis. After completion of the reaction, the reaction was quenched with H₂O. The aqueous layer was extracted with EtOAc, and the organic phases were dried over anhydrous Na₂SO₄, filtered, and evaporated under reduced pressure. The crude product was purified by silica gel column chromatography to give the corresponding product **5**.

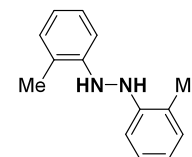
Hydrazobenzene^[6] (**5a**):

94% yield; a colorless solid; mp 125.3-126.0 °C; ¹H NMR (500 MHz, CDCl₃) δ 5.53 (br, 2H), 6.81-6.84 (m, 6H), 7.18-7.21 (m, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 112.3, 119.8, 129.3, 148.8; MS (FAB): *m/z* 274 (M⁺+H).



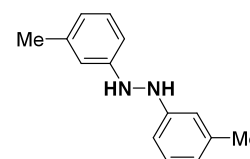
2,2'-Dimethylhydrazobenzene^[7] (**5b**):

63% yield; a pale yellow solid; mp 161.8-162.6 °C; ¹H NMR (500 MHz, CDCl₃) δ 2.25 (s, 6H), 5.51 (br, 2H), 6.76-6.79 (m, 2H), 6.88-6.90 (m, 2H), 7.07-7.11 (m, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 17.1, 111.0, 119.4, 121.1, 127.2, 130.4, 146.2; MS (EI): *m/z* 212 (M⁺), 91 (100%).



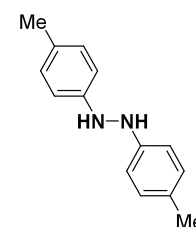
3,3'-Dimethylhydrazobenzene^[8] (**5c**):

41% yield; a pale orange solid; mp 39.6-40.5 °C; ¹H NMR (500 MHz, CDCl₃) δ 2.27 (s, 6H), 5.50 (br, 2H), 6.63-6.67 (m, 6H), 7.07-7.12 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 21.6, 109.4, 112.9, 120.7, 129.2, 139.2, 149.0; MS (FAB): *m/z* 211 (M-H), 73 (100%).



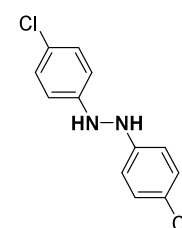
4,4'-Dimethylhydrazobenzene^[7] (**5d**):

82% yield; a yellow solid; mp 113.3-113.9 °C; ¹H NMR (500 MHz, CDCl₃) δ 2.29 (s, 6H), 5.50 (br, 2H), 6.78 (d, *J* = 8.0 Hz, 4H), 7.05 (d, *J* = 8.0 Hz, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 20.5, 112.4, 129.0, 129.8, 146.7; MS (EI): *m/z* 212 (M⁺), 210 (100%).



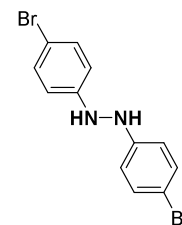
4,4'-Dichlorohydrazobenzene^[2] (**5e**):

80% yield; a pale brown needles; mp 125.7-128.0 °C; ¹H NMR (500 MHz, CDCl₃) δ 5.62 (br, 2H), 6.75 (d, *J* = 9.0 Hz, 4H), 7.16 (d, *J* = 9.0 Hz, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 113.5, 124.7, 129.3, 147.0; MS (FAB): *m/z* 252 (M⁺), 254 (M⁺+2).



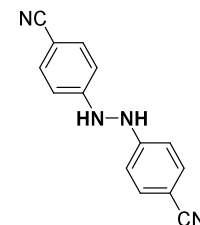
4,4'-Dibromohydrazobenzene^[2] (5f):

72% yield; a pale yellow solid; mp 123.4-125.1 °C; ¹H NMR (300 MHz, CDCl₃) δ 5.61 (br, 2H), 6.69 (d, *J* = 9.0 Hz, 4H), 7.29 (d, *J* = 9.0 Hz, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 111.8, 113.9, 132.1, 147.4; MS (EI): *m/z* 342 (M⁺).



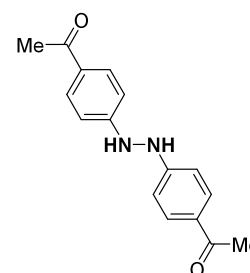
4,4'-Dicyanohydrazobenzene^[3] (5g):

81% yield; a colorless solid; mp 188.1-188.5 °C; ¹H NMR (500 MHz, CDCl₃) δ 6.12 (br, 2H), 6.85 (d, *J* = 8.5 Hz, 4H), 7.51 (d, *J* = 8.5 Hz, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 103.1, 112.1, 119.4, 134.0, 151.1; MS (FAB): *m/z* 234 (M⁺), 81 (100%).



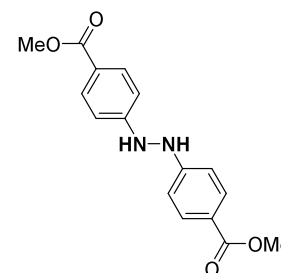
4,4'-Diacetylhydrazobenzene^[9] (5h):

81% yield; a brown solid; mp 163.0-165.1 °C; ¹H NMR (500 MHz, CDCl₃) δ 2.41 (s, 6H), 6.39 (br, 2H), 6.73 (d, *J* = 8.5 Hz, 4H), 7.75 (d, *J* = 8.5 Hz, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 26.1, 111.1, 129.3, 130.7, 152.3, 196.7; MS (FAB): *m/z* 269 (M+H, 100%).



Dimethylhydrazobenzene-4,4'-dicarboxylate (5i):

64% yield; a pale brown solid; mp 174.7-177.2 °C; ¹H NMR (300 MHz, CDCl₃) δ 3.86 (s, 6H), 6.07 (br, 2H), 6.82 (d, *J* = 9.0 Hz, 4H), 7.91 (d, *J* = 9.0 Hz, 4H); ¹³C NMR (75 MHz, CDCl₃) δ 51.7, 111.2, 131.6, 151.9, 166.9; MS (EI): *m/z* 300 (M⁺), 73 (100%), HRMS (FAB): Calcd for C₁₆H₁₆N₂O₄: 300.1110, Found: 300.1110

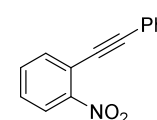


Procedure for synthesis of 2-(Phenylethynyl)nitroaniline (6)

To a solution of 1-nitro-2-iodobenzene (1.0 g, 4 mmol) in triethylamine (20 ml) at room temperature was added cis-PdCl₂(PPh₃)₂ (0.056 g, 0.2 mmol). The resultant suspension was stirred for 30 min followed by addition of phenylacetylene (6 mmol) and then CuI (0.0076 g, 0.2 mmol). This mixture was then stirred for 24 h. The reaction mixture was washed with 1N HCl aq., saturated NaHCO₃ aq. and brine, dried over Na₂SO₄, filtered, and evaporated under reduced pressure. The crude product was purified by silica gel column chromatography to give the compound 6.

2-(Phenylethynyl)nitroaniline^[10] (6):

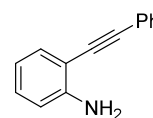
68% yield; a brown oil; ¹H NMR (500 MHz, CDCl₃) δ 7.37-7.38 (m, 3H), 7.43-7.46 (m, 1H), 7.57-7.60 (m, 3H), 7.71 (m, 1H), 8.07 (m, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 84.7, 97.1, 118.7,



122.3, 124.7, 128.4, 128.5, 129.2, 132.0, 132.8, 134.5, 149.6; MS (FAB): m/z 223 (M^+ , 100%)

2-(Phenylethynyl)aniline^[11] (7):

83% yield; pale yellow prisms; ^1H NMR (500 MHz, CDCl_3) δ 4.26 (br, 2H), 6.70-6.72 (m 2H), 7.11-7.15 (m, 1H), 7.30-7.37 (m, 4H), 7.51-7.53 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 85.9, 94.7, 107.9, 114.3, 117.9, 123.3, 128.2, 128.3, 129.7, 131.4, 132.1, 147.7; MS (FAB): m/z 194 ($M+H$, 100%)

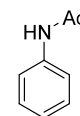


General procedure for indium-catalyzed reductive synthesis of aromatic amide derivatives

To a 5 mL screw-capped vial containing freshly distilled CHCl_3 (0.6 mL) were successively added under N_2 aromatic nitro compound **1** (0.6 mmol), InI_3 (0.03 mmol) and 1, 1', 3, 3'-tetramethyldisiloxane (1.2 mmol). The resulting mixture was stirred at room temperature, and monitored by TLC analysis. After completion of the reaction, AcCl (0.6 mmol) and Et_3N (0.6 mmol) were added to the reaction mixture, followed by an additional stirring for 30 min. After completion of the reaction, the reaction was quenched with Na_2CO_3 aq. The aqueous layer was extracted with CHCl_3 , the organic phases were dried over anhydrous Na_2SO_4 , filtered, and evaporated under reduced pressure. The crude product was purified by silica gel column chromatography to give the corresponding product.

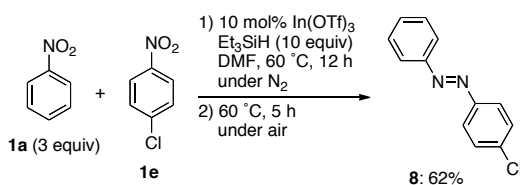
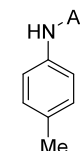
Acetanilide^[12] (2a'):

99% yield; a white powder; mp 88.4-89.5 °C; ^1H NMR (500 MHz, CDCl_3) δ 2.12 (s, 3H), 7.06-7.09 (m, 1H), 7.25-7.29 (m, 2H), 7.50-7.51 (m, 2H), 8.25 (br, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 24.3, 120.1, 124.2, 128.8, 138.0, 167.0; MS (FAB): m/z 136 ($M+H$, 100%).



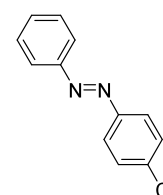
***p*-Acetotoluidine^[13] (2d'):**

82% yield; a white powder; mp 105.9-106.4 °C; ^1H NMR (500 MHz, CDCl_3) δ 2.13 (s, 3H), 2.30 (s, 4H), 7.09 (d, $J=8.0$ Hz, 2H), 7.37 (d, $J=8.5$ Hz, 2H), 7.71 (br, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 20.8, 24.3, 120.1, 129.4, 133.8, 135.4, 168.5; MS (FAB): m/z 150 ($M+H$, 100%).



4-Chloroazobenzene^[14] (8):

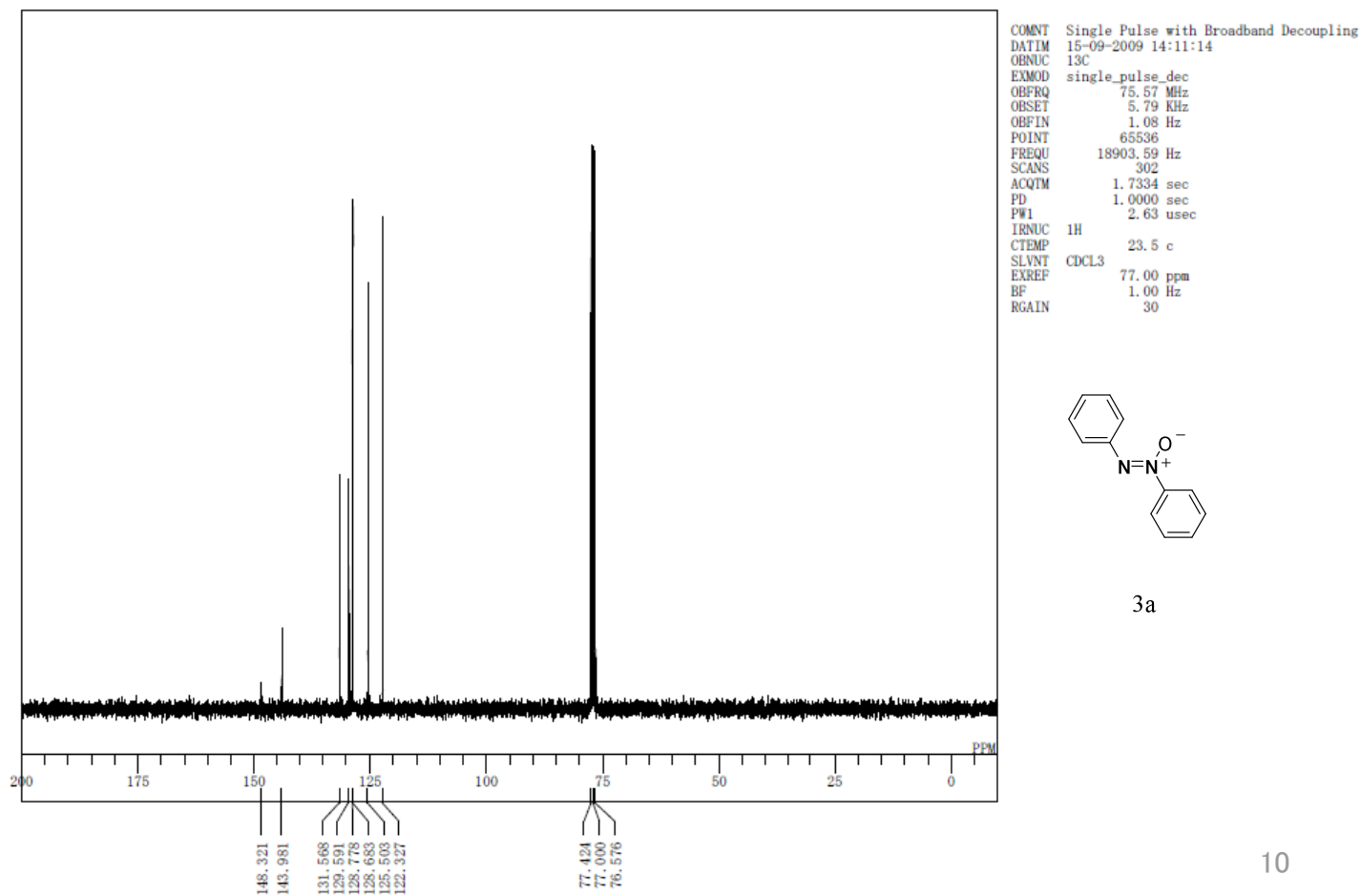
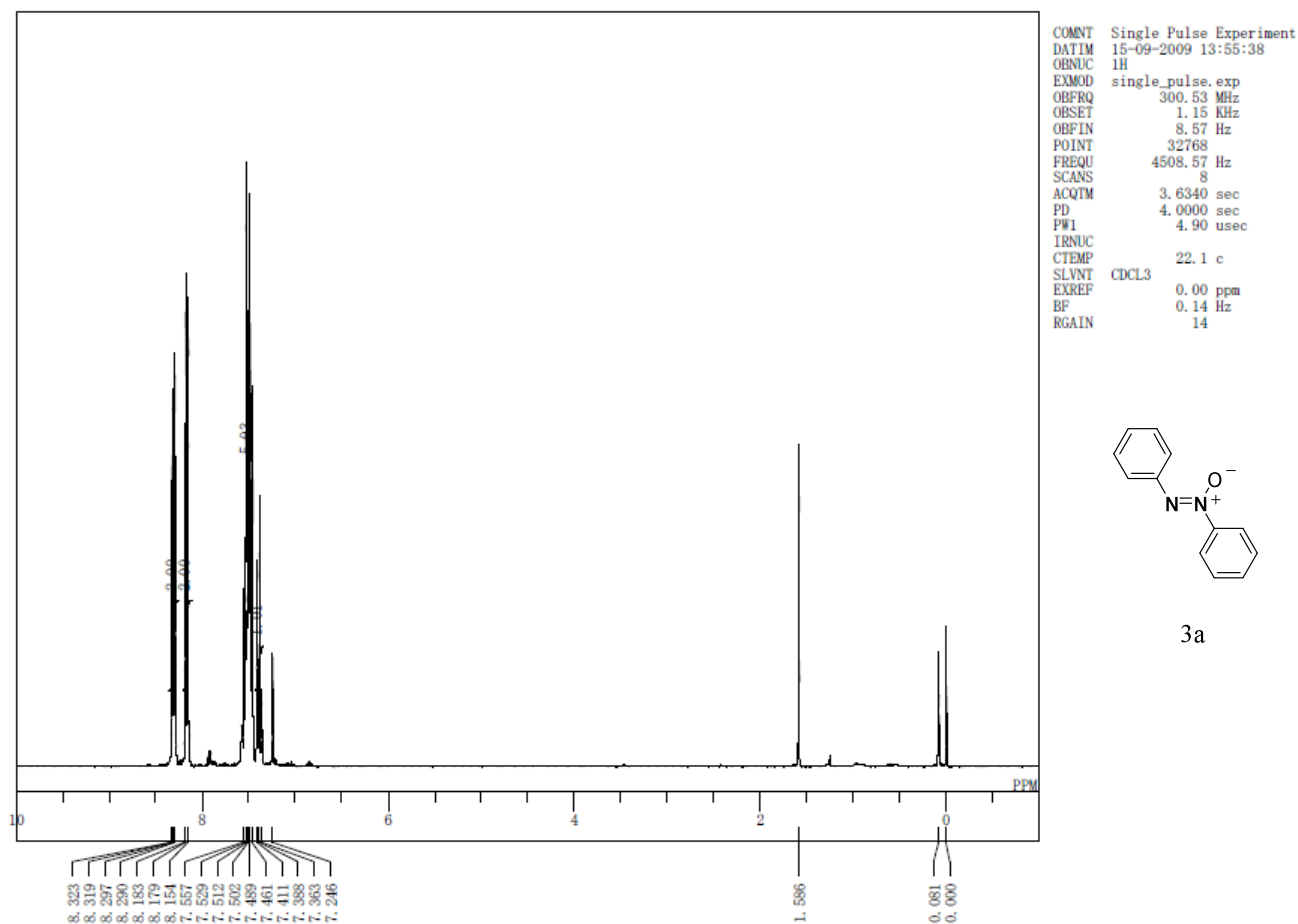
62% yield; an orange solid; ^1H NMR (300 MHz, CDCl_3) δ 7.45-7.54 (m, 5H), 7.85-7.92 (m 4H); ^{13}C NMR (75 MHz, CDCl_3) δ 122.9, 124.1, 129.1, 129.3, 131.3, 136.9, 150.9, 152.4;



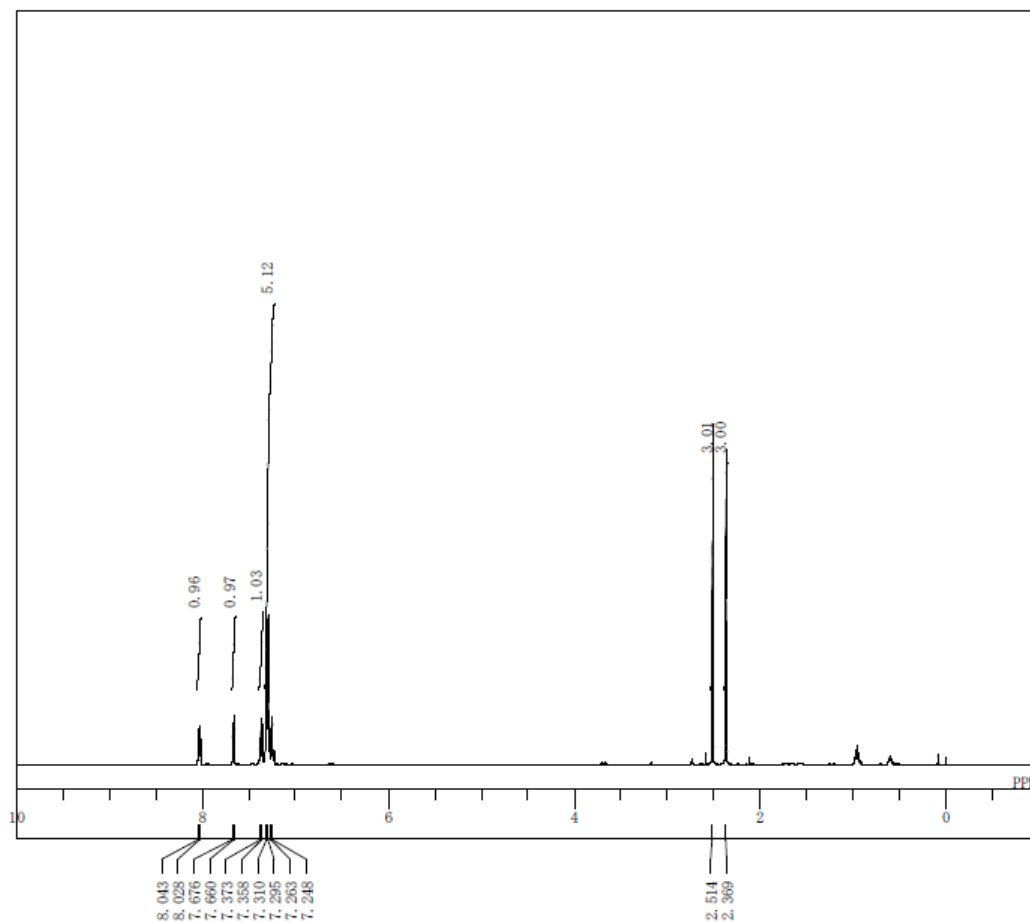
MS (FAB): m/z 216 (M^+ , 100%). HRMS (FAB): Calcd for $\text{C}_{12}\text{H}_9\text{ClN}_2$: 216.0454, Found: 216.0453

References

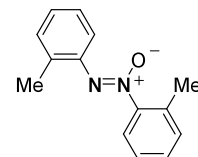
- [1] S. Wada, M. Urano, H. Suzuki, *J. Org. Chem.* **2002**, *67*, 8254.
- [2] K. Ohe, S. Uemura, N. Sugita, H. Masuda, T. Taga, *J. Org. Chem.* **1989**, *54*, 4169.
- [3] K. K. Park, S. Y. Han, *Tetrahedron Lett.* **1996**, *37*, 6721.
- [4] A. Gorrane, A. Corma, H. Garcia, *Science* **2008**, *322*, 1661.
- [5] B. Priewisch, K. Ruck-Braun, *J. Org. Chem.* **2005**, *70*, 2350.
- [6] J. M. Khurana, A. Ray, *Bull. Chem. Soc. Jpn.* **1996**, *69*, 407.
- [7] F. A. Khan, J. Dash, C. Sudheer, R. K. Gupta, *Tetrahedron Lett.* **2003**, *44*, 7783.
- [8] L. C. Snyder, *J. Am. Chem. Soc.* **1962**, *84*, 340.
- [9] M. B. Sridhara, G. R. Srinivasa, D. C. Gowda, *J. Chem. Res.* **2004**, 74.
- [10] Y. Yamane, X. Liu, A. Hamasaki, T. Ishida, M. Haruta, T. Yokoyama, M. Tokunaga, *Org. Lett.* **2009**, *11*, 5162.
- [11] N. Sakai, K. Annaka, T. Konakahara, *Org. Lett.* **2004**, *6*, 1527.
- [12] N. Sakai, K. Fujii, T. Konakahara, *Tetrahedron Lett.* **2008**, *49*, 6873.
- [13] E. Campaigne, R. D. Lake, *J. Org. Chem.* **1959**, *24*, 478.
- [14] D. Seyferth, W. Tronich, H.-M. Shih, *J. Org. Chem.* **1974**, *39*, 158.



Single Pulse Experiment

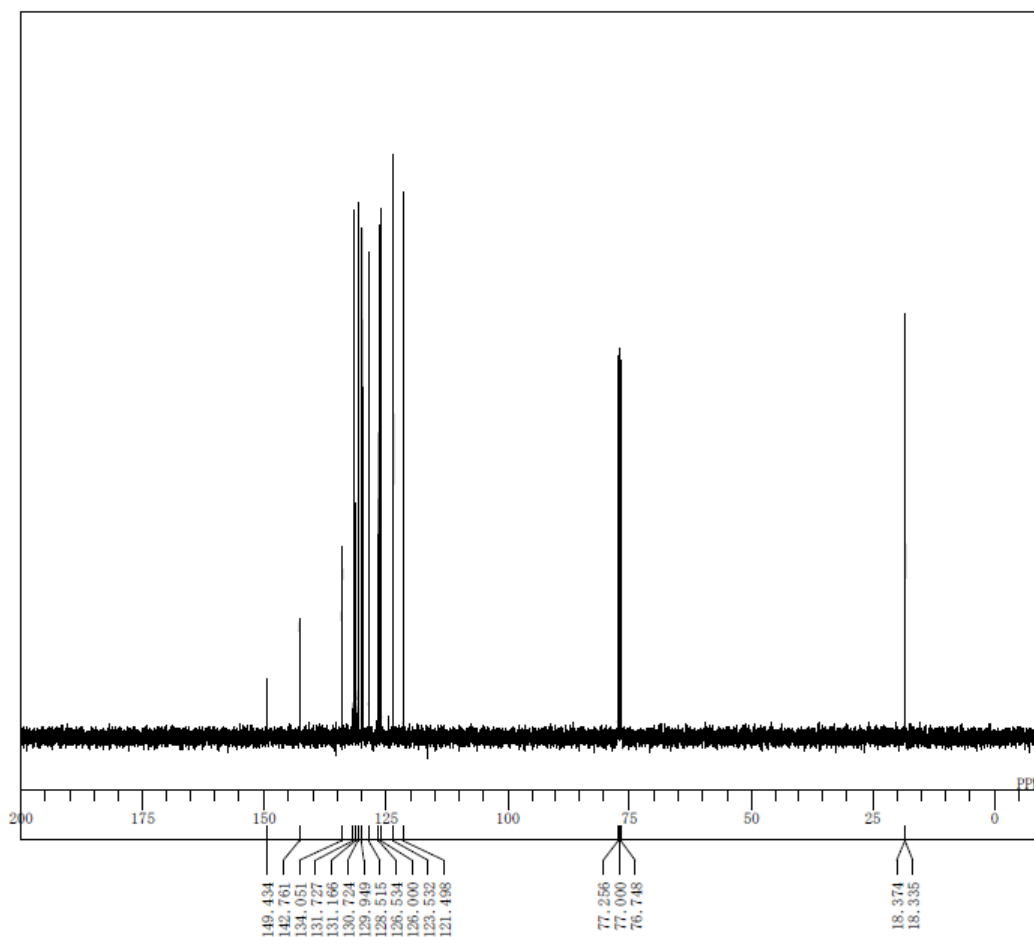


DFILE KF-546-1H.1
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DATIM 16-10-2008 17:02:02
OBNUC 1H
EXMOD single_pulse_exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 32768
FREQU 7507.51 Hz
SCANS 8
ACQTM 2.1823 sec
PD 4.0000 sec
PW1 7.00 usec
IRNUC
CTEMP 23.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.23 Hz
RGAIN 12

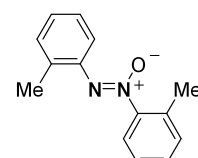


3b

Single Pulse with Broadband Decoupling

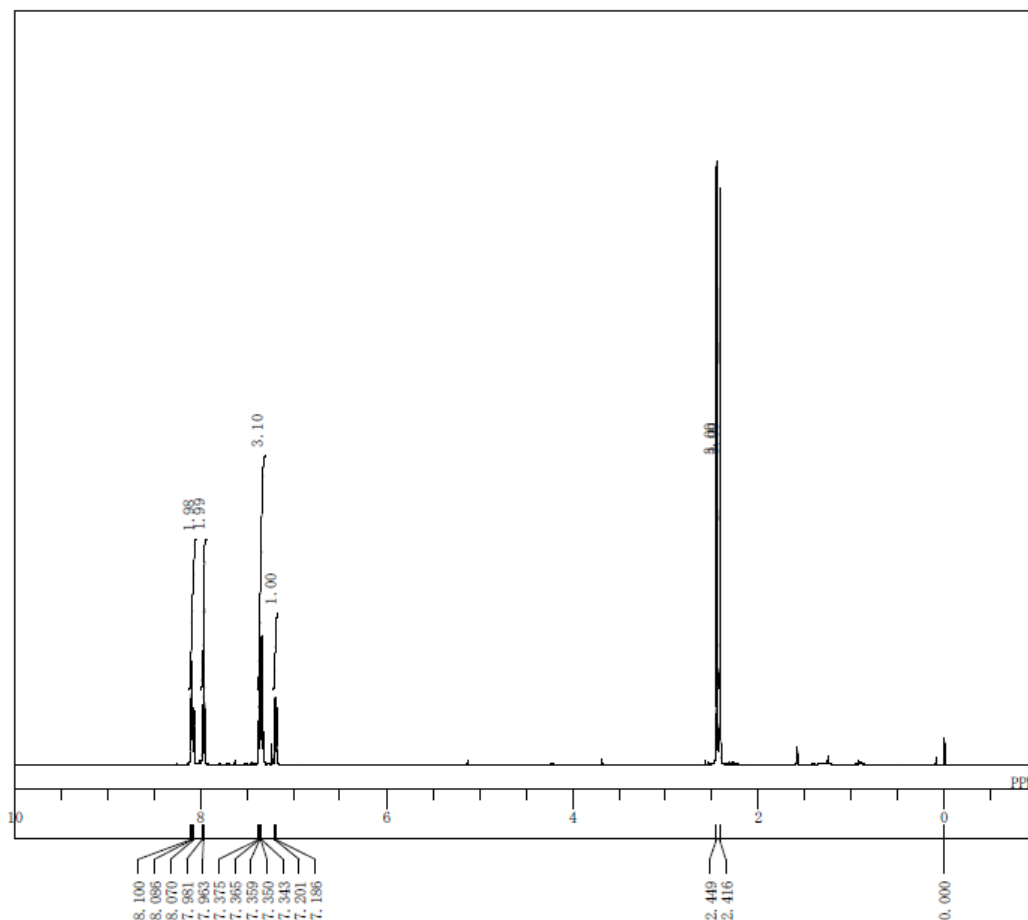


DFILE KF-546-13C.1
COMNT Single Pulse with Broadband Decoupling
DATIM 16-10-2008 17:09:37
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 65536
FREQU 31446.54 Hz
SCANS 197
ACQTM 1.0420 sec
PD 1.0000 sec
PW1 4.17 usec
IRNUC 1H
CTEMP 27.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 30

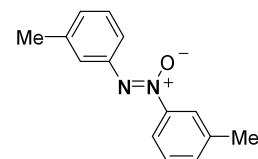


3b

Single Pulse Experiment

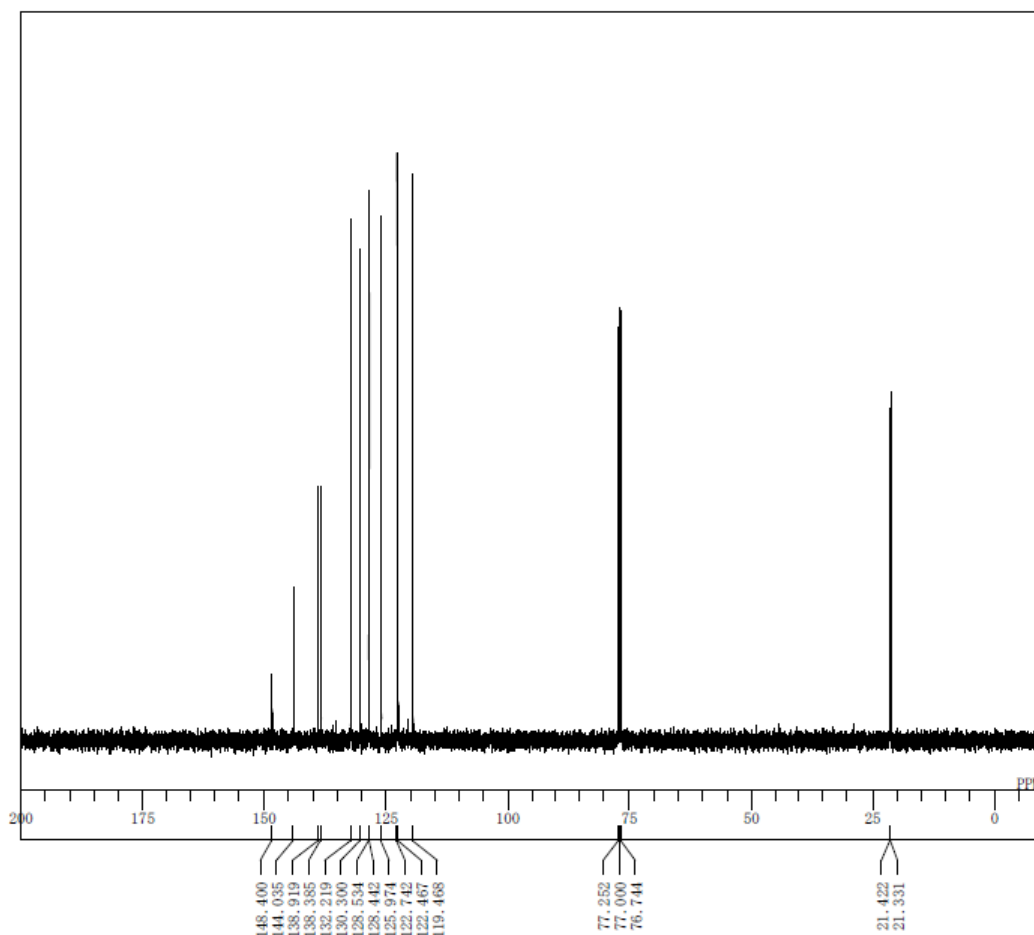


DFILE KF-547-1-1H.1
COMNT Single Pulse Experiment
DATIM 15-10-2008 15:55:17
OBNUC 1H
EXMOD single_pulse_exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 32768
FREQU 7507.61 Hz
SCANS 8
ACQTM 2.1823 sec
PD 4.0000 sec
PW1 7.00 usec
IRNUC
CTEMP 25.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.23 Hz
RGAIN 13

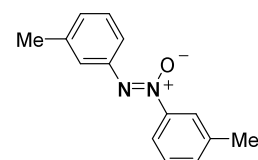


3c

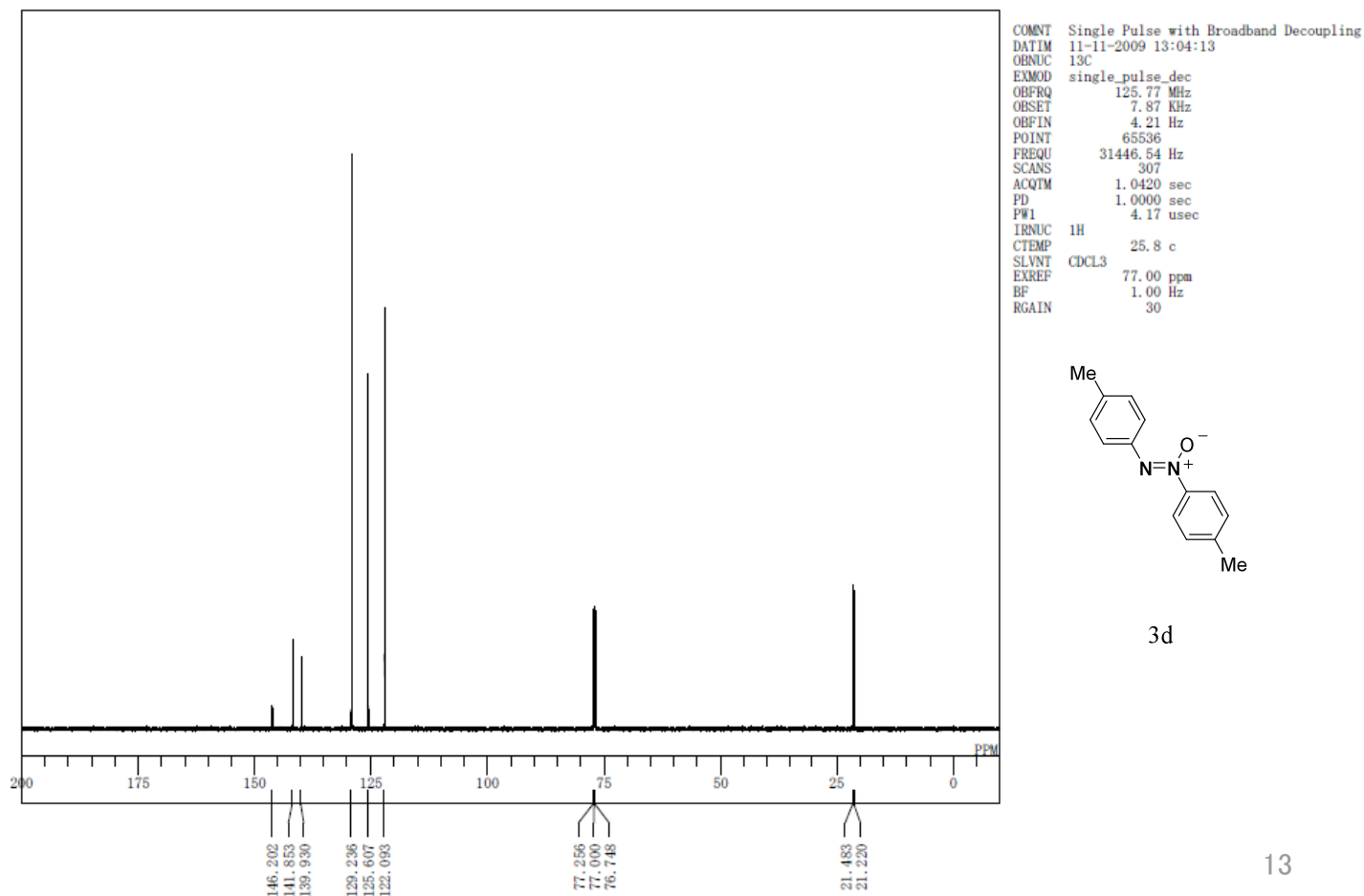
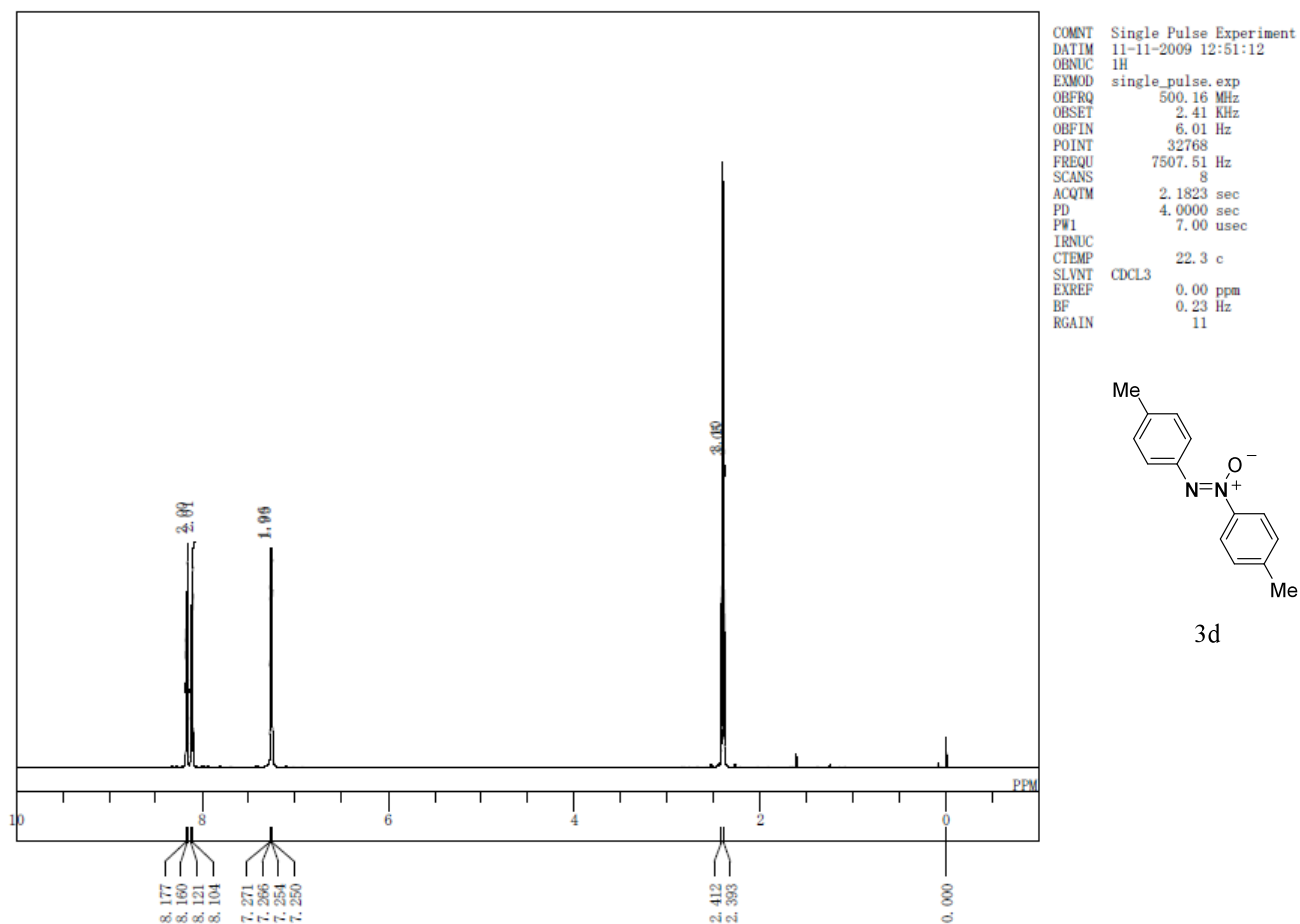
Single Pulse with Broadband Decoupling

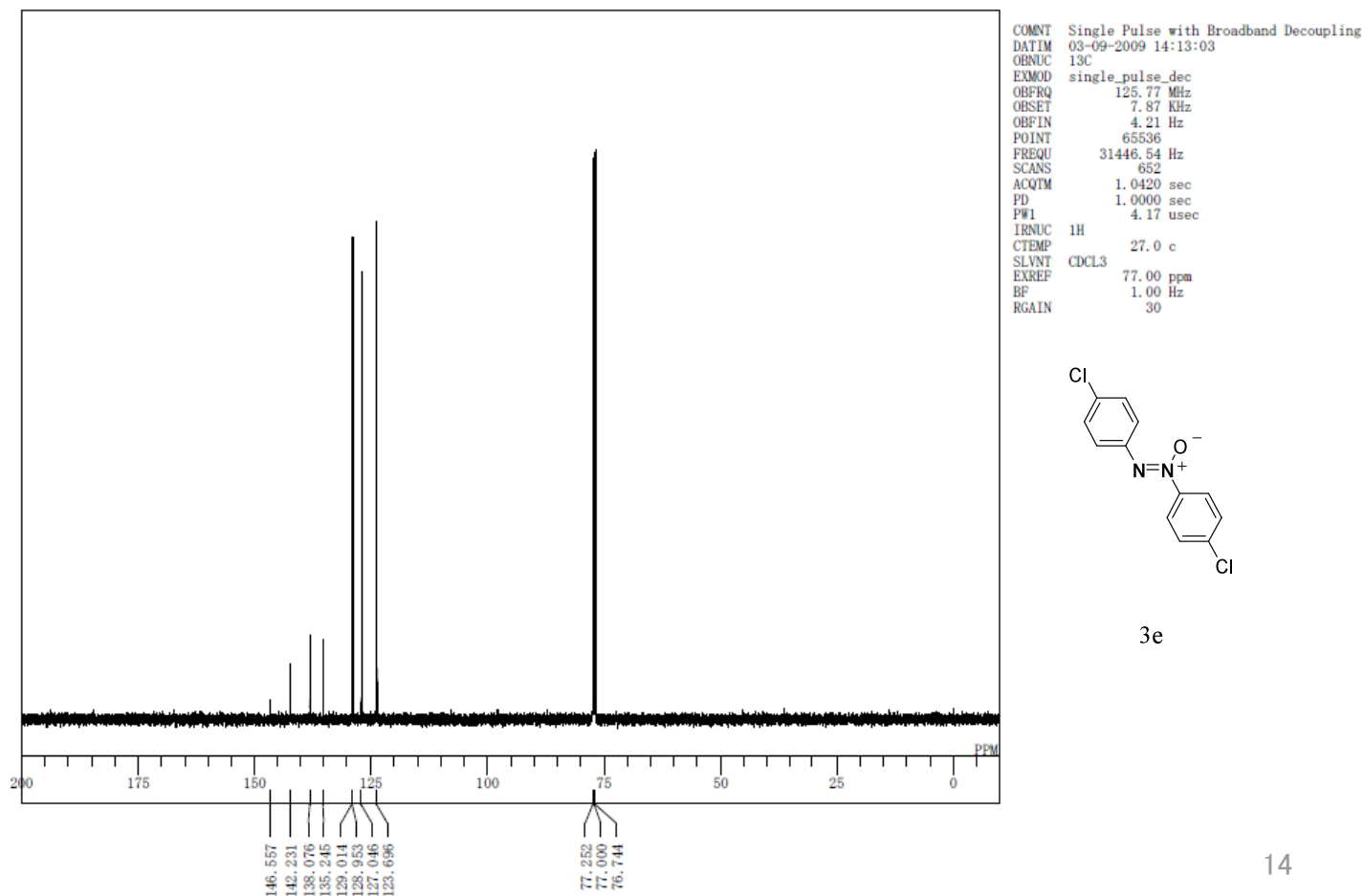
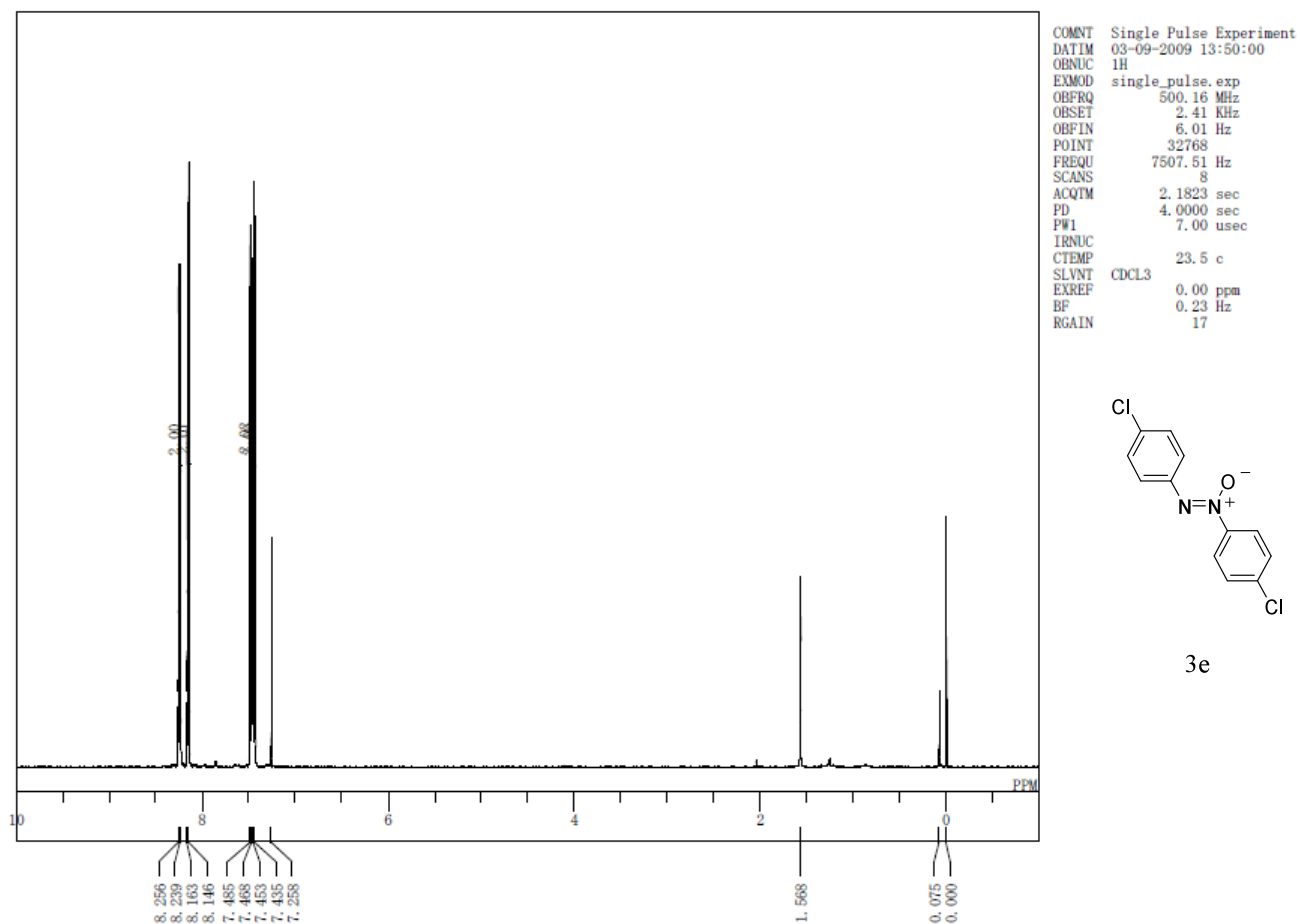


DFILE KF-547-1-13C.1
COMNT Single Pulse with Broadband Decoupling
DATIM 15-10-2008 16:01:41
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 65536
FREQU 31446.54 Hz
SCANS 163
ACQTM 1.0420 sec
PD 1.0000 sec
PW1 4.17 usec
IRNUC 1H
CTEMP 26.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 30

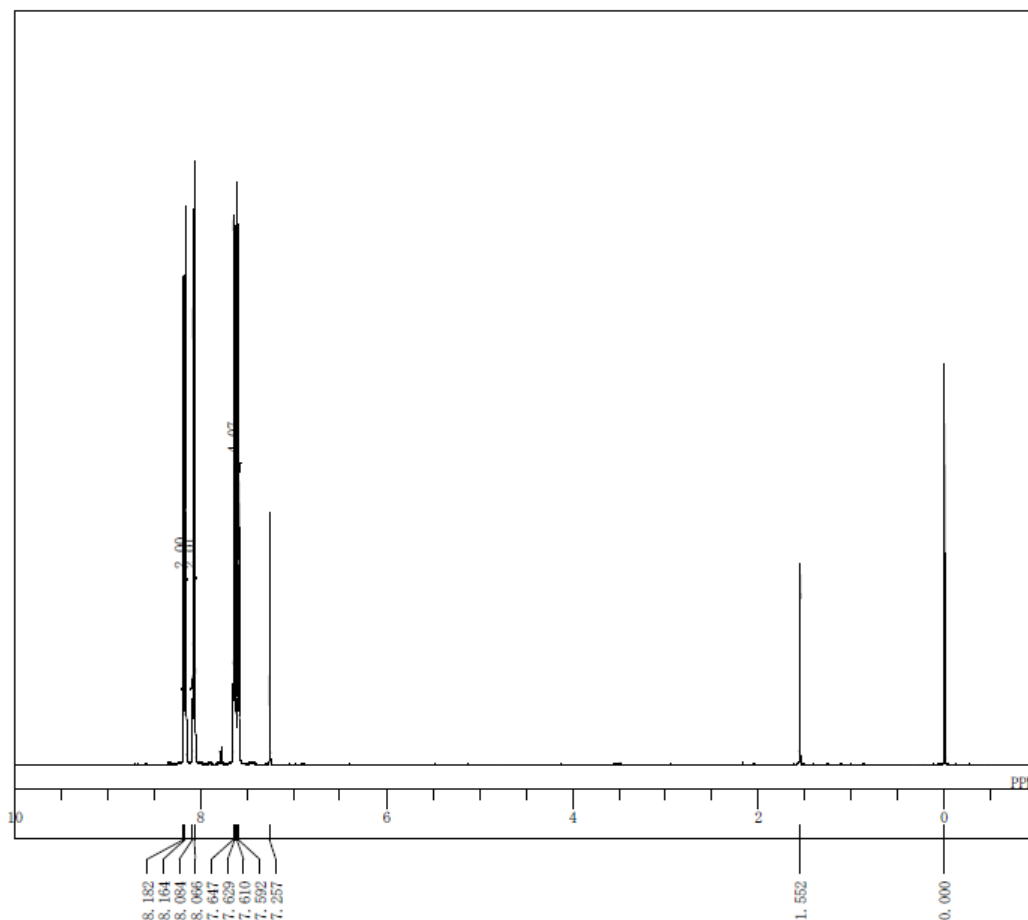


3c

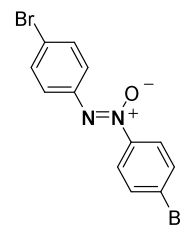




Single Pulse Experiment

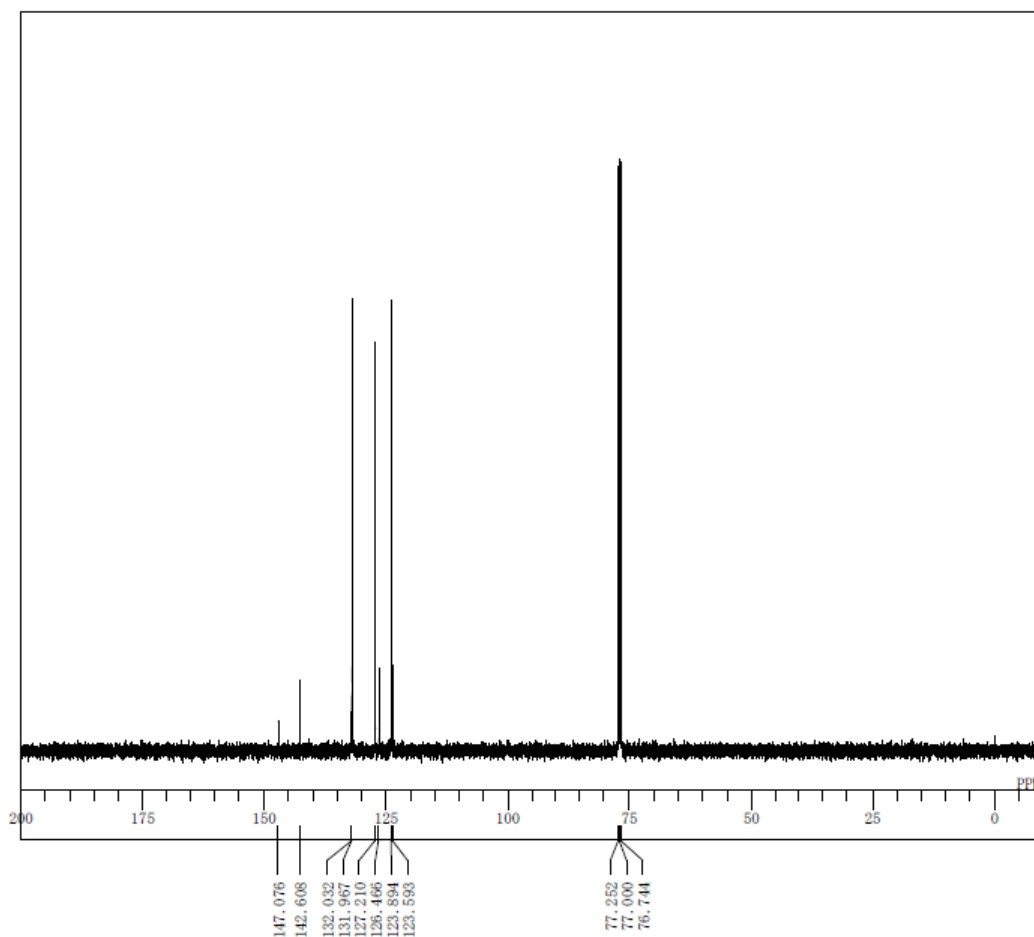


DFILE KF-563-t-1H.1
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DATIM 01-11-2008 14:06:15
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EXMOD single_pulse.exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 32768
FREQU 7507.61 Hz
SCANS 8
AQTM 2.1823 sec
PD 4.0000 sec
PW1 7.00 usec
IRNUC
CTEMP 24.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.23 Hz
RGAIN 18

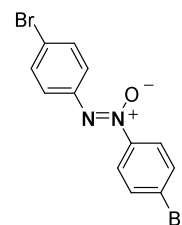


3f

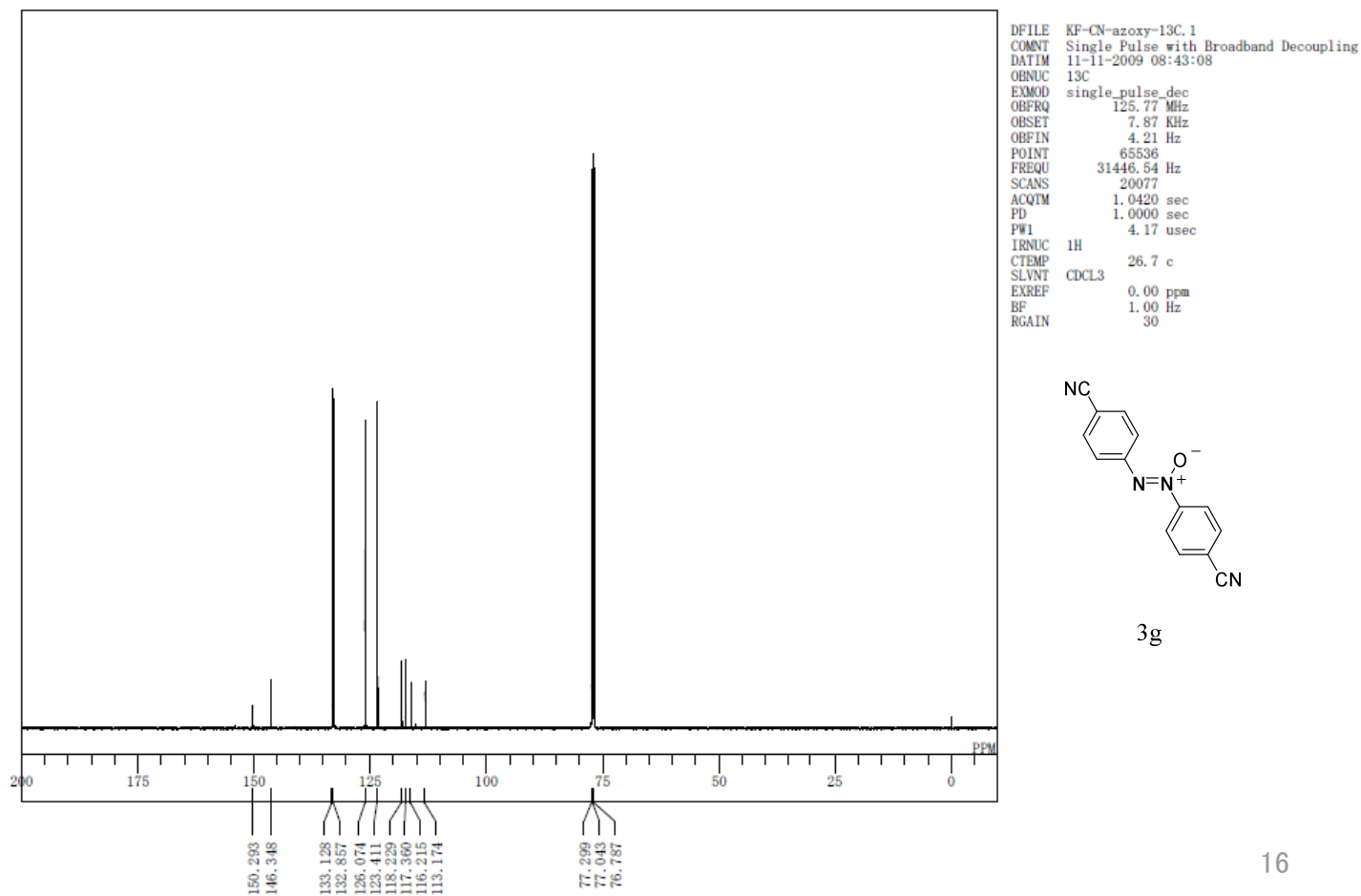
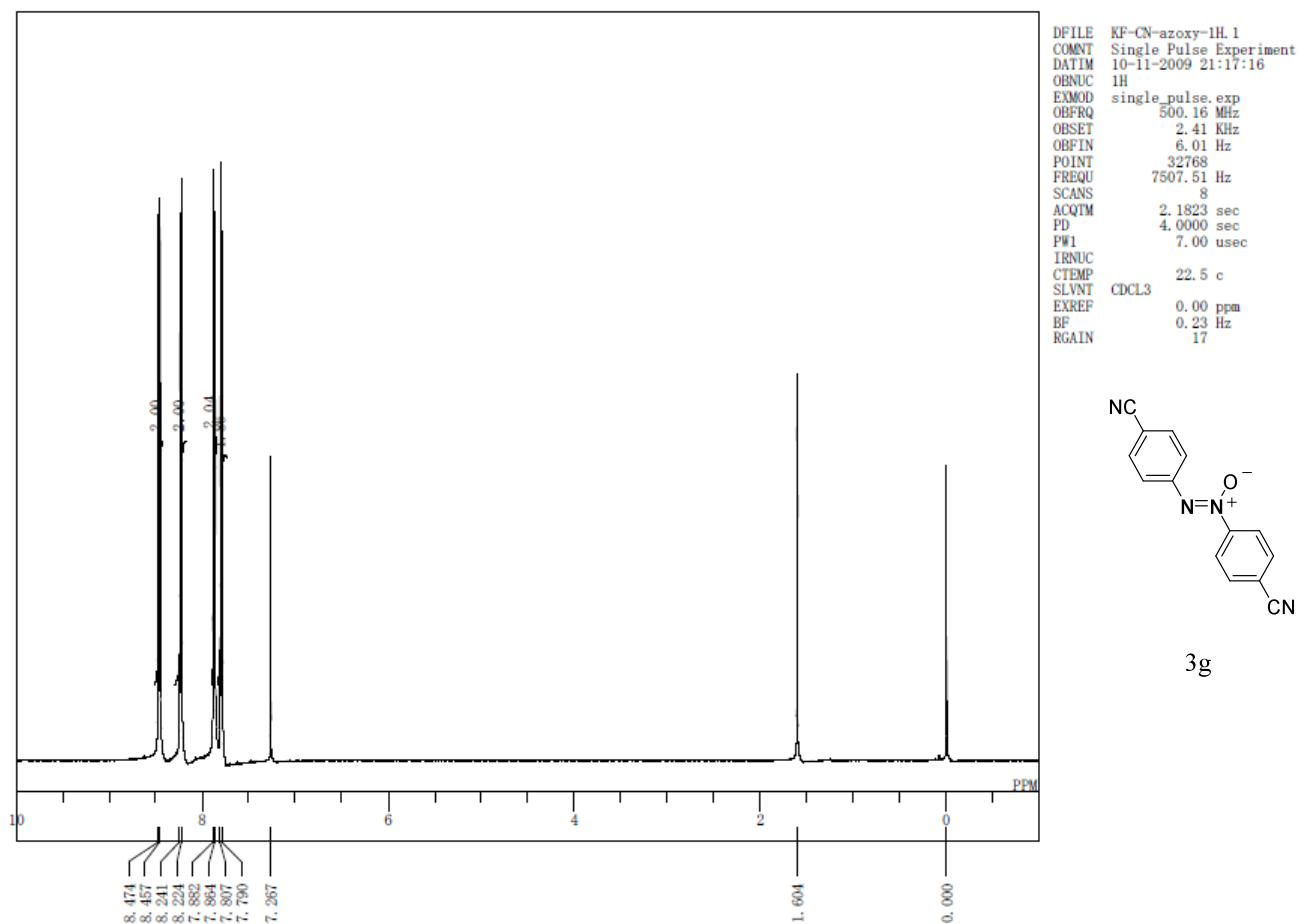
Single Pulse with Broadband Decoupling



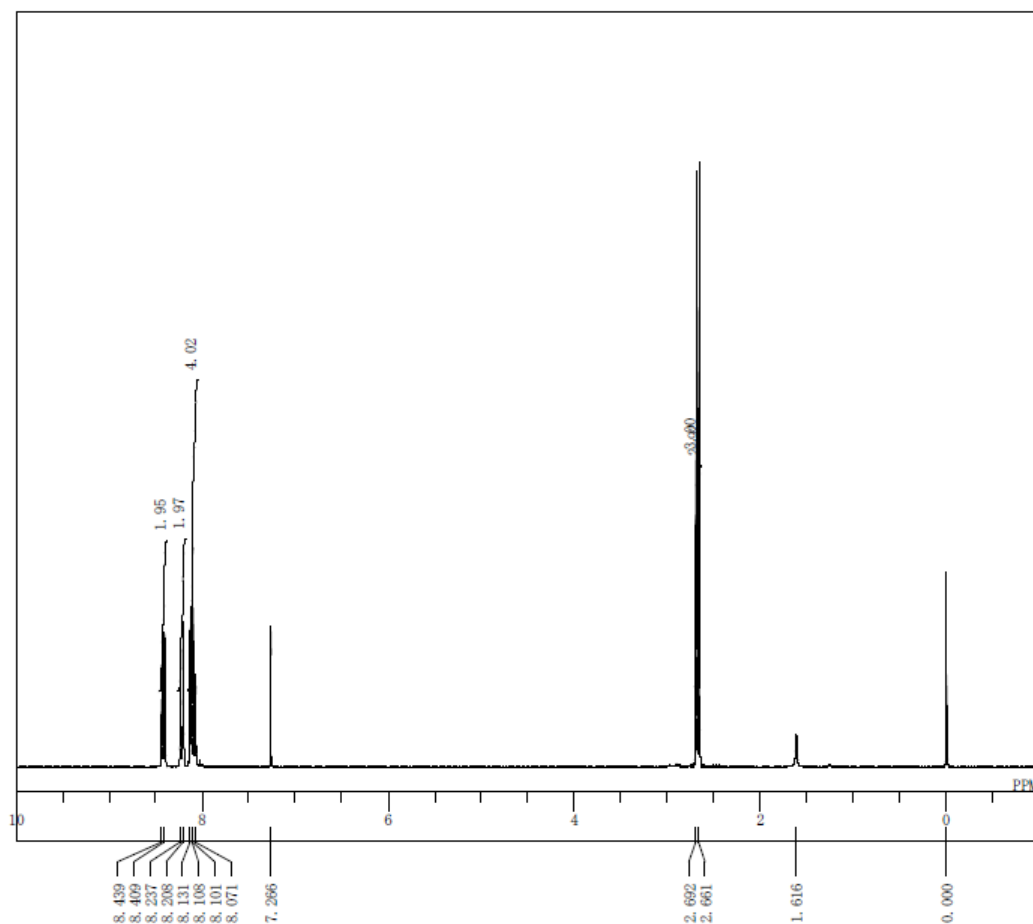
DFILE KF-563-t-2-13C.1
COMNT Single Pulse with Broadband Decoupling
DATIM 19-11-2008 16:23:53
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 65536
FREQU 31446.54 Hz
SCANS 323
AQTM 1.0420 sec
PD 1.0000 sec
PW1 4.17 usec
IRNUC 1H
CTEMP 27.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 30



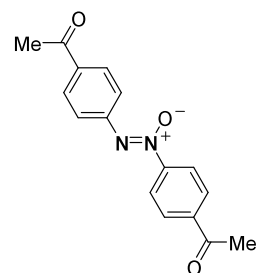
3f



Single Pulse Experiment

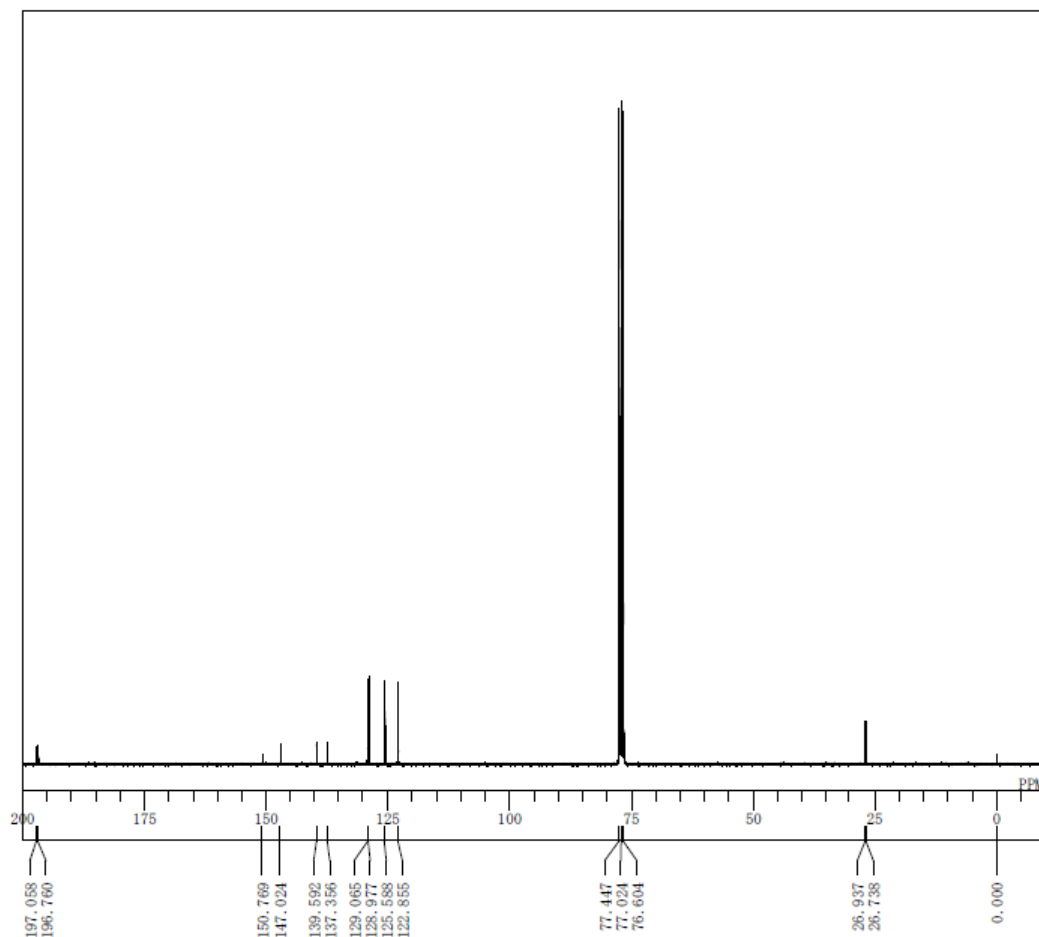


DFILE KF-AcAzoxy-1H.1
COMNT Single Pulse Experiment
DATIM 05-11-2009 22:00:44
1H
EXMOD single_pulse_exp
OBFRQ 300.53 MHz
OBSET 1.15 KHz
OBFIN 8.57 Hz
POINT 32768
FREQU 4508.57 Hz
SCANS 8
ACQTM 3.6340 sec
PD 4.0000 sec
PW1 4.90 usec
IRNUC
CTEMP 21.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.14 Hz
RGAIN 18

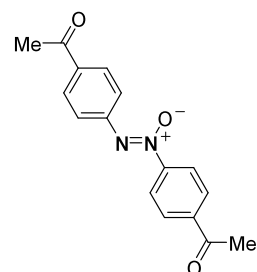


3h

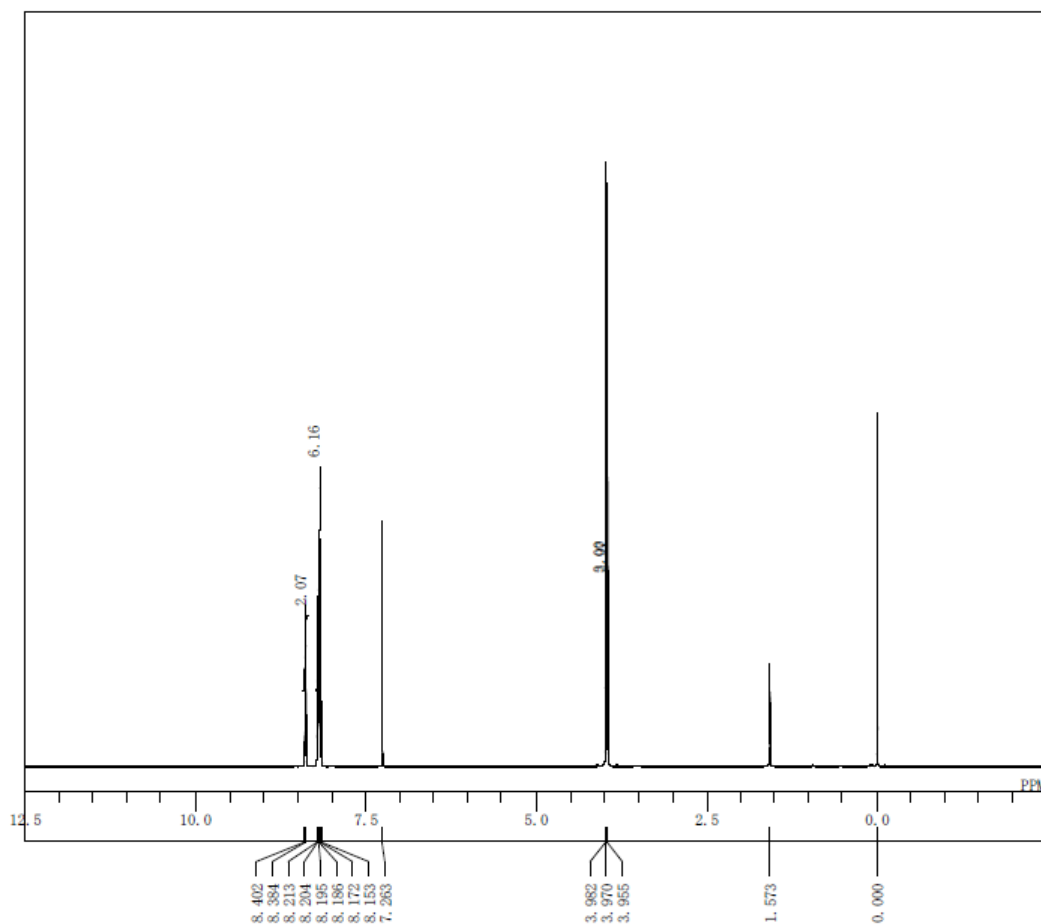
Single Pulse with Broadband Decoupling



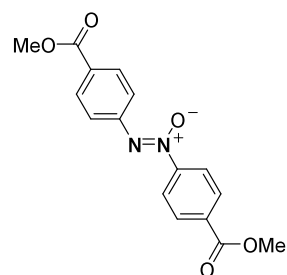
DFILE KF-AcAzoxy-13C.1
COMNT Single Pulse with Broadband Decoupling
DATIM 06-11-2009 07:53:21
13C
EXMOD single_pulse_dec
OBFRQ 75.57 MHz
OBSET 5.79 KHz
OBFIN 1.08 Hz
POINT 65536
FREQU 18903.59 Hz
SCANS 12959
ACQTM 1.7334 sec
PD 1.0000 sec
PW1 2.63 usec
IRNUC 1H
CTEMP 23.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 30



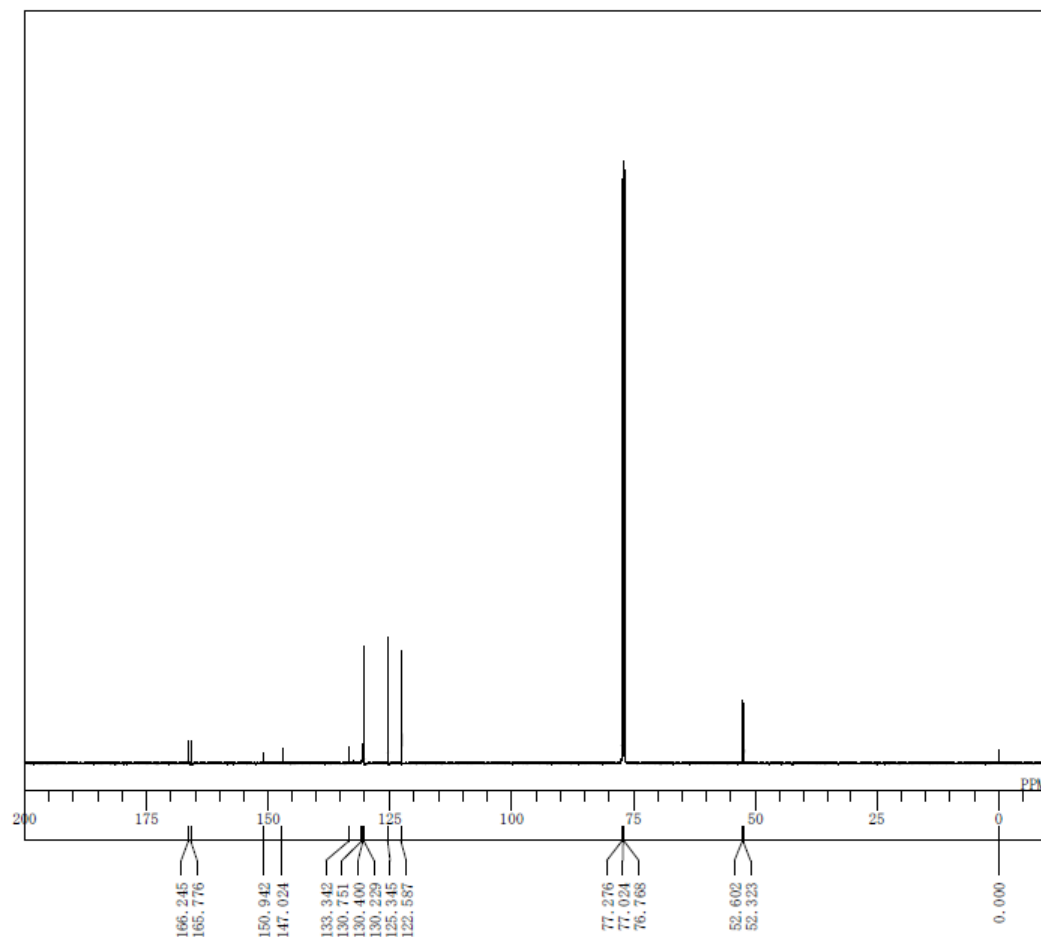
3h



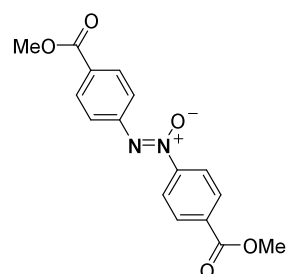
DFILE SN-ester-azoxy-re-1H.1
 COMNT Single Pulse Experiment
 DATIM 12-11-2009 22:25:48
 OBNUC 1H
 EXMOD single_pulse_exp
 OBFREQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 32768
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 2.1823 sec
 PD 4.0000 sec
 PW1 7.00 usec
 IRNUC
 CTEMP 22.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 21



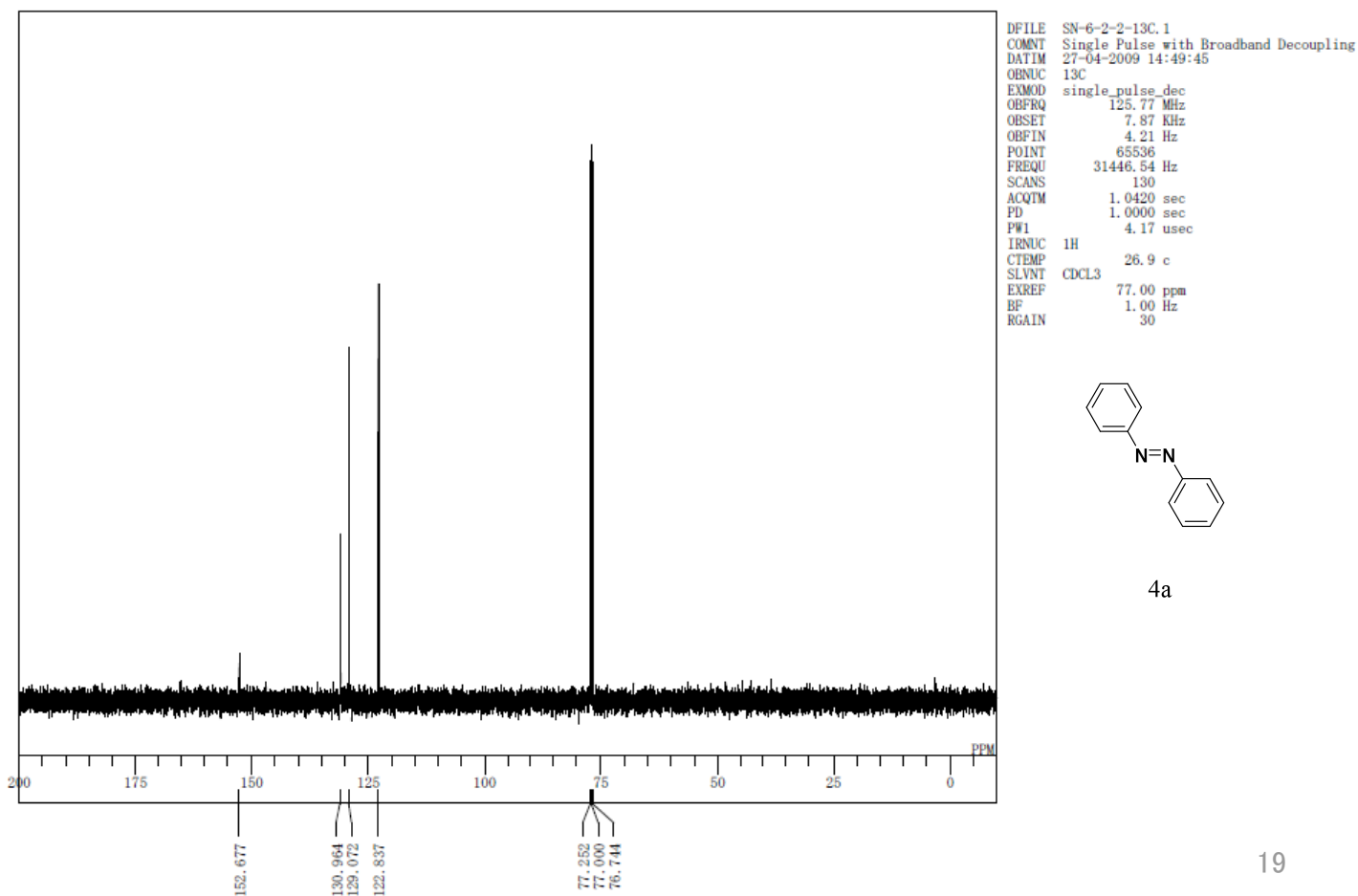
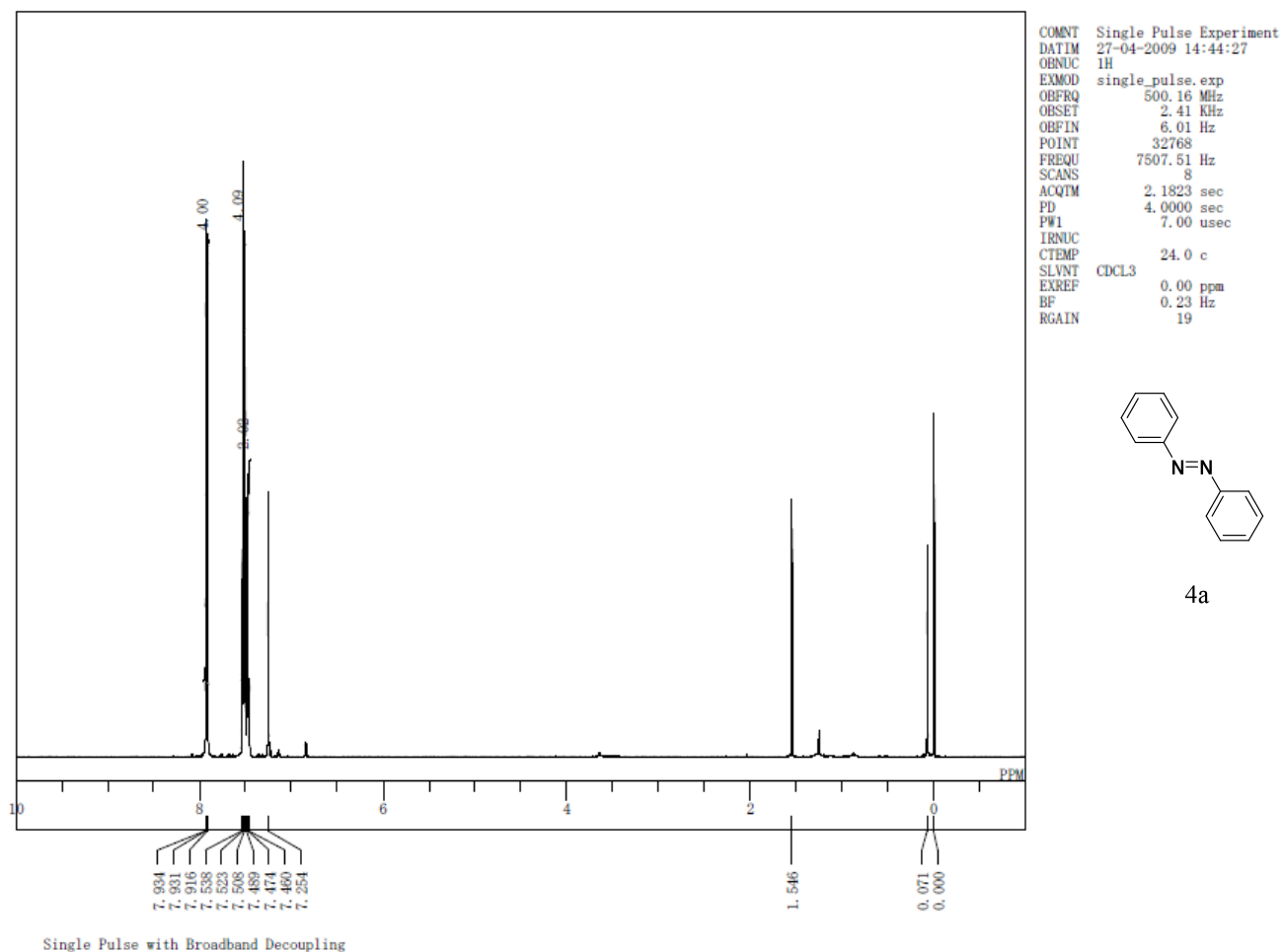
3i

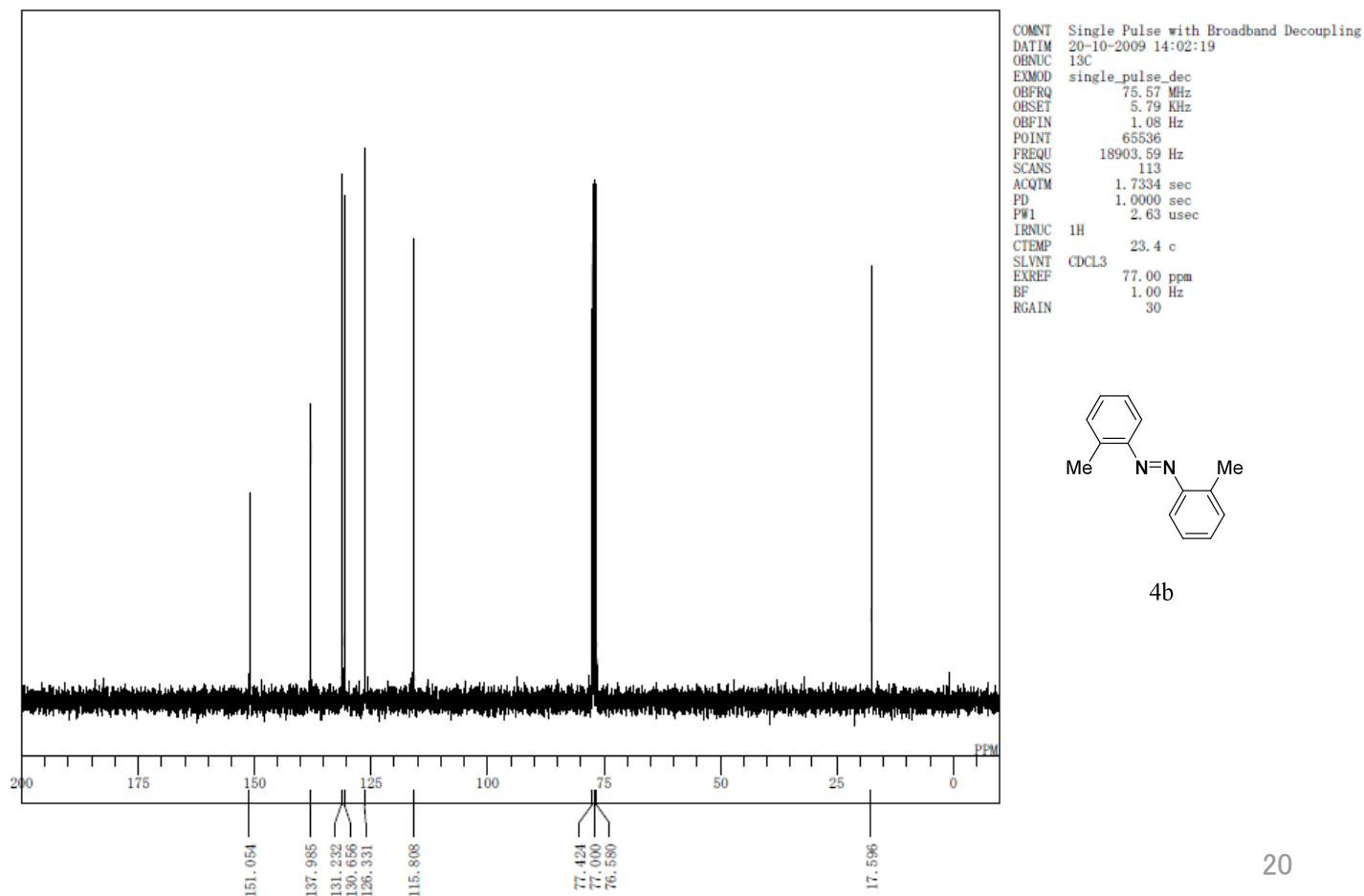
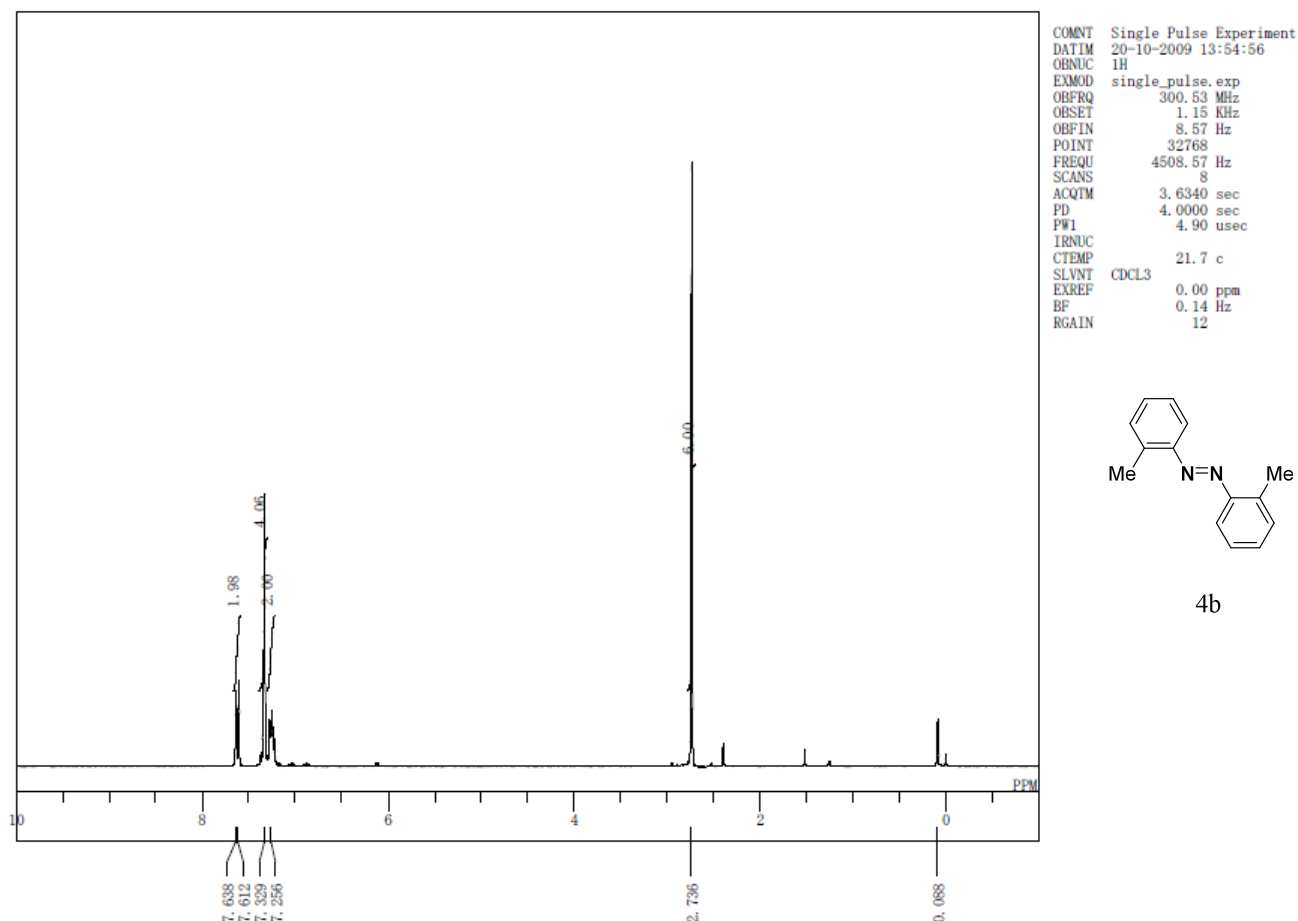


DFILE SN-ester-azoxy-re-13C.1
 COMNT Single Pulse with Broadband Decoupling
 DATIM 13-11-2009 08:23:27
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFREQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 65536
 FREQU 31446.54 Hz
 SCANS 17487
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 26.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 1.00 Hz
 RGAIN 30

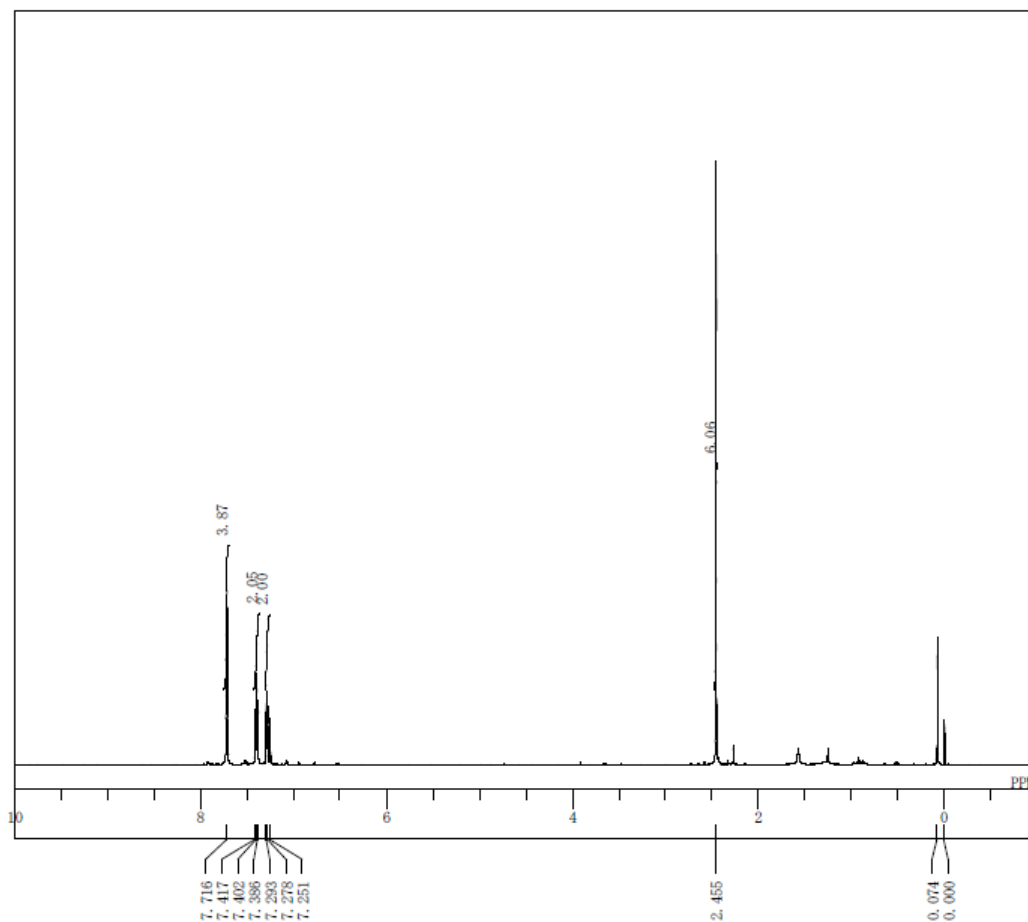


3i

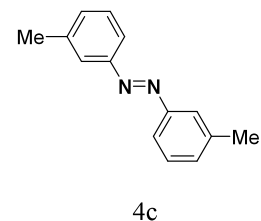




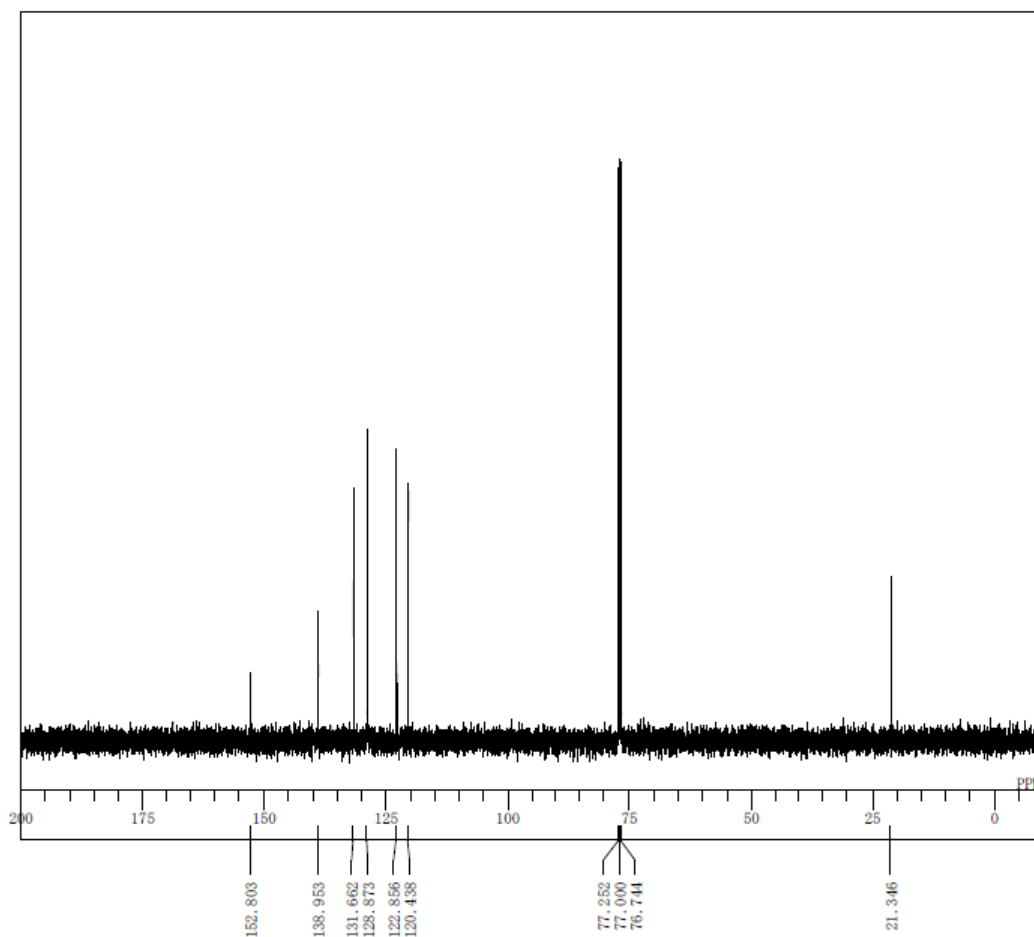
Single Pulse Experiment



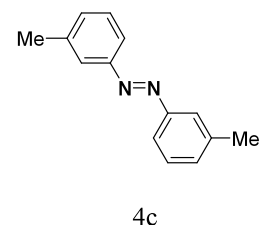
DFILE SN-21-1-1H.1
COMNT Single Pulse Experiment
DATIM 29-05-2009 17:30:40
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EXMOD single_pulse_exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 32768
FREQU 7507.61 Hz
SCANS 8
ACQTM 2.1823 sec
PD 4.0000 sec
PW1 7.00 usec
IRNUC
CTEMP 23.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.23 Hz
RGAIN 16



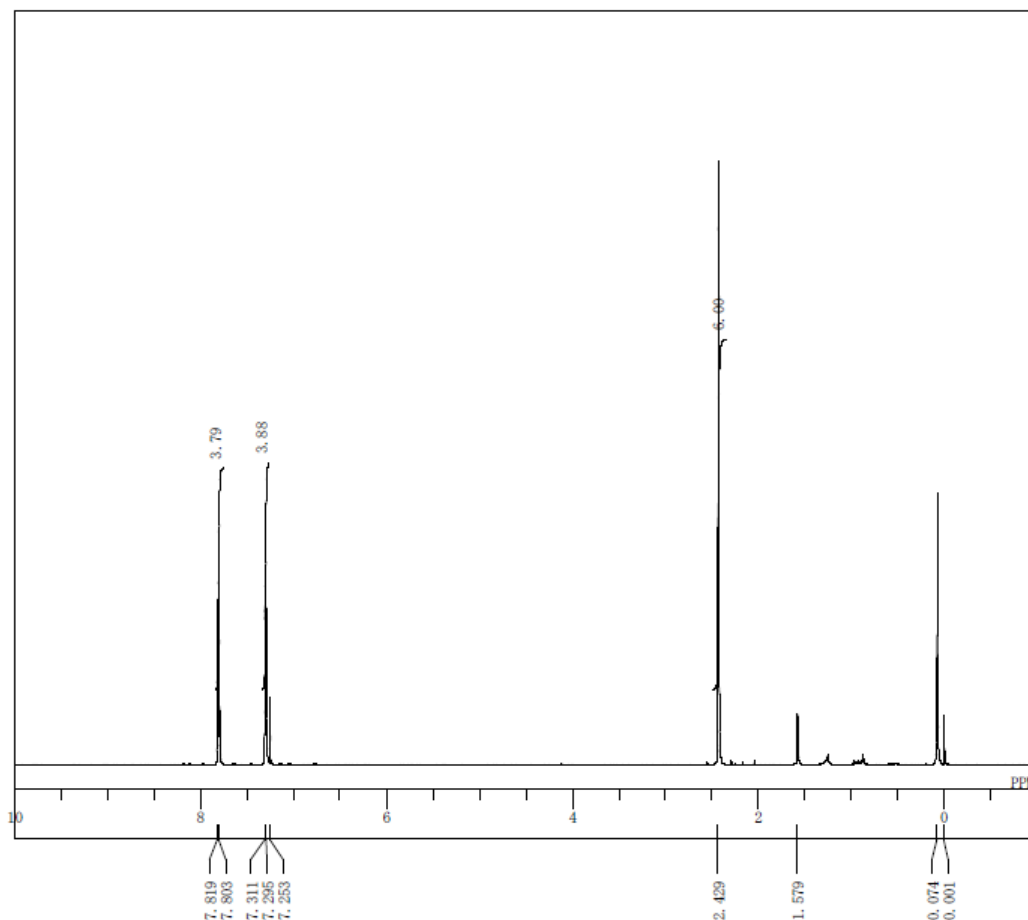
Single Pulse with Broadband Decoupling



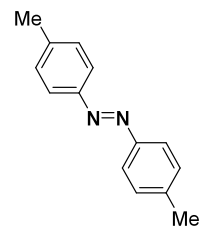
DFILE SN-21-1-13C.1
COMNT Single Pulse with Broadband Decoupling
DATIM 29-05-2009 17:35:39
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EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 65536
FREQU 31446.54 Hz
SCANS 106
ACQTM 1.0420 sec
PD 1.0000 sec
PW1 4.17 usec
IRNUC 1H
CTEMP 26.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 30



Single Pulse Experiment

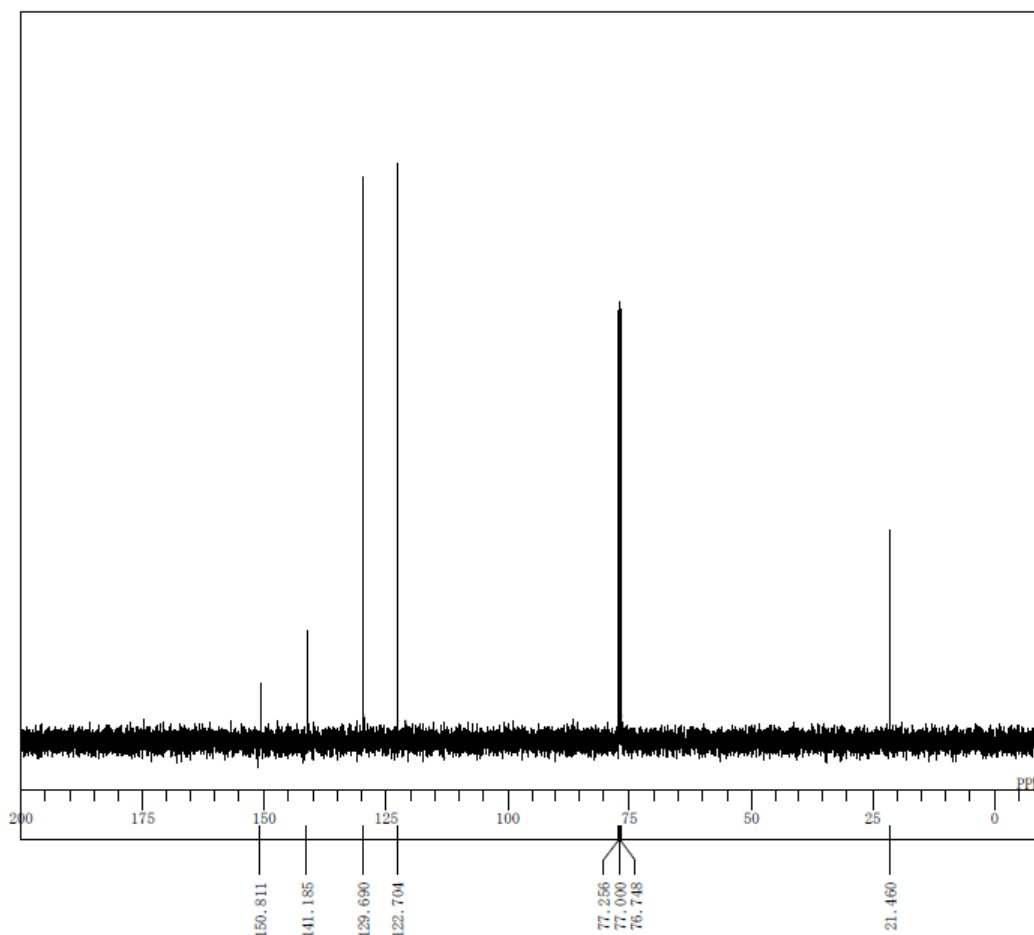


DFILE KF-530-before-1H.1
COMNT Single Pulse Experiment
DATIM 12-09-2008 16:10:34
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EXMOD single_pulse.exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 32768
FREQU 7507.51 Hz
SCANS 8
ACQTM 2.1823 sec
PD 4.0000 sec
PW1 7.00 usec
IRNUC
CTEMP 21.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.23 Hz
RGAIN 15

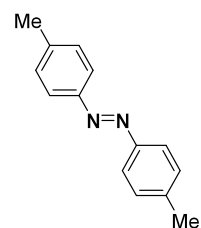


4d

Single Pulse with Broadband Decoupling

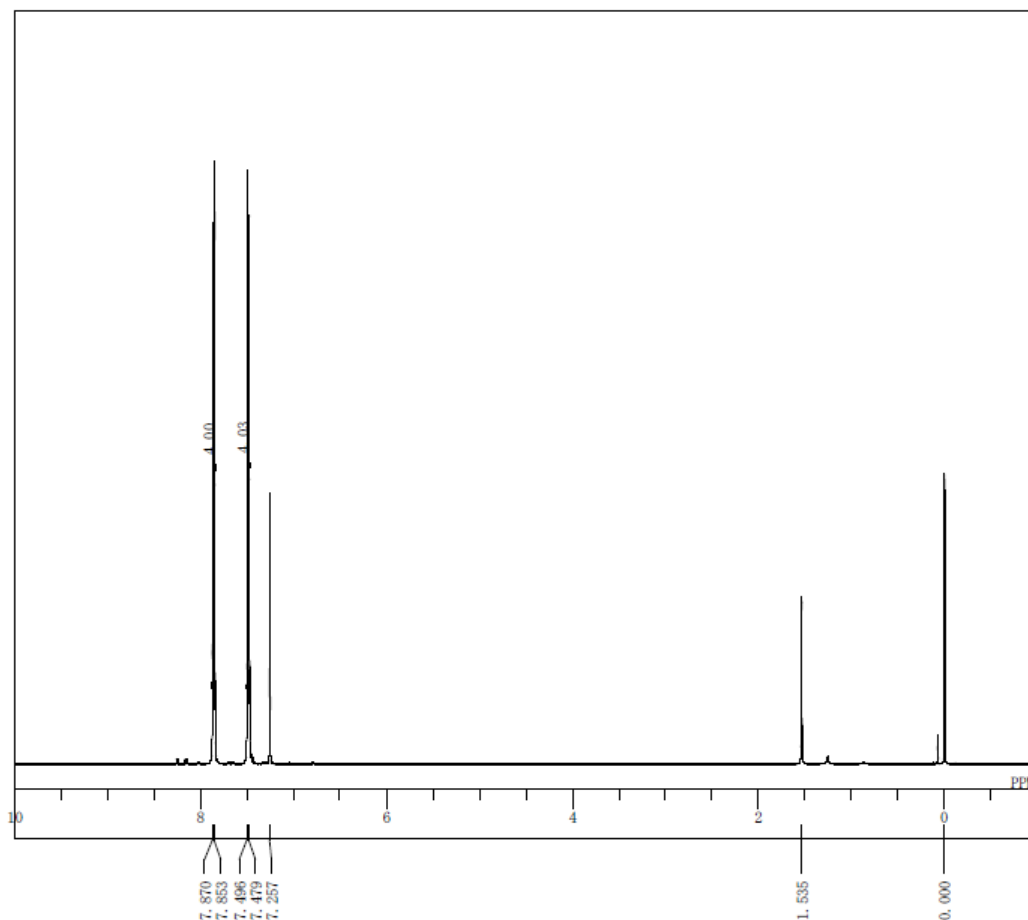


DFILE KF-530-before-13C.1
COMNT Single Pulse with Broadband Decoupling
DATIM 12-09-2008 16:15:02
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 65536
FREQU 31446.54 Hz
SCANS 106
ACQTM 1.0420 sec
PD 1.0000 sec
PW1 4.17 usec
IRNUC 1H
CTEMP 24.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 30

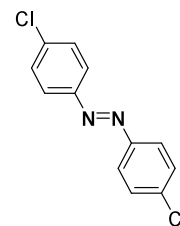


4d

Single Pulse Experiment

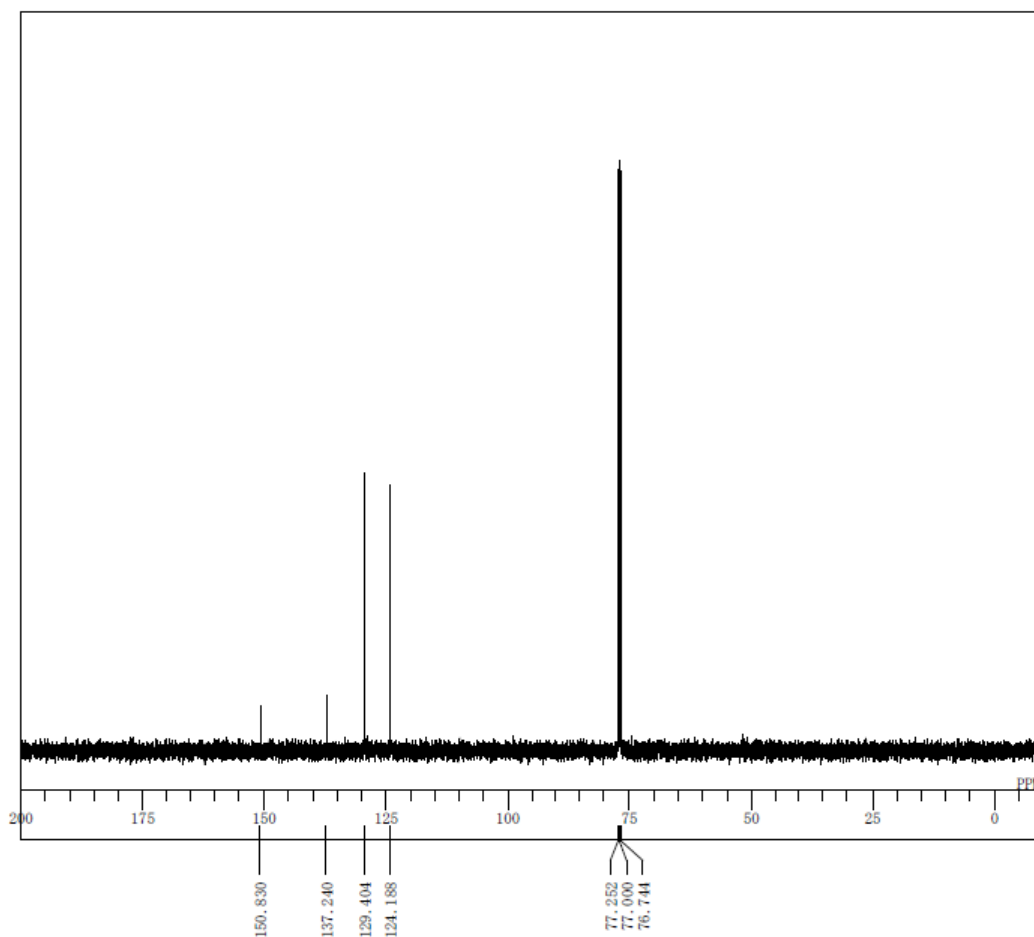


DFILE KF-599-t-2-1H.1
COMNT Single Pulse Experiment
DATIM 09-01-2009 16:08:50
OBNUC 1H
EXMOD single_pulse.exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 32768
FREQU 7507.61 Hz
SCANS 8
ACQTM 2.1823 sec
PD 4.0000 sec
PW1 7.00 usec
IRNUC
CTEMP 27.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.23 Hz
RGAIN 22

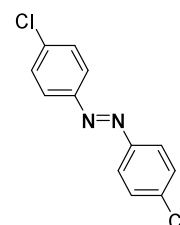


4e

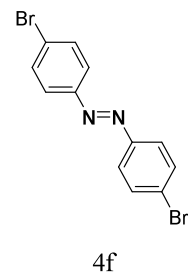
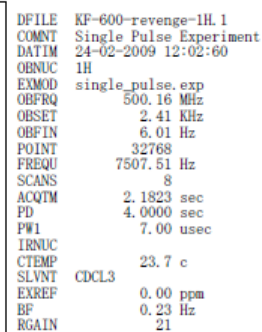
Single Pulse with Broadband Decoupling



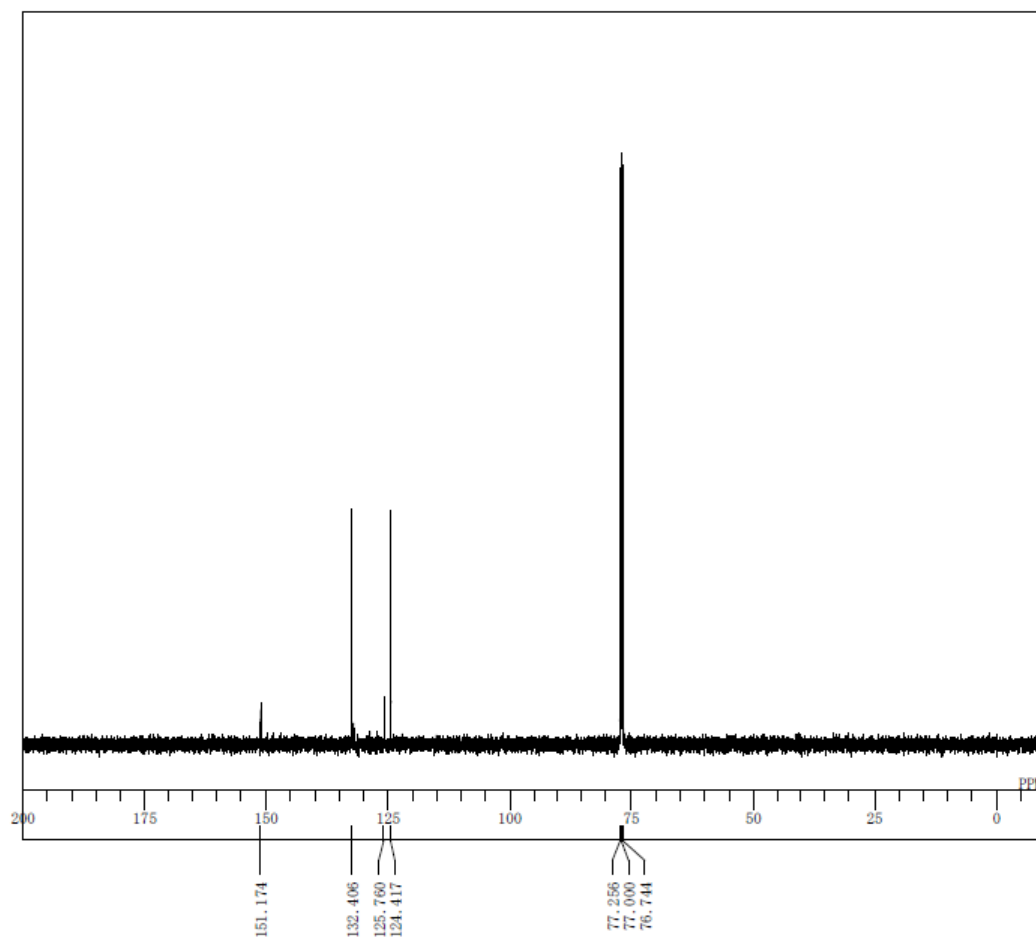
DFILE KF-599-t-2-13C.1
COMNT Single Pulse with Broadband Decoupling
DATIM 09-01-2009 16:16:34
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 65536
FREQU 31446.54 Hz
SCANS 202
ACQTM 1.0420 sec
PD 1.0000 sec
PW1 4.17 usec
IRNUC 1H
CTEMP 29.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 30



4e



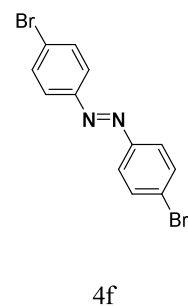
Single Pulse with Broadband Decoupling



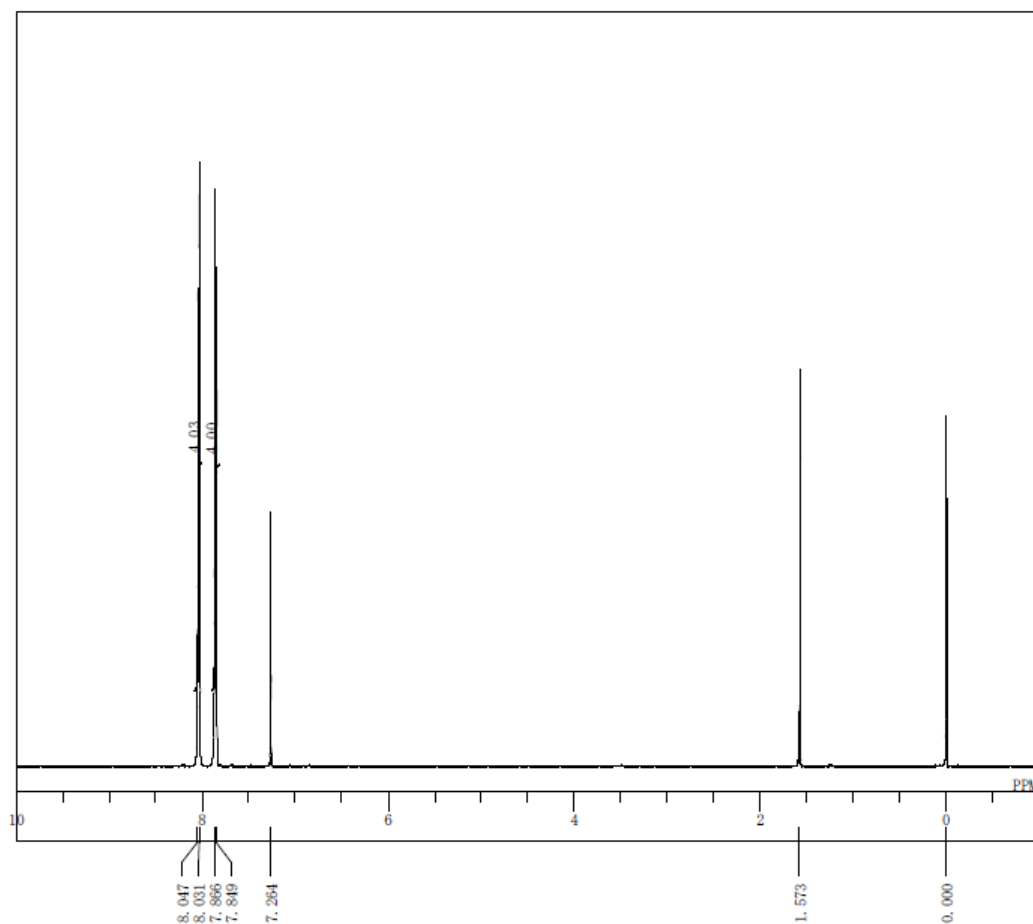
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DFILE      600-revenge-13C.1
COMNT      Single Pulse with Broadband Decoupling
DATUM      26-02-2009 12:12:32
ORNB       13C
EXMOD      single_pulse_dec
OBFRQ      125.77 MHz
OBSET      7.87 KHz
OBFIN      4.21 Hz
POINT      65536
FREQU      31446.54 Hz
SCANS      675
AQTM       1.0420 sec
PD          1.0000 sec
PW1         4.17 usec
IRNUC      1H
CTEMP      27.1 c
SLVNT      CDCL3
EXREF      77.00 ppm
BF          1.00 Hz
RGAIN      30

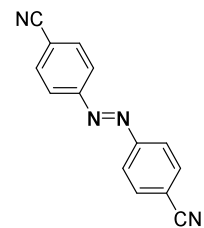
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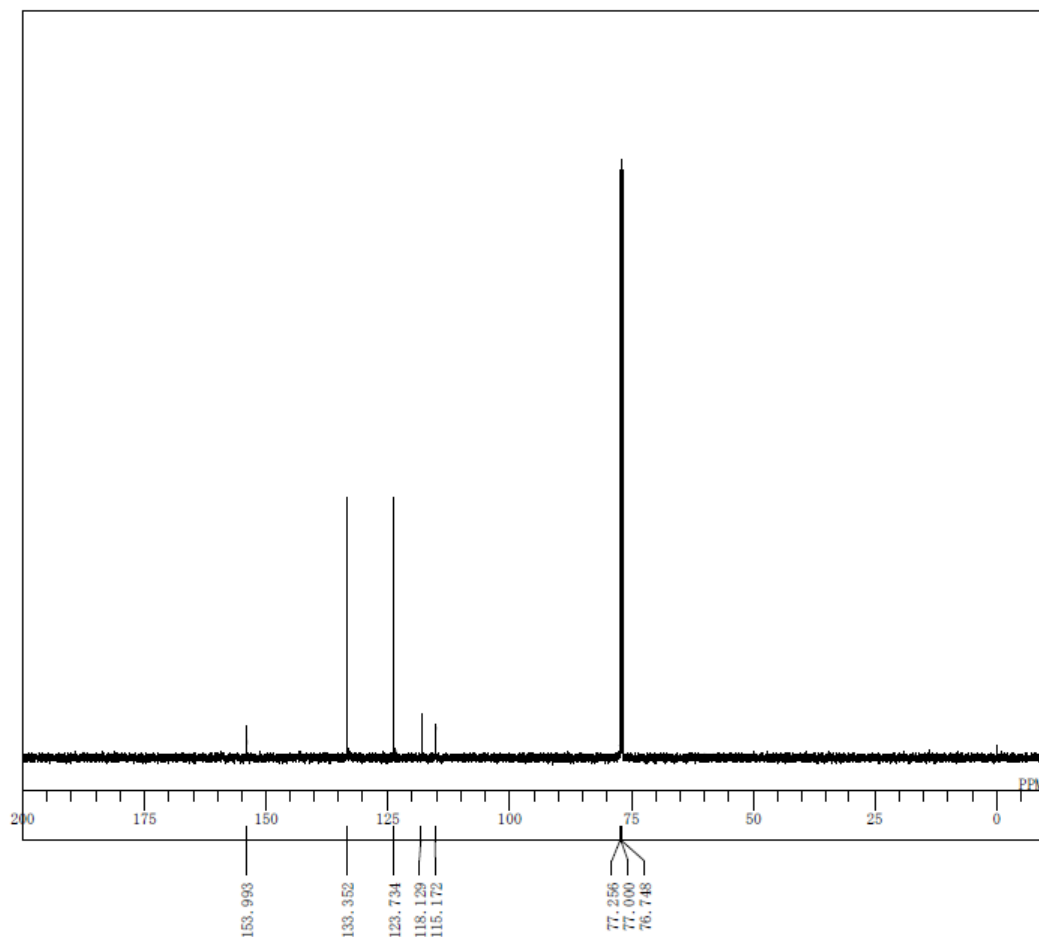
Single Pulse Experiment



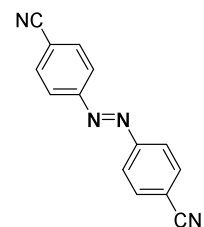
DFILE KF-CN-azo-1H.1
COMNT Single Pulse Experiment
DATIM 05-11-2009 18:16:56
OBNUC 1H
EXMOD single_pulse.exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 32768
FREQU 7507.61 Hz
SCANS 8
AQTM 2.1823 sec
PD 4.0000 sec
PW1 7.00 usec
IRNUC
CTEMP 22.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.23 Hz
RGAIN 21



4g

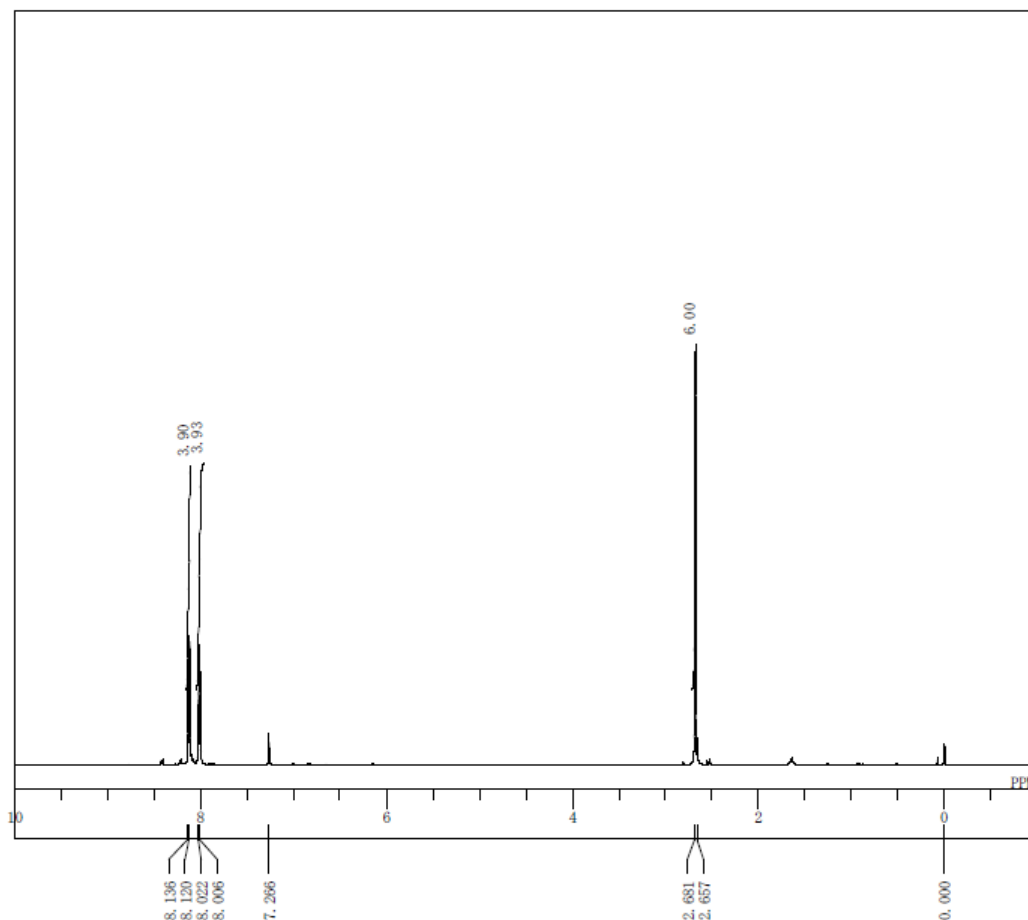


COMNT Single Pulse with Broadband Decoupling
DATIM 19-09-2009 09:51:07
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 65536
FREQU 31446.54 Hz
SCANS 1345
AQTM 1.0420 sec
PD 1.0000 sec
PW1 4.17 usec
IRNUC 1H
CTEMP 26.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 30

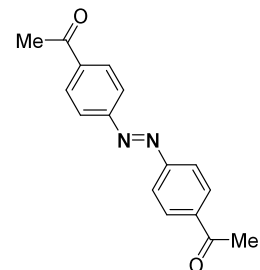


4g

Single Pulse Experiment

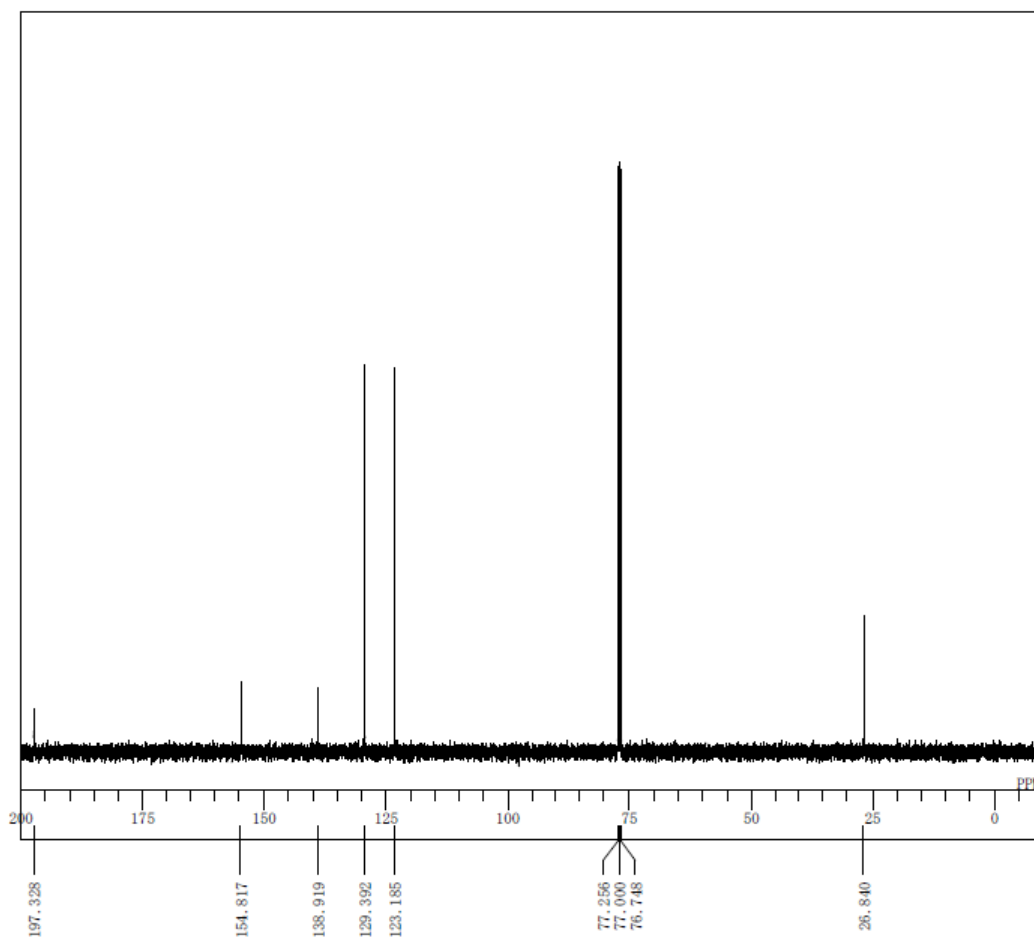


DFILE KF-602-t-1-1H.1
COMNT Single Pulse Experiment
DATIM 26-02-2009 17:11:42
OBNUC 1H
EXMOD single_pulse_exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 32768
FREQU 7507.61 Hz
SCANS 8
ACQTM 2.1823 sec
PD 4.0000 sec
PW1 7.00 usec
IRNUC
CTEMP 23.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.23 Hz
RGAIN 17

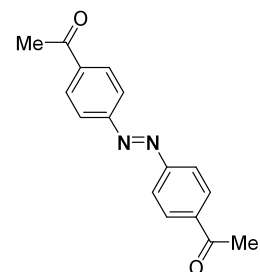


4h

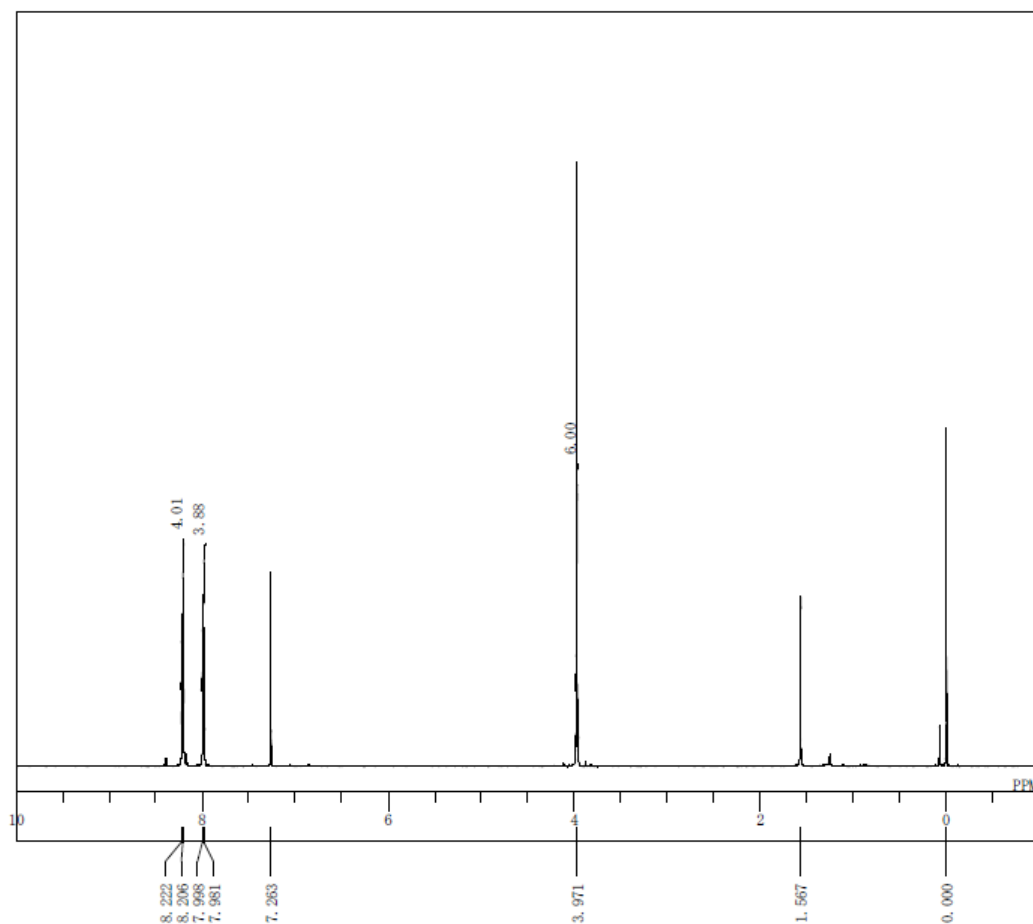
Single Pulse with Broadband Decoupling



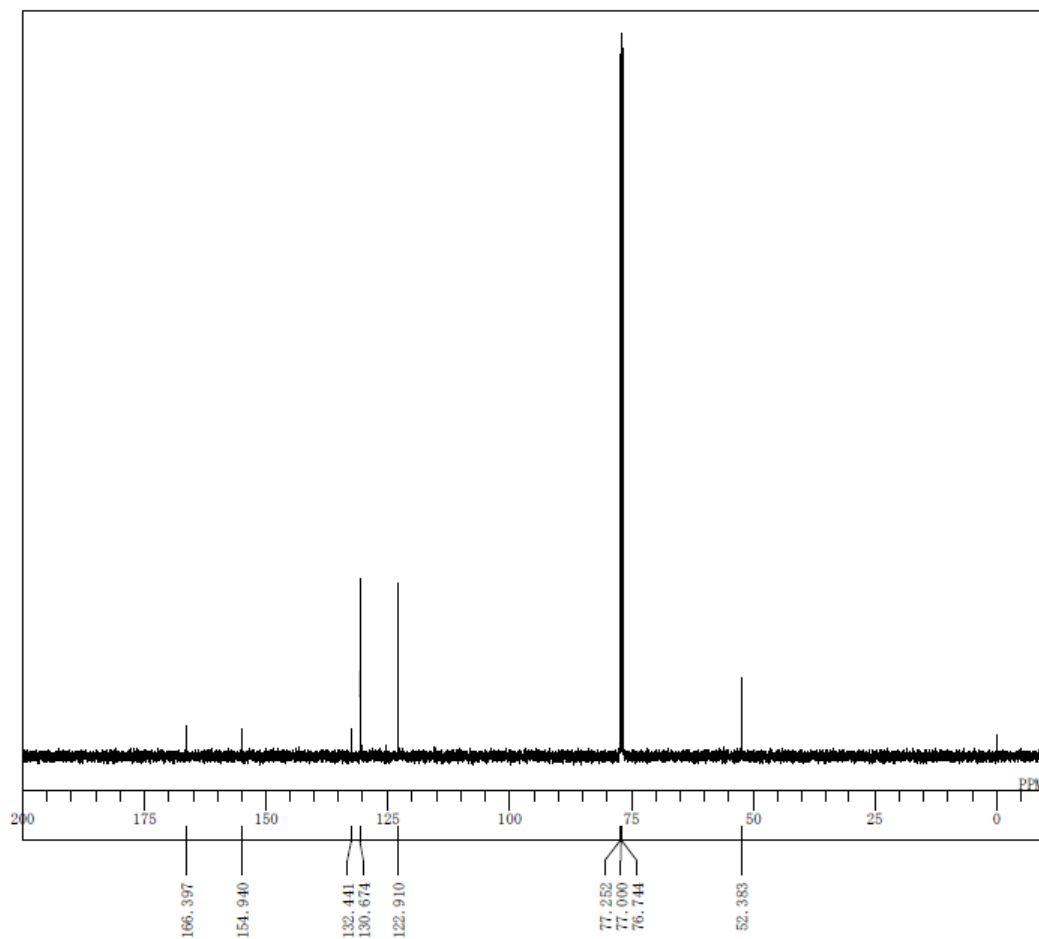
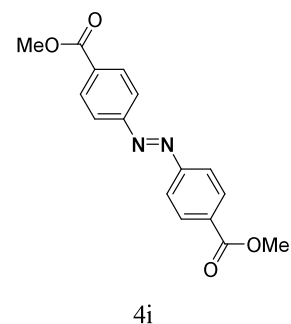
DFILE KF-602-t-1-13C.1
COMNT Single Pulse with Broadband Decoupling
DATIM 26-02-2009 17:19:29
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 65536
FREQU 31446.54 Hz
SCANS 203
ACQTM 1.0420 sec
PD 1.0000 sec
PW1 4.17 usec
IRNUC 1H
CTEMP 26.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 30



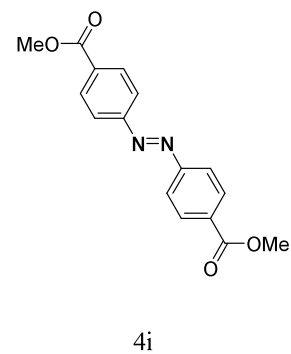
4h

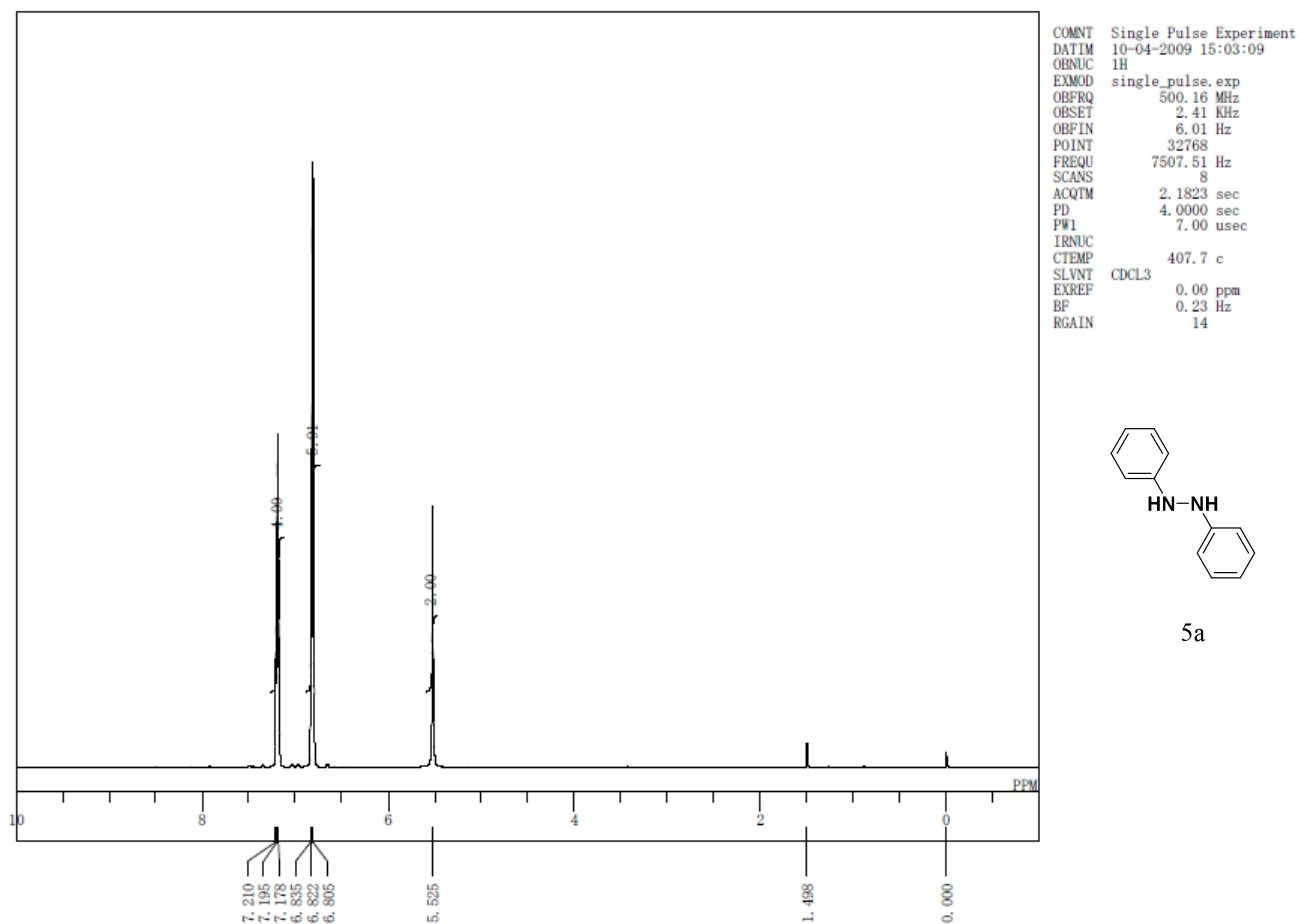


COMNT Single Pulse Experiment
DATIM 11-11-2009 13:15:28
OBNUC 1H
EXMOD single_pulse.exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 32768
FREQU 7507.51 Hz
SCANS 8
AQTM 2.1823 sec
PD 4.0000 sec
PW1 7.00 usec
IRNUC
CTEMP 22.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.23 Hz
RGAIN 22

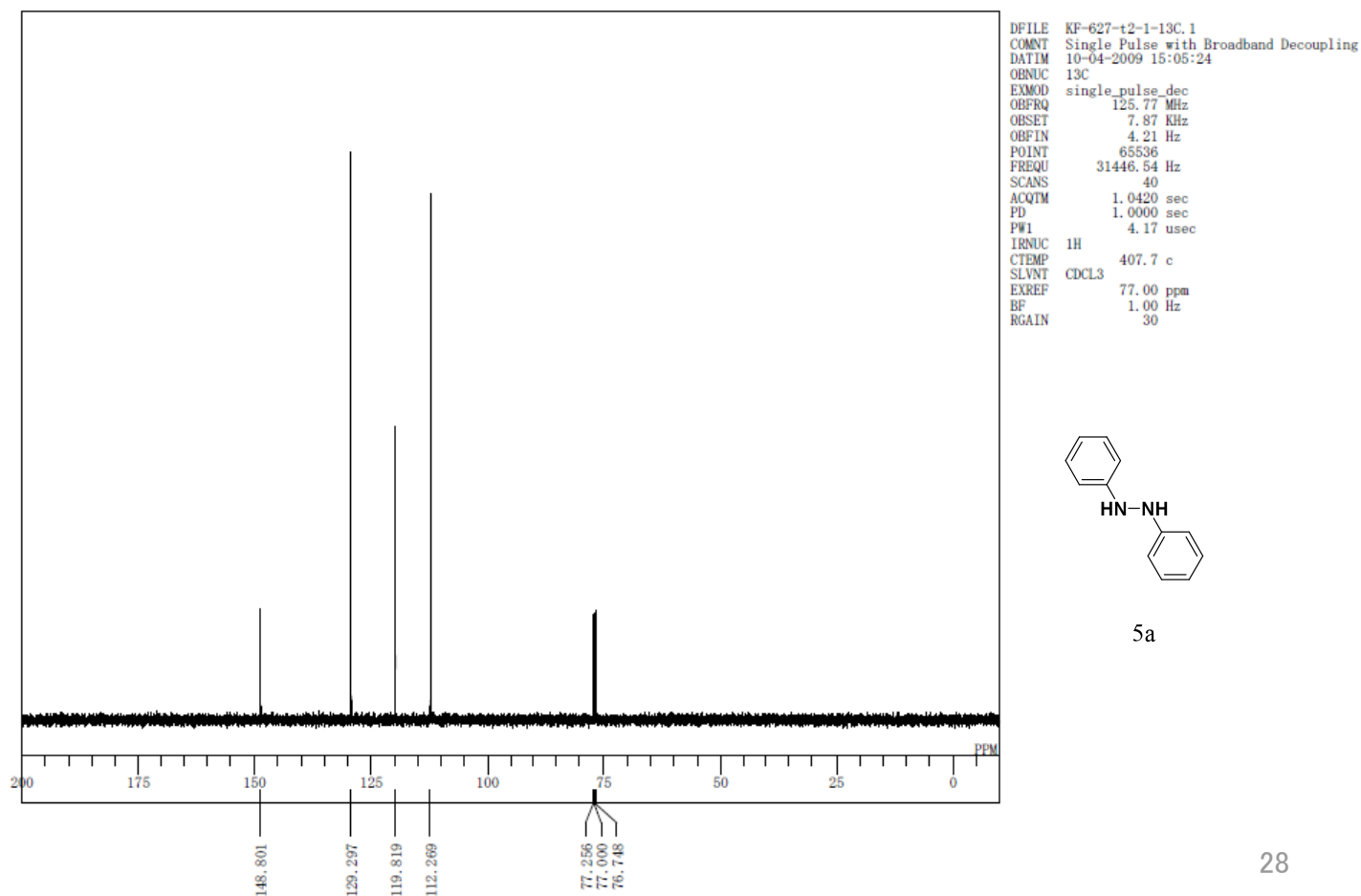


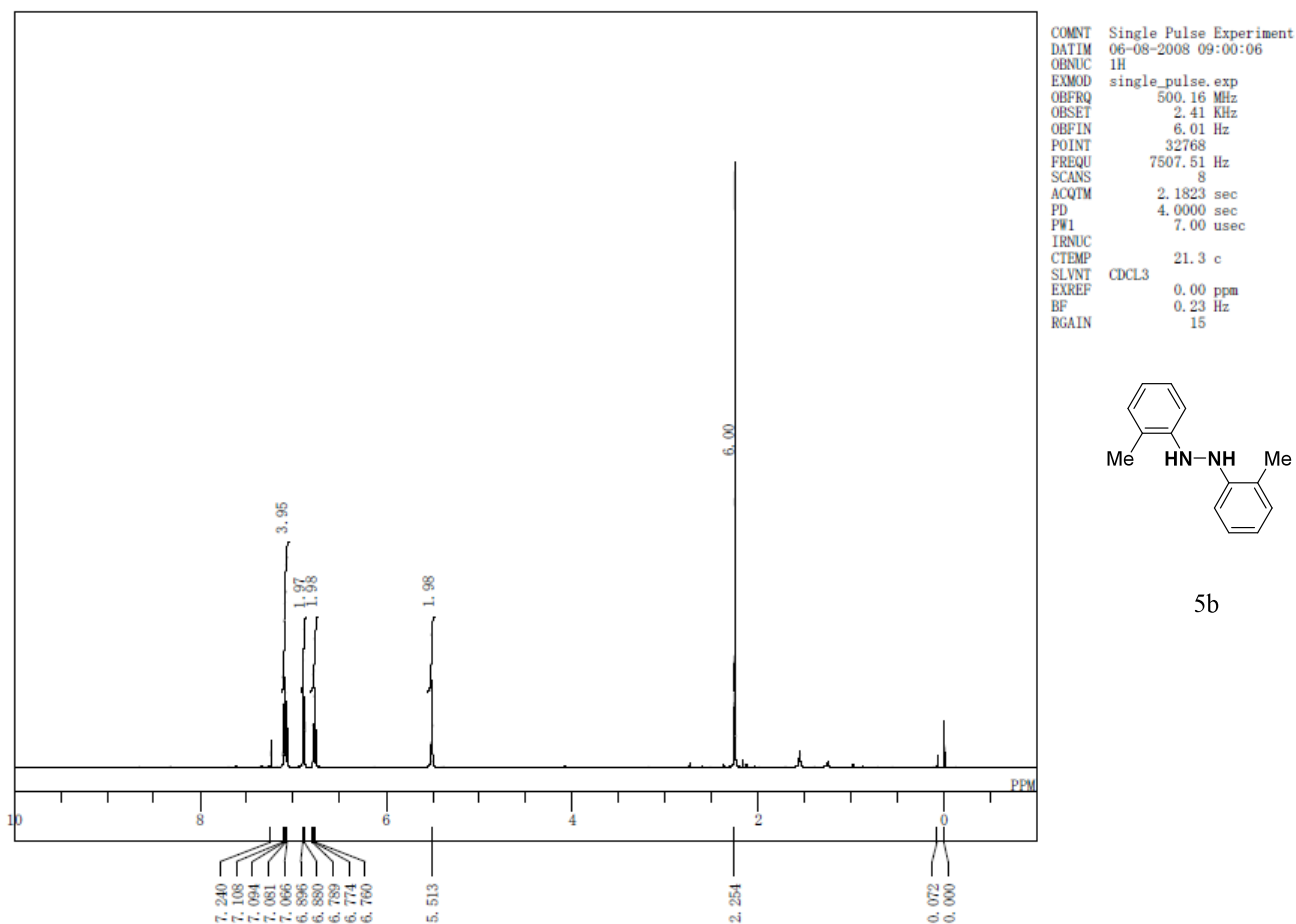
COMNT Single Pulse with Broadband Decoupling
DATIM 11-11-2009 13:50:27
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 65536
FREQU 31446.54 Hz
SCANS 1002
AQTM 1.0420 sec
PD 1.0000 sec
PW1 4.17 usec
IRNUC 1H
CTEMP 26.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 30



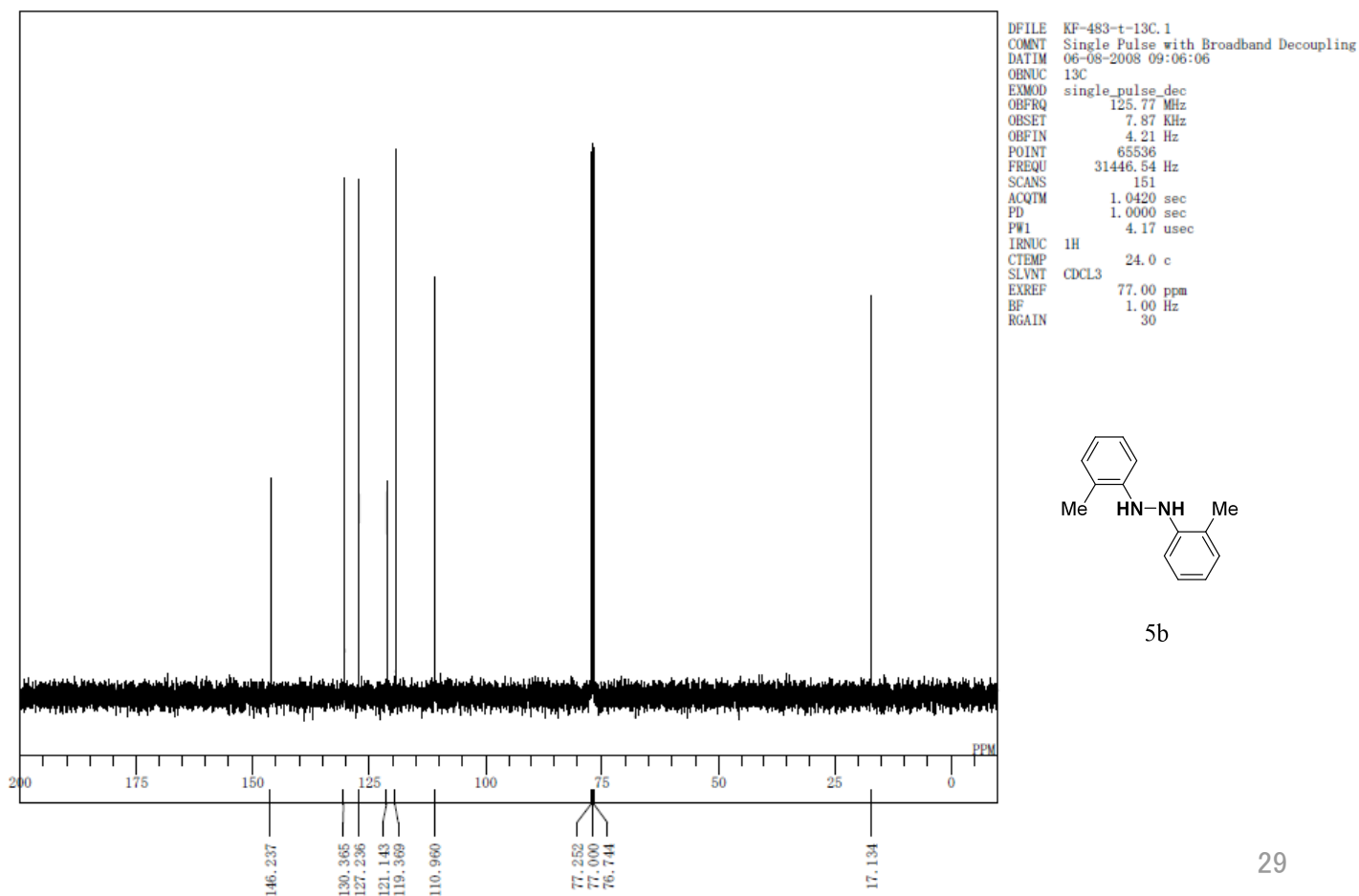


Single Pulse with Broadband Decoupling

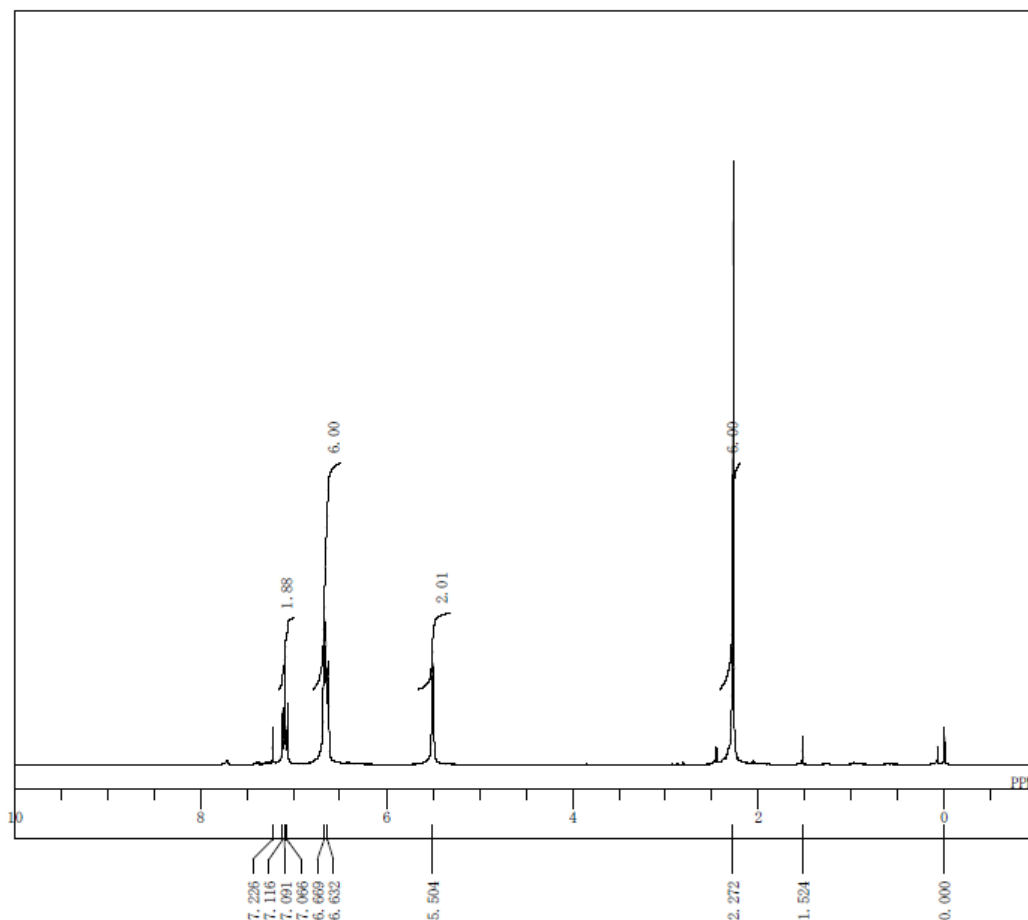




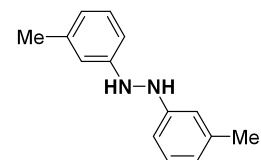
Single Pulse with Broadband Decoupling



Single Pulse Experiment

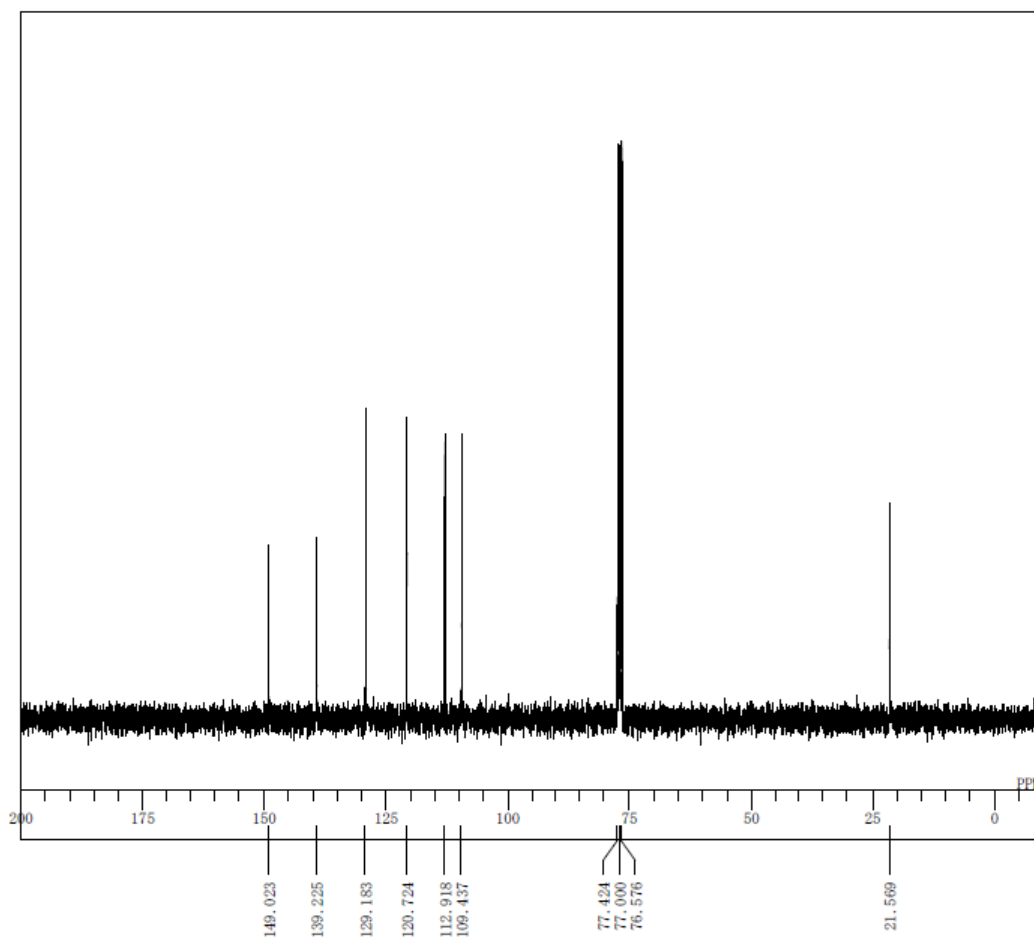


DFILE KF-620-t-2-1H.1
COMNT Single Pulse Experiment
DATIM 13-03-2009 16:20:13
1H
OBNUC
EXMOD single_pulse.exp
OBFRQ 300.53 MHz
OBSET 1.15 KHz
OBFIN 8.57 Hz
POINT 32768
FREQU 4508.57 Hz
SCANS 8
ACQTM 3.6340 sec
PD 4.0000 sec
PW1 4.90 usec
IRNUC
CTEMP 23.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.14 Hz
RGAIN 14

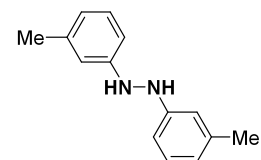


5c

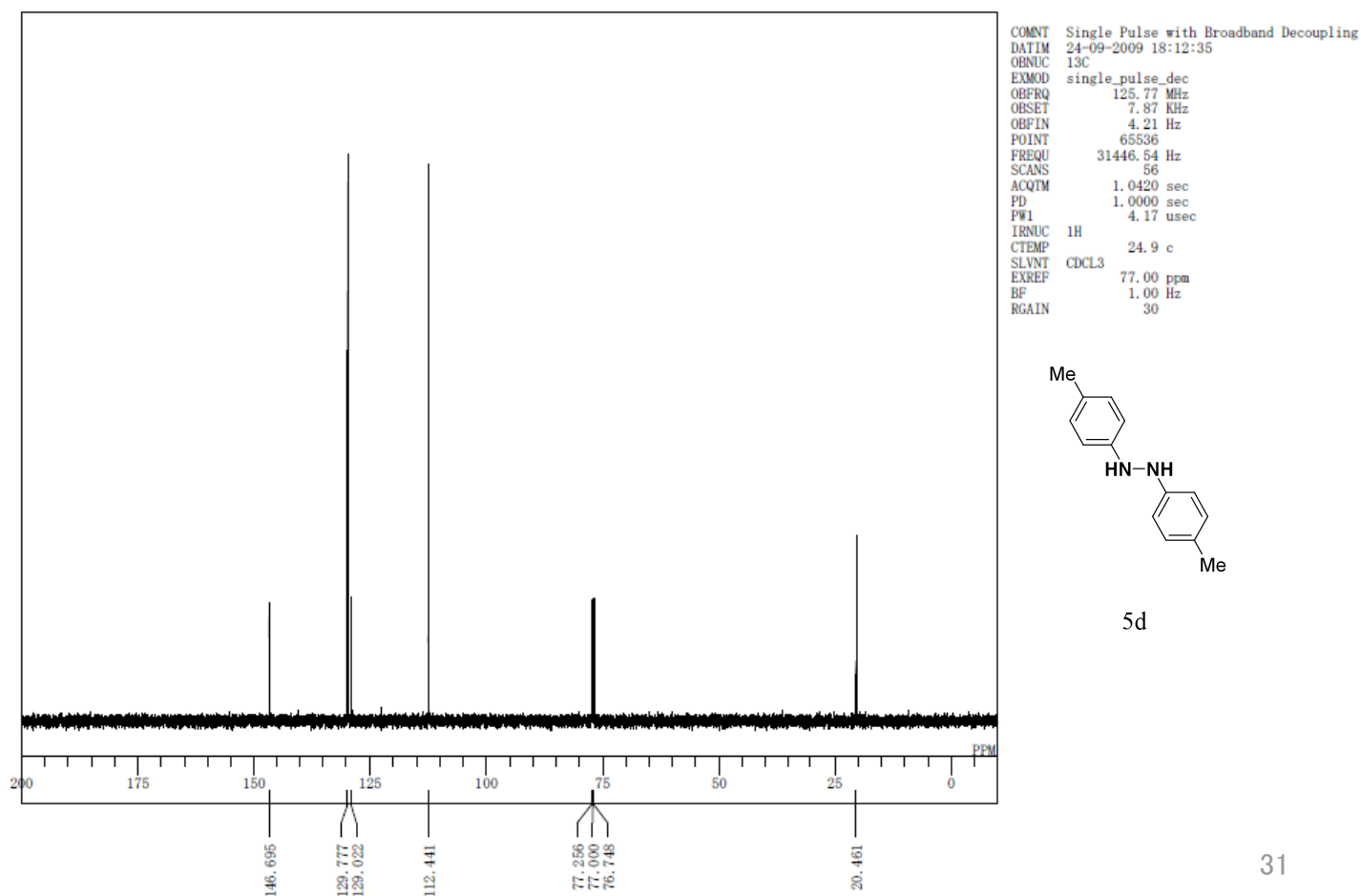
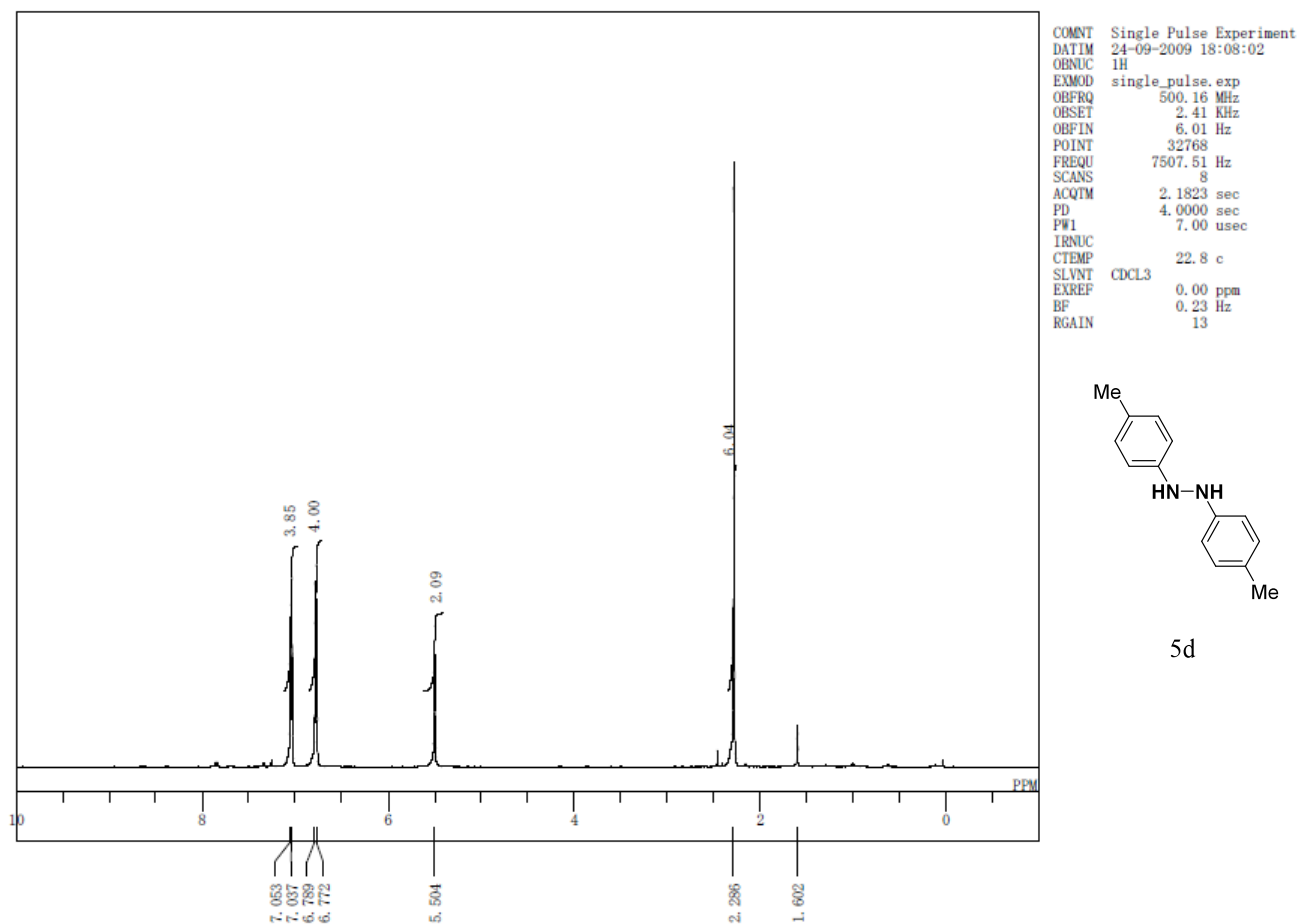
Single Pulse with Broadband Decoupling



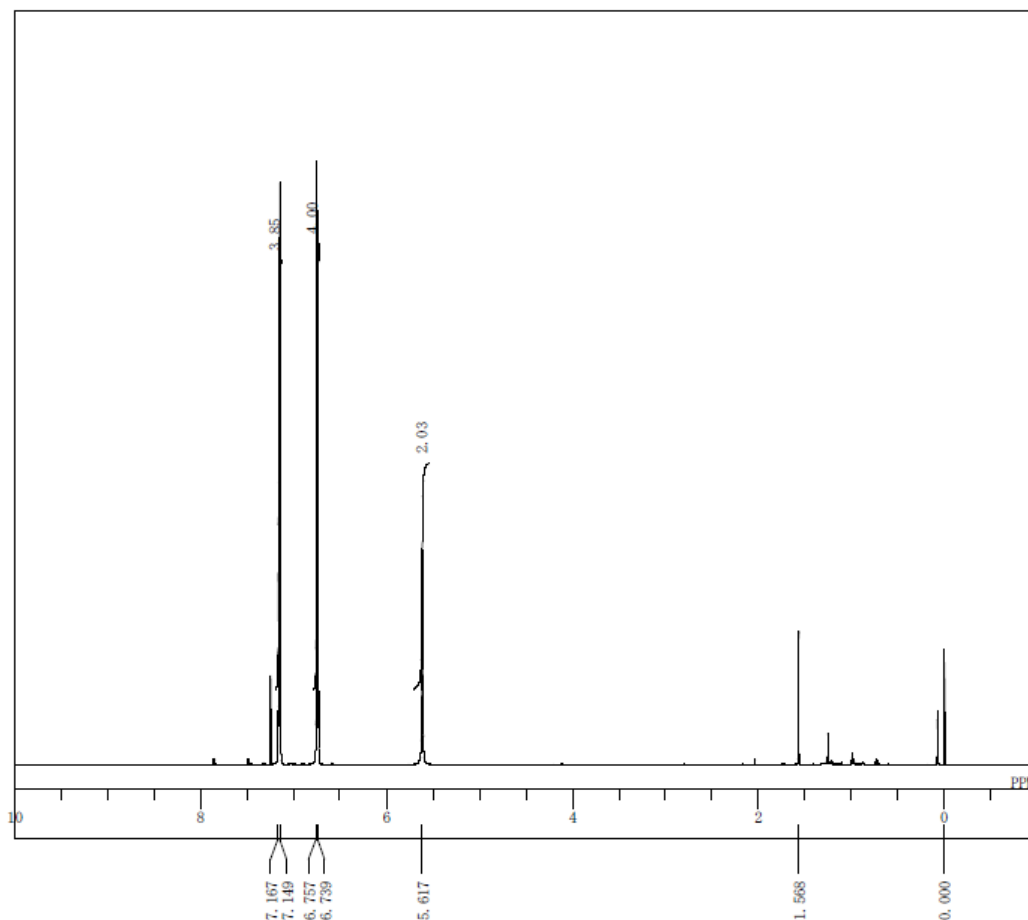
DFILE KF-620-t-2-13C.1
COMNT Single Pulse with Broadband Decoupling
DATIM 13-03-2009 16:32:19
13C
OBNUC
EXMOD single_pulse_dec
OBFRQ 75.57 MHz
OBSET 5.79 KHz
OBFIN 1.08 Hz
POINT 65536
FREQU 18903.59 Hz
SCANS 206
ACQTM 1.7334 sec
PD 1.0000 sec
PW1 2.63 usec
IRNUC 1H
CTEMP 25.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 30



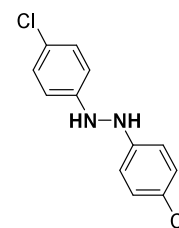
5c



Single Pulse Experiment

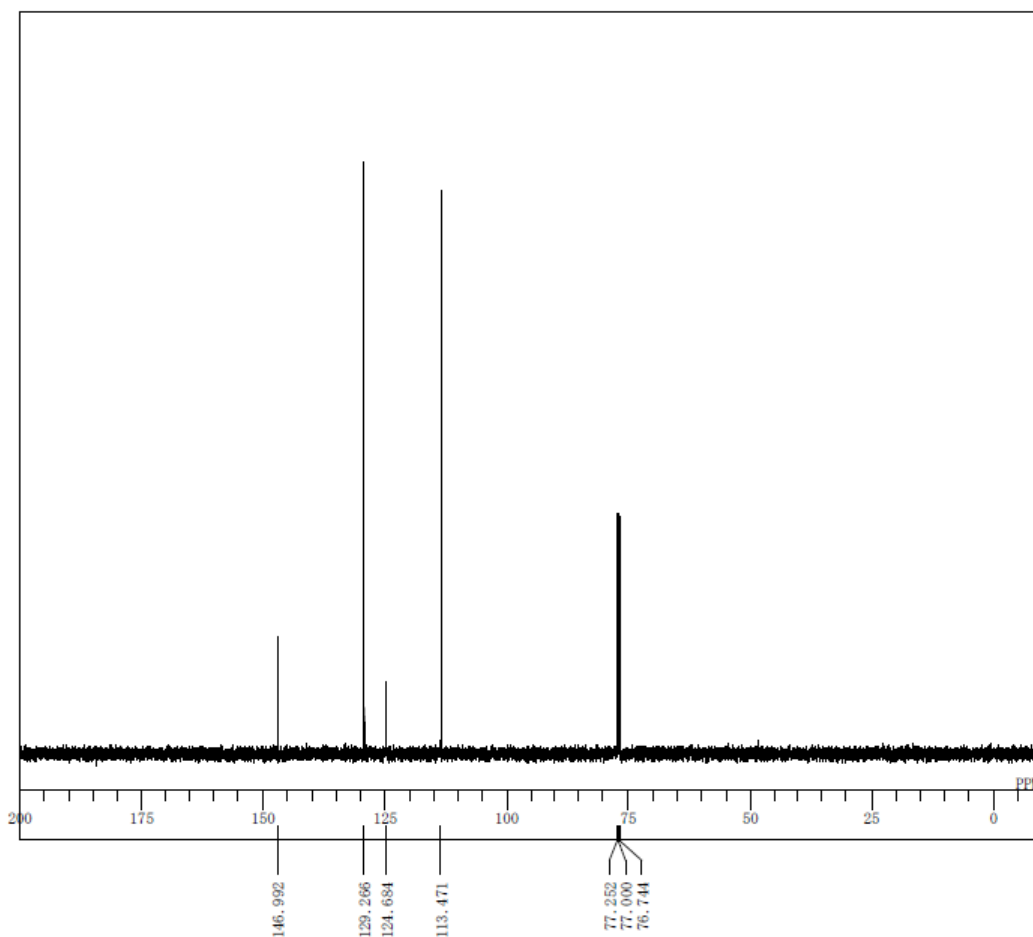


DFILE KF-534-1-1H.1
COMNT Single Pulse Experiment
DATIM 18-09-2008 17:57:29
OBNUC 1H
EXMOD single_pulse_exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 32768
FREQU 7507.51 Hz
SCANS 8
AQTM 2.1823 sec
PD 4.0000 sec
PW1 7.00 usec
IRNUC
CTEMP 22.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.23 Hz
RGAIN 15

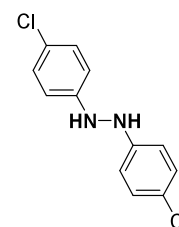


5e

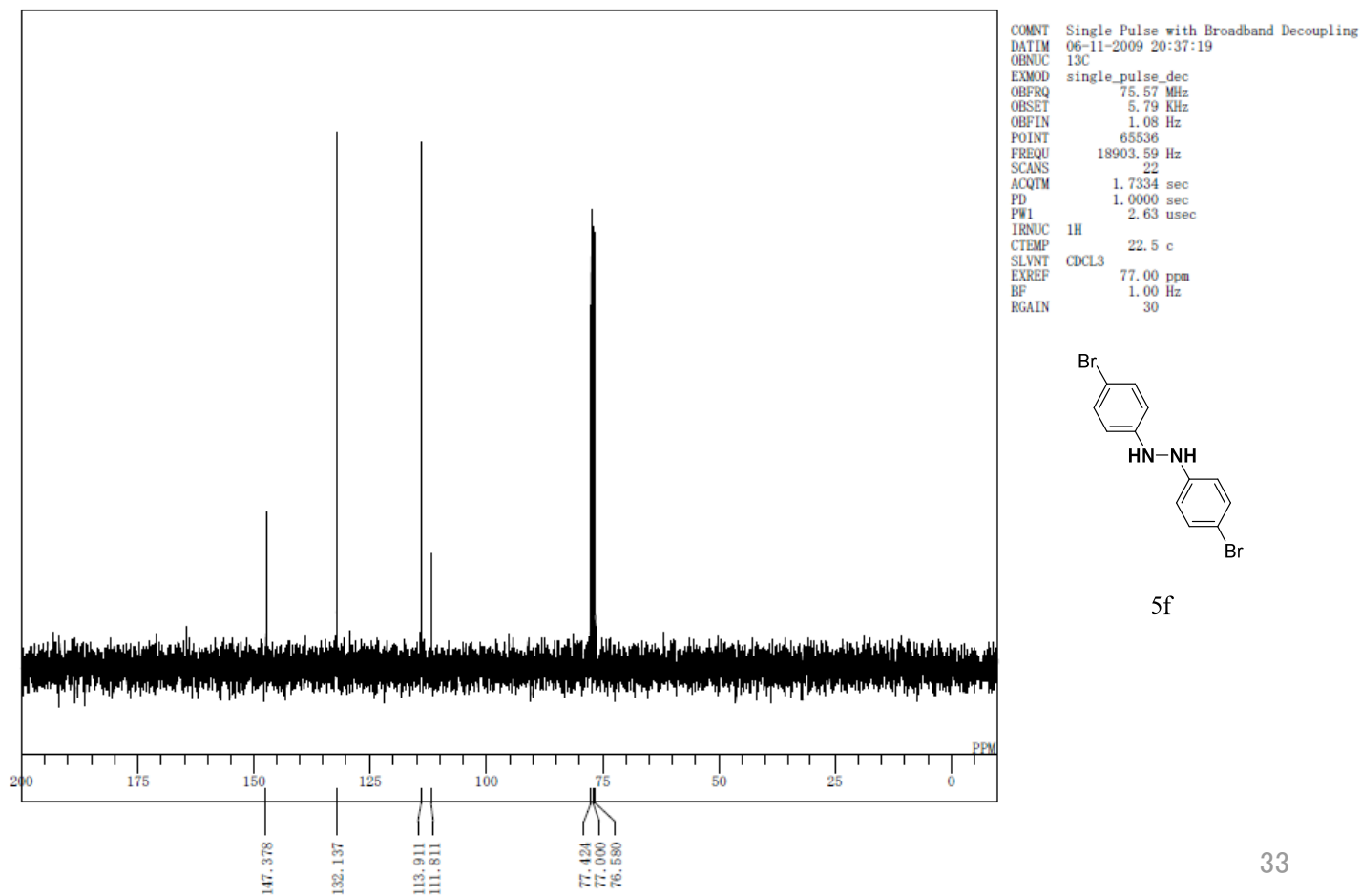
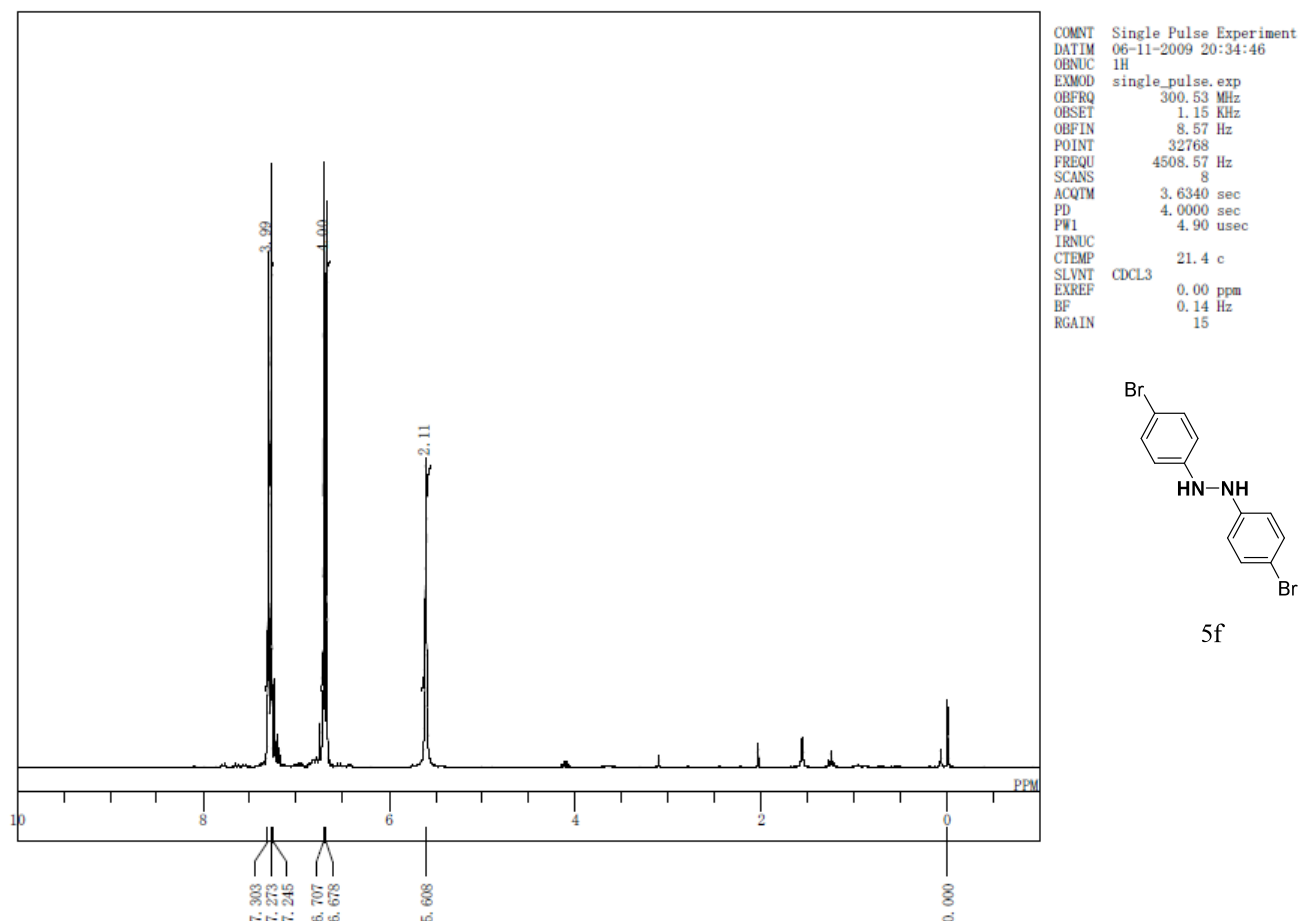
Single Pulse with Broadband Decoupling



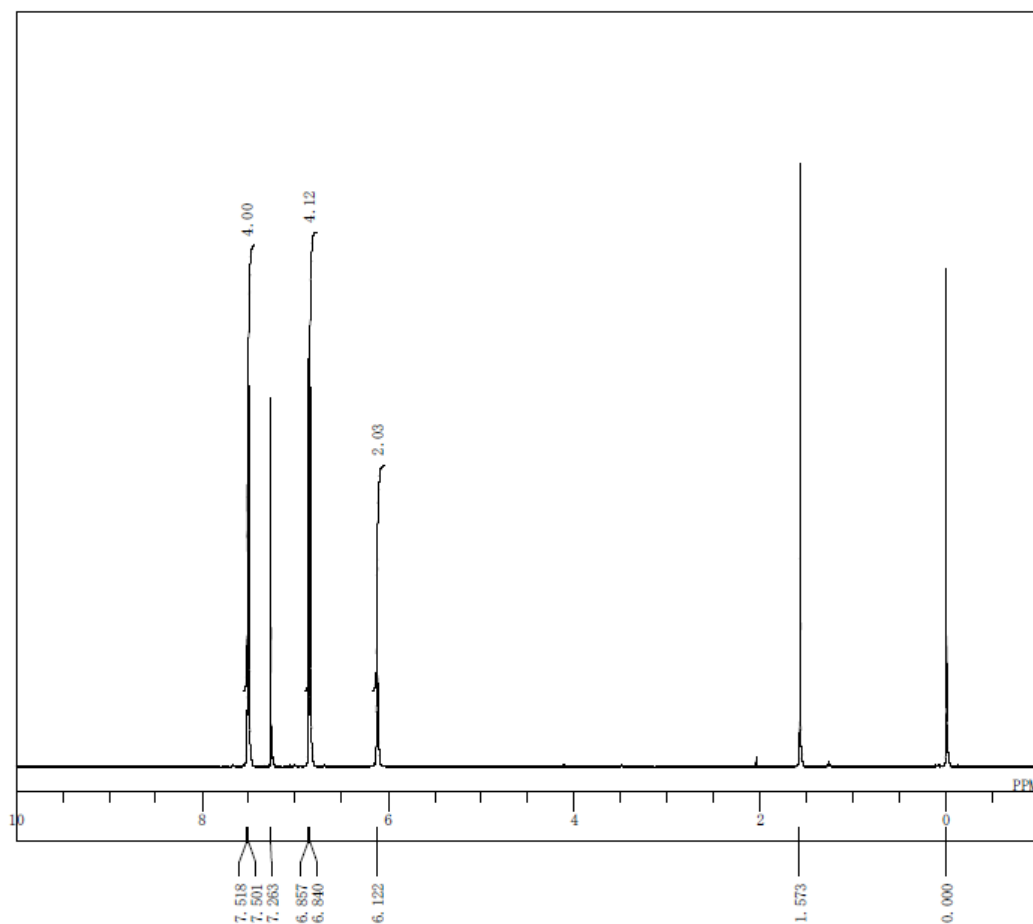
DFILE KF-534-1-13C.1
COMNT Single Pulse with Broadband Decoupling
DATIM 18-09-2008 18:00:55
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 65536
FREQU 31446.54 Hz
SCANS 75
AQTM 1.0420 sec
PD 1.0000 sec
PW1 4.17 usec
IRNUC 1H
CTEMP 25.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 30



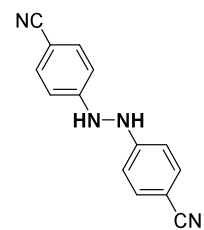
5e



Single Pulse Experiment

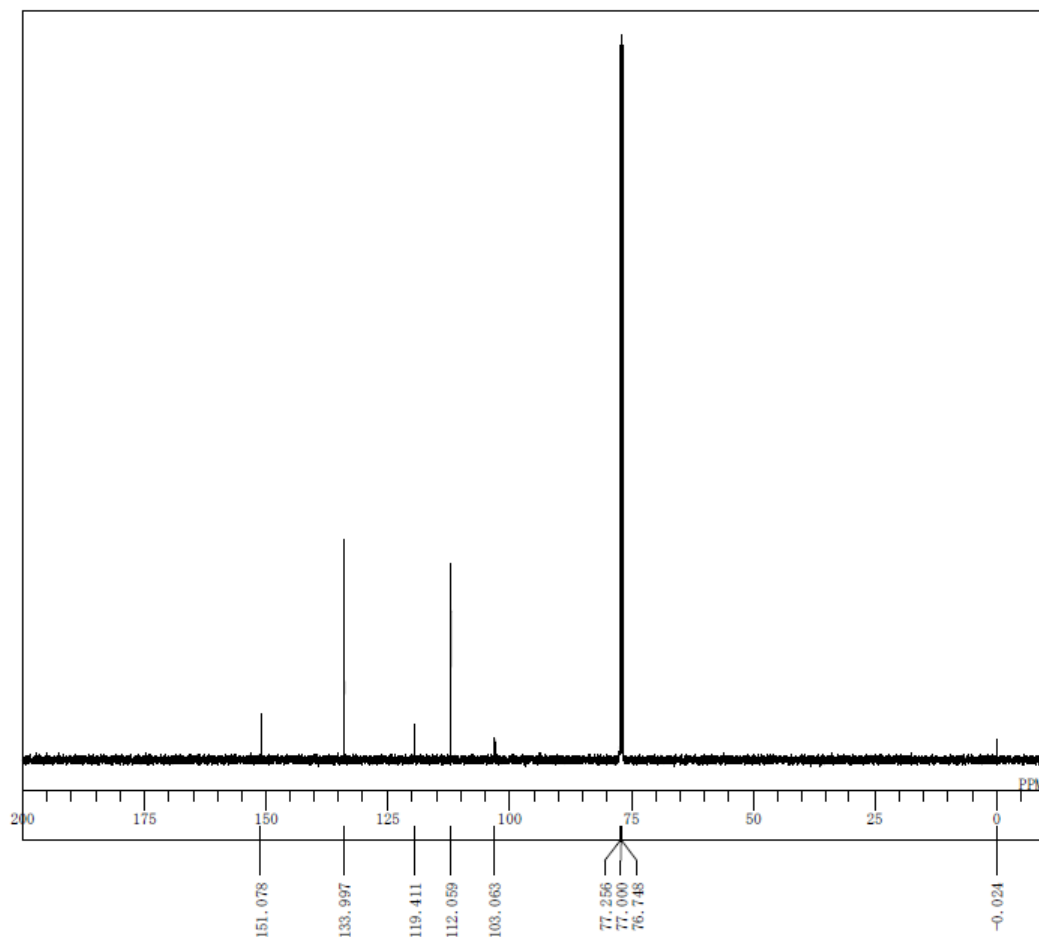


DFILE KF-848-t3-3-1H.1
COMNT Single Pulse Experiment
DATIM 07-11-2009 18:03:02
OBNUC 1H
EXMOD single_pulse_exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 32768
FREQU 7507.51 Hz
SCANS 8
ACQTM 2.1823 sec
PD 4.0000 sec
PW1 7.00 usec
IRNUC
CTEMP 22.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.23 Hz
RGAIN 23

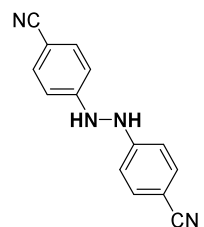


5g

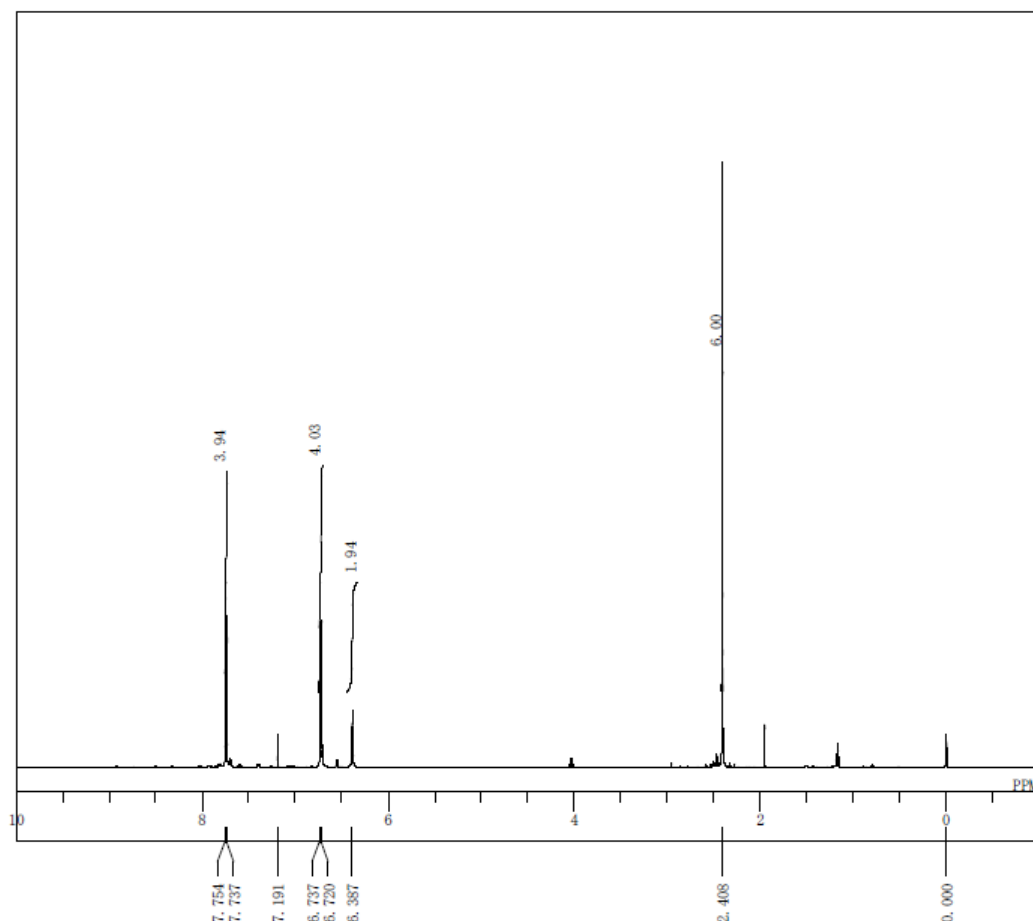
Single Pulse with Broadband Decoupling



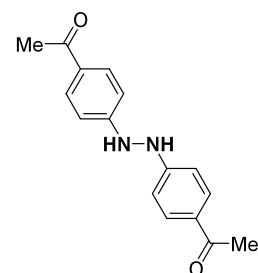
DFILE KF-848-t3-3-2-13C.1
COMNT Single Pulse with Broadband Decoupling
DATIM 07-11-2009 19:50:21
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 65536
FREQU 31446.54 Hz
SCANS 2067
ACQTM 1.0420 sec
PD 1.0000 sec
PW1 4.17 usec
IRNUC 1H
CTEMP 26.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 30



5g

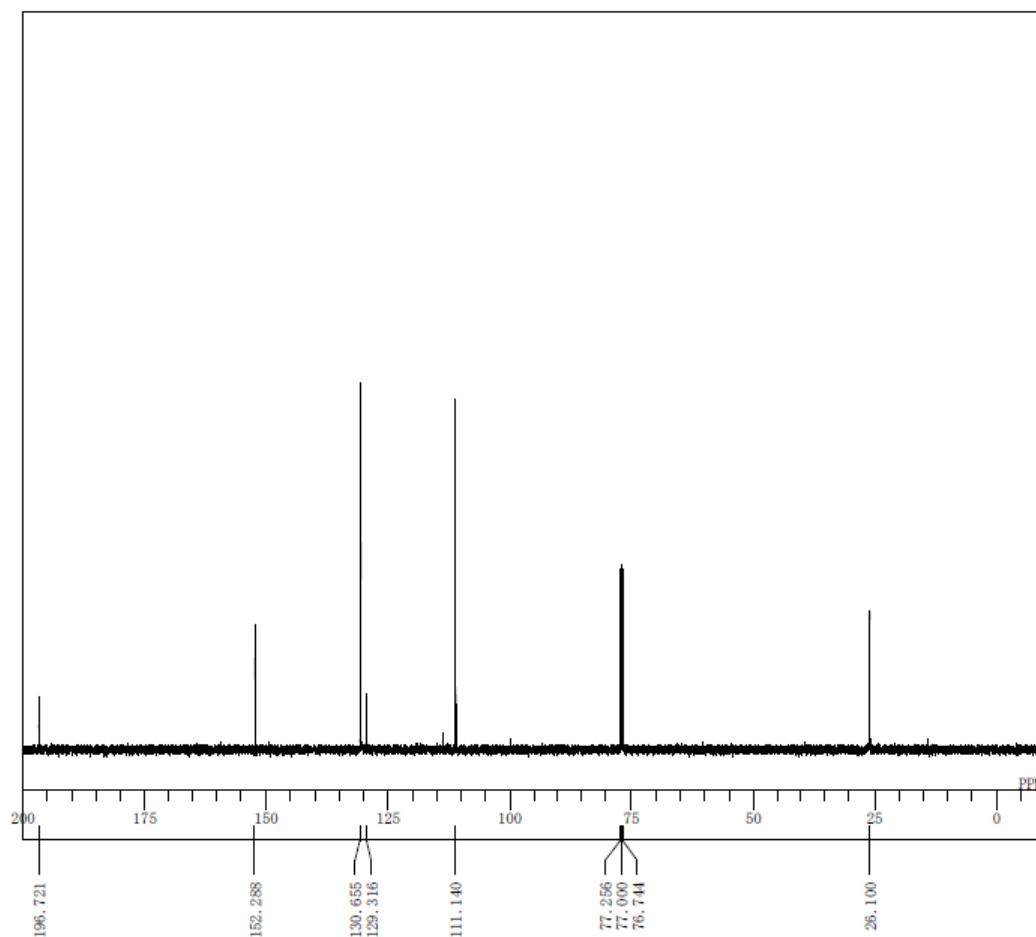


COMET Single Pulse Experiment
DATIM 25-09-2008 00:14:49
OBNUC 1H
EXMOD single_pulse.exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 32768
FREQU 7507.51 Hz
SCANS 8
AQTM 2.1823 sec
PD 4.0000 sec
PW1 7.00 usec
IRNUC
CTEMP 25.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.23 Hz
RGAIN 13

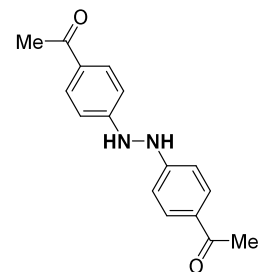


5h

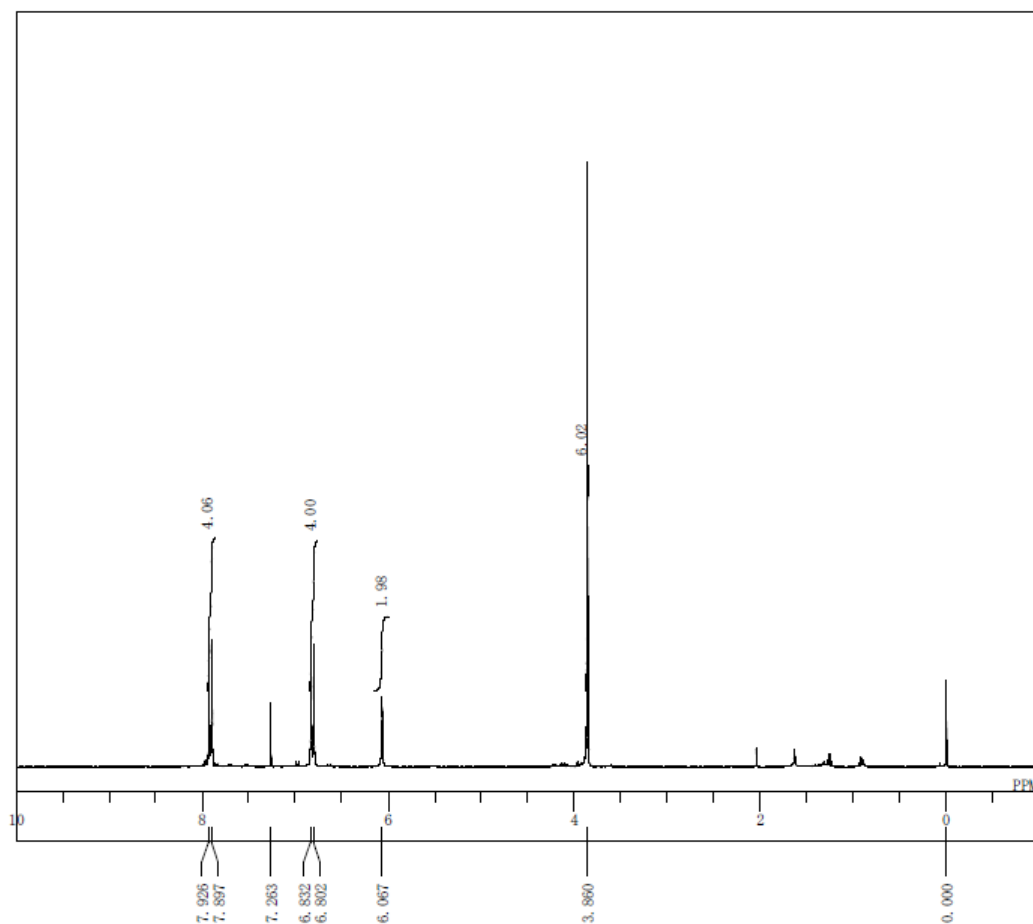
Single Pulse with Broadband Decoupling



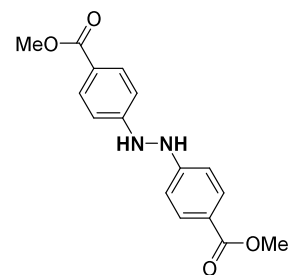
DFILE KF-538-1-2-13C.1
COMET Single Pulse with Broadband Decoupling
DATIM 25-09-2008 00:18:38
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 65536
FREQU 31446.54 Hz
SCANS 87
AQTM 1.0420 sec
PD 1.0000 sec
PW1 4.17 usec
IRNUC 1H
CTEMP 27.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 30



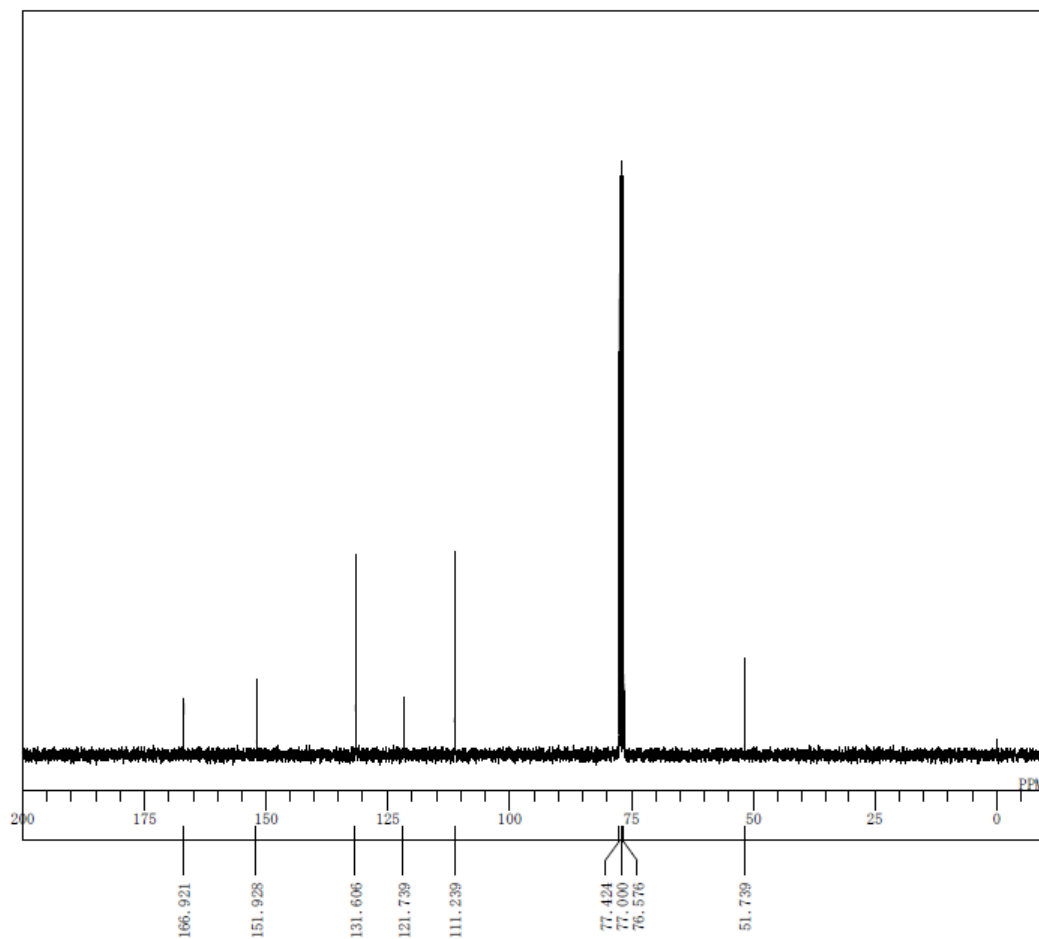
5h



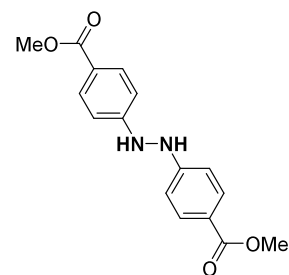
COMNT Single Pulse Experiment
DATIM 11-09-2009 12:51:44
OBNUC 1H
EXMOD single_pulse.exp
OBFRQ 300.53 MHz
OBSET 1.15 KHz
OBFIN 8.57 Hz
POINT 32768
FREQU 4508.57 Hz
SCANS 8
AQTM 3.6340 sec
PD 4.0000 sec
PW1 4.90 usec
IRNUC
CTEMP 22.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.14 Hz
RGAIN 17



5i

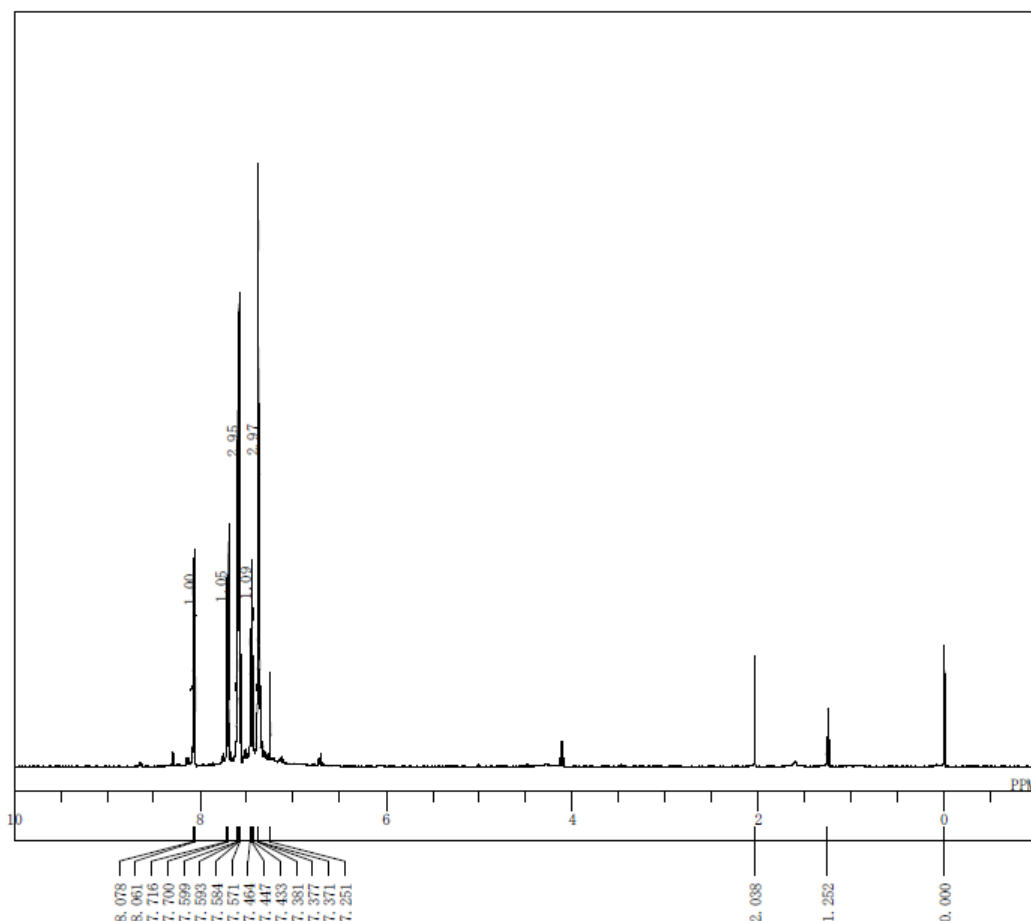


COMNT Single Pulse with Broadband Decoupling
DATIM 11-09-2009 13:25:07
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 75.57 MHz
OBSET 5.79 KHz
OBFIN 1.08 Hz
POINT 65536
FREQU 18903.59 Hz
SCANS 675
AQTM 1.7334 sec
PD 1.0000 sec
PW1 2.63 usec
IRNUC 1H
CTEMP 23.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 30

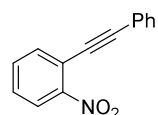


5i

Single Pulse Experiment

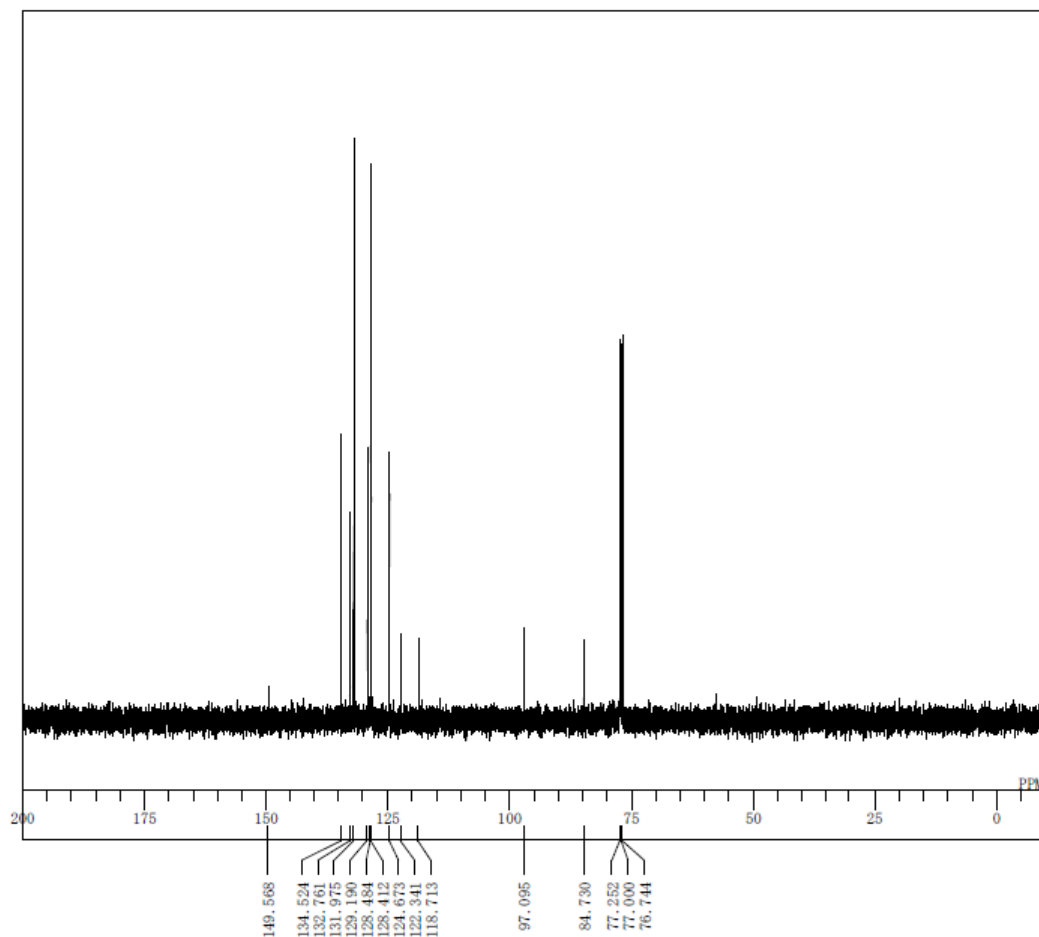


DFILE KF-825-t-3-1H.1
COMNT Single Pulse Experiment
DATIM 22-10-2009 15:15:39
OBNUC 1H
EXMOD single_pulse_exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 32768
FREQU 7507.61 Hz
SCANS 8
ACQTM 2.1823 sec
PD 4.0000 sec
PW1 7.00 usec
IRNUC
CTEMP 22.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.23 Hz
RGAIN 13

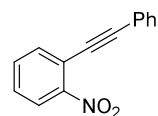


6

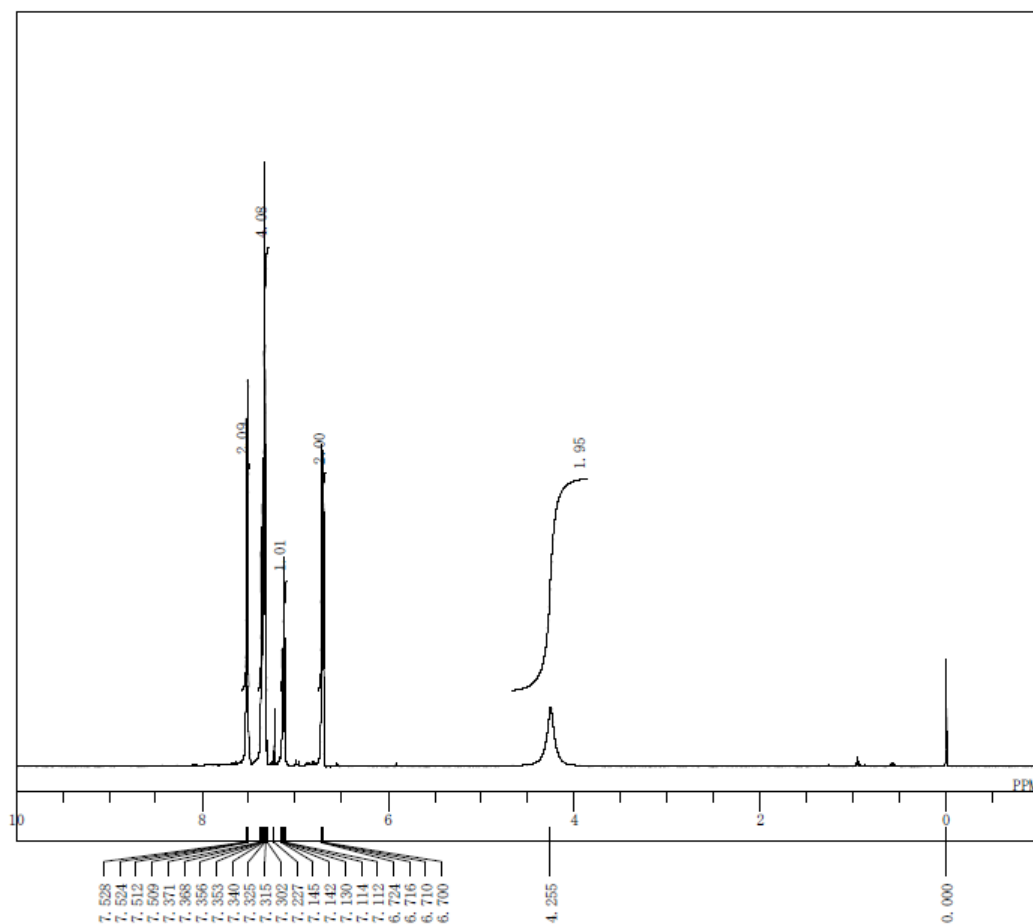
Single Pulse with Broadband Decoupling



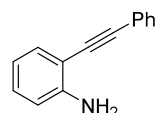
DFILE KF-825-t-3-13C.1
COMNT Single Pulse with Broadband Decoupling
DATIM 22-10-2009 15:19:33
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 65536
FREQU 31446.54 Hz
SCANS 38
ACQTM 1.0420 sec
PD 1.0000 sec
PW1 4.17 usec
IRNUC 1H
CTEMP 24.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 30



Single Pulse Experiment

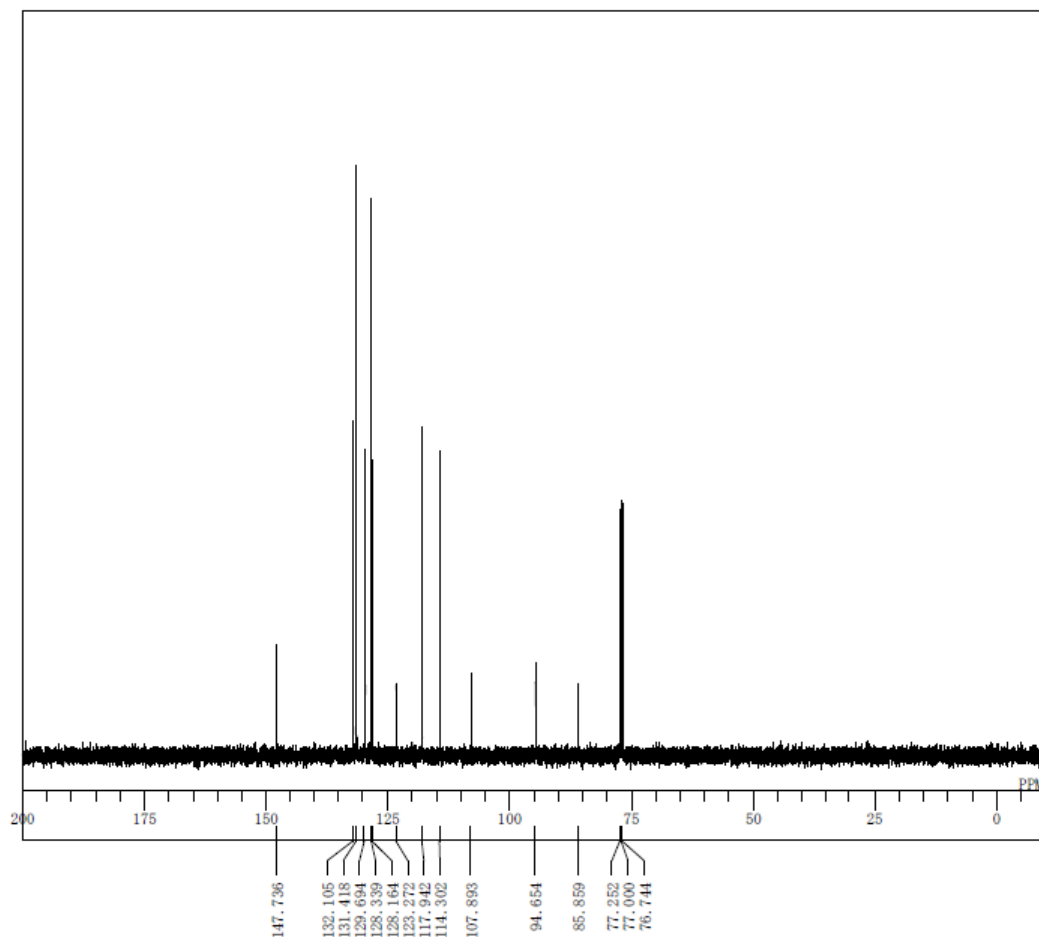


DFILE KF-836-t0-1H.1
COMNT Single Pulse Experiment
DATIM 26-10-2009 11:09:44
OBNUC 1H
EXMOD single_pulse_exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 32768
FREQU 7507.61 Hz
SCANS 8
AQTM 2.1823 sec
PD 4.0000 sec
PW1 7.00 usec
IRNUC
CTEMP 22.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.23 Hz
RGAIN 13

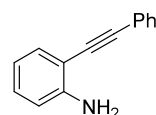


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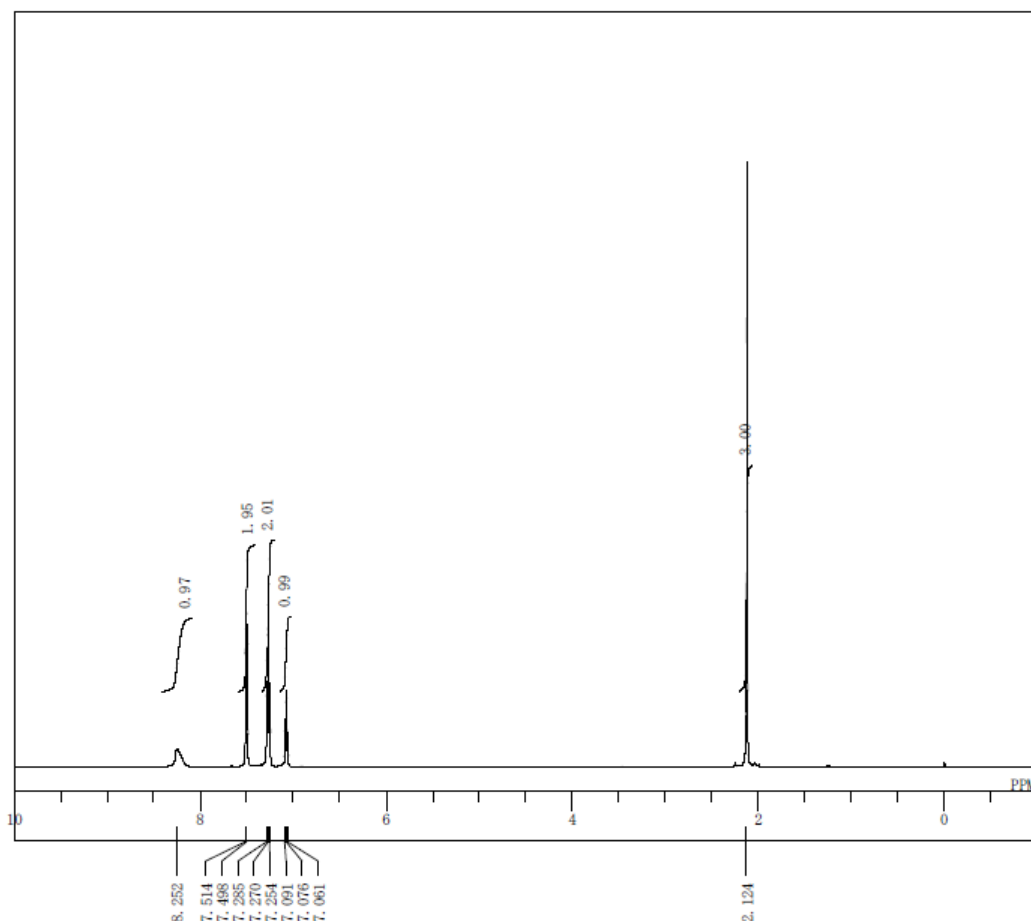
Single Pulse with Broadband Decoupling



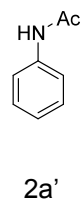
DFILE KF-836-t0-13C.1
COMNT Single Pulse with Broadband Decoupling
DATIM 26-10-2009 11:16:10
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 65536
FREQU 31446.54 Hz
SCANS 108
AQTM 1.0420 sec
PD 1.0000 sec
PW1 4.17 usec
IRNUC 1H
CTEMP 25.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 30



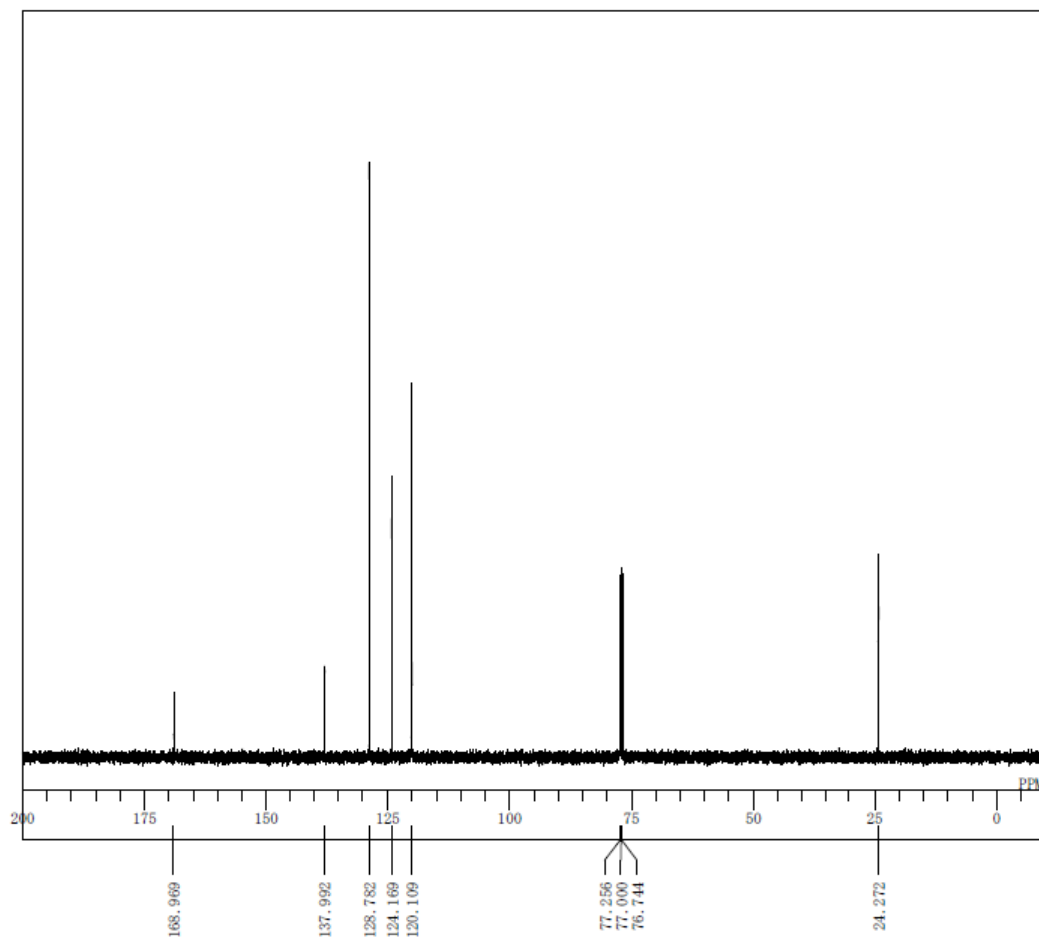
Single Pulse Experiment



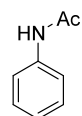
DFILE KF-312-1H.1
COMNT Single Pulse Experiment
DATIM 13-02-2008 17:20:41
OBNUC 1H
EXMOD single_pulse.exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 32768
FREQU 7507.61 Hz
SCANS 8
AQTM 2.1823 sec
PD 4.0000 sec
PW1 7.00 usec
IRNUC
CTEMP 24.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.23 Hz
RGAIN 12



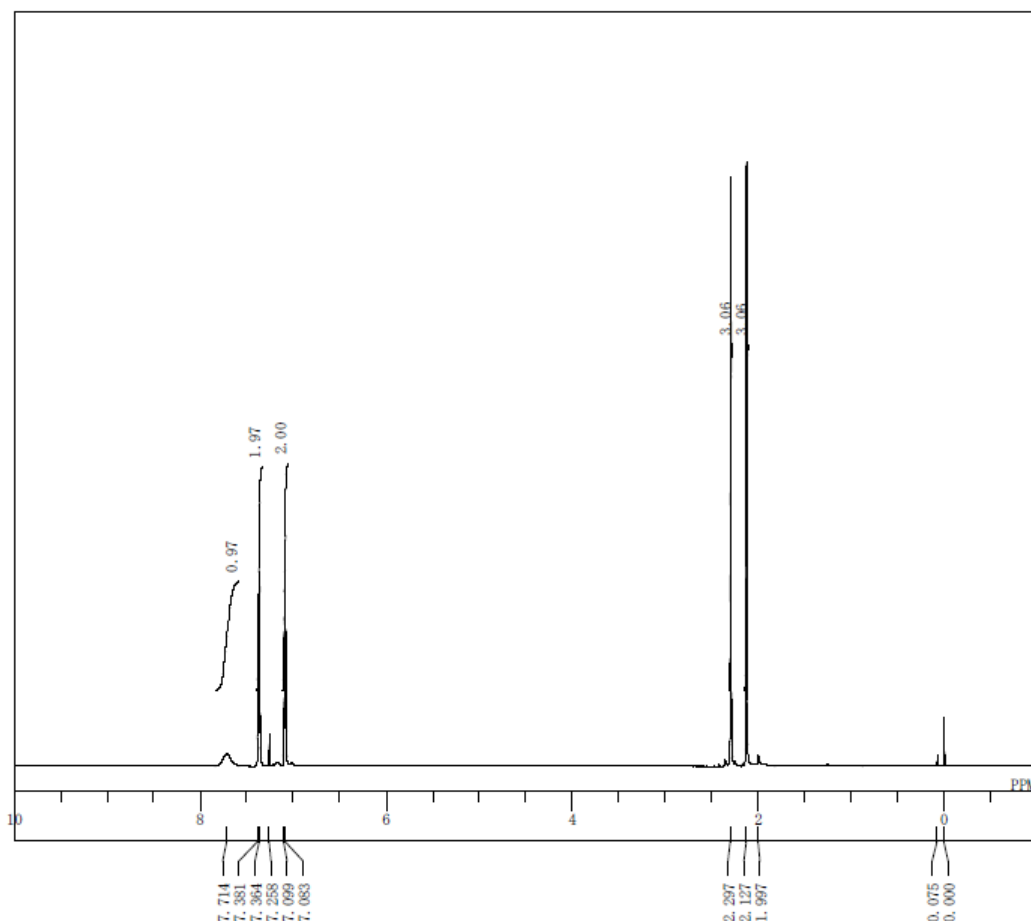
Single Pulse with Broadband Decoupling



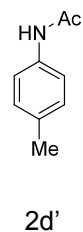
DFILE KF-312-13C.1
COMNT Single Pulse with Broadband Decoupling
DATIM 13-02-2008 17:26:28
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 65536
FREQU 31446.54 Hz
SCANS 101
AQTM 1.0420 sec
PD 1.0000 sec
PW1 4.17 usec
IRNUC 1H
CTEMP 26.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 30



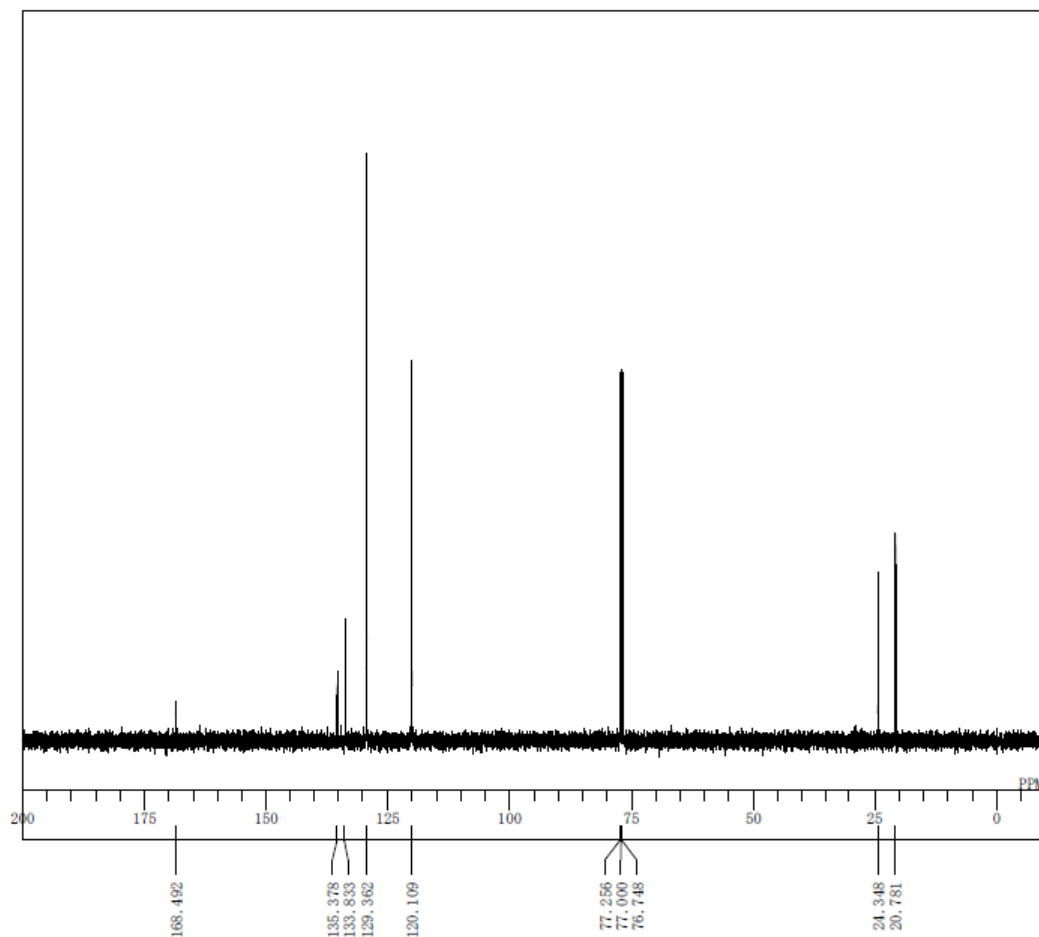
Single Pulse Experiment



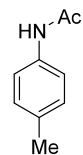
DFILE SN-145-1-1H.1
 COMNT Single Pulse Experiment
 DATIM 19-11-2009 16:26:11
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 32768
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 2.1823 sec
 PD 4.0000 sec
 PW1 7.00 usec
 IRNUC
 CTEMP 22.7 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 13



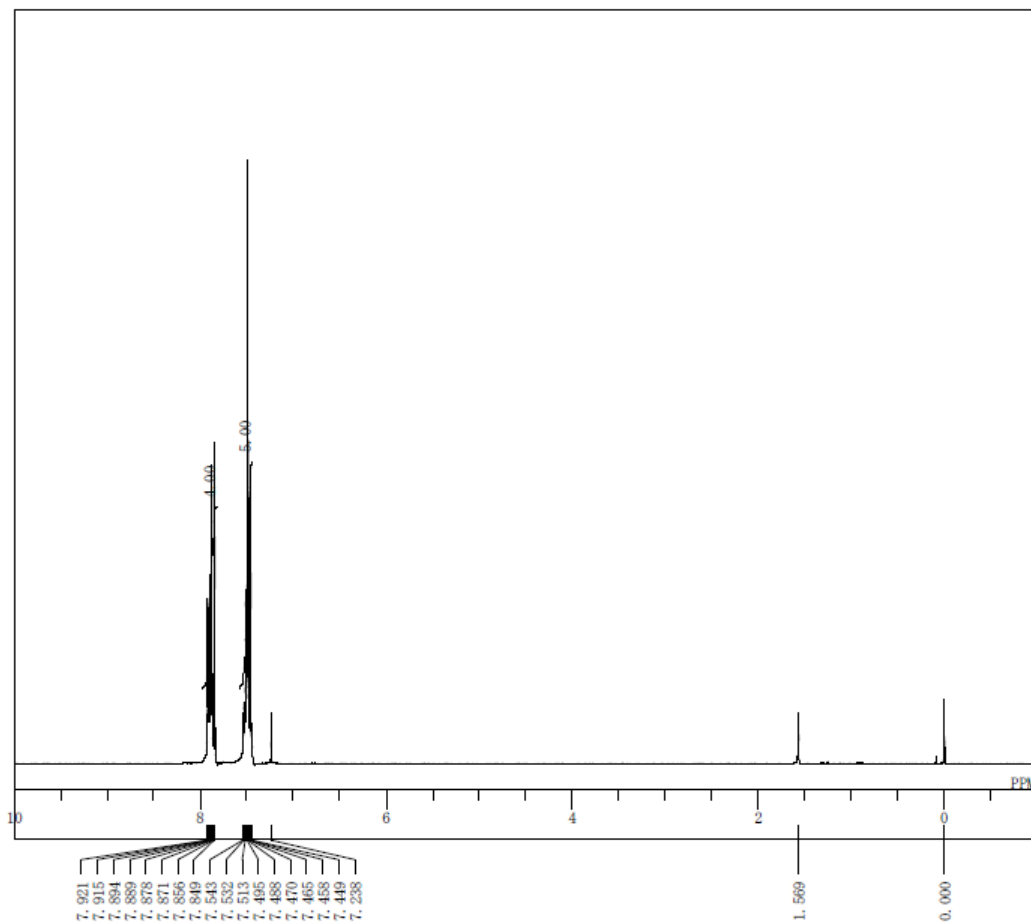
Single Pulse with Broadband Decoupling



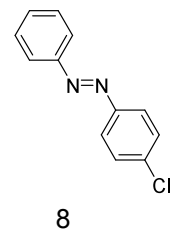
DFILE SN-145-1-13C.1
 COMNT Single Pulse with Broadband Decoupling
 DATIM 19-11-2009 16:34:09
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 65536
 FREQU 31446.54 Hz
 SCANS 208
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 26.1 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 30



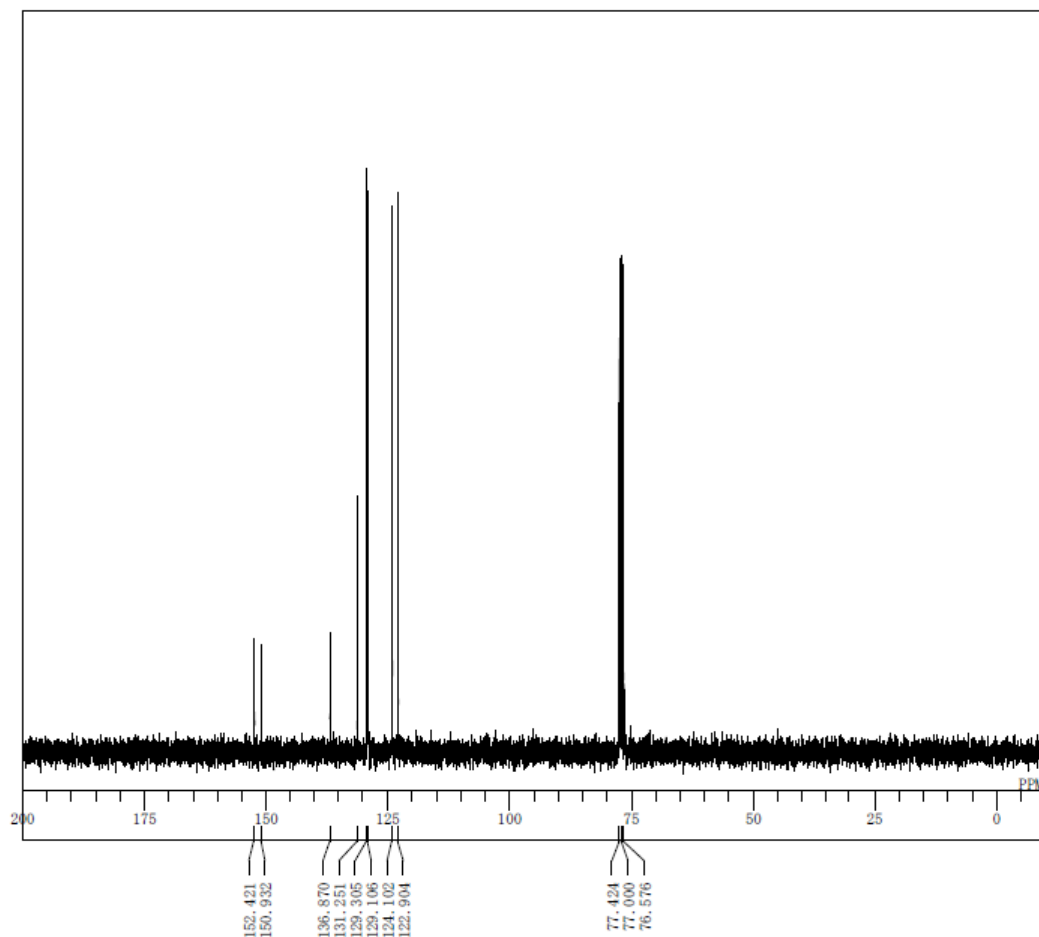
Single Pulse Experiment



DFILE KF-814-t-2-1H.1
COMNT Single Pulse Experiment
DATIM 30-09-2009 16:29:36
OBNUC 1H
EXMOD single_pulse_exp
OBFRQ 300.53 MHz
OBSET 1.15 KHz
OBFIN 8.57 Hz
POINT 32768
FREQU 4508.57 Hz
SCANS 8
ACQTM 3.6340 sec
PD 4.0000 sec
PW1 4.90 usec
IRNUC
CTEMP 22.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.14 Hz
RGAIN 13



Single Pulse with Broadband Decoupling



DFILE KF-814-t-2-13C.1
COMNT Single Pulse with Broadband Decoupling
DATIM 30-09-2009 17:26:55
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 75.57 MHz
OBSET 5.79 KHz
OBFIN 1.08 Hz
POINT 65536
FREQU 18903.59 Hz
SCANS 95
ACQTM 1.7334 sec
PD 1.0000 sec
PW1 2.63 usec
IRNUC 1H
CTEMP 23.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 30

