

***Copper-Catalyzed [5+1] Annulation of 2-Ethynylanilines with an N,O-Acetal
Leading to Construction of Quinoline Derivatives***

Supporting Information

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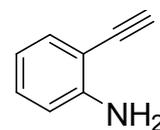
Experimental Section

General Methods. All reactions were carried out under a N₂ atmosphere, unless otherwise noted. Copper or acid catalysts were commercially available and were basically used without further purification. 2-Ethynylanilines were prepared from commercially available 2-iodo(bromo)anilines and a silylacetylene. Dichloroethane (DCE) and chloroform were first distilled from P₂O₅, and the distillates were re-distilled from K₂CO₃ and dried over MS4A. Piperidine and triethylamine were distilled over CaH₂. Reactions were monitored by thin-layer chromatography (TLC) analysis. TLC was performed on silica gel 60 F₂₅₄, and the components were located by observation under UV light. Column chromatography was 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 TMS or a center peak of chloroform (77.0 ppm) as an internal standard. High-resolution mass spectra were measured using NBA (3-nitrobenzylalcohol) as a matrix.

General procedure for the synthesis of 2-ethynylaniline derivatives: To a solution of 2-iodoaniline derivative (10 mmol) and diethylamine (10 mL) were added trimethylsilylacetylene (20 mmol), PdCl₂(PPh₃)₂ (140 mg, 0.20 mmol) and CuI (76 mg, 0.40 mmol). The reaction mixture was stirred at room temperature until TLC analysis indicated the disappearance of the starting material. The reaction was quenched with AcOEt (10 mL); 1N HCl (10 mL) were added and the organic layer was separated, washed with brine, dried over MgSO₄ and concentrated under reduced pressure. The residue was dissolved in a solution (10 mL) of MeOH containing KF (1.7 g, 30 mmol) and the mixture was stirred for 12 h at room temperature. The reaction was quenched with AcOEt (10 mL) and water (10 mL) and the organic layer was separated, washed with brine, dried over MgSO₄ and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hex-AcOEt) to give the corresponding 2-ethynylaniline derivative.

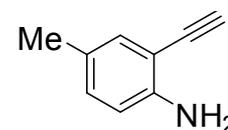
2-Ethynylaniline¹⁾ (1a)

82% yield; pale yellow oil; ¹H NMR (500 MHz, CDCl₃) δ 3.38 (s, 1H), 4.23 (brs, 2H), 6.66-6.69 (m, 2H), 7.14 (t, 1H, *J* = 8.0 Hz), 7.32 (d, 1H, *J* = 8.0 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 80.6, 82.4, 106.6, 114.3, 117.8, 130.1, 132.6, 148.5; MS (ESI): *m/z* 118 (M⁺+H); HRMS (ESI): Calcd. for C₈H₈N: 118.0657, Found: 118.0657.



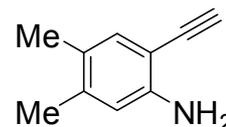
2-Ethynyl-4-methylaniline¹⁾ (1b)

66% yield; yellow oil; ¹H NMR (500 MHz, CDCl₃) δ 2.20 (s, 1H), 3.35 (s, 1H), 4.10 (brs, 2H), 6.60 (d, 1H, *J* = 8.0 Hz), 6.95 (dd, 1H, *J* = 8.0 Hz, 1.5 Hz), 7.13 (d, 1H, *J* = 1.5 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 20.2, 80.8, 82.1, 106.6, 114.5, 127.0, 130.9, 132.6, 146.1; MS (FAB): *m/z* 132 (M⁺+H); HRMS (FAB): Calcd. for C₉H₁₀N: 132.0673, Found: 132.0784.



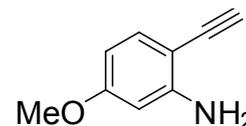
2-Ethynyl-4,5-dimethylaniline¹ (1c)

38% yield; colorless needles; mp 64-65 °C; ¹H NMR (500 MHz, CDCl₃) δ 2.09 (s, 3H), 2.15 (s, 3H), 3.31 (s, 1H), 4.03 (brs, 2H), 6.47 (s, 1H), 7.06 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 18.4, 19.9, 80.9, 81.5, 104.0, 115.8, 125.9, 132.9, 139.0, 146.5; MS (FAB): *m/z* 145 (M⁺); HRMS (FAB): Calcd. for C₁₀H₁₁N: 145.0891, Found: 145.0893.



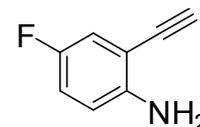
2-Ethynyl-5-methoxyaniline¹ (1d)

84% yield; brown solid; mp 42-43 °C; ¹H NMR (500 MHz, CDCl₃) δ 3.32 (s, 1H), 3.76 (s, 3H), 4.25 (brs, 2H), 6.22 (d, 1H, *J* = 3.0 Hz), 6.26 (dd, 1H, *J* = 8.5 Hz, 3.0 Hz), 7.24 (d, 1H, *J* = 8.5 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 55.1, 80.7, 81.2, 99.4, 99.5, 104.4, 133.8, 150.0, 161.3; MS (ESI): *m/z* 148 (M⁺+H); HRMS (ESI): Calcd. for C₉H₁₀NO: 148.0762, Found: 148.0787.



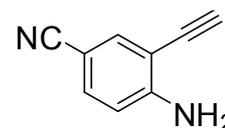
2-Ethynyl-4-fluoroaniline¹ (1e)

49% yield; yellow oil; ¹H NMR (500 MHz, CDCl₃) δ 3.40 (s, 1H), 4.11 (brs, 2H), 6.63 (dd, 1H, *J* = 9.0 Hz, *J* = 3.0 Hz), 6.88 (td, 1H, *J* = 9.0 Hz, 3.0 Hz), 7.02 (dd, 1H, *J* = 9.0 Hz, *J* = 3.0 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 79.6 (d, *J*_{C-F} = 2.9 Hz), 83.1, 107.3 (d, *J*_{C-F} = 9.6 Hz), 115.3 (d, *J*_{C-F} = 7.6 Hz), 117.4 (d, *J*_{C-F} = 22.9 Hz), 118.3 (d, *J*_{C-F} = 22.9 Hz), 145.0, 155.0 (d, *J*_{C-F} = 234.6 Hz); MS (ESI): *m/z* 136 (M⁺+H); HRMS (ESI): Calcd. for C₈H₇FN: 136.0562, Found: 136.0573.



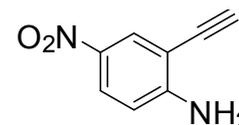
4-Amino-3-ethynylbenzonitrile¹ (1f)

44% yield; colorless solid; mp 104-106 °C; ¹H NMR (500 MHz, CDCl₃) δ 3.38 (s, 1H), 4.75 (brs, 2H), 6.62 (d, 1H, *J* = 8.0 Hz), 7.30 (d, 1H, *J* = 8.0 Hz), 7.51 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 78.1, 84.2, 99.8, 106.7, 113.9, 119.2, 133.6, 136.8, 151.7; MS (ESI): *m/z* 143 (M⁺+H); HRMS (ESI): Calcd. for C₉H₇N₂: 143.0609, Found: 143.0618.



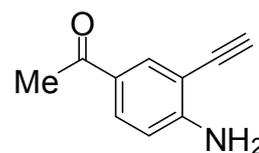
2-Ethynyl-4-nitroaniline¹ (1g)

47% yield; brown needles; mp 125-126 °C; ¹H NMR (500 MHz, CDCl₃) δ 3.48 (s, 1H), 5.04 (brs, 2H), 6.69 (d, 1H, *J* = 9.0 Hz), 8.04 (dd, 1H, *J* = 9.0 Hz, *J* = 2.5 Hz), 8.25 (d, 1H, *J* = 2.5 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 78.1, 84.2, 105.7, 112.9, 126.4, 129.3, 138.2, 153.5; MS (ESI): *m/z* 185 (M⁺+Na); HRMS (ESI): Calcd. for C₈H₆N₂NaO₂: 185.0327, Found: 185.0343.



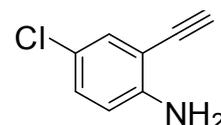
1-(4-Amino-3-ethynylphenyl)ethanone¹⁾ (1h)

65% yield; pale yellow solid; mp 77-78 °C; ¹H NMR (500 MHz, CDCl₃) δ 2.50 (s, 3H), 3.42 (s, 1H), 4.80 (brs, 2H), 6.69 (d, 1H, *J* = 9.0 Hz), 7.79 (dd, 1H, *J* = 9.0 Hz, *J* = 2.5 Hz), 7.96 (d, 1H, *J* = 2.5 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 26.0, 79.5, 83.0, 105.5, 113.3, 127.2, 130.8, 134.2, 152.4, 195.7; MS (ESI): *m/z* 160 (M⁺+H); HRMS (ESI): Calcd. for C₁₀H₁₀NO: 160.0762, Found: 160.0746.



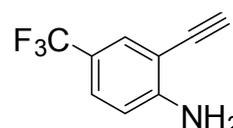
4-Chloro-2-ethynylaniline²⁾ (1i)

26% yield; brown solid; mp 51-52 °C; ¹H NMR (500 MHz, CDCl₃) δ 3.41 (s, 1H), 4.24 (brs, 2H), 6.61 (d, 1H, *J* = 8.5 Hz), 7.08 (dd, 1H, *J* = 8.5 Hz, *J* = 3.0 Hz), 7.28 (d, 1H, *J* = 3.0 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 79.3, 83.4, 107.8, 115.4, 122.0, 130.1, 131.8, 147.1; MS (ESI): *m/z* 152 (M⁺+H); HRMS (ESI): Calcd. for C₈H₇ClN: 152.0267, Found: 152.0264.



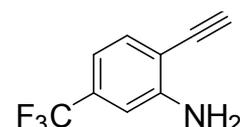
2-Ethynyl-4-(trifluoromethyl)aniline³⁾ (1j)

63% yield; brown oil; ¹H NMR (300 MHz, CDCl₃) δ 3.42 (s, 1H), 4.56 (brs, 2H), 6.71 (d, 1H, *J* = 8.4 Hz), 7.35 (dd, 1H, *J* = 8.4 Hz, 1.8 Hz), 7.57 (d, 1H, *J* = 1.8 Hz); ¹³C NMR (75 MHz, CDCl₃) δ 79.2, 83.6, 106.1, 113.7, 119.6 (q, *J*_{C-F} = 32.7 Hz), 124.3 (q, *J*_{C-F} = 268.8 Hz), 127.0 (q, *J*_{C-F} = 3.7 Hz), 130.0 (q, *J*_{C-F} = 3.7 Hz), 151.0; MS (FAB): *m/z* 185 (M⁺); HRMS (FAB): Calcd. for C₉H₆F₃N: 185.0452, Found: 185.0448.



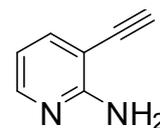
2-Ethynyl-5-(trifluoromethyl)aniline⁴⁾ (1k)

25% yield; brown oil; ¹H NMR (500 MHz, CDCl₃) δ 3.47 (s, 1H), 4.43 (brs, 2H), 6.90 (d, 1H, *J* = 8.0 Hz), 6.92 (s, 1H), 7.40 (d, 1H, *J* = 8.0 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 79.4, 84.4, 109.7, 110.7 (q, *J*_{C-F} = 3.9 Hz), 114.1 (q, *J*_{C-F} = 3.9 Hz), 123.8 (q, *J*_{C-F} = 270.9 Hz), 131.9 (q, *J*_{C-F} = 32.4 Hz), 133.1, 148.5; MS (ESI): *m/z* 184 (M⁺-H); HRMS (ESI): Calcd. for C₉H₅F₃N: 184.0374, Found: 184.0349.



2-Amino-3-ethynylpyridine⁵⁾ (1l)

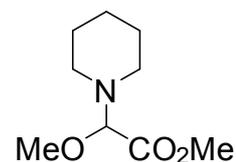
34% yield; yellow solid; mp 92-93 °C; ¹H NMR (500 MHz, CDCl₃) δ 3.42 (s, 1H), 5.16 (brs, 2H), 6.60 (ddd, 1H, *J* = 7.5 Hz, *J* = 5.0 Hz, *J* = 1.5 Hz), 7.56 (dd, 1H, *J* = 7.5 Hz, *J* = 1.5 Hz), 8.05 (dd, 1H, *J* = 5.0 Hz, *J* = 1.5 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 79.3, 83.4, 101.7, 113.3, 140.6, 148.4, 159.4; MS (ESI): *m/z* 119 (M⁺+H); HRMS (ESI): Calcd. for C₇H₇N₂: 119.0609, Found: 119.0635.



Preparation for the synthesis of *N,O*-acetal 2⁶⁾: To a freshly distilled THF (40 mL) was successively added piperidine (4.1 g, 48 mmol), triethylamine (4.8 g, 48 mmol) and methyl 2-bromo-2-methoxyacetate (7.3 g, 40 mmol). The resultant solution was stirred for 2 h at room temperature. The mixture was directly filtered without a common work-up and was evaporated under reduced pressure. The filtrate was distilled (Kugelrohr) to give the *N,O*-acetal 2.

Methyl 2-methoxy-2-(piperidin-1-yl)acetate⁶⁾ (2)

75% yield; colorless oil; bp 71-72 °C/5 mmHg; ¹H NMR (500 MHz, CDCl₃) δ 1.43-1.47 (m, 2H), 1.55-1.59 (m, 4H), 2.52-2.56 (m, 2H), 2.69-2.73 (m, 2H), 3.39 (s, 3H), 3.78 (s, 3H), 4.23 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 24.4, 26.0, 48.6, 51.6, 55.7, 94.2, 168.9; MS (ESI): *m/z* 210 (M⁺+Na); HRMS (ESI): Calcd. for C₉H₁₇NNaO₃: 210.1106, Found: 210.1126.

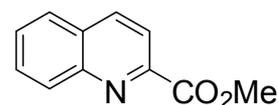


General procedure for the synthesis of quinolines with an ethynylaniline and an *N,O*-acetal:

To freshly distilled 1,2-dichloroethane (1.0 mL) were successively added CuBr₂ (6.7 mg, 0.030 mmol), a 2-ethynylaniline (0.30 mmol) and an *N,O*-acetal (0.36 mmol) under a nitrogen atmosphere. The resultant solution was refluxed and monitored by TLC until the disappearance of the starting material. The reaction was quenched with a saturated aqueous solution of Na₂CO₃. The combined organic layer was dried over Na₂SO₄ and evaporated under reduced pressure. The crude product was purified by silica gel column chromatography (hex-AcOEt).

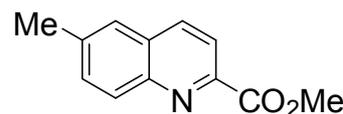
Methyl quinoline-2-carboxylate⁷⁾ (3a)

76% yield; yellow solid; mp 79-80 °C; °C; ¹H NMR (300 MHz, CDCl₃) δ 4.09 (s, 3H), 7.65 (td, 1H, *J* = 8.4 Hz, *J* = 1.5 Hz), 7.79 (td, 1H, *J* = 8.4 Hz, *J* = 1.5 Hz), 7.88 (dd, 1H, *J* = 8.4 Hz, *J* = 1.5 Hz), 8.20 (d, 1H, *J* = 8.4 Hz), 8.31 (d, 1H, *J* = 8.4 Hz); ¹³C NMR (75 MHz, CDCl₃) δ 53.2, 121.0, 127.5, 128.6, 129.3, 130.2, 130.6, 137.3, 147.4, 147.8, 165.9; MS (ESI): *m/z* 210 (M⁺+Na); HRMS (ESI): Calcd. for C₁₁H₉NNaO₂: 210.0531, Found: 210.0539.



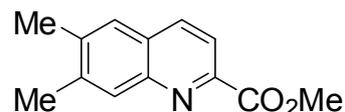
Methyl 6-methylquinoline-2-carboxylate (3b)

75% yield; pale brown crystal; mp 86-87 °C; ¹H NMR (300 MHz, CDCl₃) δ 2.57 (s, 3H), 4.08 (s, 3H), 7.61-7.64 (m, 2H), 8.15-8.23 (m, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 21.7, 53.1, 121.1, 126.3, 129.4, 130.3, 132.7, 136.5, 139.0, 146.1, 147.0, 166.0; MS (FAB): *m/z* 202 (M⁺+H); HRMS (FAB): Calcd. for C₁₂H₁₂NO₂: 202.0868, Found: 202.0887.



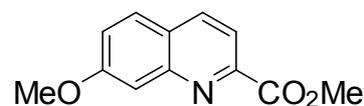
Methyl 6,7-dimethylquinoline-2-carboxylate (3c)

89% yield; colorless crystal; mp 150-151 °C; ¹H NMR (300 MHz, CDCl₃) δ 2.47 (s, 3H), 2.48 (s, 3H), 4.07 (s, 3H), 7.60 (s, 1H), 8.06-8.18 (m, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 20.2, 20.4, 53.0, 120.3, 126.6, 128.1, 129.8, 136.0, 139.1, 140.8, 146.7, 146.9, 166.2; MS (FAB): *m/z* 216 (M⁺+H); HRMS (FAB): Calcd. for C₁₃H₁₄NO₂: 216.1025, Found: 216.1028.



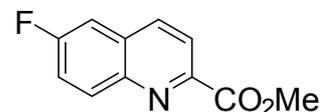
Methyl 7-methoxyquinoline-2-carboxylate (3d)

66% yield; yellow powder; mp 101-102 °C; ¹H NMR (300 MHz, CDCl₃) δ 3.96 (s, 3H), 4.08 (s, 3H), 7.31 (dd, 1H, *J* = 8.4 Hz, *J* = 2.7 Hz), 7.61 (d, 1H, *J* = 2.7 Hz), 7.76 (d, 1H, *J* = 8.4 Hz), 8.08 (d, 1H, *J* = 8.4 Hz), 8.22 (d, 1H, *J* = 8.4 Hz); ¹³C NMR (75 MHz, CDCl₃) δ 53.1, 55.7, 108.1, 119.1, 122.3, 124.8, 128.4, 136.8, 147.9, 149.4, 161.2, 166.1; MS (FAB): *m/z* 218 (M⁺+H); HRMS (FAB): Calcd. for C₁₂H₁₂NO₃: 218.0817, Found: 218.0811.



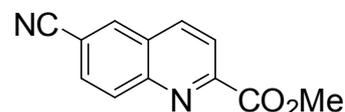
Methyl 6-fluoroquinoline-2-carboxylate (3e)

81% yield; pale yellow crystal; mp 112-113 °C; ¹H NMR (300 MHz, CDCl₃) δ 4.09 (s, 3H), 7.50 (dd, 1H, *J* = 9.0 Hz, *J* = 3.0 Hz), 7.57 (td, 1H, *J* = 9.0 Hz, *J* = 3.0 Hz), 8.20-8.34 (m, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 53.2, 110.6 (d, *J*_{C-F} = 22.4 Hz), 120.9 (d, *J*_{C-F} = 25.4 Hz), 121.7, 130.2 (d, *J*_{C-F} = 10.6 Hz), 133.3 (d, *J*_{C-F} = 9.3 Hz), 136.6 (d, *J*_{C-F} = 5.6 Hz), 144.6, 147.3 (d, *J*_{C-F} = 3.2 Hz), 161.6 (d, *J*_{C-F} = 251.8 Hz), 165.6; MS (FAB): *m/z* 206 (M⁺+H); HRMS (FAB): Calcd. for C₁₁H₉FNO₂: 206.0617, Found: 206.0590.



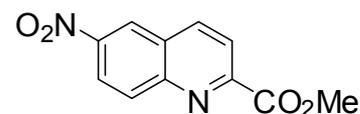
Methyl 6-cyanoquinoline-2-carboxylate (3f)

48% yield; pale yellow solid; mp 184-185 °C; ¹H NMR (300 MHz, CDCl₃) δ 4.12 (s, 3H), 7.94 (d, 1H, *J* = 8.7 Hz), 8.31-8.34 (m, 2H), 8.40-8.43 (m, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 53.5, 112.4, 118.0, 122.6, 128.4, 130.9, 132.1, 133.8, 137.9, 148.3, 150.5, 165.2; MS (FAB): *m/z* 213 (M⁺+H); HRMS (FAB): Calcd. for C₁₂H₉N₂O₂: 213.0664, Found: 213.0670.

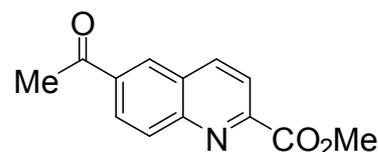


Methyl 6-nitroquinoline-2-carboxylate⁸⁾ (3g)

48% yield; yellow powder; mp 235-237 °C; ¹H NMR (500 MHz, CD₃OD+CDCl₃+d₆-DMSO) δ 4.24 (s, 3H), 8.23 (d, 1H, *J* = 8.5 Hz), 8.32 (d, 1H, *J* = 8.5 Hz), 8.44 (d, 1H, *J* = 8.5 Hz), 8.57 (d, 1H, *J* = 8.5 Hz), 8.84 (s, 1H); ¹³C NMR (125 MHz, CD₃OD+CDCl₃+d₆-DMSO) δ 54.6, 123.9, 125.0, 125.8, 129.7, 133.5, 141.4, 148.1, 150.6, 152.2, 166.4; MS (FAB): *m/z* 233 (M⁺+H); HRMS (FAB): Calcd. for C₁₁H₉N₂O₄: 233.0562, Found: 233.0581.

**Methyl 6-acetylquinoline-2-carboxylate (3h)**

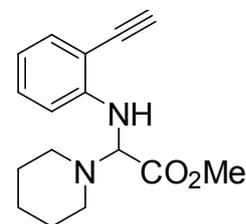
46% yield; yellow needles; mp 134-135 °C; ¹H NMR (300 MHz, CDCl₃) δ 2.77 (s, 3H), 4.11 (s, 3H), 8.27 (d, 1H, *J* = 8.4 Hz), 8.31-8.39 (m, 2H), 8.45 (d, 1H, *J* = 8.4 Hz), 8.50 (s, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 26.8, 53.4, 121.8, 128.4, 128.5, 129.3, 131.2, 136.3, 138.9, 149.2, 149.8, 165.5, 197.1; MS (FAB): *m/z* 230 (M⁺+H); HRMS (FAB): Calcd. for C₁₃H₁₂NO₃: 230.0817, Found: 230.0802.



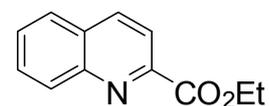
General procedure for the direct synthesis of quinolines with ethynylanilines and ethyl glyoxylate: To a solution of 1,2-dichloroethane (1.5 mL) were added TFA (5.7 mg, 0.050 mmol), Na₂SO₄ (2.0 mmol), ethyl glyoxylate (0.50 mmol), piperidine (43 mg, 0.50 mmol) and ethynylanilines (0.50 mmol). Subsequently, CuBr₂ (11 mg, 0.050 mmol) was added to this mixture. The resultant mixture was refluxed and monitored by TLC until the disappearance of the starting material. After the reaction, the reaction mixture was directly subjected to silica gel column chromatography (hex-AcOEt) without the usual work-up to give the corresponding quinoline derivative.

Methyl 2-(2-ethynylphenylamino)-2-(piperidin-1-yl)acetate (4a)

62% yield; colorless oil; ¹H NMR (500 MHz, CDCl₃) δ 1.42-1.45 (m, 2H), 1.50-1.57 (m, 4H), 2.43-2.47 (m, 2H), 2.72-2.76 (m, 2H), 3.82 (s, 3H), 4.58 (d, 1H, *J* = 6.0 Hz), 5.82 (d, 1H, *J* = 6.0 Hz), 6.67 (t, 1H, *J* = 7.5 Hz), 6.77 (d, 1H, *J* = 7.5 Hz), 7.20 (td, 1H, *J* = 7.5 Hz, *J* = 1.5 Hz), 7.34 (dd, 1H, *J* = 7.5 Hz, *J* = 1.5 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 24.7, 25.9, 26.1, 48.5, 50.2, 51.8, 74.6, 80.3, 82.9, 107.1, 111.7, 117.2, 130.0, 132.4, 147.6, 169.7; MS (ESI): *m/z* 295 (M⁺+Na); HRMS (ESI): Calcd. for C₁₆H₂₀N₂NaO₂: 295.1422, Found: 295.1396.

**Ethyl quinoline-2-carboxylate⁹⁾ (5a)**

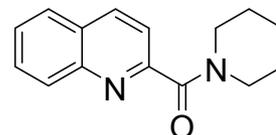
78% yield; brown oil; ¹H NMR (500 MHz, CDCl₃) δ 1.42 (t, 3H, *J* = 7.0 Hz), 4.49 (q, 2H, *J* = 7.0 Hz), 7.58 (td, 1H, *J* = 8.5 Hz, *J* = 1.5 Hz), 7.73 (td, 1H, *J* = 8.5 Hz, *J* = 1.5 Hz), 7.81 (d, 1H, *J* = 8.5 Hz), 8.12 (d, 1H, *J* = 8.5 Hz),



8.22-8.26 (m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 14.4, 62.2, 121.0, 127.5, 128.5, 129.3, 130.2, 130.8, 137.2, 147.6, 148.2, 165.4; MS (ESI): m/z 224 (M^+Na); HRMS (ESI): Calcd. for $\text{C}_{12}\text{H}_{11}\text{NNaO}_2$: 224.0688, Found: 224.0703.

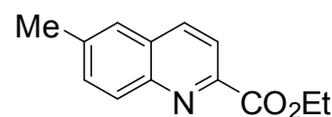
Piperidin-1-yl(quinolin-2-yl)methanone¹⁰ (5a')

48% yield; brown oil; ^1H NMR (500 MHz, CDCl_3) δ 1.57-1.62 (m, 2H), 1.69-1.76 (m, 4H), 3.52 (t, 2H, $J = 5.5$ Hz), 3.81 (t, 2H, $J = 5.5$ Hz), 7.59 (td, 1H, $J = 8.0$ Hz, $J = 1.5$ Hz), 7.66 (d, 1H, $J = 8.0$ Hz), 7.75 (td, 1H, $J = 8.0$ Hz, $J = 1.5$ Hz), 7.85 (d, 1H, $J = 8.0$ Hz), 8.11 (d, 1H, $J = 8.0$ Hz), 8.25 (d, 1H, $J = 8.0$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 24.6, 25.6, 26.5, 43.3, 48.3, 120.4, 127.3, 127.6, 127.9, 129.7, 129.9, 137.0, 146.8, 154.4, 167.7; MS (ESI): m/z 263 (M^+Na); HRMS (ESI): Calcd. for $\text{C}_{15}\text{H}_{16}\text{N}_2\text{NaO}$: 263.1160, Found: 263.1152.



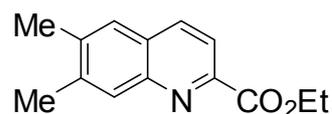
Ethyl 6-methylquinoline-2-carboxylate¹¹ (5b)

73% yield; brown oil; ^1H NMR (500 MHz, CDCl_3) δ 1.40 (t, 3H, $J = 6.5$ Hz), 2.46 (s, 3H), 4.46 (q, 2H, $J = 6.5$ Hz), 7.50-7.54 (m, 2H), 8.06-8.12 (m, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 14.3, 21.7, 62.0, 121.0, 126.2, 129.3, 130.3, 132.5, 136.3, 138.7, 146.1, 147.3, 165.4; MS (FAB): m/z 216 (M^+H); HRMS (FAB): Calcd. for $\text{C}_{13}\text{H}_{14}\text{NO}_2$: 216.1025, Found: 216.1027.



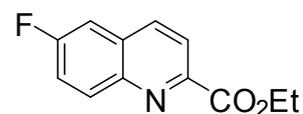
Ethyl 6,7-dimethylquinoline-2-carboxylate (5c)

66% yield; brown solid; mp 91-92 °C; ^1H NMR (500 MHz, CDCl_3) δ 1.48 (t, 3H, $J = 7.0$ Hz), 2.45 (s, 3H), 2.47 (s, 3H), 4.55 (q, 2H, $J = 7.0$ Hz), 7.58 (s, 1H), 8.07 (d, 1H, $J = 7.5$ Hz), 8.10 (s, 1H), 8.14 (d, 1H, $J = 7.5$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 14.3, 20.1, 20.3, 62.0, 120.2, 126.5, 127.9, 129.9, 135.8, 138.9, 140.5, 146.7, 147.2, 165.6; MS (FAB): m/z 230 (M^+H); HRMS (FAB): Calcd. for $\text{C}_{14}\text{H}_{16}\text{NO}_2$: 230.1181, Found: 230.1174.



Ethyl 6-fluoroquinoline-2-carboxylate (5d)

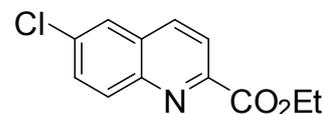
73% yield; brown solid; mp 76-77 °C; ^1H NMR (500 MHz, CDCl_3) δ 1.49 (t, 3H, $J = 7.0$ Hz), 4.56 (q, 2H, $J = 7.0$ Hz), 7.49 (d, 1H, $J = 8.0$ Hz), 7.56 (t, 1H, $J = 8.0$ Hz), 8.20 (dd, 1H, $J = 9.0$ Hz, $J = 2.5$ Hz), 8.25 (dd, 1H, $J = 9.0$ Hz, $J = 2.5$ Hz), 8.31-8.34 (m, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ 14.4, 62.3, 110.5 (d, $J_{\text{C-F}} = 21.1$ Hz), 120.7 (d, $J_{\text{C-F}} = 26.4$ Hz), 121.8, 130.1 (d, $J_{\text{C-F}} = 10.6$ Hz), 133.5 (d, $J_{\text{C-F}} = 8.7$ Hz), 136.5 (d, $J_{\text{C-F}} = 5.8$ Hz), 144.7, 147.8 (d, $J_{\text{C-F}} = 2.9$ Hz), 161.7 (d, $J_{\text{C-F}} = 264.1$ Hz), 165.2; MS (FAB): m/z 220 (M^+); HRMS (FAB): Calcd. for $\text{C}_{12}\text{H}_{11}\text{FNO}_2$:



220.0774, Found: 220.0775.

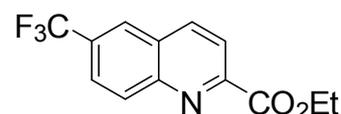
Ethyl 6-chloroquinoline-2-carboxylate (5e)

73% yield; yellow solid; mp 94-95 °C; ¹H NMR (500 MHz, CDCl₃) δ 1.50 (t, 3H, *J* = 7.0 Hz), 4.56 (q, 2H, *J* = 7.0 Hz), 7.71 (dd, 1H, *J* = 9.0 Hz, *J* = 2.5 Hz), 7.85 (d, 1H, *J* = 2.5 Hz), 8.18-8.22 (m, 2H), 8.25 (d, 1H, *J* = 9.0 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 14.3, 62.3, 121.8, 126.1, 129.7, 131.2, 132.2, 134.4, 136.2, 145.8, 148.4, 165.0; MS (FAB): *m/z* 236 (M⁺); HRMS (FAB): Calcd. for C₁₂H₁₁ClNO₂: 236.0478, Found: 236.0475.



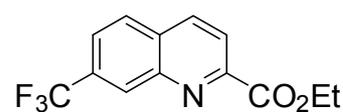
Ethyl 6-(trifluoromethyl)quinoline-2-carboxylate (5f)

56% yield; colorless solid; 105-106 °C; ¹H NMR (500 MHz, CDCl₃) δ 1.43 (t, 3H, *J* = 7.5 Hz), 4.51 (q, 2H, *J* = 7.5 Hz), 7.88 (dd, 1H, *J* = 9.0 Hz, *J* = 1.5 Hz), 8.13 (s, 1H), 8.21 (dd, 1H, *J* = 9.0 Hz, *J* = 1.5 Hz), 8.34 (d, 1H, *J* = 9.0 Hz), 8.36 (d, 1H, *J* = 9.0 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 14.3, 62.5, 122.1, 123.7 (q, *J*_{C-F} = 270.9 Hz), 125.5 (q, *J*_{C-F} = 4.8 Hz), 125.8 (q, *J*_{C-F} = 2.9 Hz), 128.2, 130.2 (q, *J*_{C-F} = 32.4 Hz), 132.0, 138.2, 148.5, 150.3, 165.0; MS (FAB): *m/z* 270 (M⁺); HRMS (FAB): Calcd. for C₁₃H₁₁F₃NO₂: 270.0742, Found: 270.0745.



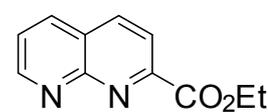
Ethyl 7-(trifluoromethyl)quinoline-2-carboxylate (5g)

67% yield; yellow solid; mp 58-59 °C; ¹H NMR (500 MHz, CDCl₃) δ 1.51 (t, 3H, *J* = 7.0 Hz), 4.59 (q, 2H, *J* = 7.0 Hz), 7.83 (dd, 1H, *J* = 8.5 Hz, *J* = 2.0 Hz), 8.03 (d, 1H, *J* = 8.0 Hz), 8.31, (d, 1H, *J* = 8.5 Hz), 8.39 (d, 1H, *J* = 8.0 Hz), 8.67 (d, 1H, *J* = 2.0 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 14.4, 62.6, 122.9, 123.7 (q, *J*_{C-F} = 271.9 Hz), 124.2 (q, *J*_{C-F} = 2.9 Hz), 128.7 (q, *J*_{C-F} = 4.8 Hz), 128.8, 130.6, 132.1 (q, *J*_{C-F} = 7.5 Hz), 137.3, 146.6, 149.7, 165.0; MS (FAB): *m/z* 270 (M⁺+H); HRMS (FAB): Calcd. for C₁₃H₁₁F₃NO₂: 270.0742, Found: 270.0733.



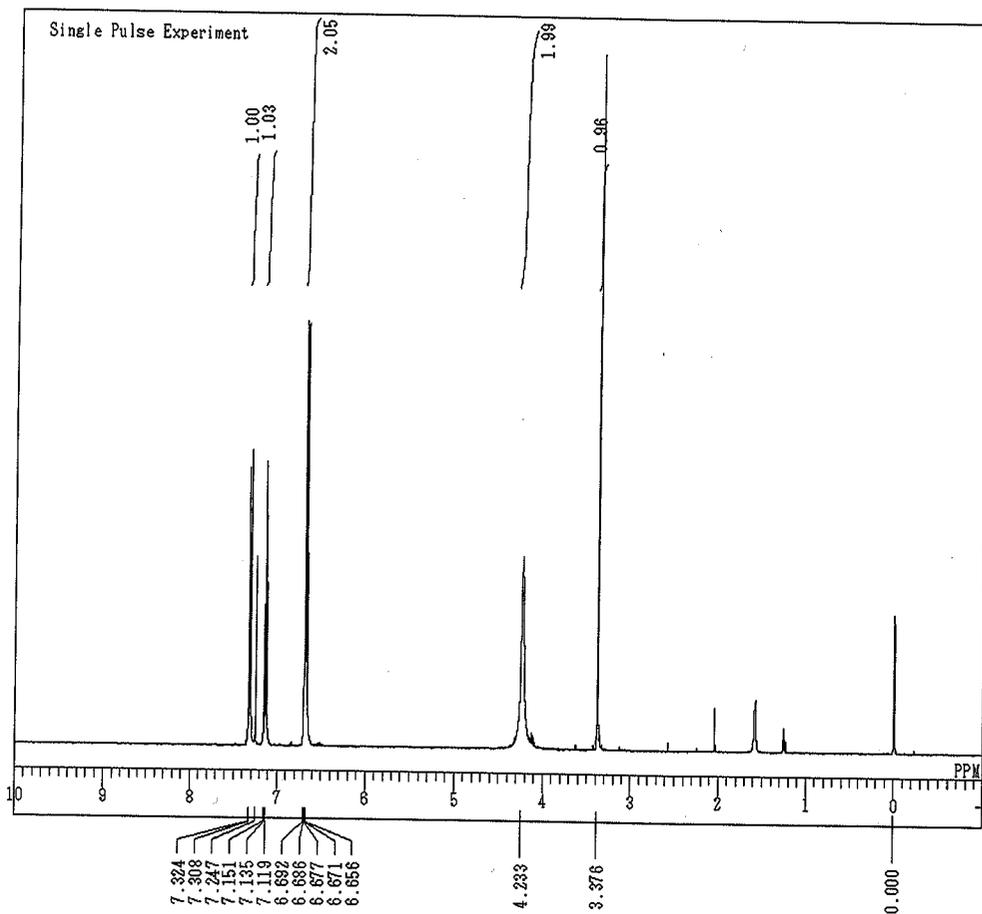
Ethyl 1,8-naphthyridine-2-carboxylate (5h)

56% yield; brown oil; ¹H NMR (500 MHz, CDCl₃) δ 1.42 (t, 3H, *J* = 7.5 Hz), 4.47 (q, 2H, *J* = 7.5 Hz), 7.52 (dd, 1H, *J* = 8.0 Hz, *J* = 4.0 Hz), 8.21 (dd, 1H, *J* = 8.0 Hz, *J* = 2.0 Hz), 8.23 (d, 1H, *J* = 8.0 Hz), 8.30 (d, 1H, *J* = 8.0 Hz), 9.16 (dd, 1H, *J* = 4.0 Hz, *J* = 2.0 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 14.2, 62.3, 121.9, 123.5, 124.1, 136.7, 138.4, 151.2, 154.7, 155.2, 165.2; MS (ESI): *m/z* 225 (M⁺+Na); HRMS (ESI): Calcd. for C₁₁H₁₀N₂NaO₂: 225.0640, Found: 225.0633.

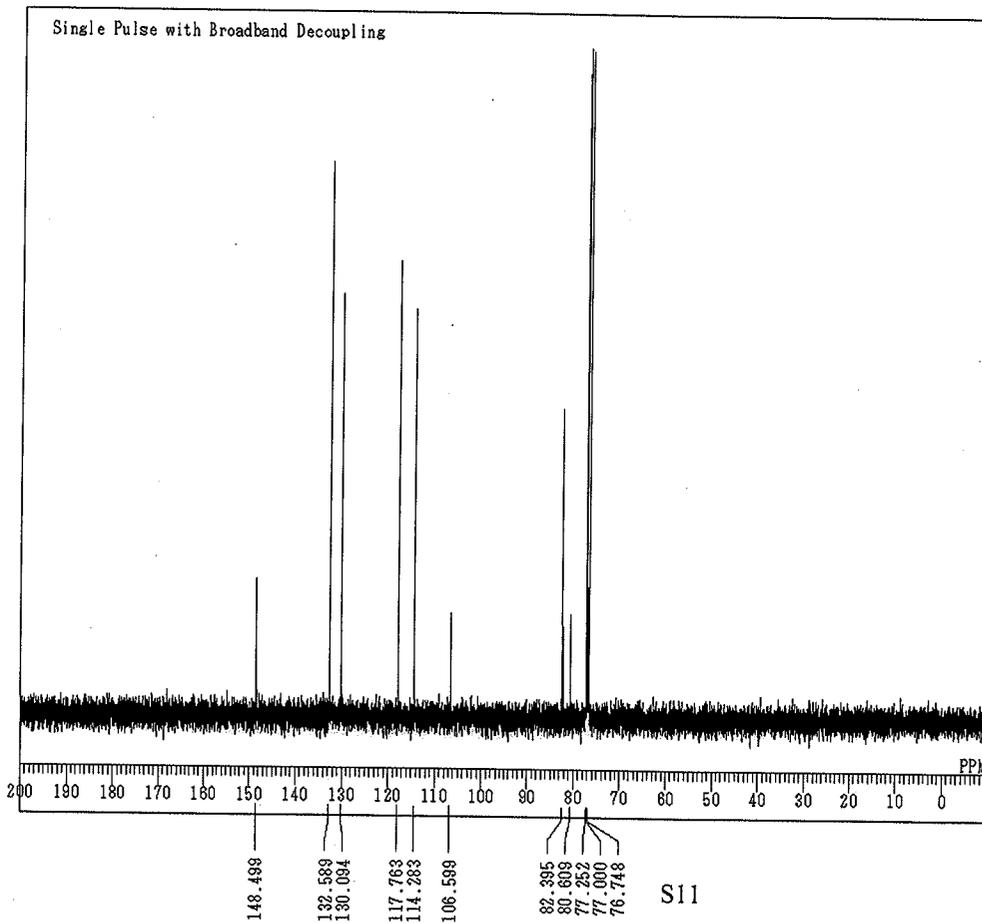
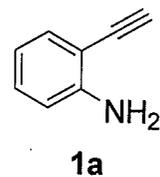


References

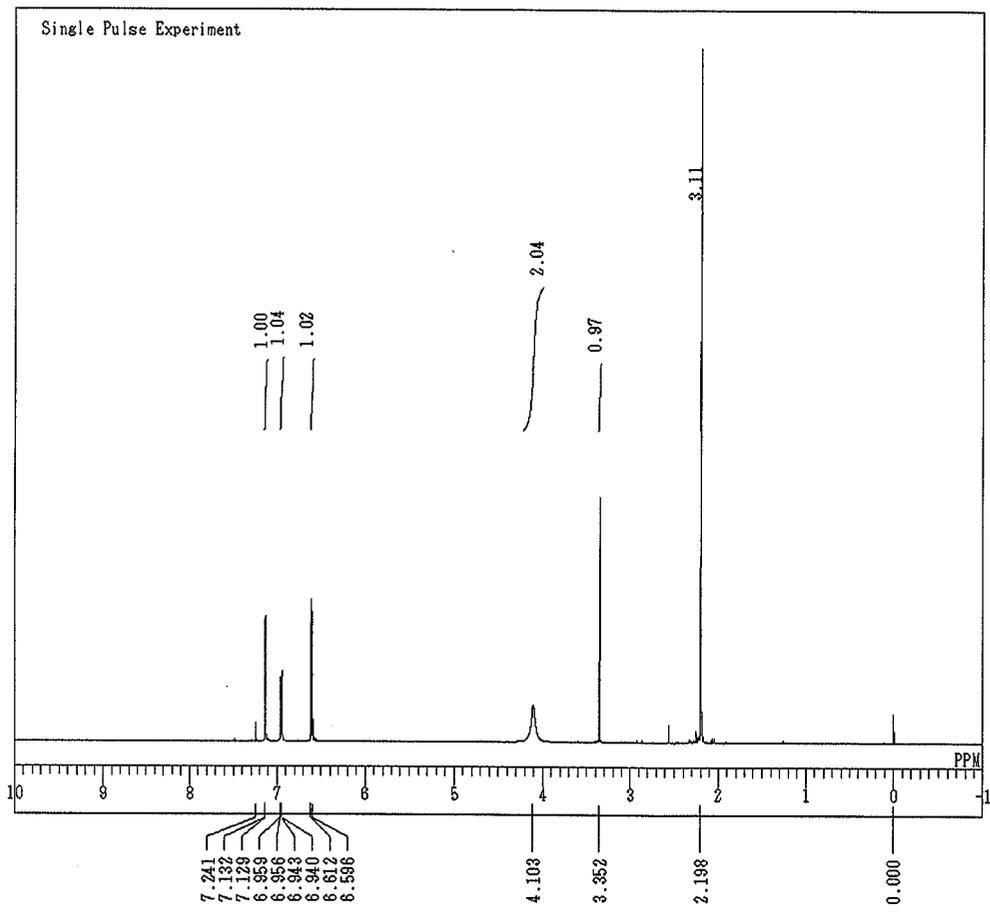
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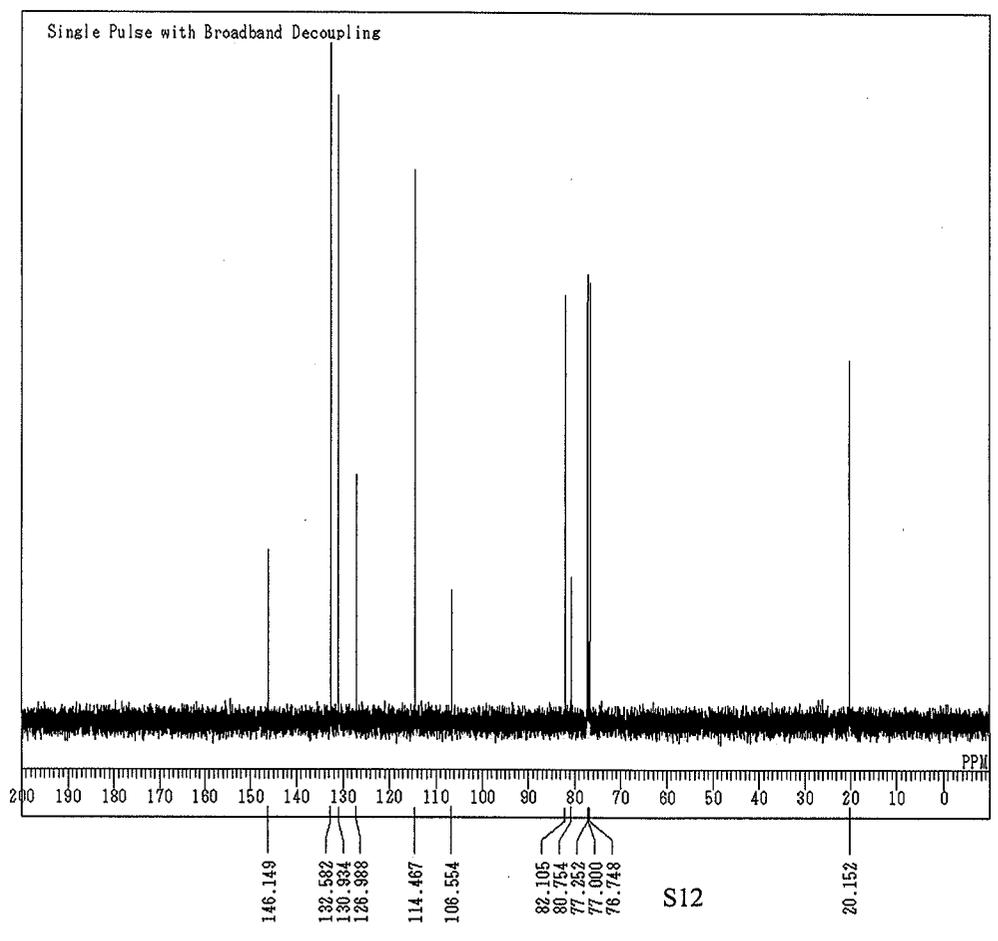
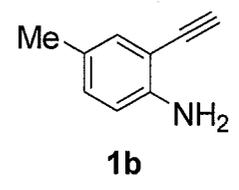
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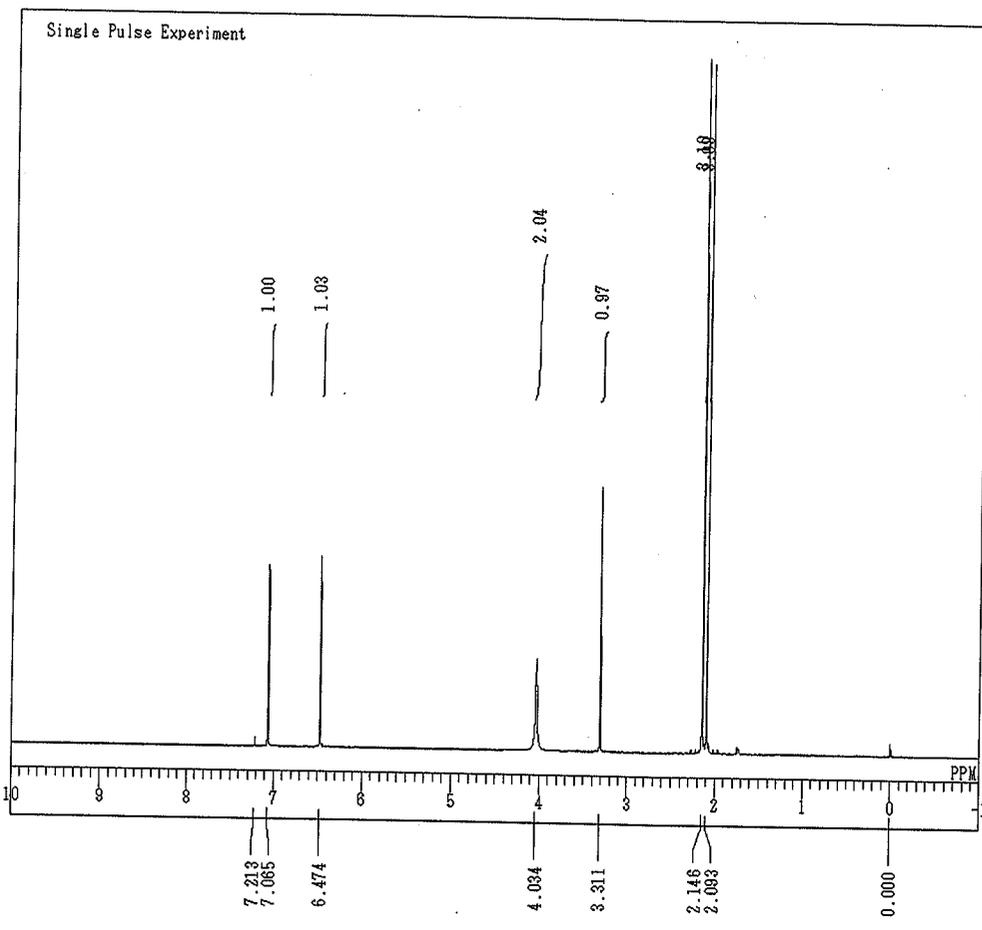
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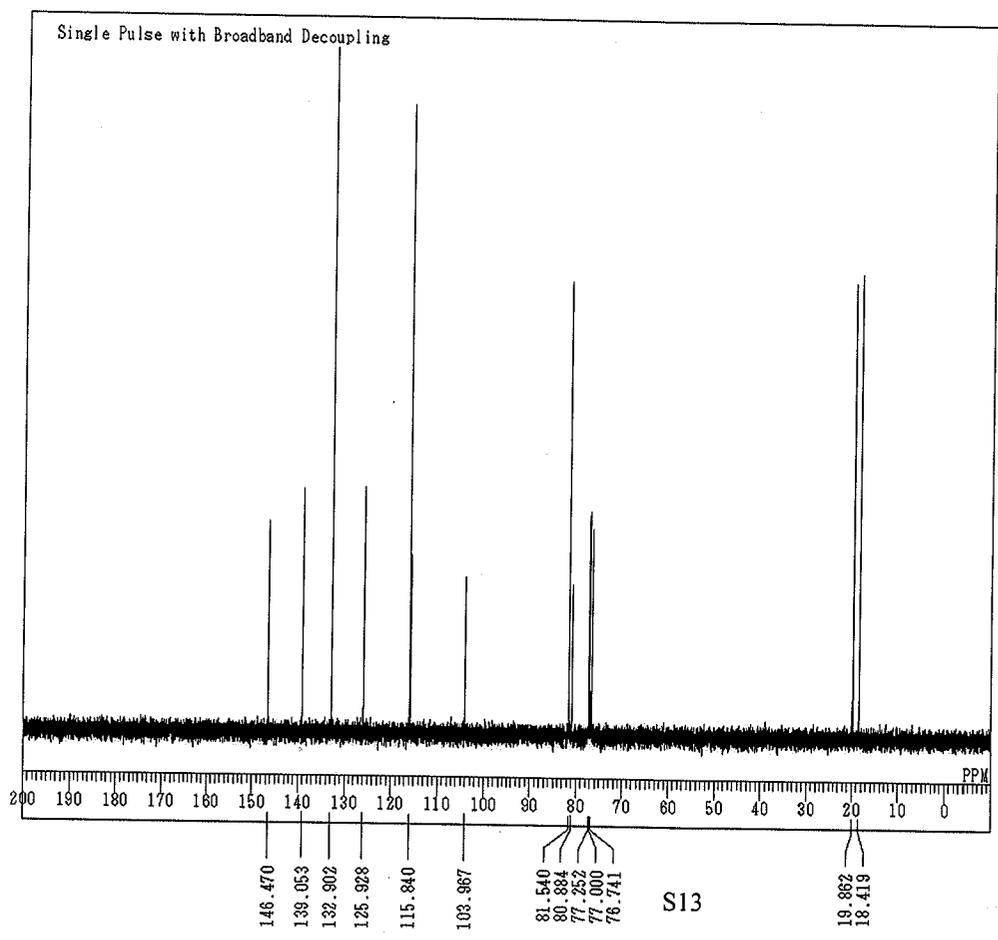
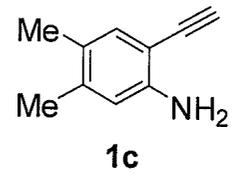
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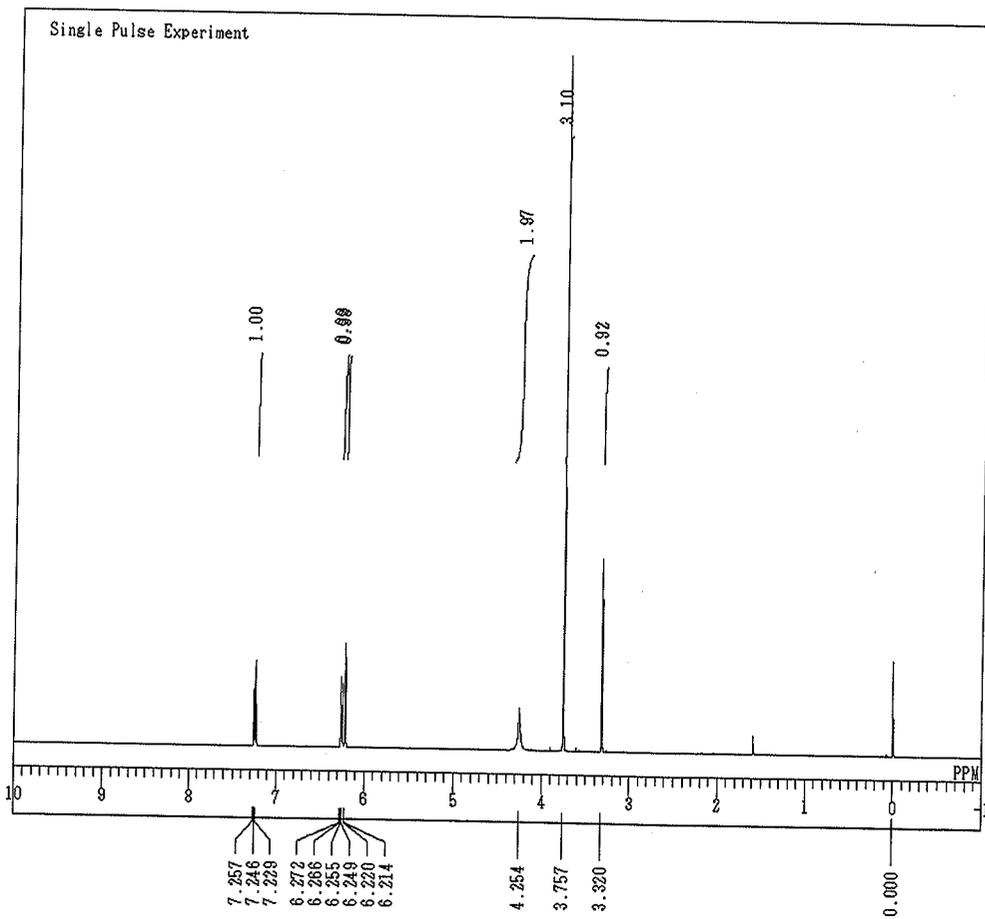
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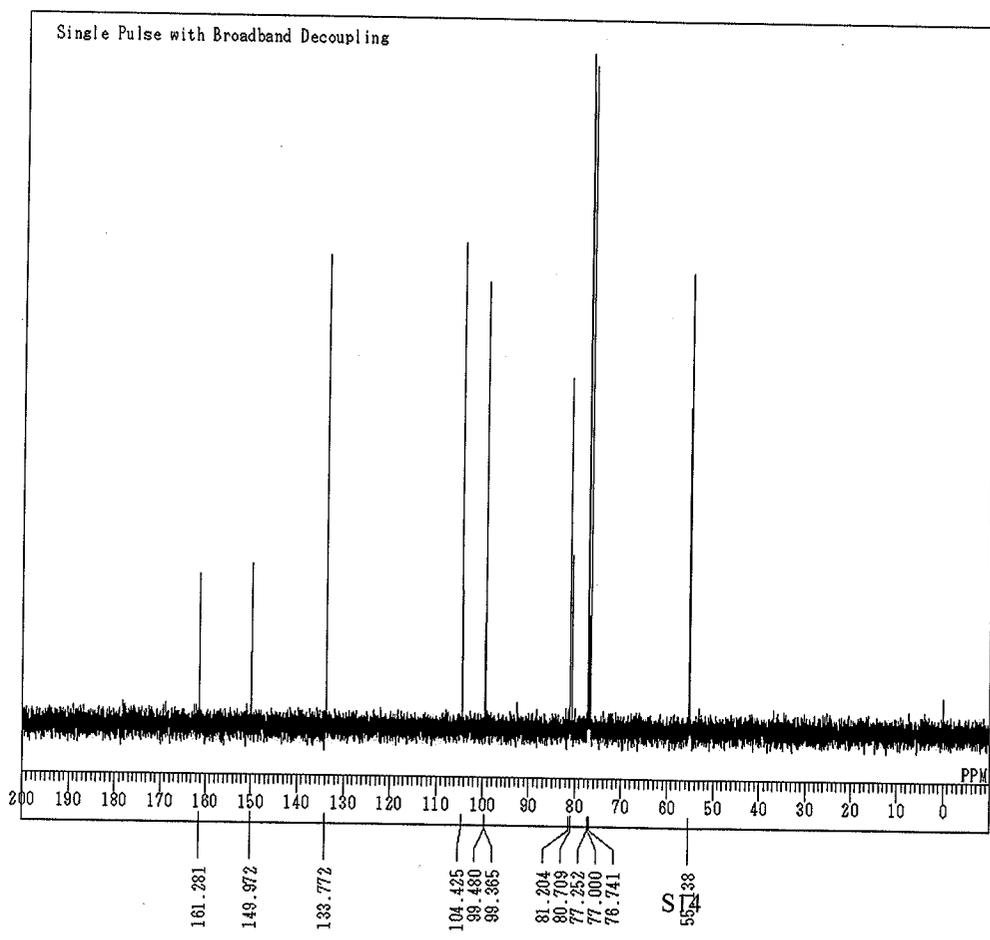
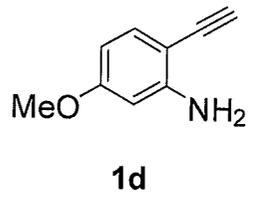
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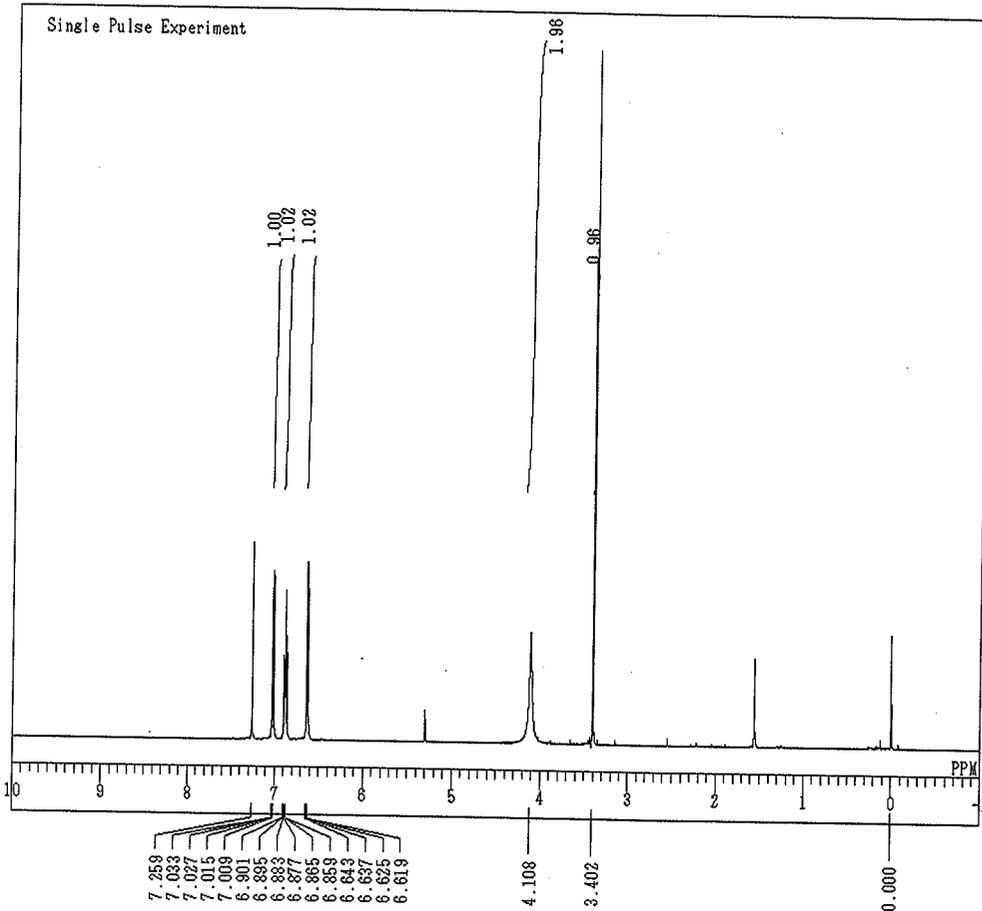
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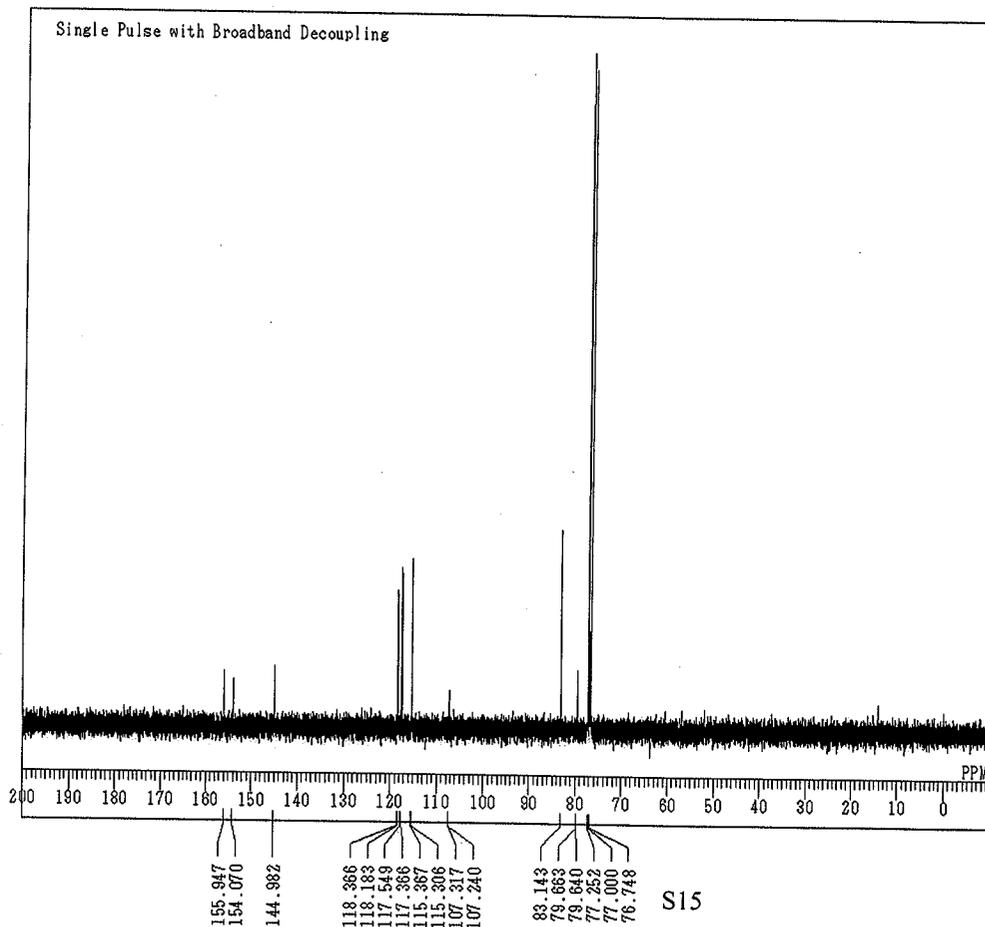
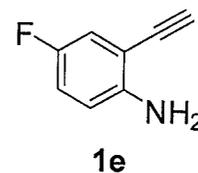
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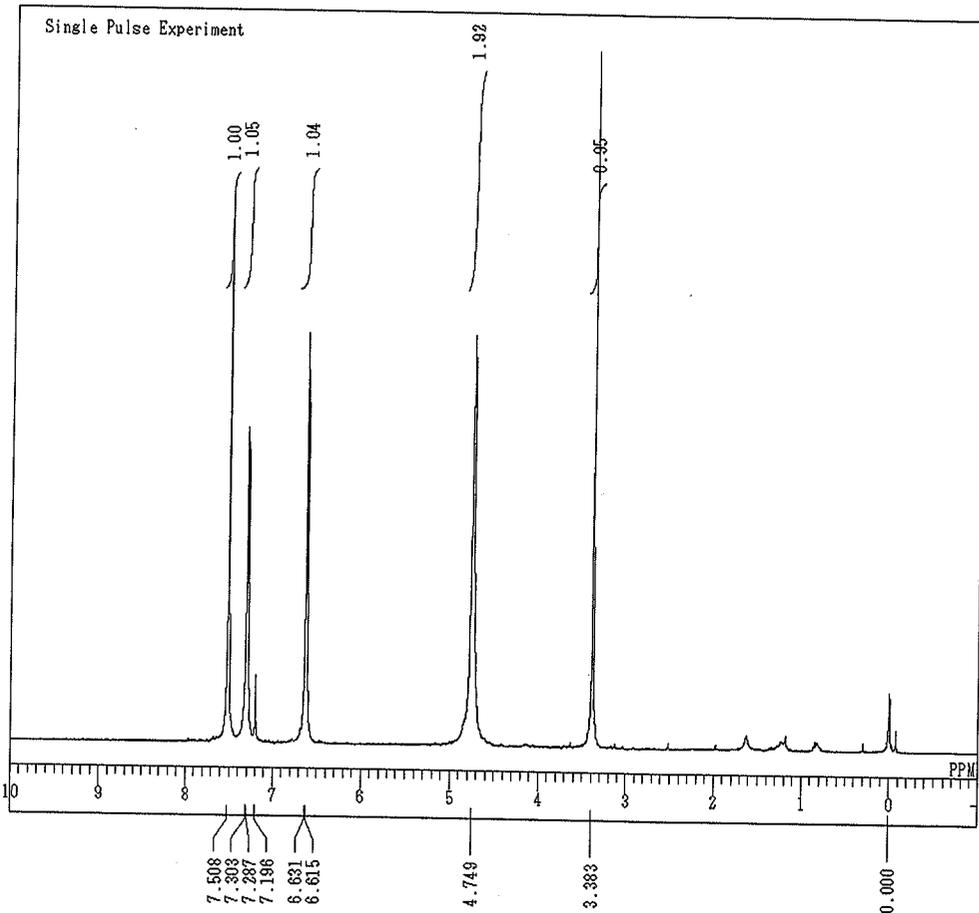
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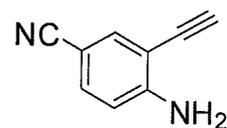
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 BF 0.23 Hz
 RGAIN 21



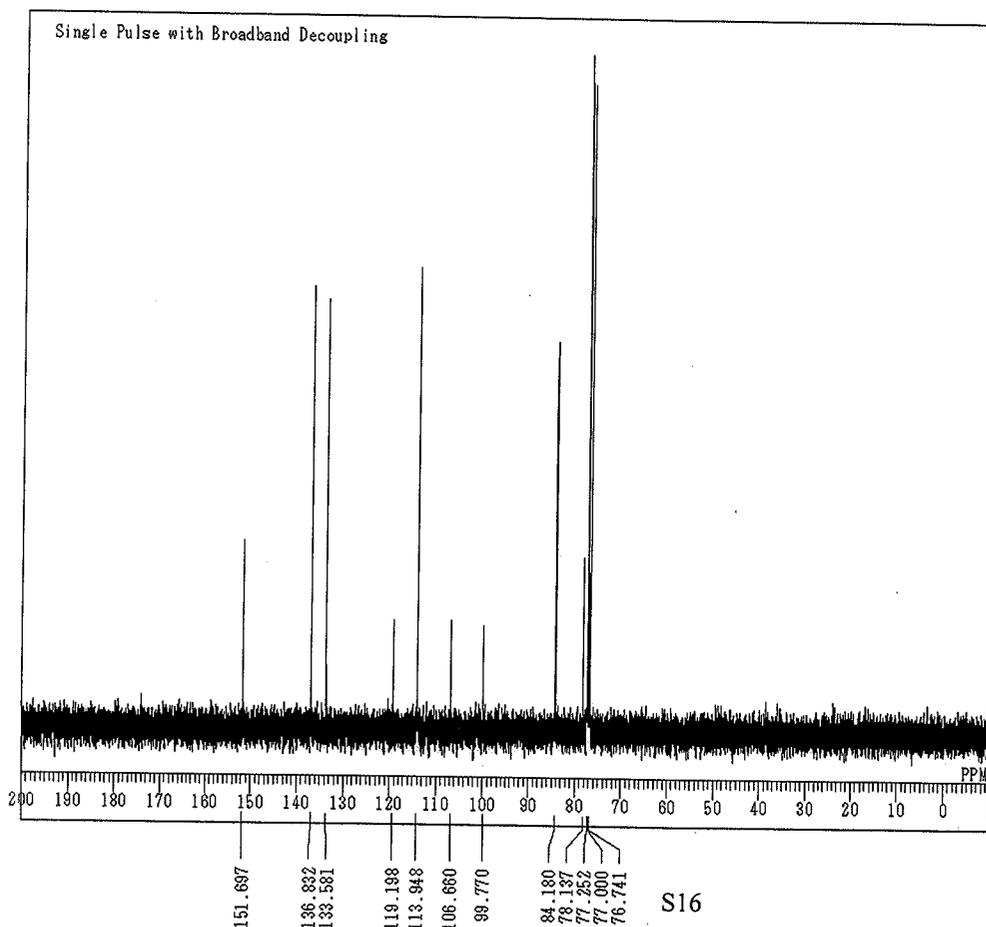
COMNT Single Pulse with Broadband D
 DATIM 01-11-2011 06:35:24
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFREQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 31446.54 Hz
 SCANS 223
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 25.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.98 Hz
 RGAIN 30



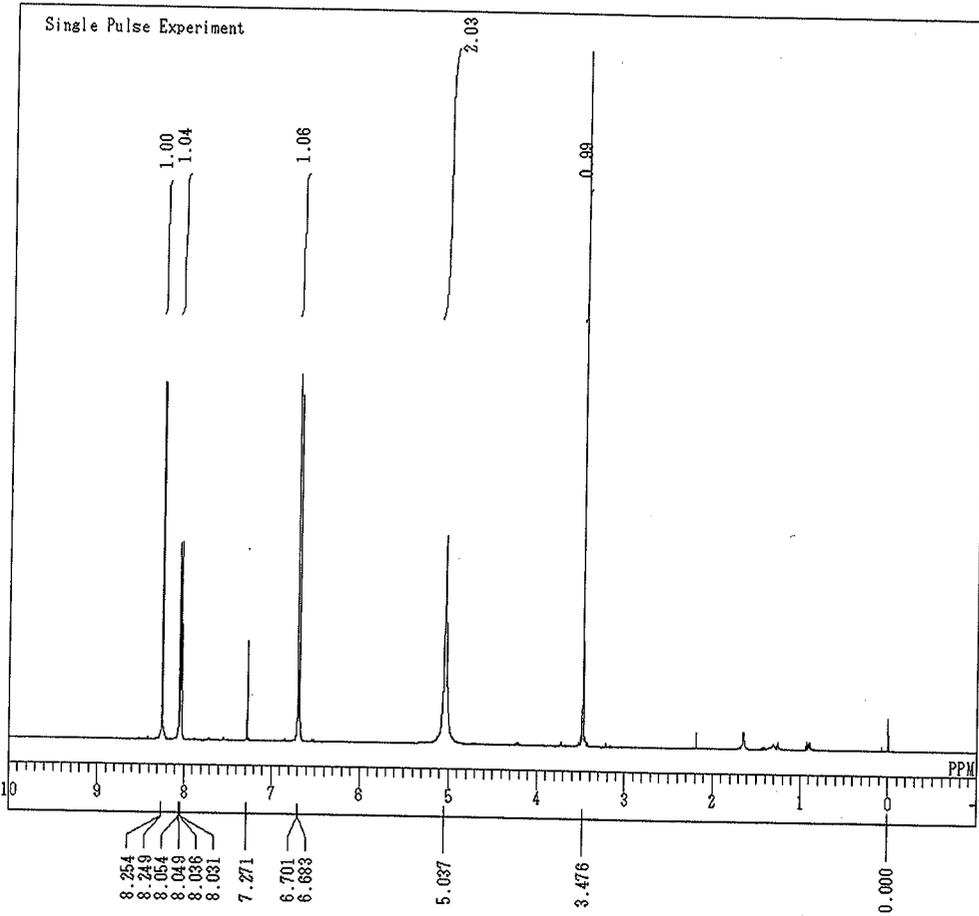
COMNT Single Pulse Experiment
 DATIM 06-12-2011 08:38:47
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFREQ 500.16 MHz
 OBSETE 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16384
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 2.1823 sec
 PD 4.0000 sec
 PW1 7.00 usec
 IRNUC
 CTEMP 18.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 15



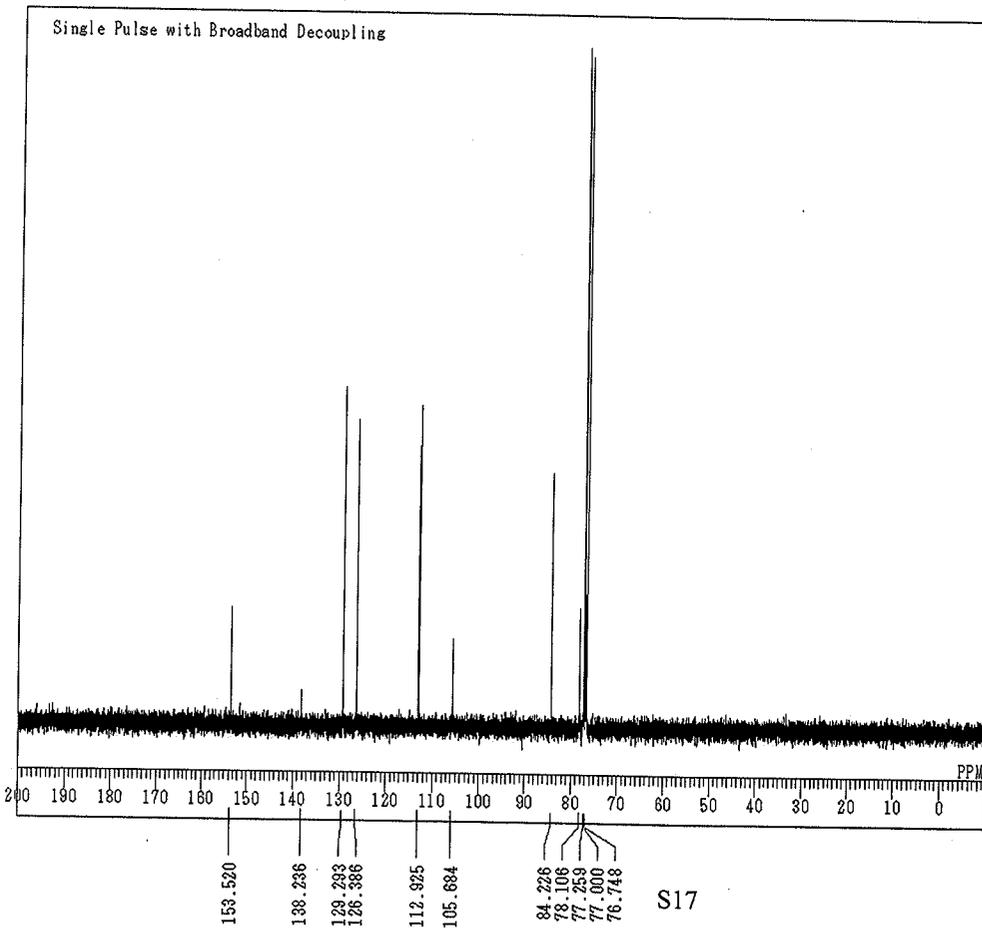
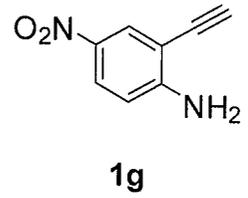
1f



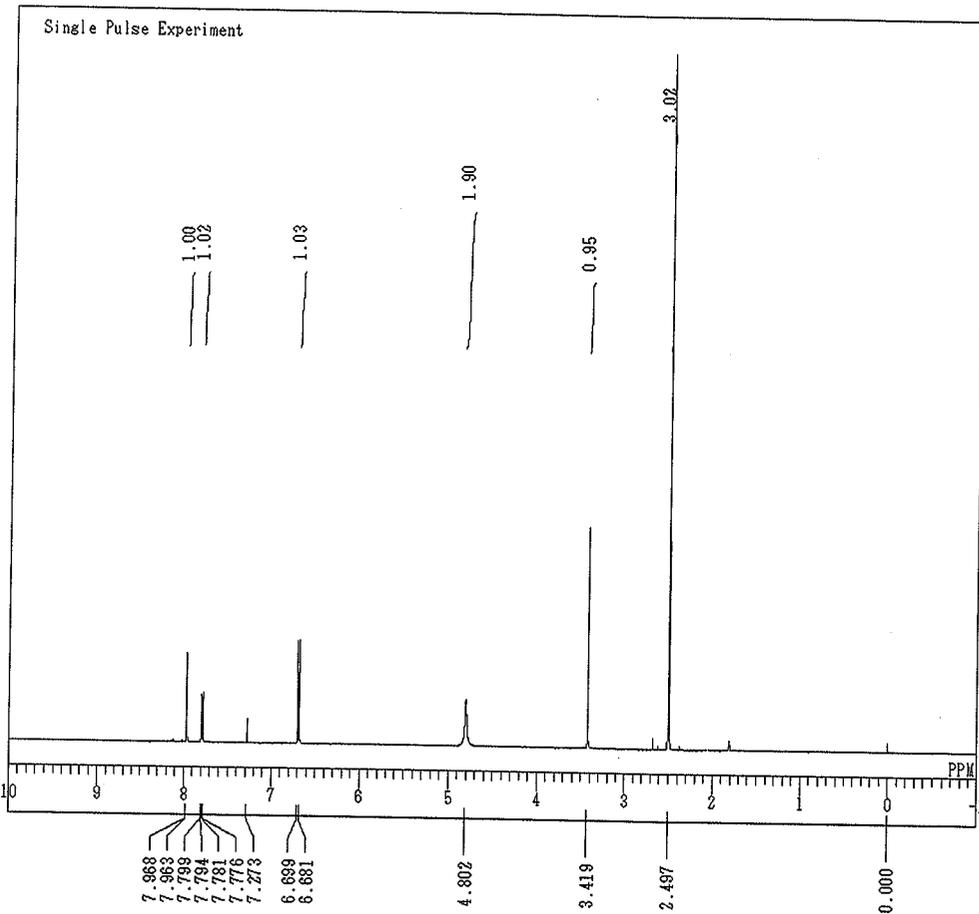
COMNT Single Pulse with Broadband D
 DATIM 06-12-2011 08:47:13
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFREQ 125.77 MHz
 OBSETE 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 31446.54 Hz
 SCANS 144
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 21.0 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.96 Hz
 RGAIN 30



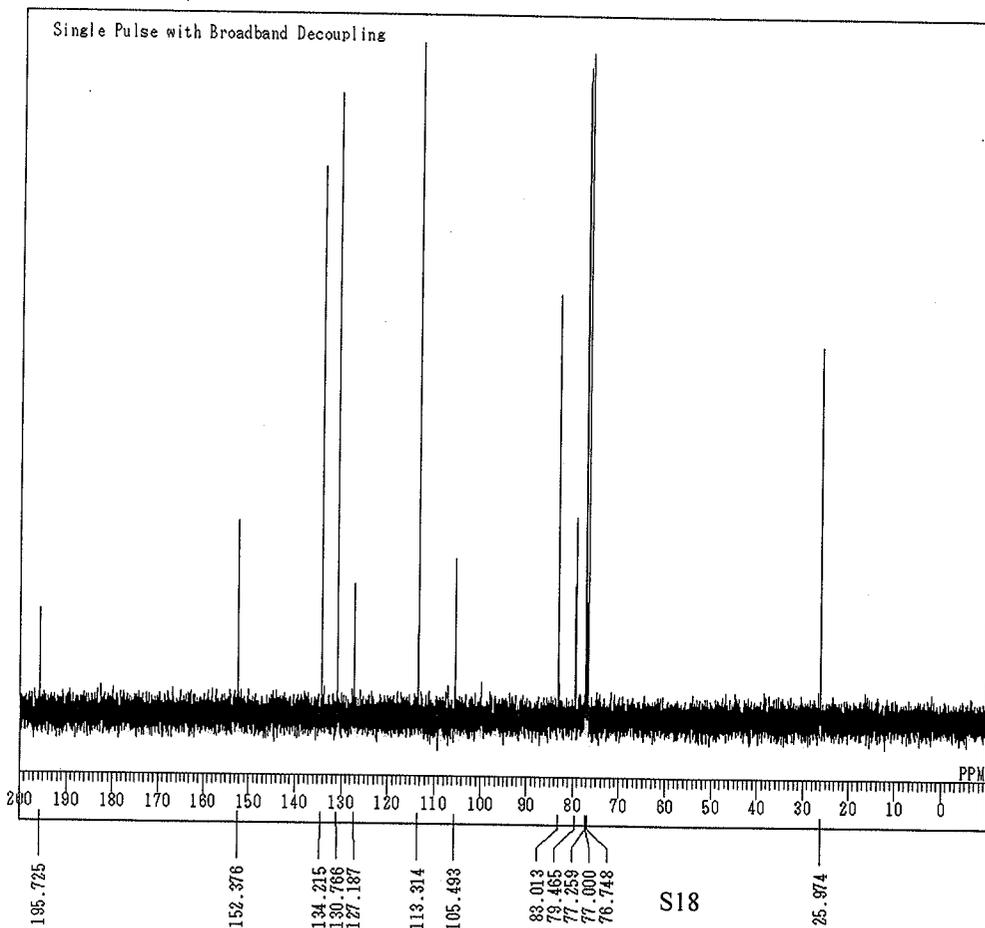
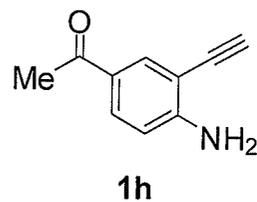
COMNT Single Pulse Experiment
 DATIM 06-12-2011 08:53:25
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFRQ 500.16 MHz
 OBSET 2.41 kHz
 OBFIN 8.01 Hz
 POINT 16384
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 2.1823 sec
 PD 4.0000 sec
 PW1 7.00 usec
 IRNUC
 CTEMP 19.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 17



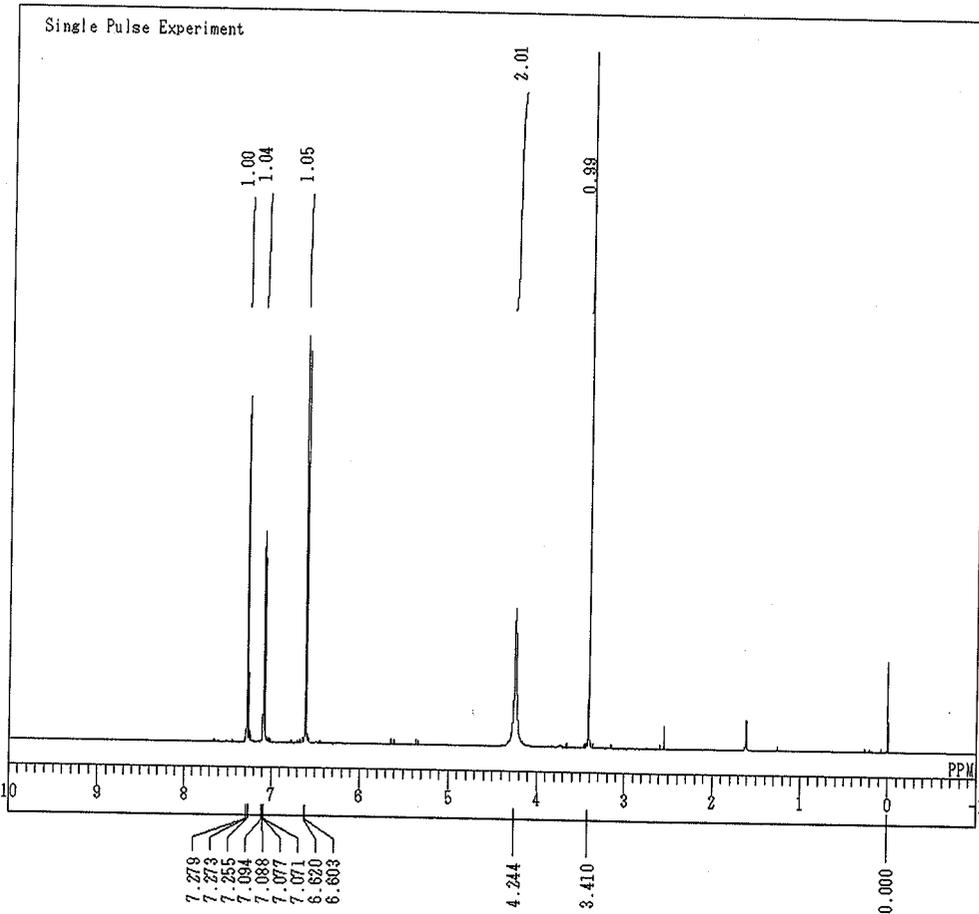
COMNT Single Pulse with Broadband D
 DATIM 06-12-2011 09:03:34
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.77 MHz
 OBSET 7.87 kHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 31446.54 Hz
 SCANS 215
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 21.3 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.96 Hz
 RGAIN 30



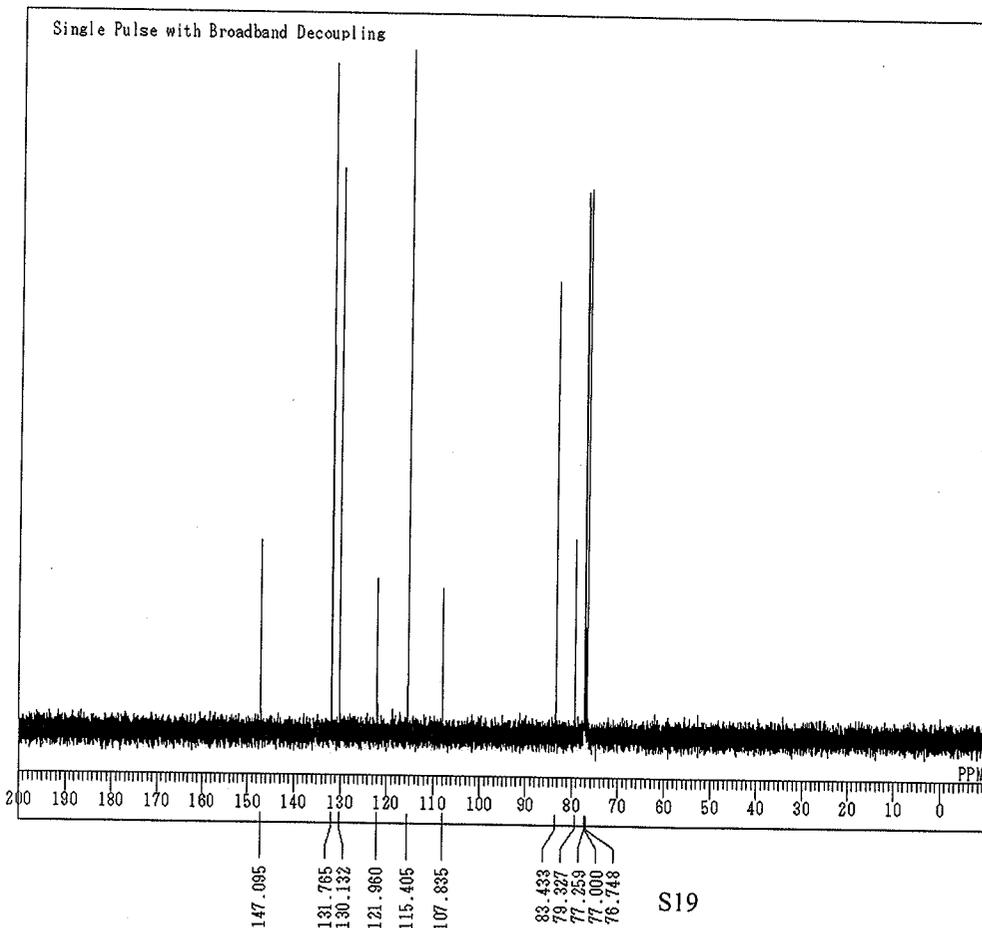
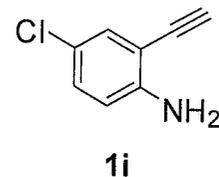
COMNT Single Pulse Experiment
 DATIM 04-11-2011 22:02:08
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 8.01 Hz
 POINT 16384
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 2.1823 sec
 PD 4.0000 sec
 PW1 7.00 usec
 IRNUC
 CTEMP 24.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 15



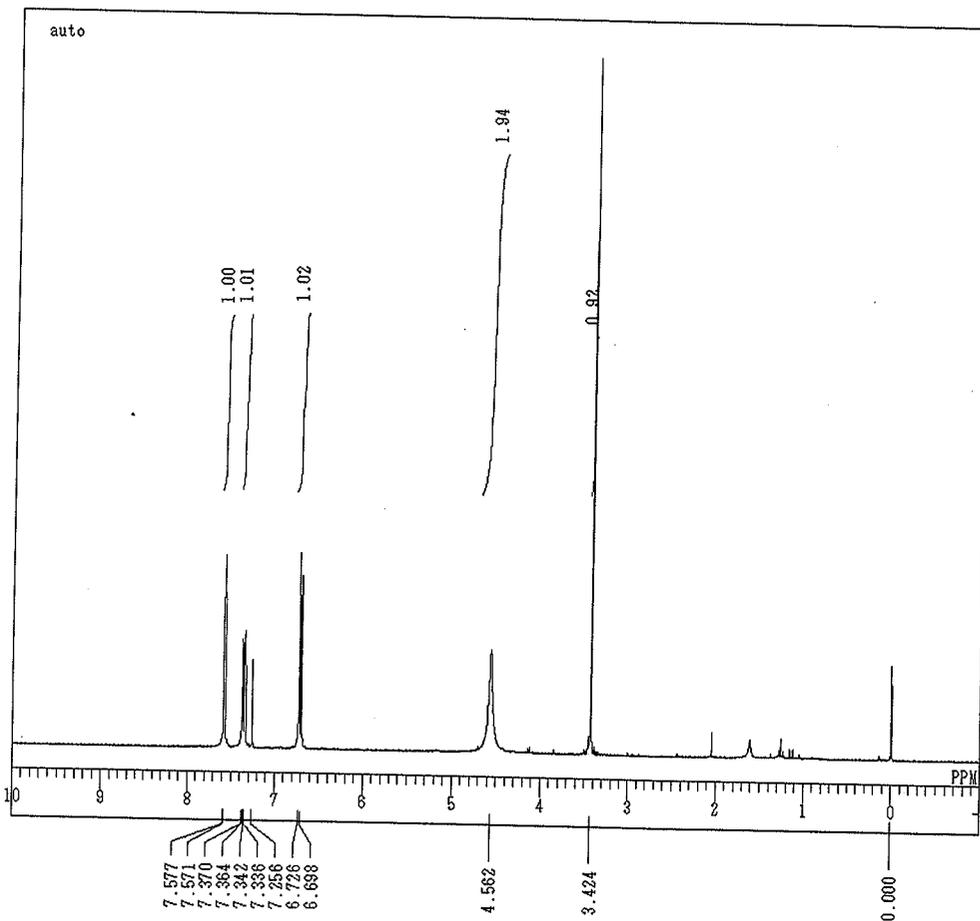
COMNT Single Pulse with Broadband D
 DATIM 04-11-2011 22:08:34
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 31448.54 Hz
 SCANS 102
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 25.7 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.98 Hz
 RGAIN 30



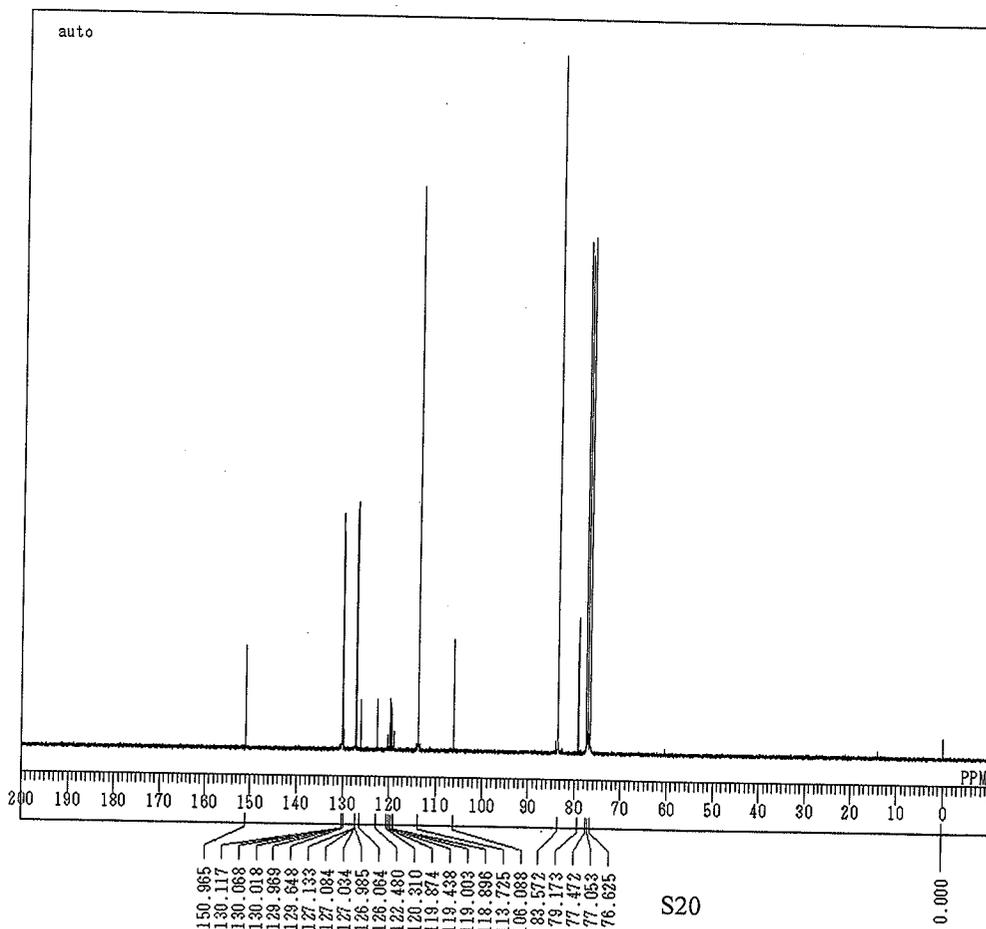
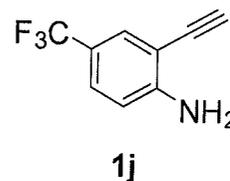
COMNT Single Pulse Experiment
 DATIM 05-08-2011 01:12:33
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIM 6.01 Hz
 POINT 16384
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 2.1823 sec
 PD 4.0000 sec
 PW1 7.00 usec
 IRNUC
 CTEMP 21.0 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 15



COMNT Single Pulse with Broadband D
 DATIM 05-08-2011 01:19:38
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIM 4.21 Hz
 POINT 32768
 FREQU 31446.54 Hz
 SCANS 157
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 23.0 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.96 Hz
 RGAIN 30

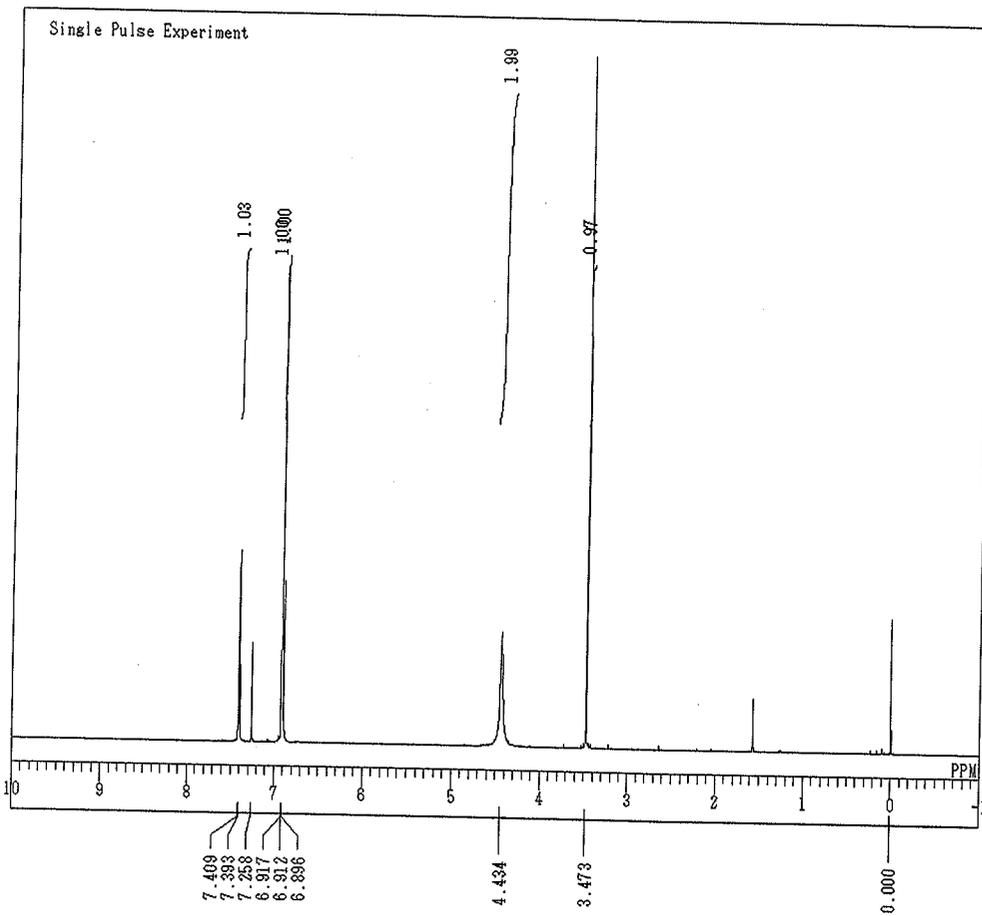


COMNT auto
 DATIM Tue Jun 28 22:55:08 2011
 OBNUC 1H
 EXMOD NON
 OBFREQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.00 Hz
 POINT 16384
 FREQU 6006.01 Hz
 SCANS 8
 ACQTH 2.7279 sec
 PD 4.2720 sec
 PW1 5.50 usec
 IRNUC 1H
 CTEMP 20.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.18 Hz
 RGAIN 17

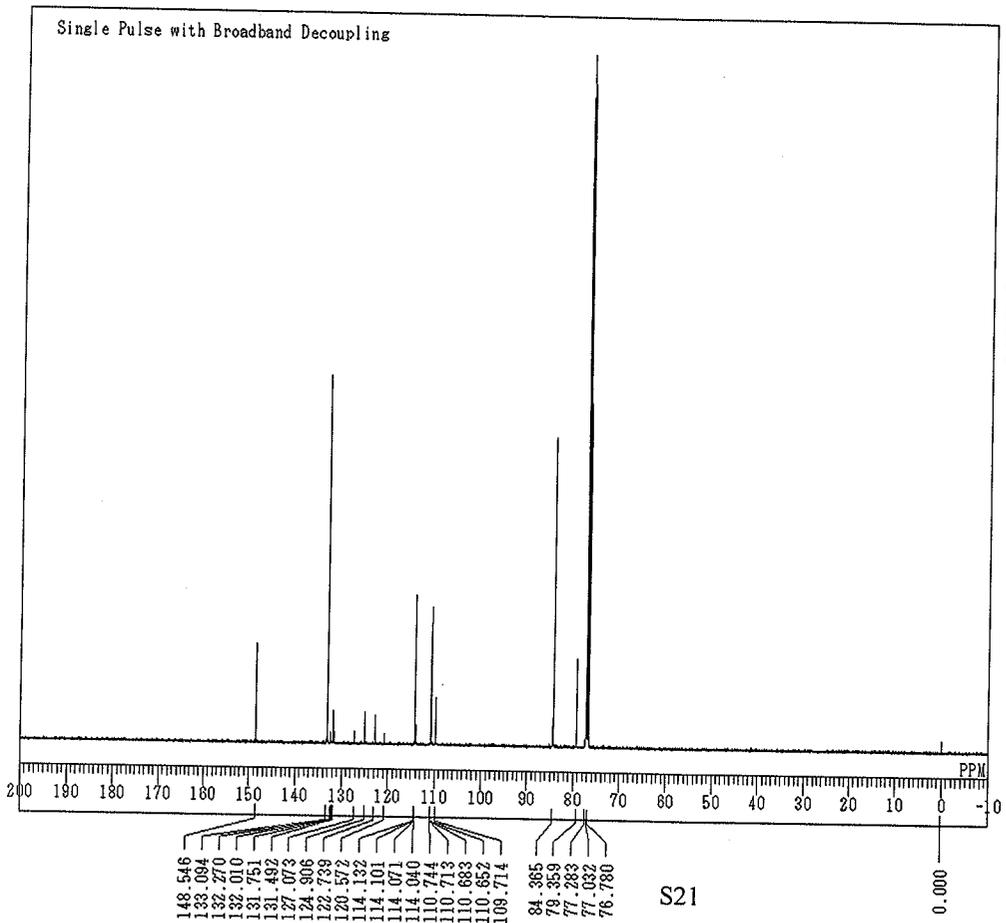
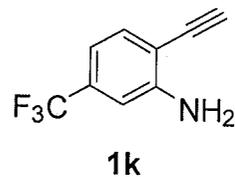


COMNT auto
 DATIM Wed Jun 29 09:52:26 2011
 OBNUC 13C
 EXMOD BCM
 OBFREQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.00 Hz
 POINT 32768
 FREQU 20356.23 Hz
 SCANS 13092
 ACQTH 1.6097 sec
 PD 1.3900 sec
 PW1 4.00 usec
 IRNUC 1H
 CTEMP 20.3 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.62 Hz
 RGAIN 27

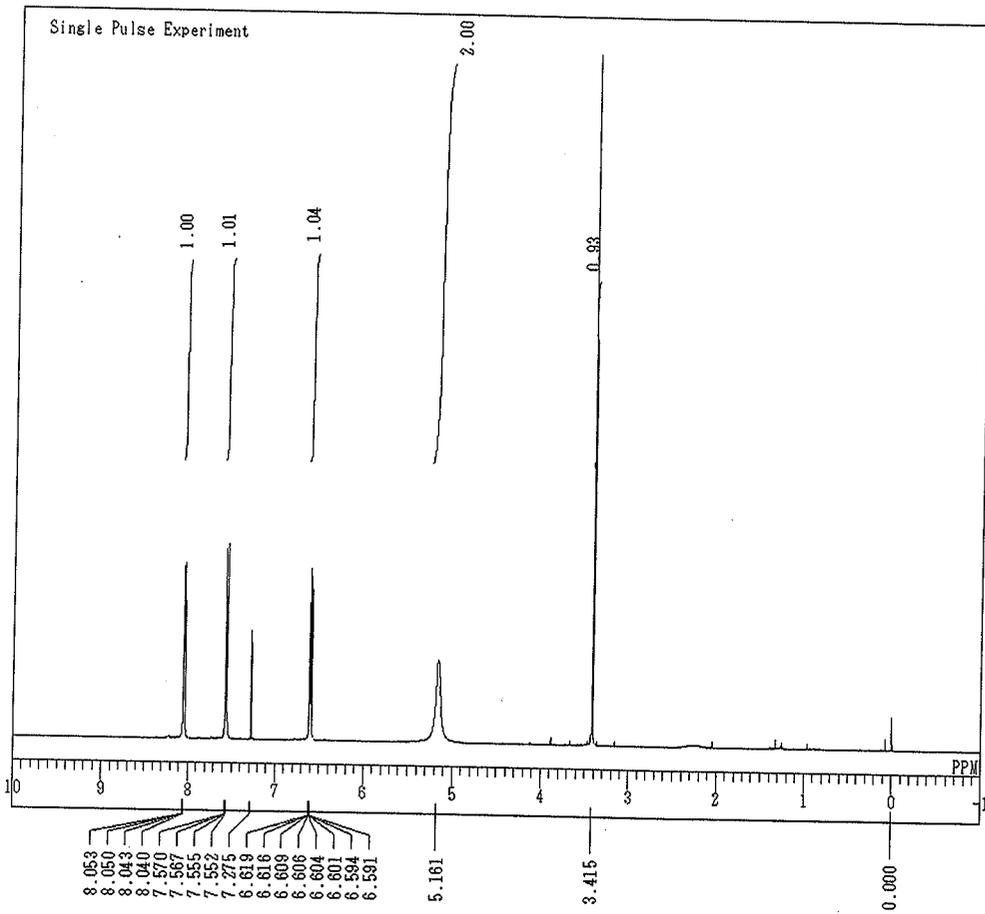
S20



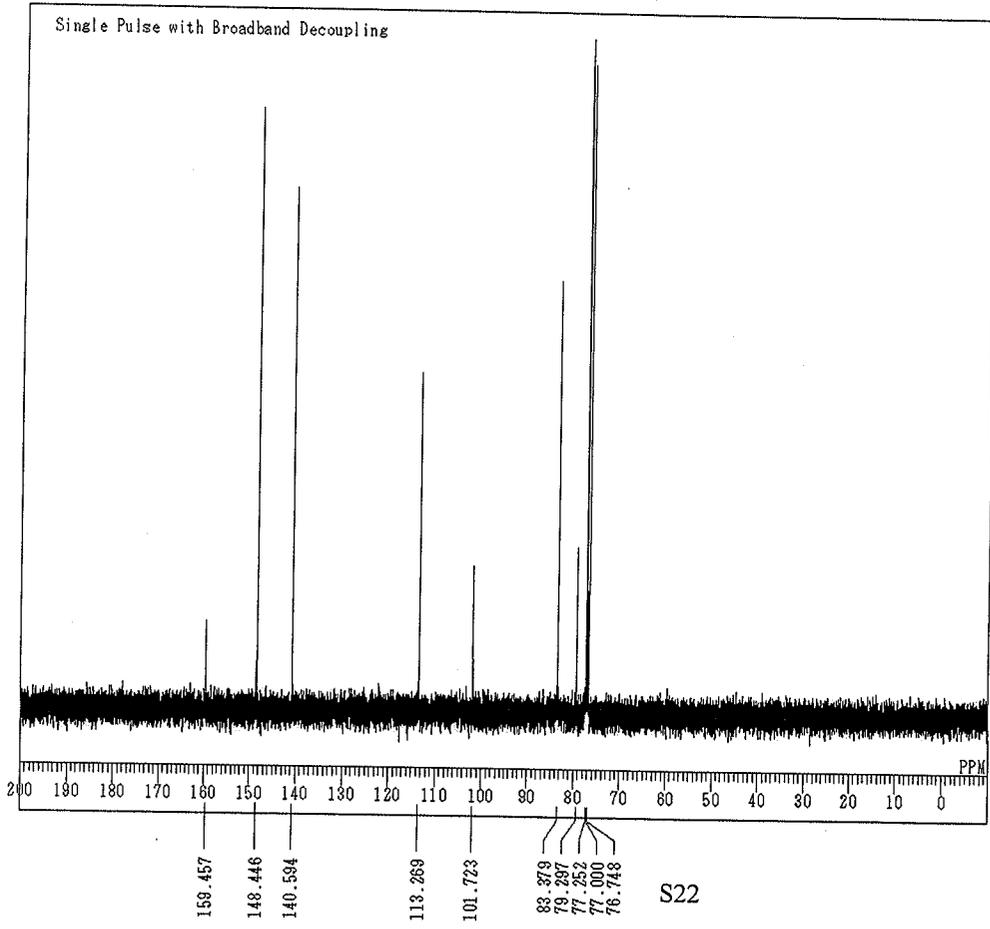
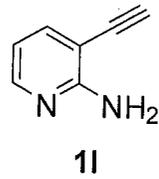
COMNT Single Pulse Experiment
 DATIM 12-12-2011 09:11:37
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFREQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 8.01 Hz
 POINT 16384
 FREQ 7507.51 Hz
 SCANS 8
 ACQTM 2.1823 sec
 PD 4.0000 sec
 PW1 7.00 usec
 IRNUC
 CTEMP 19.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 17



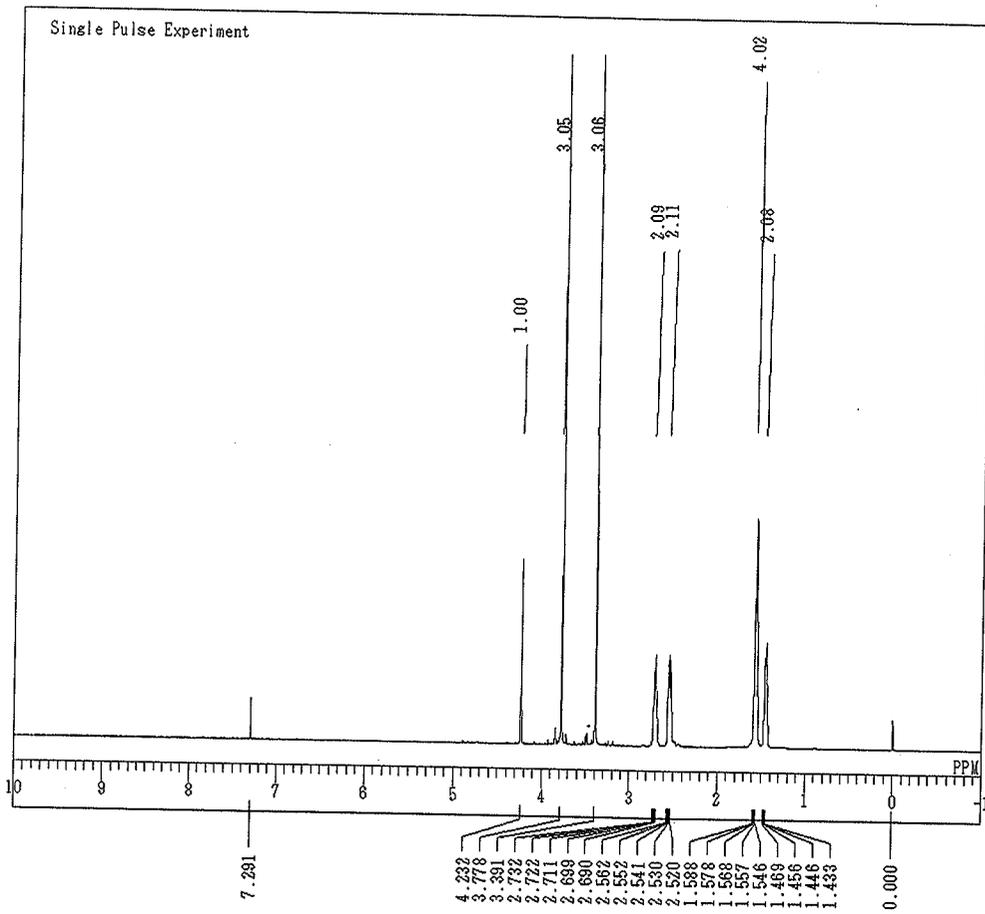
COMNT Single Pulse with Broadband D
 DATIM 12-12-2011 18:48:58
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFREQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQ 31446.54 Hz
 SCANS 16886
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 22.1 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.96 Hz
 RGAIN 30



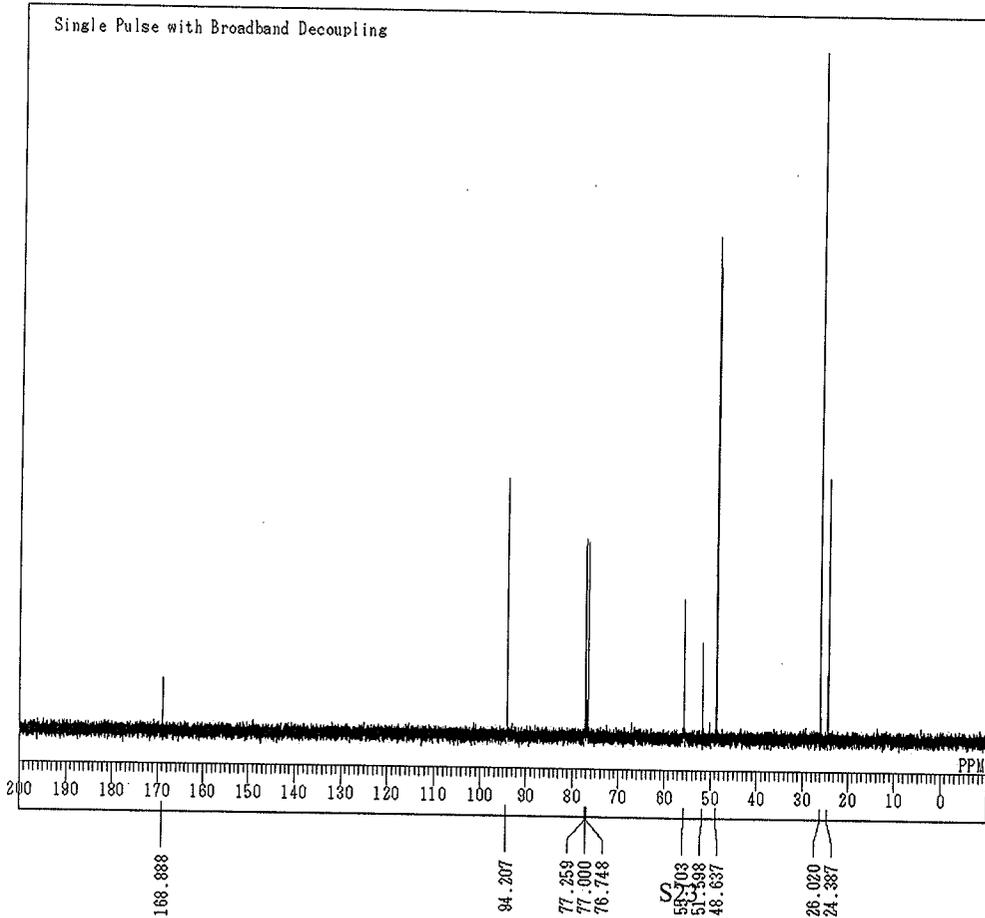
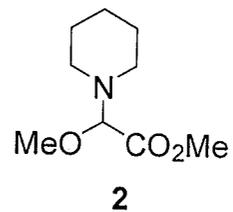
COMNT Single Pulse Experiment
 DATIM 04-11-2011 21:48:53
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFREQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16384
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 2.1823 sec
 PD 4.0000 sec
 PW1 7.00 usec
 IRNUC
 CTEMP 24.1 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 17



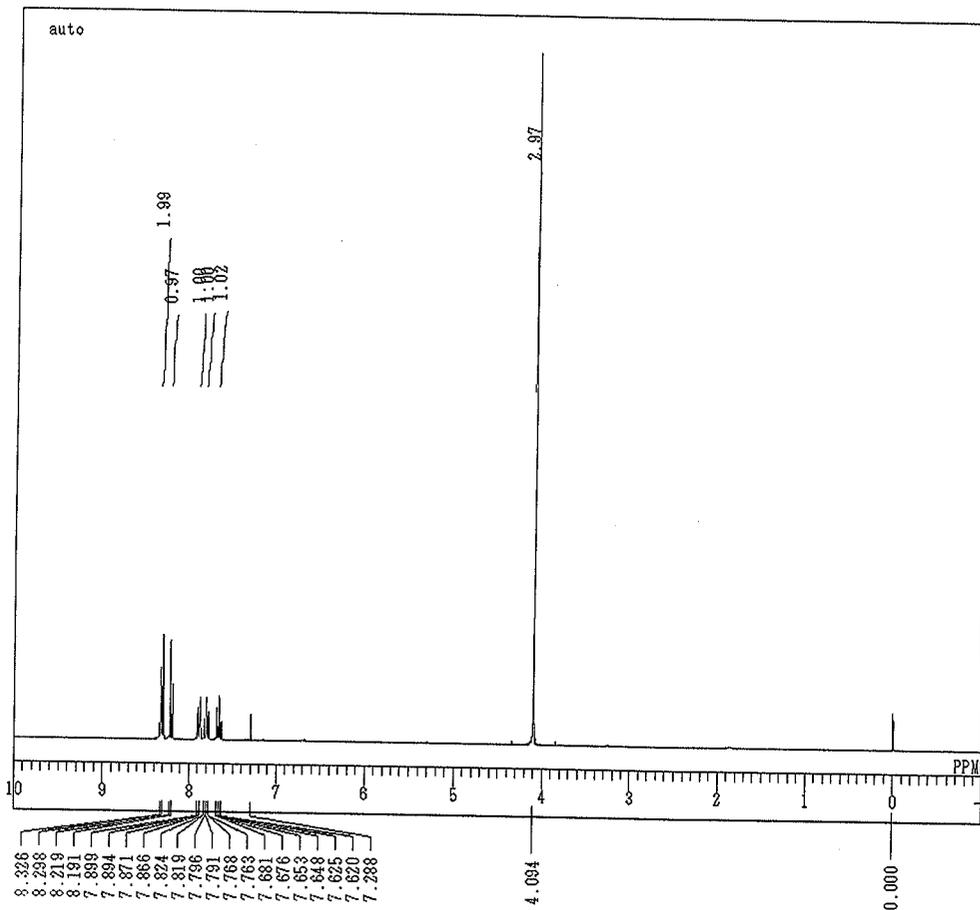
COMNT Single Pulse with Broadband D
 DATIM 04-11-2011 21:57:05
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFREQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 31446.54 Hz
 SCANS 203
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 28.4 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.96 Hz
 RGAIN 30



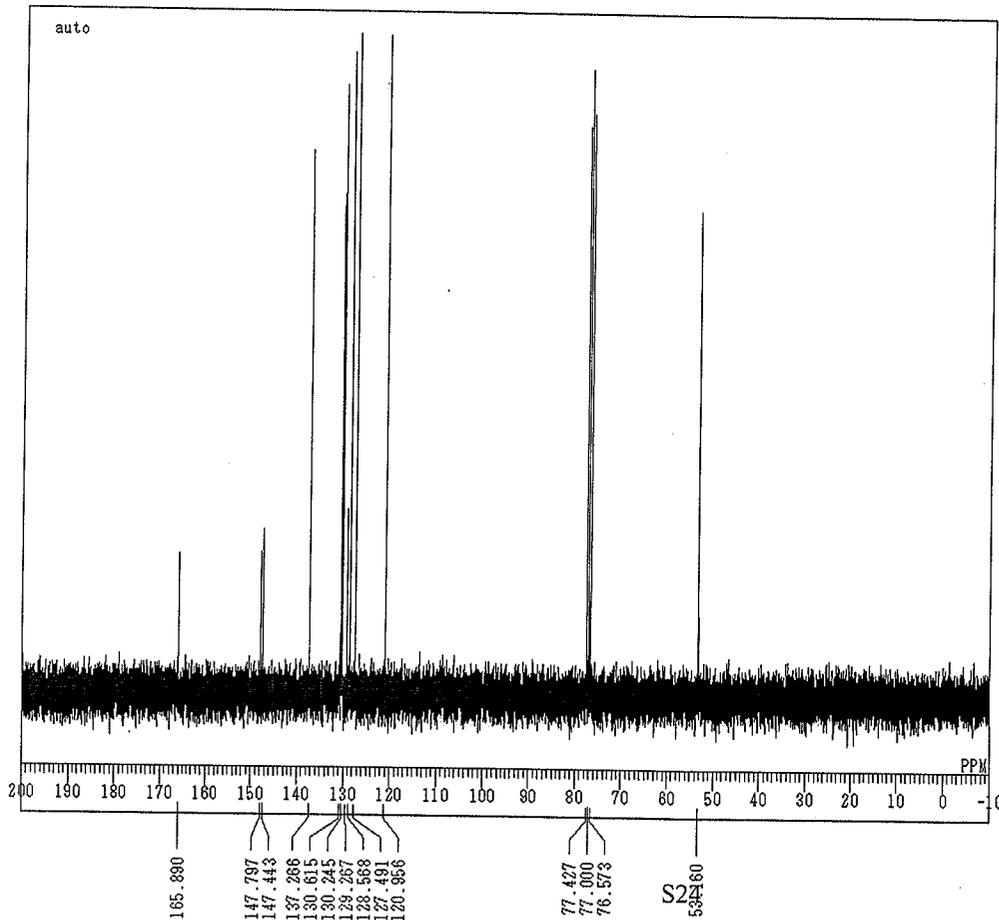
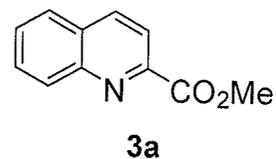
COMNT Single Pulse Experiment
 DATIM 27-06-2009 14:41:16
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFREQ 500.16 MHz
 OBSSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16384
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 2.1823 sec
 PD 4.0000 sec
 PW1 7.00 usec
 IRNUC
 CTEMP 22.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 11



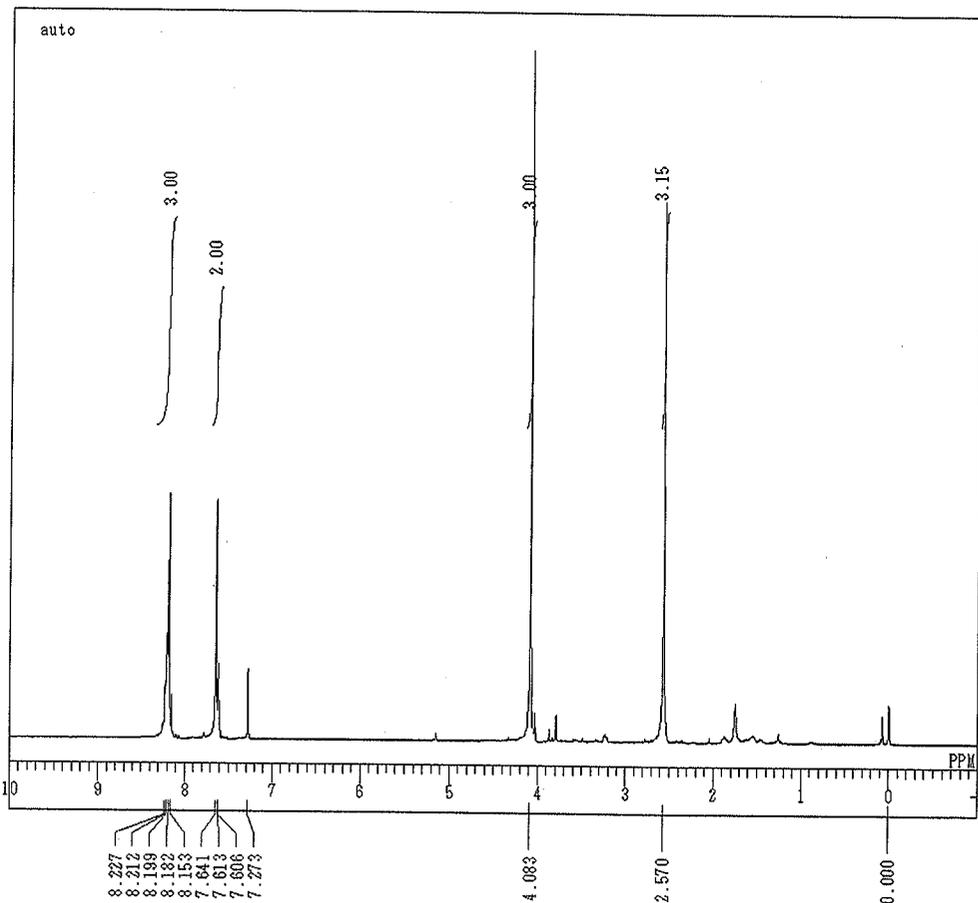
COMNT Single Pulse with Broadband D
 DATIM 27-06-2009 14:47:27
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFREQ 125.77 MHz
 OBSSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 31446.54 Hz
 SCANS 101
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 25.4 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.96 Hz
 RGAIN 30



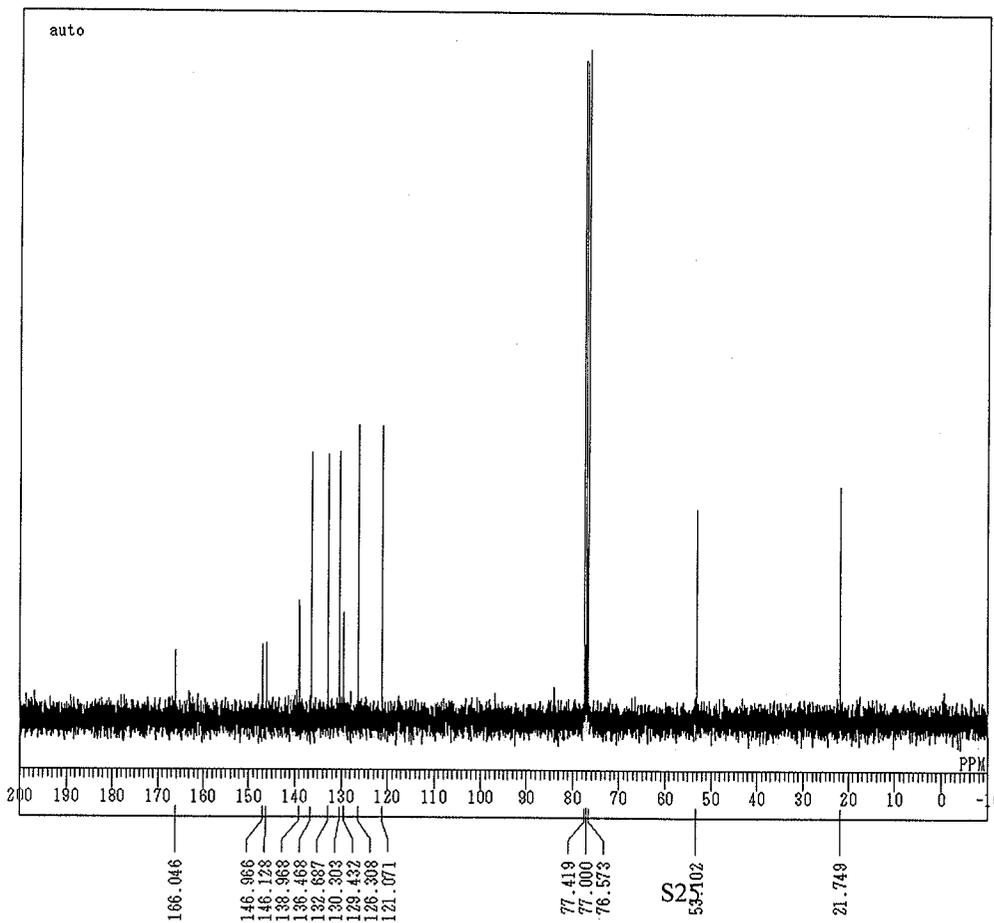
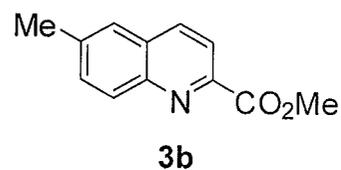
COMNT auto
 DATIM Thu Mar 18 17:00:29 2010
 OBNUC 1H
 EXMOD NON
 OBFRQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.00 Hz
 POINT 18384
 FREQU 6006.01 Hz
 SCANS 8
 ACQTM 2.7279 sec
 PD 4.2720 sec
 PW1 5.50 usec
 IRNUC 1H
 CTEMP 20.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.18 Hz
 RGAIN 16



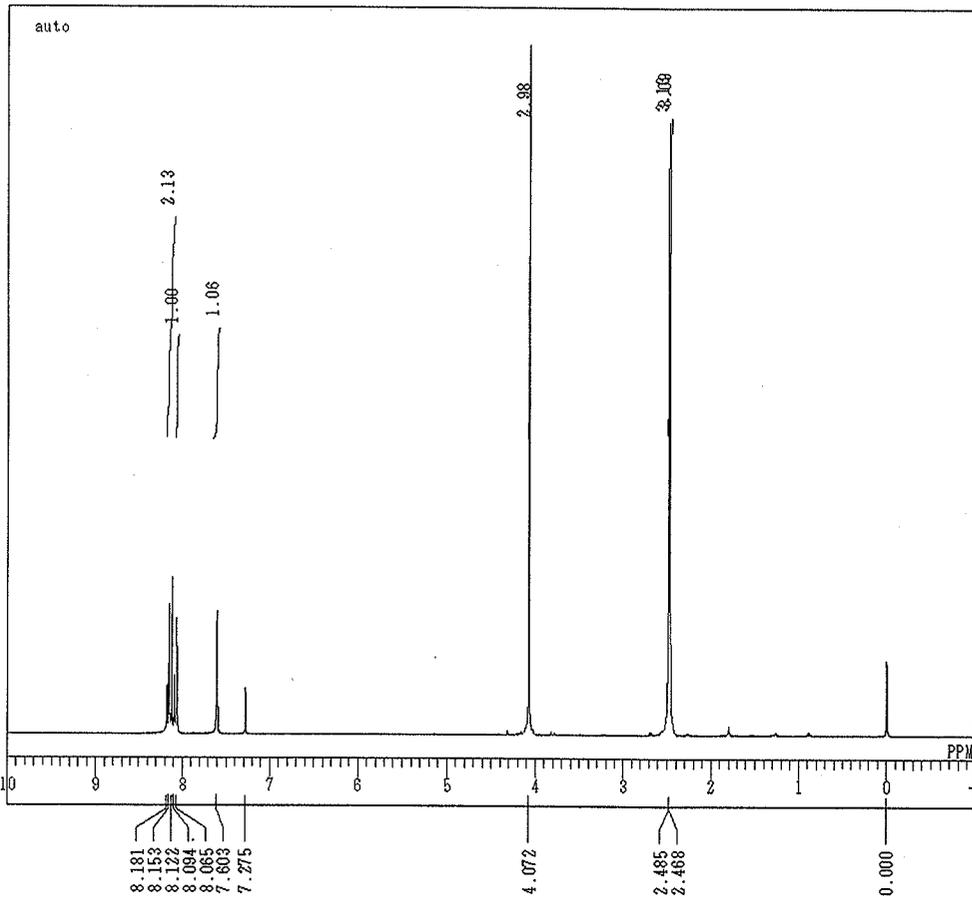
COMNT auto
 DATIM Thu Mar 18 17:03:52 2010
 OBNUC 13C
 EXMOD BCM
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.00 Hz
 POINT 32768
 FREQU 20356.23 Hz
 SCANS 55
 ACQTM 1.6097 sec
 PD 1.3900 sec
 PW1 4.00 usec
 IRNUC 1H
 CTEMP 21.1 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.62 Hz
 RGAIN 28



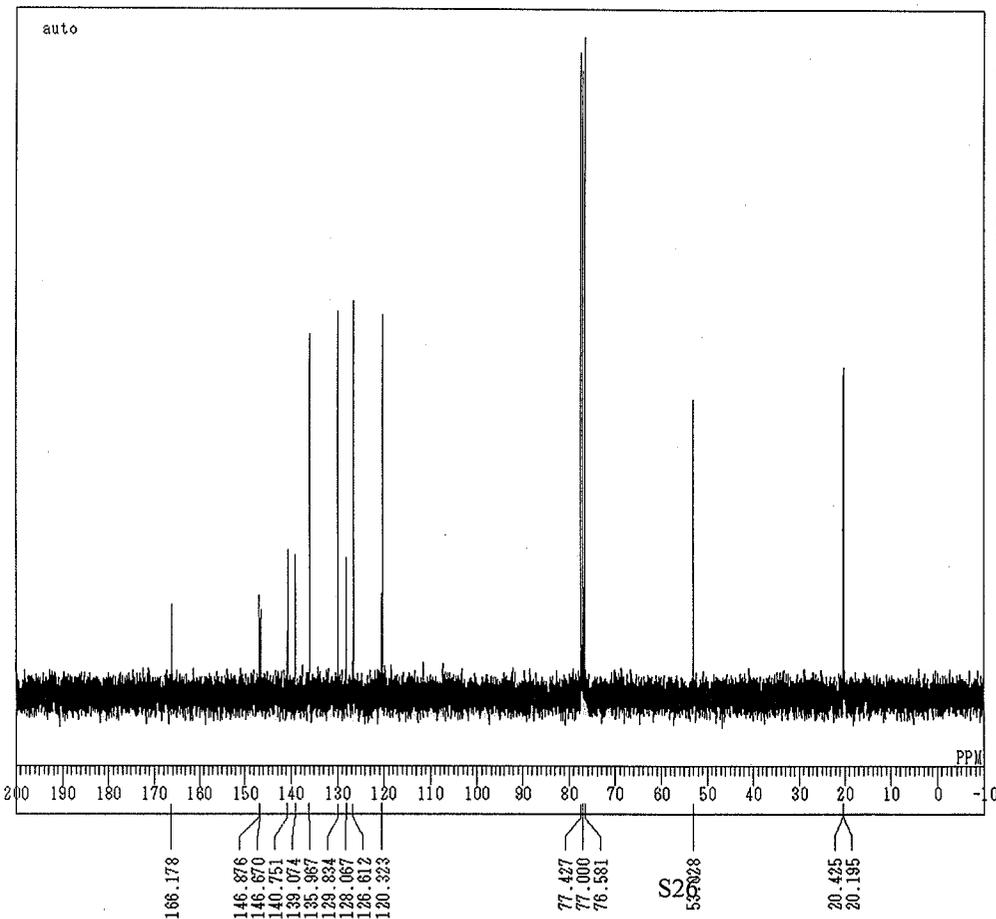
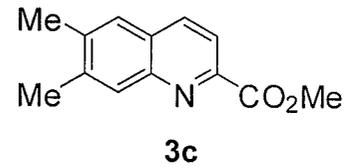
COMNT auto
 DATIM Thu Mar 18 15:04:33 2010
 OBNUC 1H
 EXMOD NON
 OBFREQ 300.40 MHz
 OBSSET 130.00 KHz
 OBFIN 1150.00 Hz
 POINT 16384
 FREQU 6006.01 Hz
 SCANS 8
 ACQTM 2.7273 sec
 PD 4.2720 sec
 PW1 5.50 usec
 IRNUC 1H
 CTEMP 21.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.18 Hz
 RGAIN 18



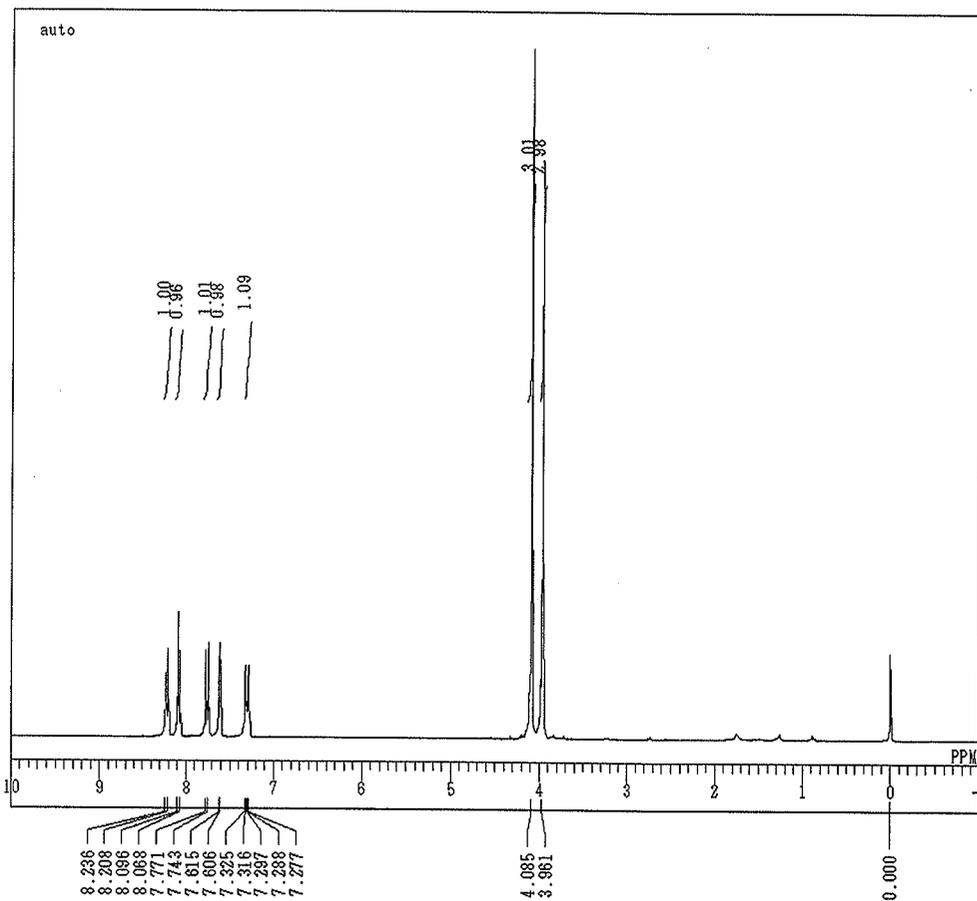
COMNT auto
 DATIM Thu Mar 18 15:15:41 2010
 OBNUC 13C
 EXMOD BCM
 OBFREQ 75.45 MHz
 OBSSET 124.00 KHz
 OBFIN 1840.00 Hz
 POINT 32768
 FREQU 20356.23 Hz
 SCANS 210
 ACQTM 1.6097 sec
 PD 1.3900 sec
 PW1 4.00 usec
 IRNUC 1H
 CTEMP 22.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.62 Hz
 RGAIN 28



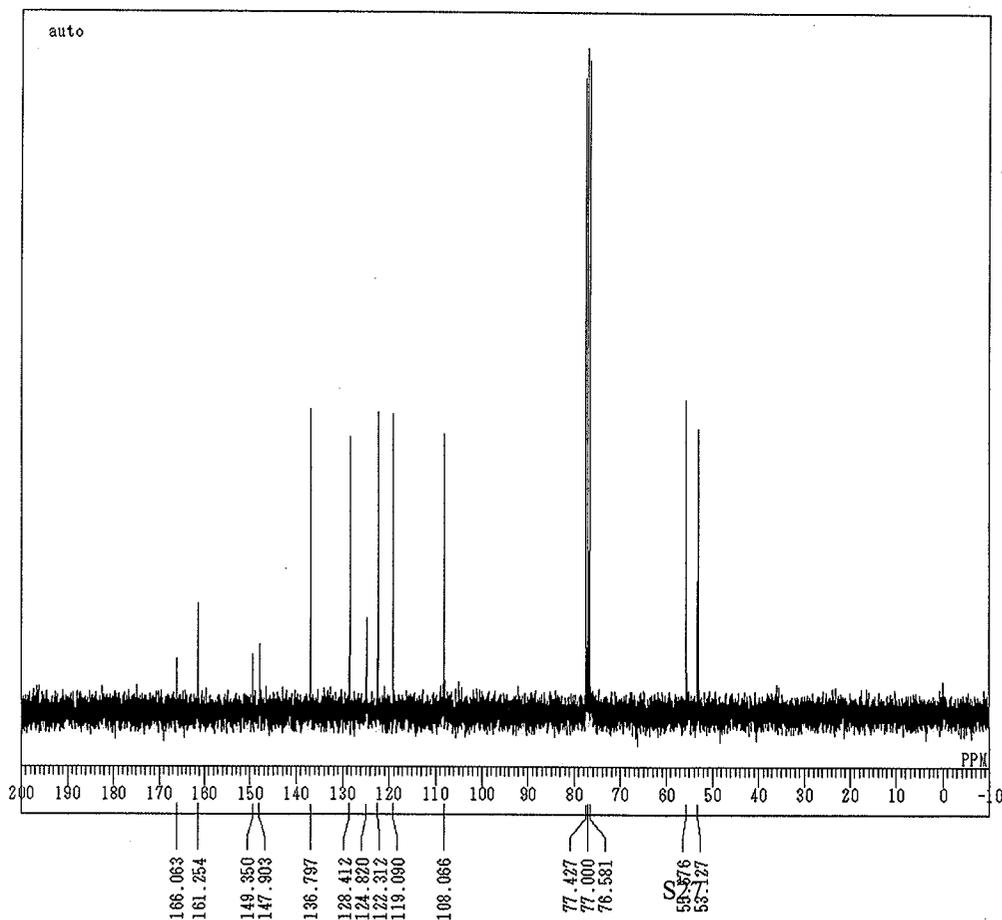
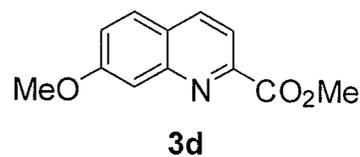
COMNT auto
 DATIM Tue Mar 23 18:33:59 2010
 OBNUC 1H
 EXMOD NON
 OBFRQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.00 Hz
 POINT 16384
 FREQU 6006.01 Hz
 SCANS 8
 ACQTM 2.7278 sec
 PD 4.2720 sec
 PW1 5.50 usec
 IRNUC 1H
 CTEMP 22.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.18 Hz
 RGAIN 15



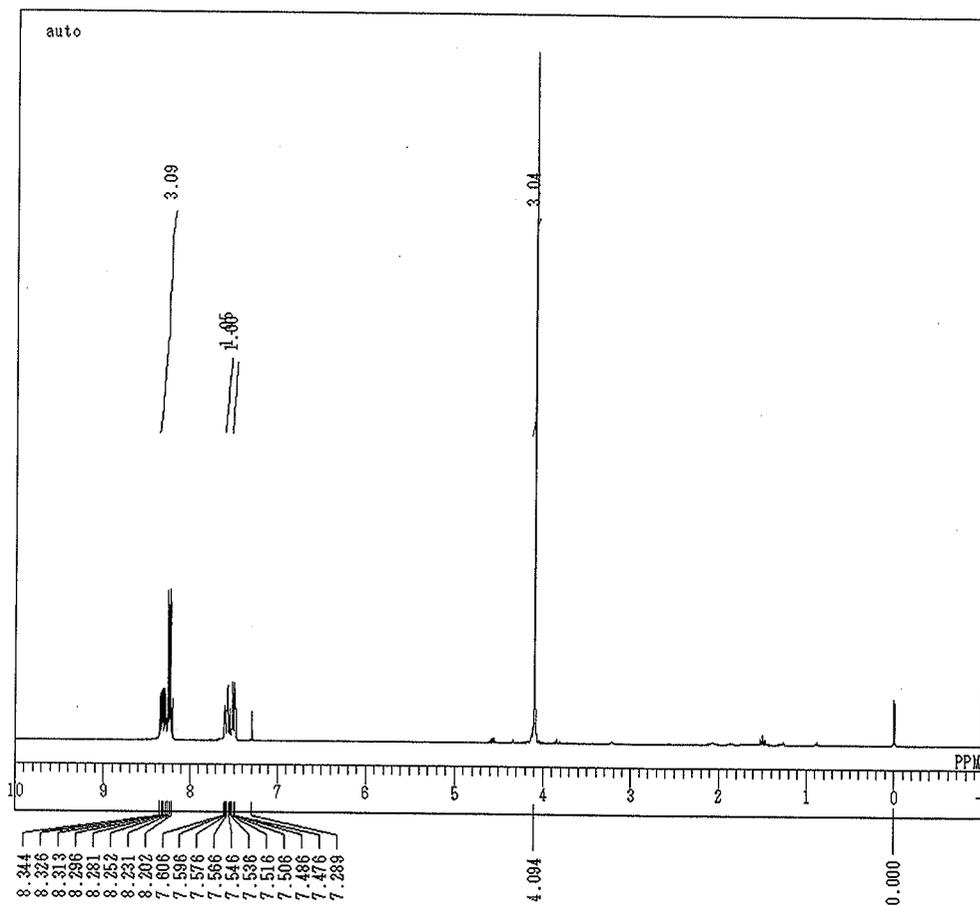
COMNT auto
 DATIM Tue Mar 23 18:40:02 2010
 OBNUC 13C
 EXMOD BCM
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.00 Hz
 POINT 32768
 FREQU 20356.23 Hz
 SCANS 109
 ACQTM 1.6087 sec
 PD 1.3900 sec
 PW1 4.00 usec
 IRNUC 1H
 CTEMP 22.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.62 Hz
 RGAIN 28



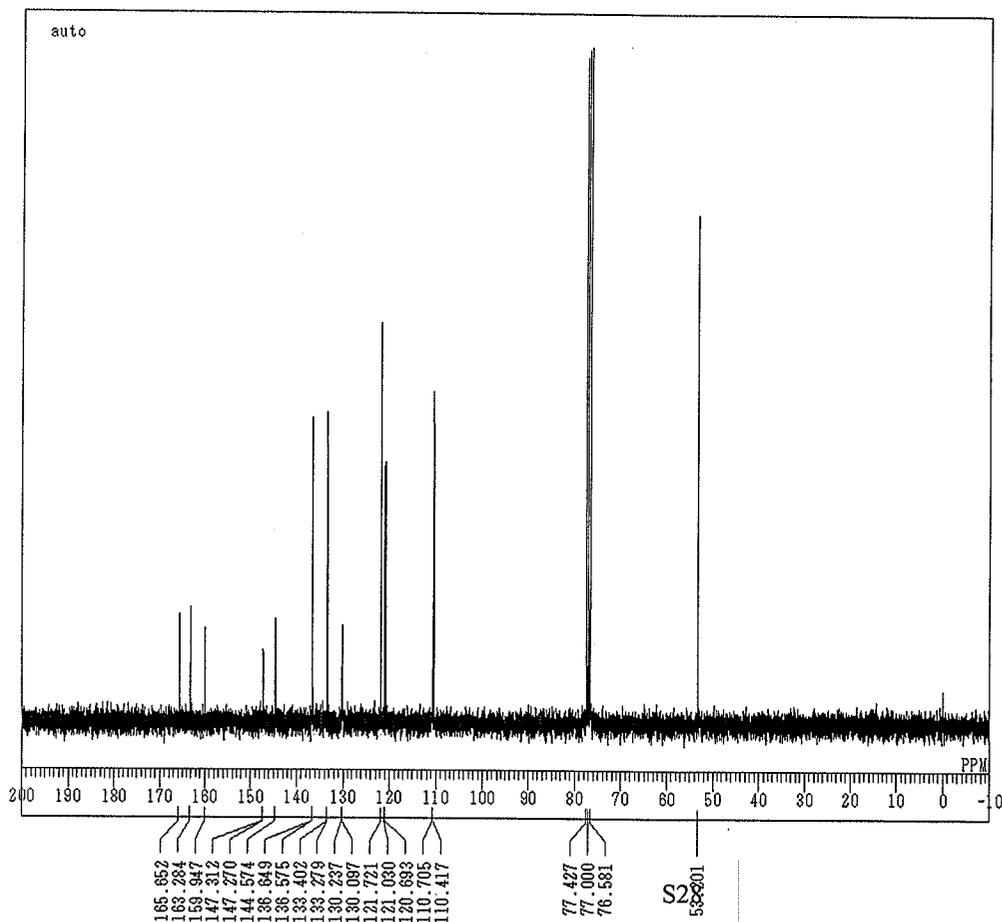
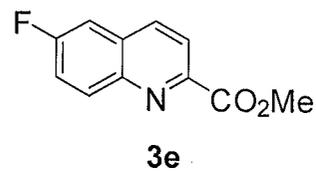
COMNT auto
 DATIM Tue Mar 23 18:18:31 2010
 OBNUC 1H
 EXMOD NON
 OBFREQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.00 Hz
 POINT 16384
 FREQ 6006.01 Hz
 SCANS 8
 ACQTM 2.7279 sec
 PD 4.2720 sec
 PW1 5.50 usec
 IRNUC 1H
 CTEMP 21.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.18 Hz
 RGAIN 16



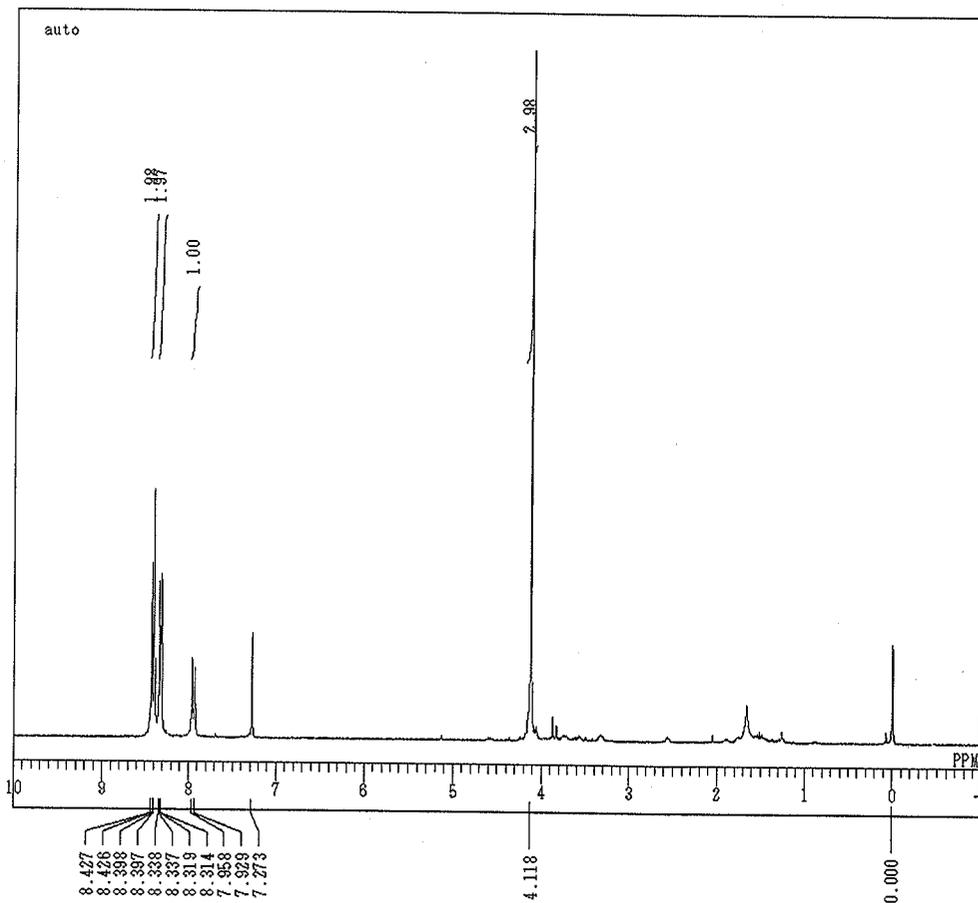
COMNT auto
 DATIM Tue Mar 23 18:29:31 2010
 OBNUC 13C
 EXMOD BCM
 OBFREQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.00 Hz
 POINT 32768
 FREQ 20356.23 Hz
 SCANS 208
 ACQTM 1.8097 sec
 PD 1.3900 sec
 PW1 4.00 usec
 IRNUC 1H
 CTEMP 23.0 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.62 Hz
 RGAIN 28



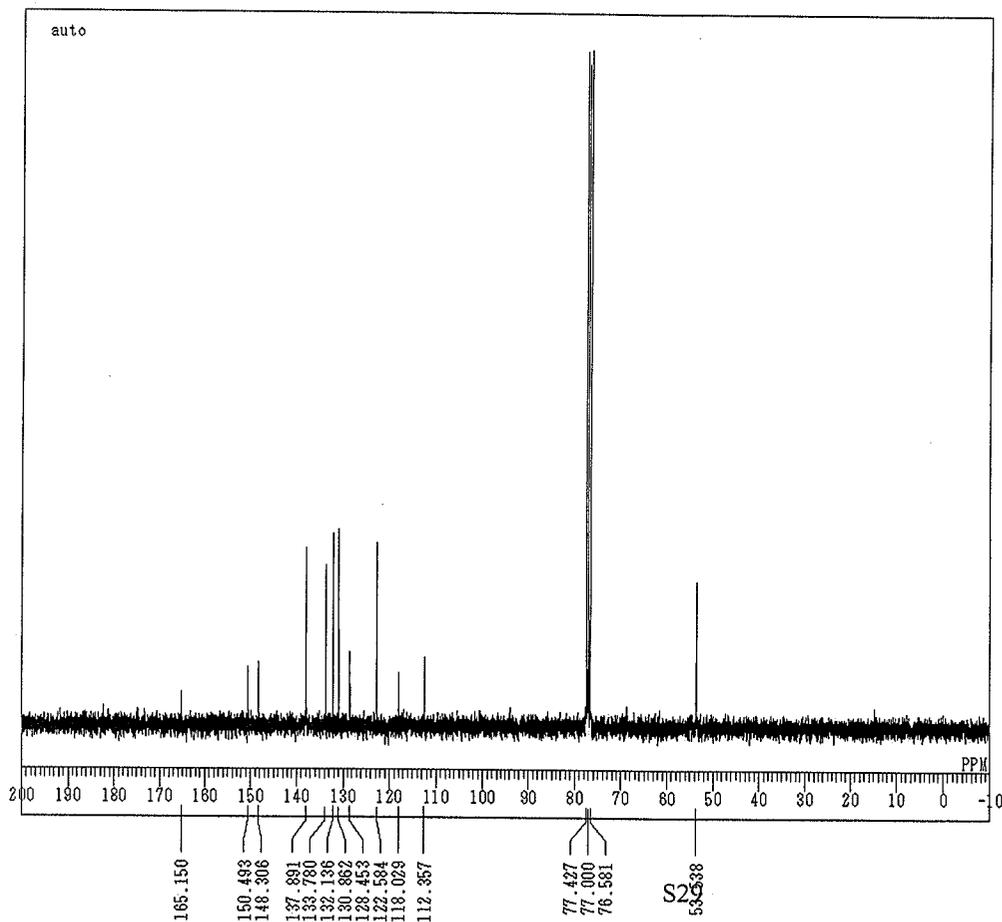
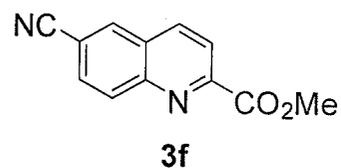
COMNT auto
 DATIM Tue Mar 23 18:44:19 2010
 OBNUC 1H
 EXMOD NON
 OBFRQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.00 Hz
 POINT 16384
 FREQU 6006.01 Hz
 SCANS 8
 ACQTM 2.7279 sec
 PD 4.2720 sec
 PW1 5.50 usec
 IRNUC 1H
 CTEMP 21.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.18 Hz
 RGAIN 15



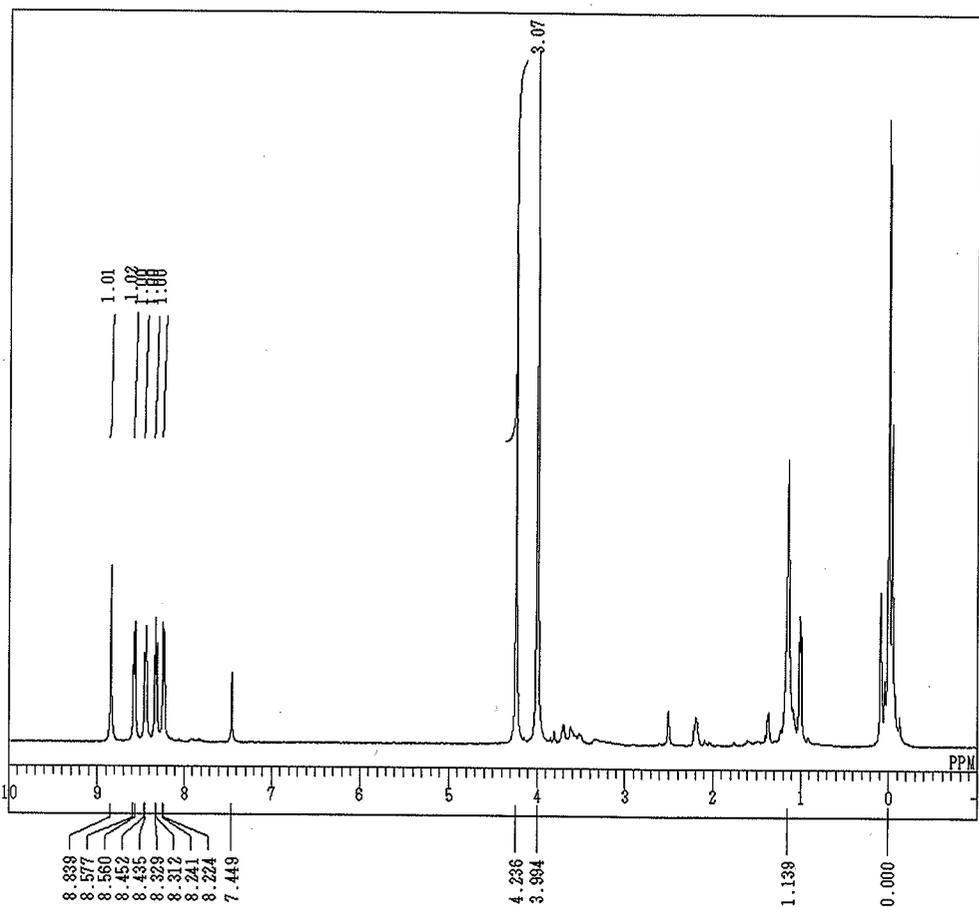
COMNT auto
 DATIM Tue Mar 23 18:55:20 2010
 OBNUC 13C
 EXMOD BCM
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.00 Hz
 POINT 32768
 FREQU 20356.23 Hz
 SCANS 209
 ACQTM 1.6097 sec
 PD 1.3900 sec
 PW1 4.00 usec
 IRNUC 1H
 CTEMP 22.1 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.62 Hz
 RGAIN 28



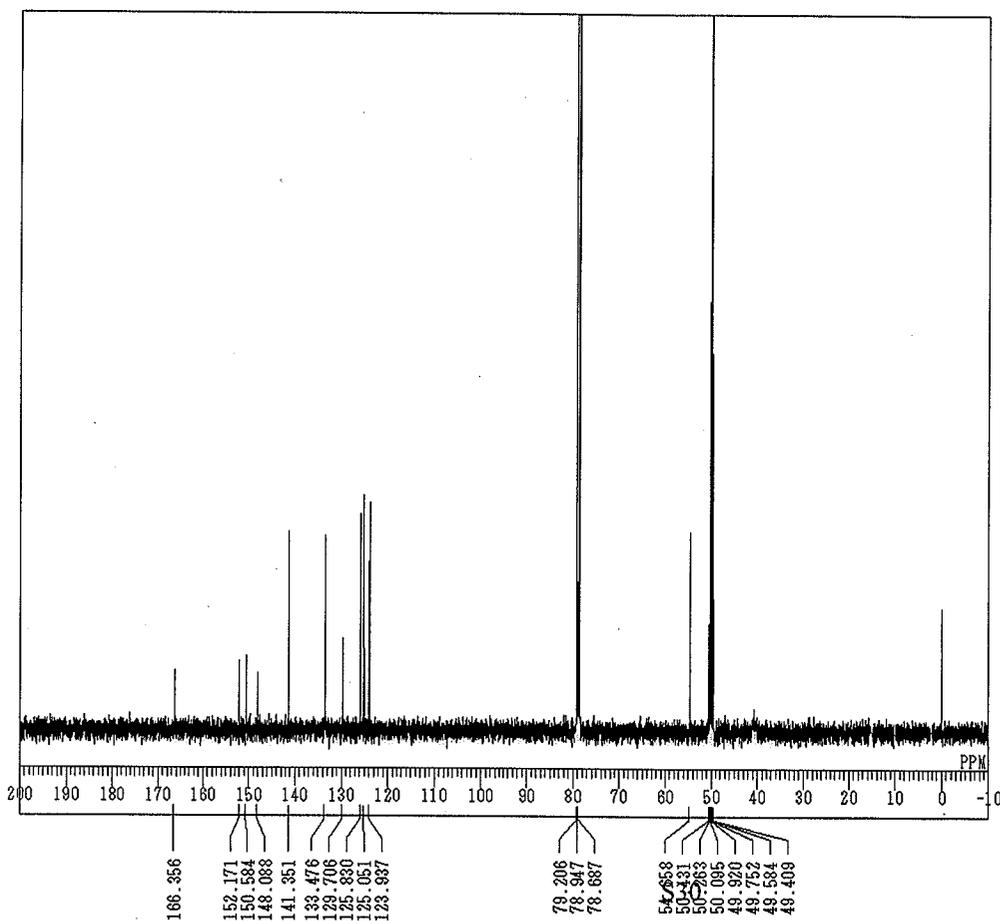
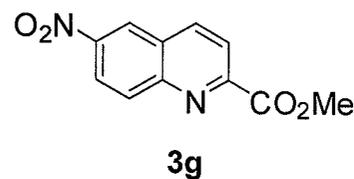
COMNT auto
 DATIM Thu Mar 18 15:36:28 2010
 OBNUC 1H
 EXMOD NON
 OBFRQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.00 Hz
 POINT 16384
 FREQU 6006.01 Hz
 SCANS 8
 ACQTM 2.7279 sec
 PD 4.2720 sec
 PW1 5.50 usec
 IRNUC 1H
 CTEMP 21.4 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.18 Hz
 RGAIN 20



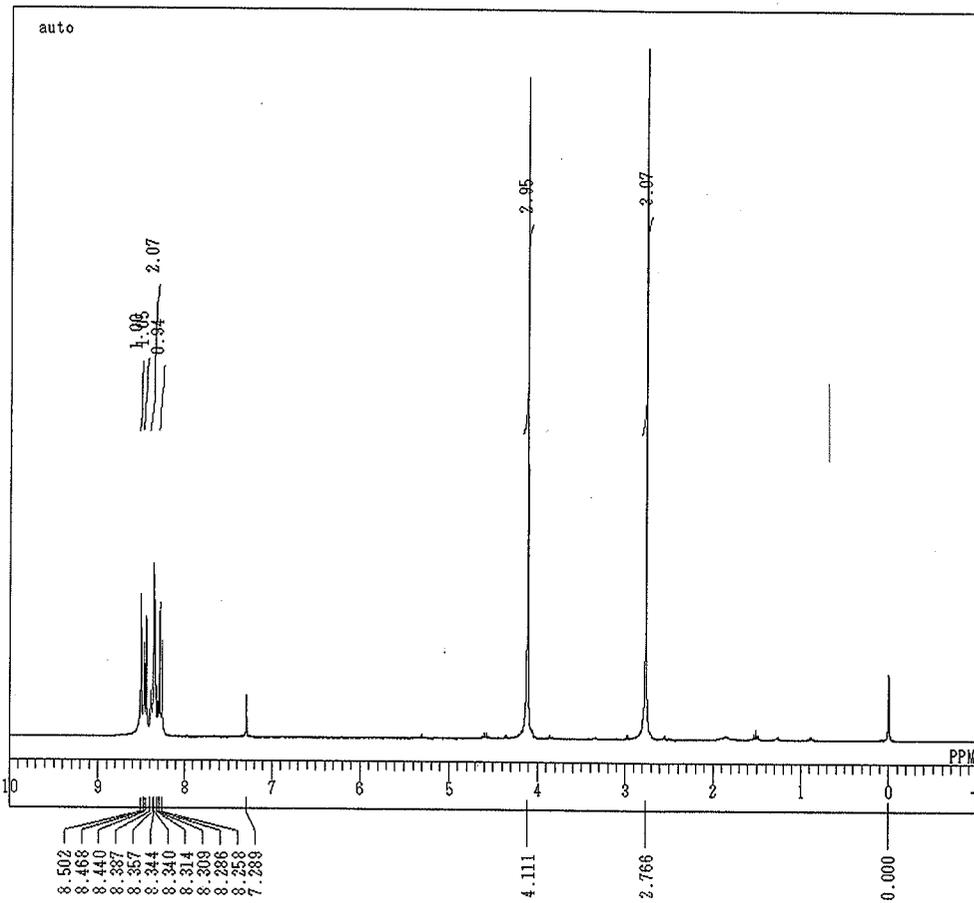
COMNT auto
 DATIM Thu Mar 18 15:57:23 2010
 OBNUC 13C
 EXMOD BCM
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.00 Hz
 POINT 32768
 FREQU 20356.23 Hz
 SCANS 406
 ACQTM 1.6097 sec
 PD 1.3900 sec
 PW1 4.00 usec
 IRNUC 1H
 CTEMP 21.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.62 Hz
 RGAIN 26



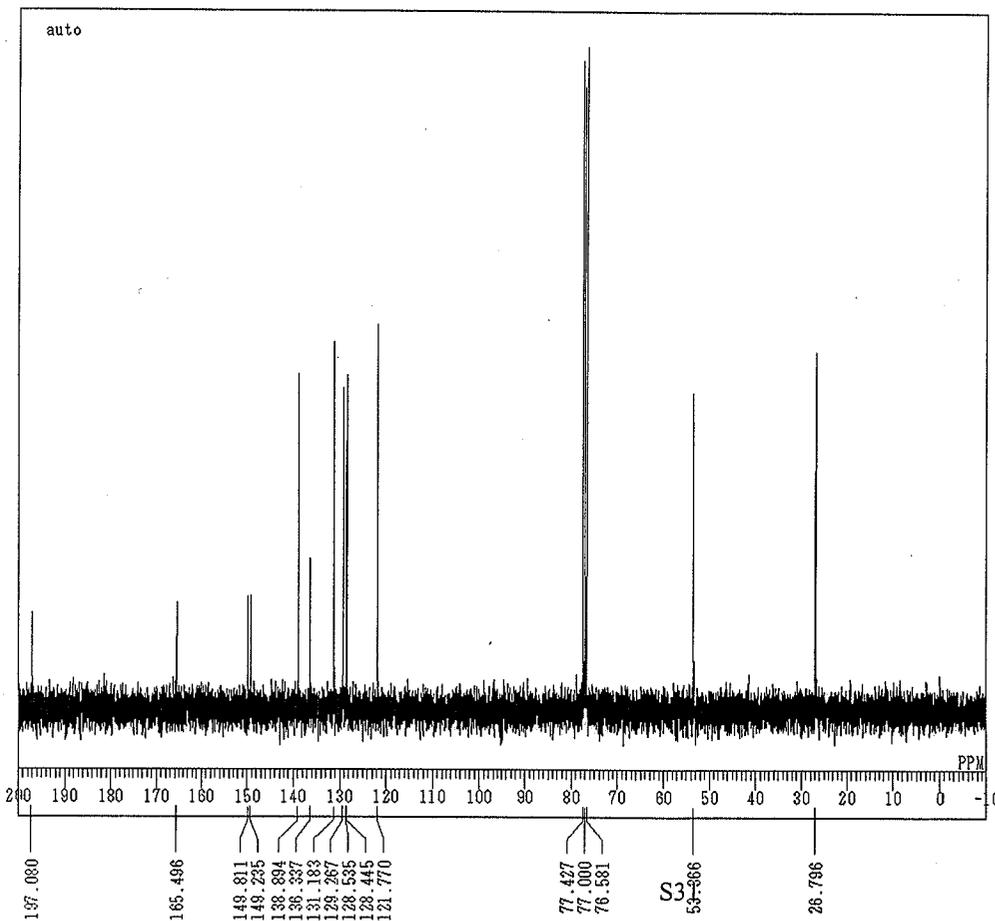
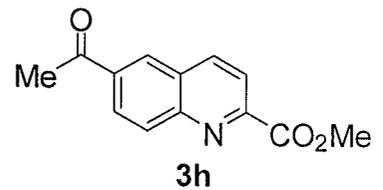
COMNT Single Pulse Experiment
 DATIM 15-01-2012 00:39:01
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFREQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 18384
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 2.1823 sec
 PD 4.0000 sec
 PW1 7.00 usec
 IRNUC
 CTEMP 19.0 c
 SLVNT CD3OD + CDCl₃ + d₆-DMSO
 EREF 0.00 ppm
 BF 0.23 Hz (ν_N = 2.5:2.0 = 0.5)
 RGAIN 19



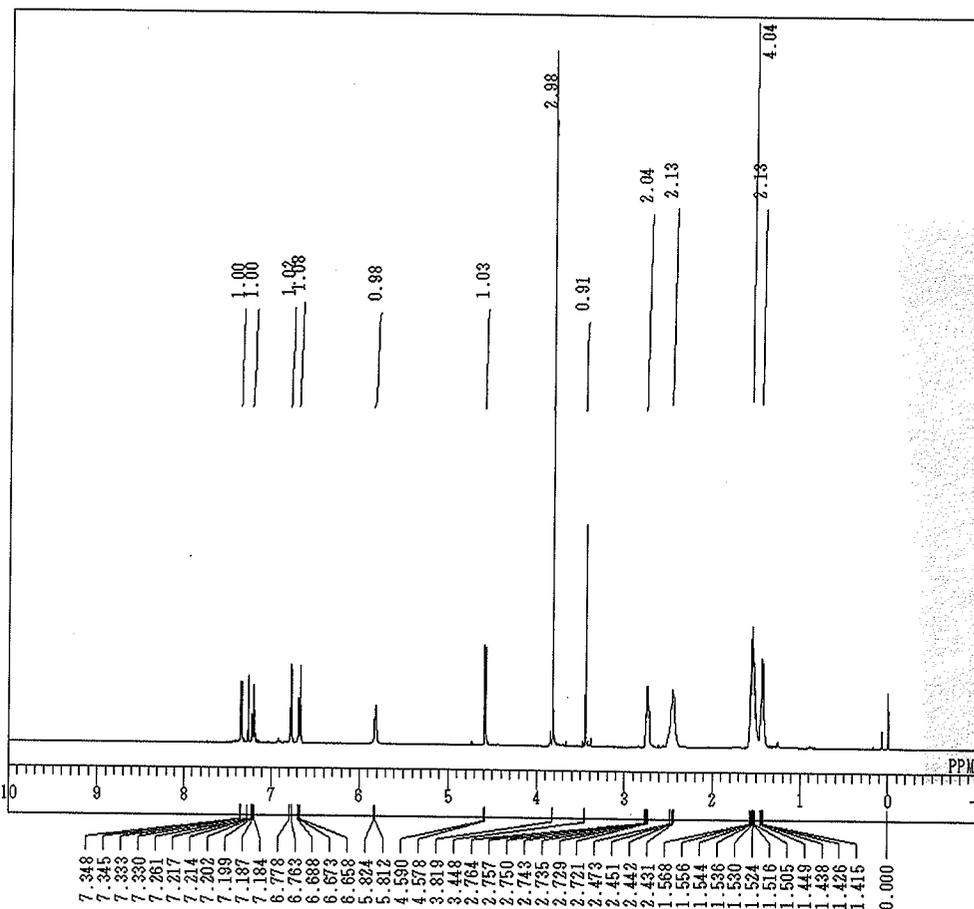
COMNT Single Pulse with Broadband D
 DATIM 15-01-2012 02:09:12
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFREQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 31446.54 Hz
 SCANS 1000
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 21.5 c
 SLVNT CD3OD + CDCl₃ + d₆-DMSO
 EREF 0.00 ppm
 BF 0.96 Hz
 RGAIN 30



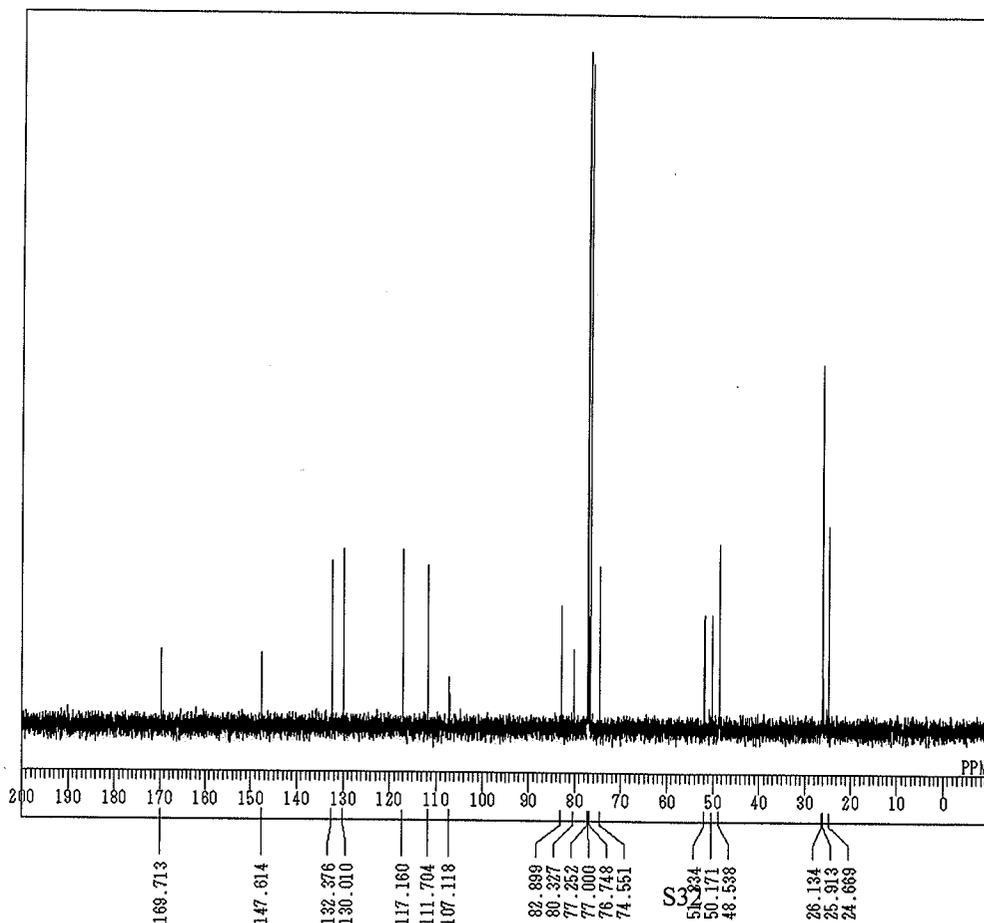
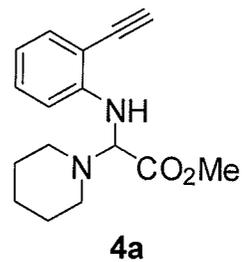
COMNT auto
 DATIM Tue Mar 23 19:10:18 2010
 OBNUC 1H
 EXMOD NON
 OBFRQ 300.40 MHz
 OBSET 130.00 KHz
 OBFIN 1150.00 Hz
 POINT 16384
 FREQU 6006.01 Hz
 SCANS 8
 ACQTM 2.7279 sec
 PD 4.2720 sec
 PW1 5.50 usec
 IRNUC 1H
 CTEMP 21.1 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.18 Hz
 RGAIN 17



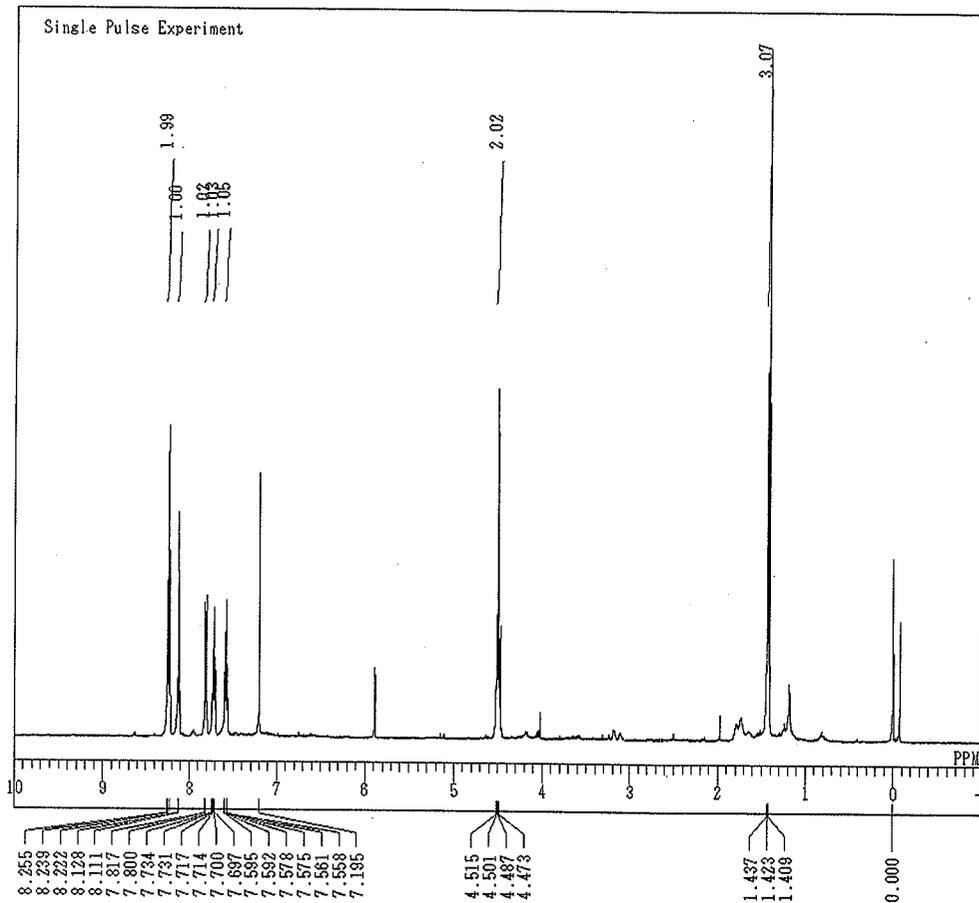
COMNT auto
 DATIM Tue Mar 23 19:17:02 2010
 OBNUC 13C
 EXMOD BCM
 OBFRQ 75.45 MHz
 OBSET 124.00 KHz
 OBFIN 1840.00 Hz
 POINT 32768
 FREQU 20356.23 Hz
 SCANS 123
 ACQTM 1.6097 sec
 PD 1.3900 sec
 PW1 4.00 usec
 IRNUC 1H
 CTEMP 21.9 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.62 Hz
 RGAIN 26



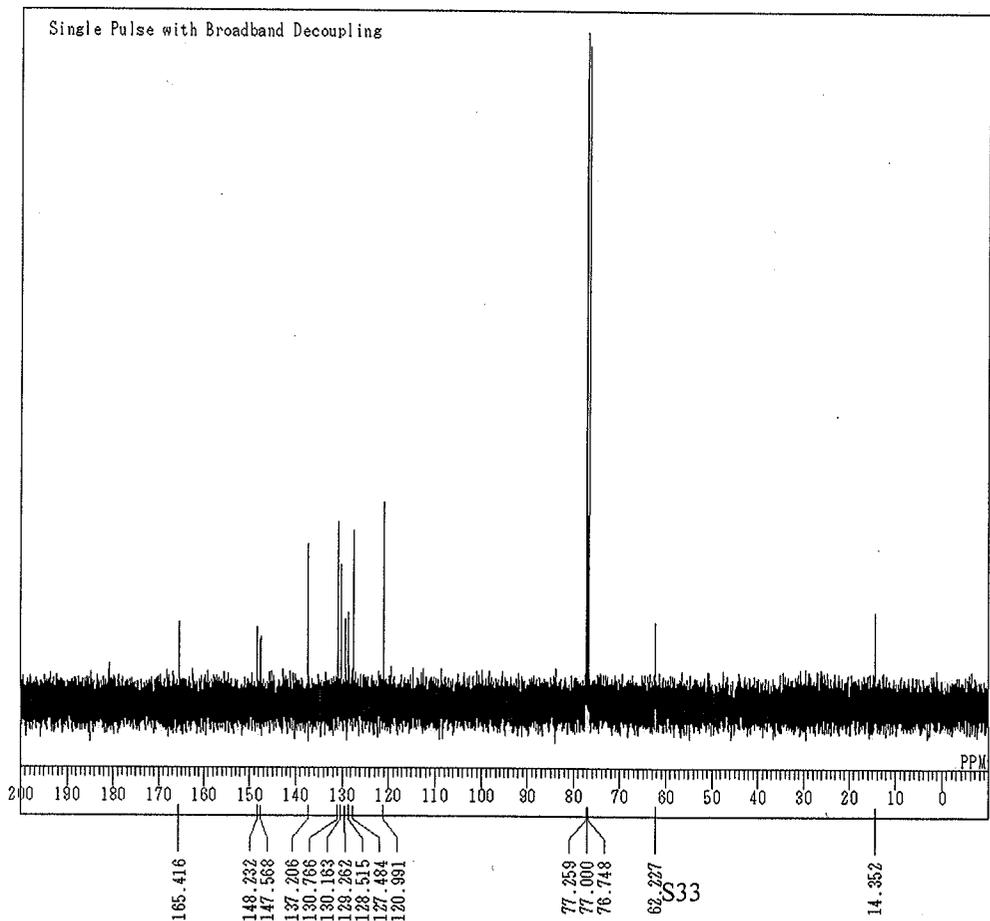
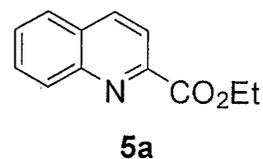
COMNT Single Pulse Experiment
 DATIM 12-12-2011 08:58:07
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFREQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 8.01 Hz
 POINT 16384
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 2.1823 sec
 PD 4.0000 sec
 PW1 7.00 usec
 IRNUC
 CTEMP 19.6 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 15



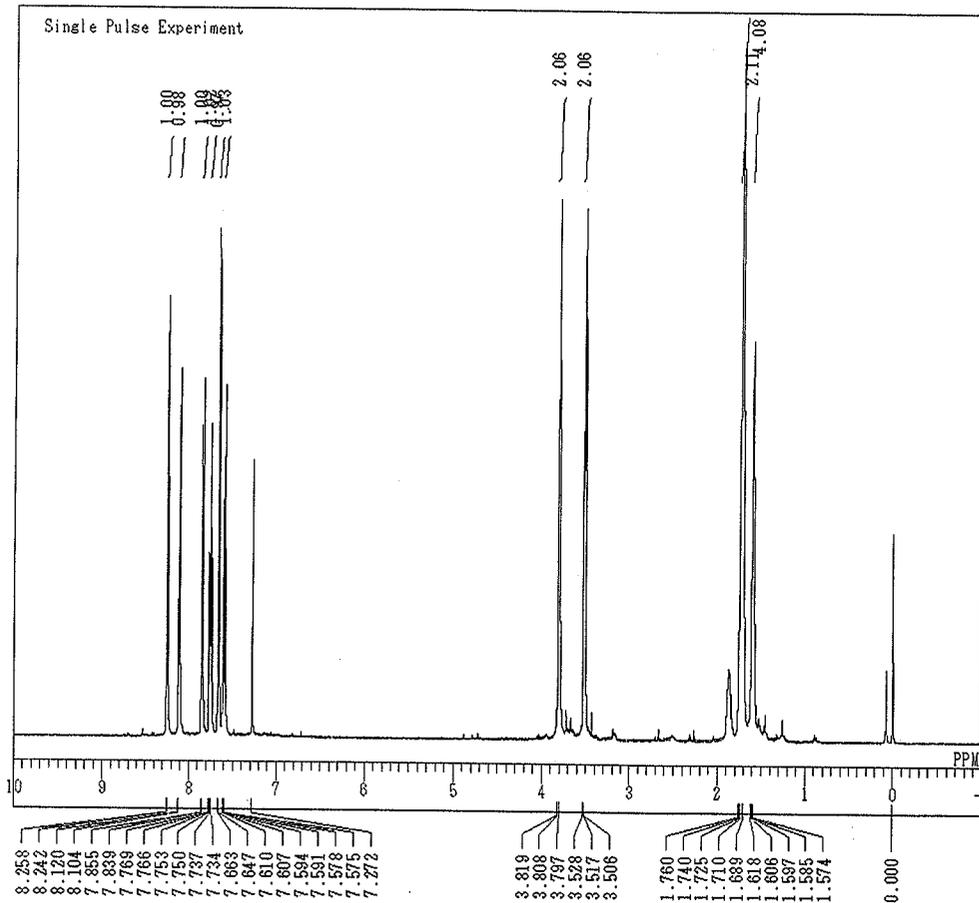
COMNT Single Pulse with Broadband D
 DATIM 12-12-2011 09:06:29
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFREQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 31446.54 Hz
 SCANS 203
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 21.4 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.96 Hz
 RGAIN 30



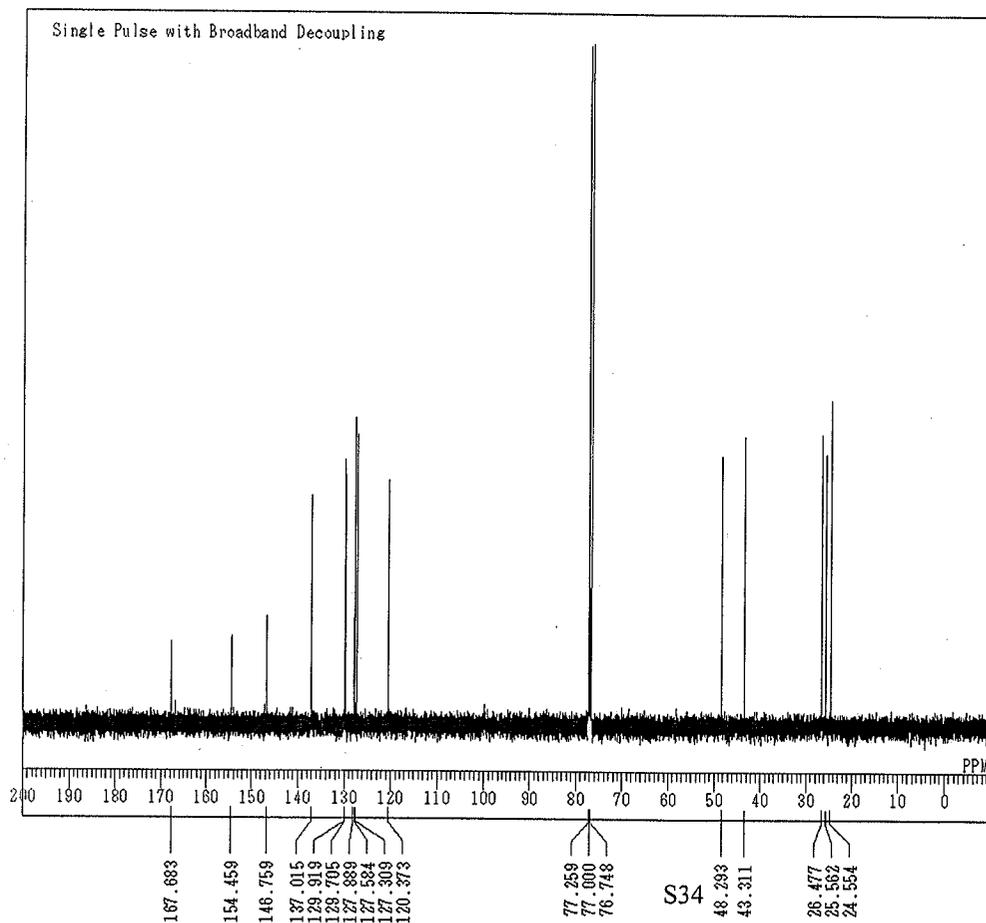
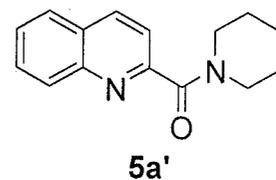
COMNT Single Pulse Experiment
 DATIM 27-05-2010 11:41:16
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16384
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 2.1823 sec
 PD 4.0000 sec
 PW1 7.00 usec
 IRNUC
 CTEMP 22.3 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 17



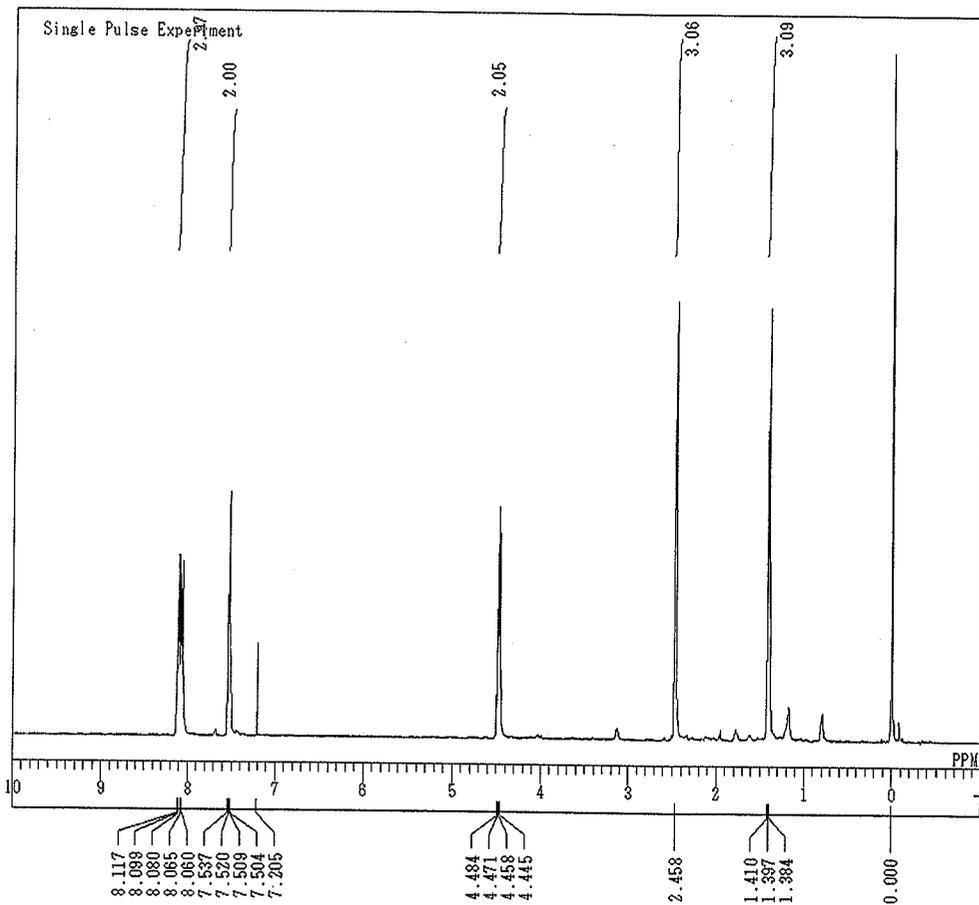
COMNT Single Pulse with Broadband D
 DATIM 27-05-2010 11:48:19
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 31446.54 Hz
 SCANS 103
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 23.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.96 Hz
 RGAIN 30



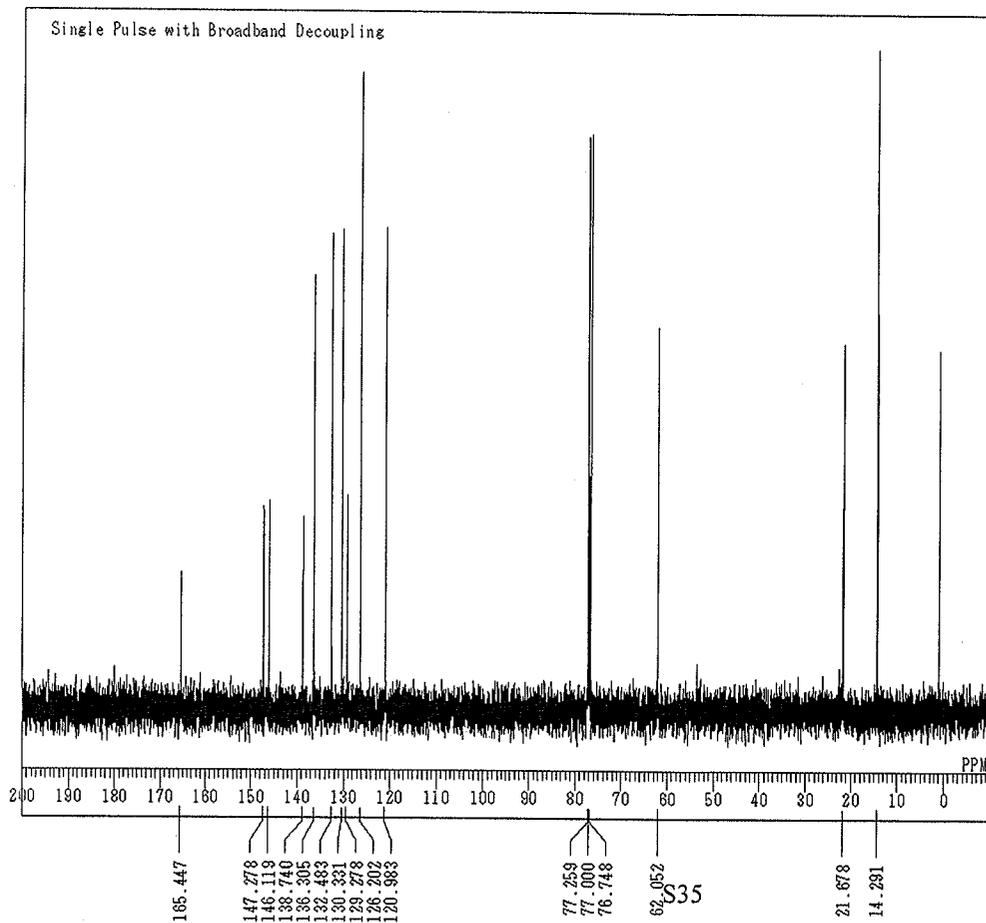
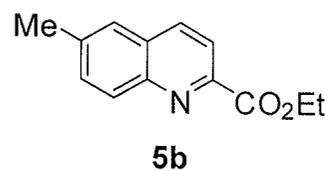
COMNT Single Pulse Experiment
 DATIM 16-07-2009 10:49:15
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16384
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 2.1823 sec
 PD 4.0000 sec
 PW1 7.00 usec
 IRNUC
 CTEMP 23.3 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 15



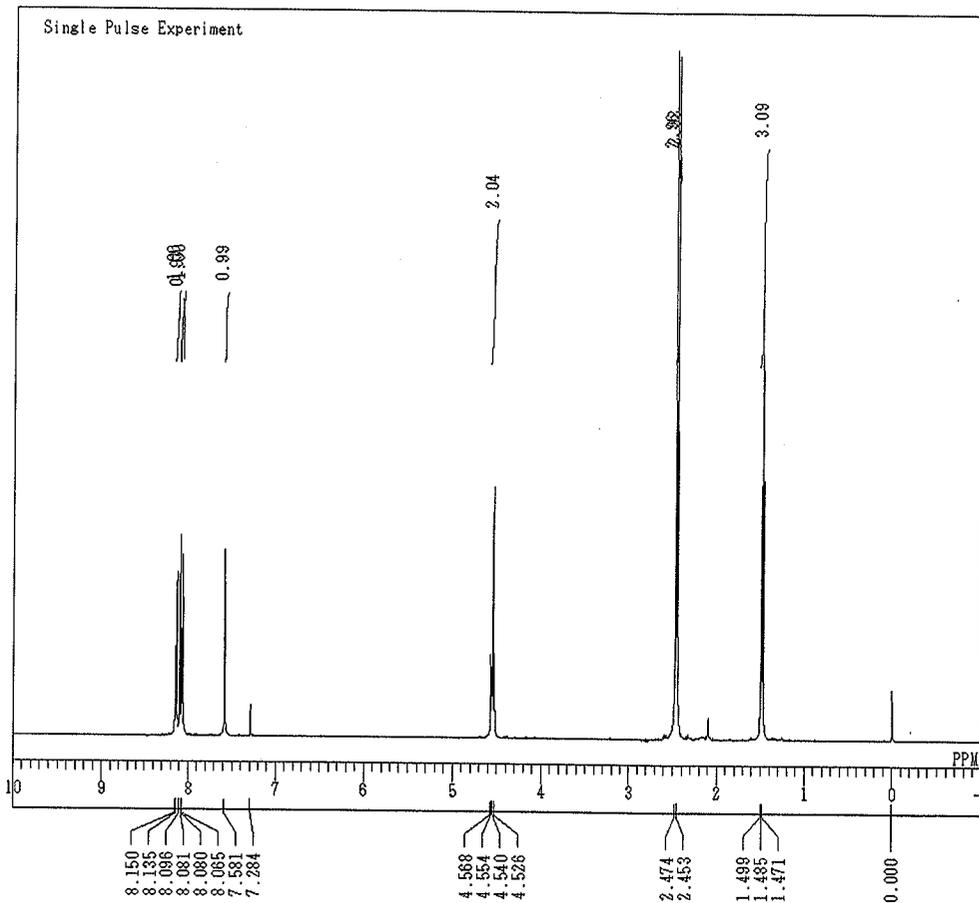
COMNT Single Pulse with Broadband D
 DATIM 16-07-2009 11:00:42
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 31446.54 Hz
 SCANS 311
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 26.4 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.96 Hz
 RGAIN 30



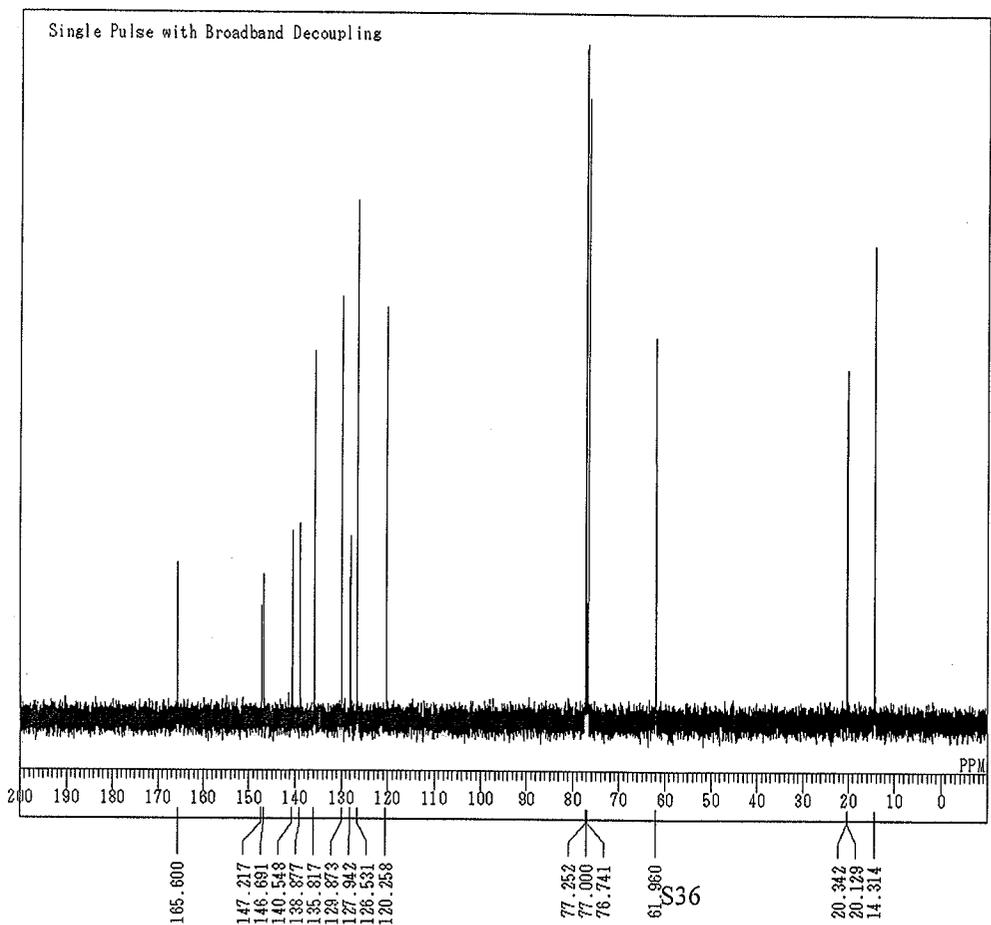
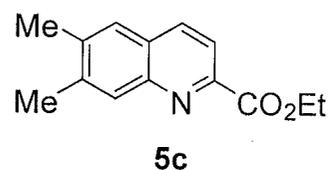
COMNT Single Pulse Experiment
 DATIM 12-12-2011 07:36:27
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFREQ 500.16 MHz
 OBSSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16384
 FREQ 7507.51 Hz
 SCANS 8
 ACQTM 2.1823 sec
 PD 4.0000 sec
 PW1 7.00 usec
 IRNUC
 CTEMP 19.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 13



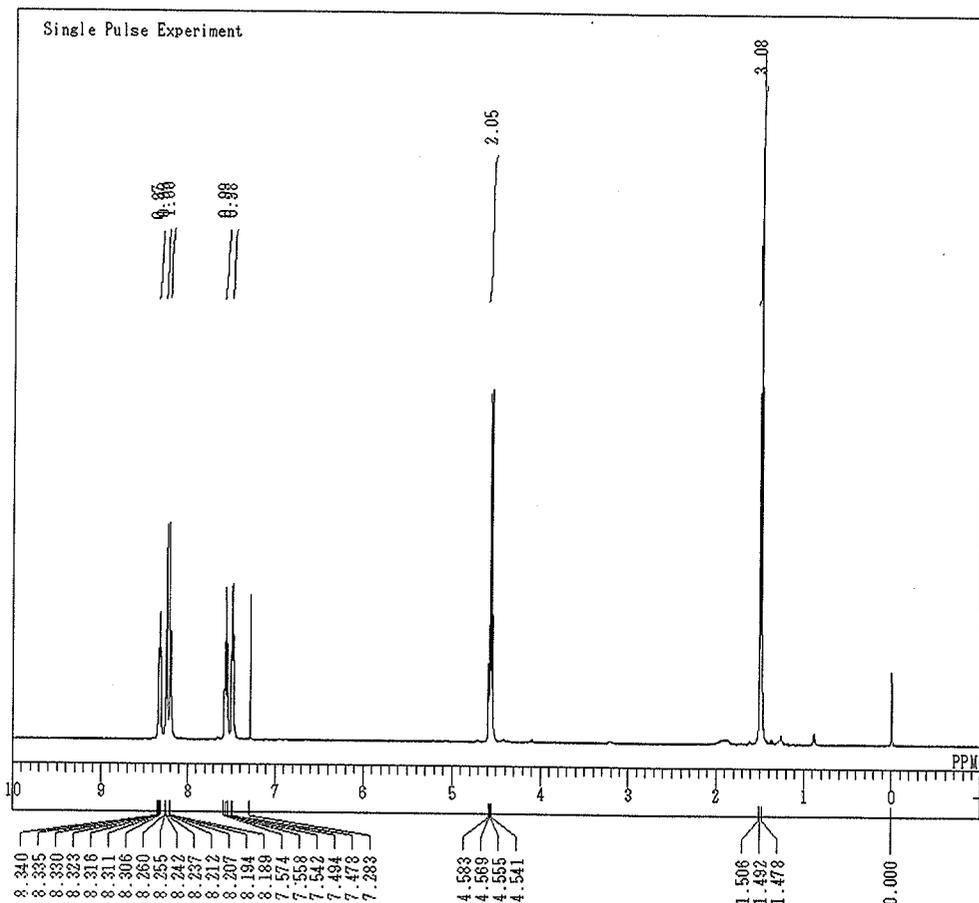
COMNT Single Pulse with Broadband D
 DATIM 12-12-2011 07:42:52
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFREQ 125.77 MHz
 OBSSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQ 31446.54 Hz
 SCANS 102
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 21.2 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.96 Hz
 RGAIN 30



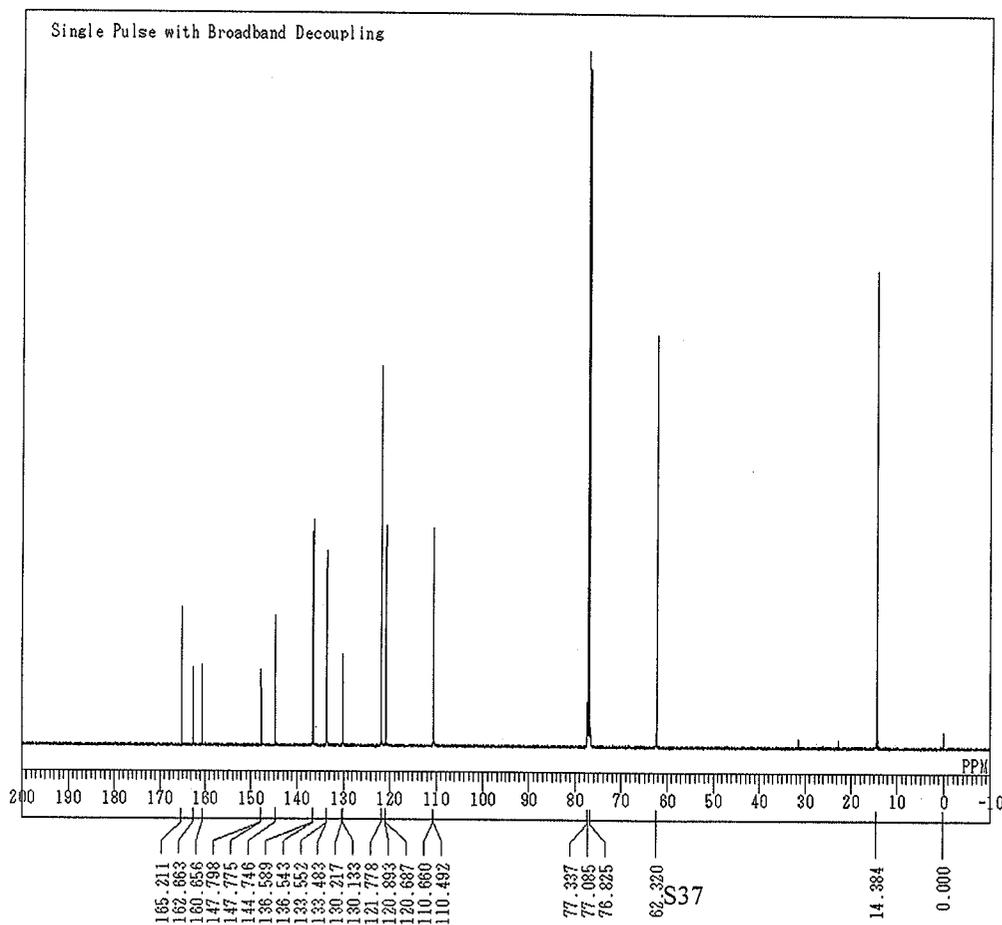
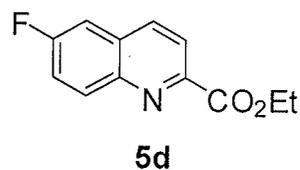
COMNT Single Pulse Experiment
 DATIM 09-08-2011 04:49:56
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFREQ 500.16 MHz
 OBSSET 2.41 KHz
 OBFIN 8.01 Hz
 POINT 16384
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 2.1823 sec
 PD 4.0000 sec
 PW1 7.00 usec
 IRNUC
 CTEMP 22.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 13



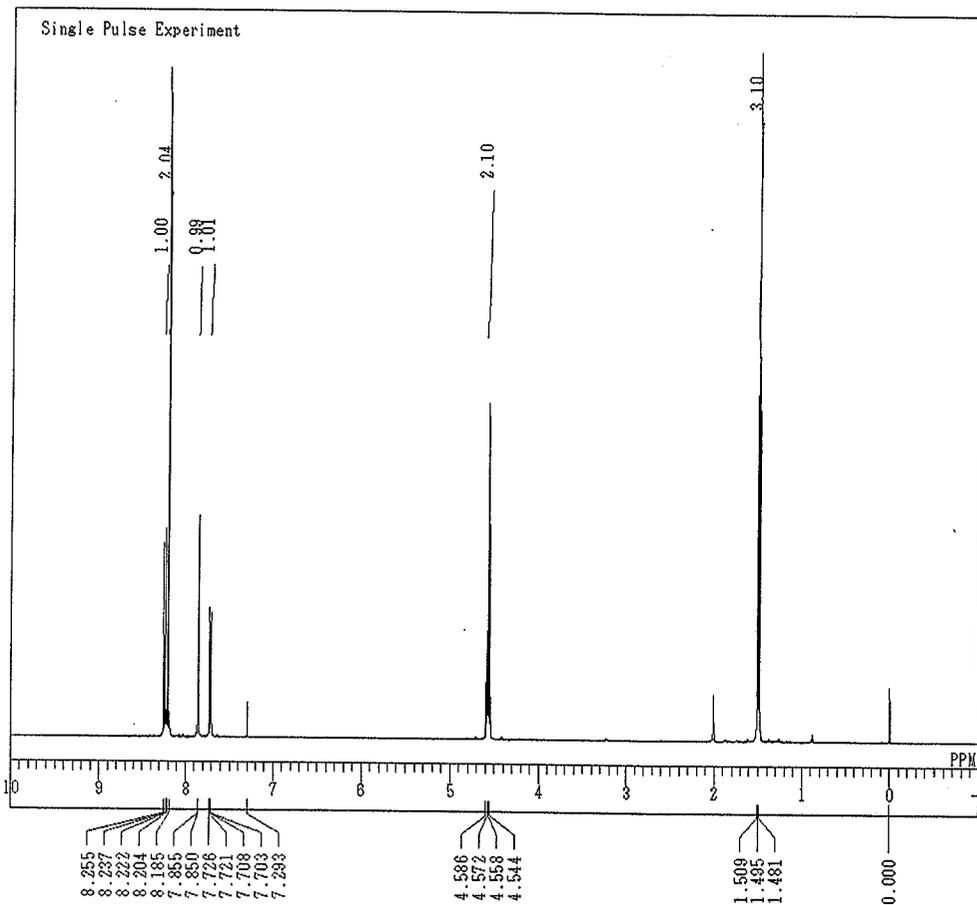
COMNT Single Pulse with Broadband D
 DATIM 09-08-2011 04:58:31
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFREQ 125.77 MHz
 OBSSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 31446.54 Hz
 SCANS 136
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 24.1 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.96 Hz
 RGAIN 30



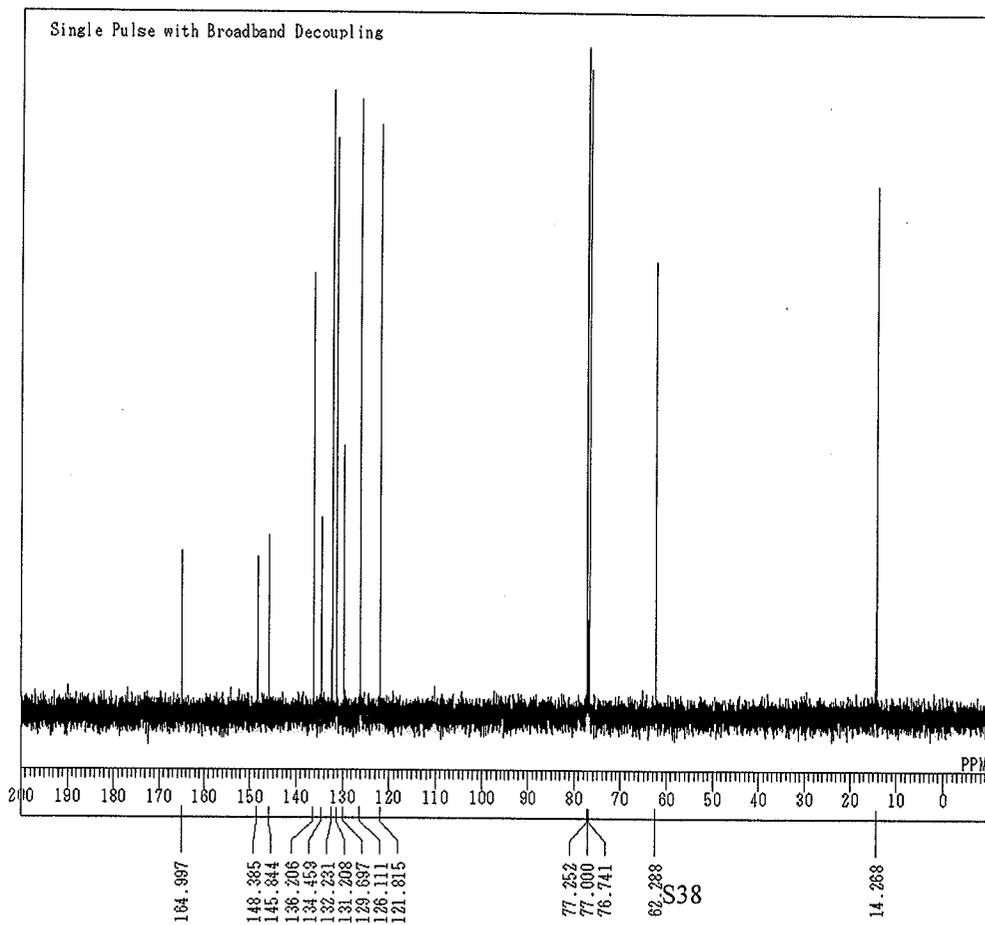
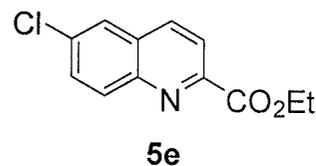
COMNT Single Pulse Experiment
 DATIM 16-11-2011 08:20:05
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFREQ 500.16 MHz
 OBSSET 2.41 KHz
 OBFIN 8.01 Hz
 POINT 16384
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 2.1823 sec
 PD 4.0000 sec
 PW1 7.00 usec
 IRNUC
 CTEMP 24.3 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 15



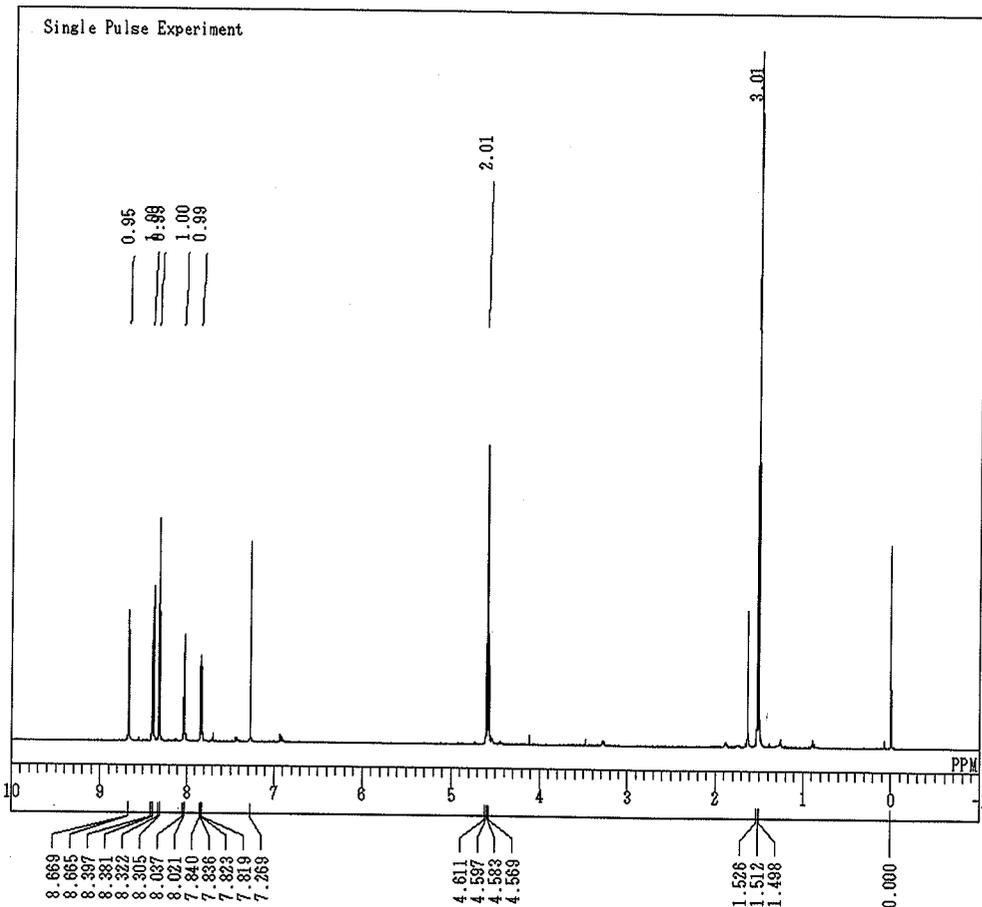
COMNT Single Pulse with Broadband D
 DATIM 16-11-2011 18:01:15
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFREQ 125.77 MHz
 OBSSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 31446.54 Hz
 SCANS 16971
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 26.2 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.96 Hz
 RGAIN 30



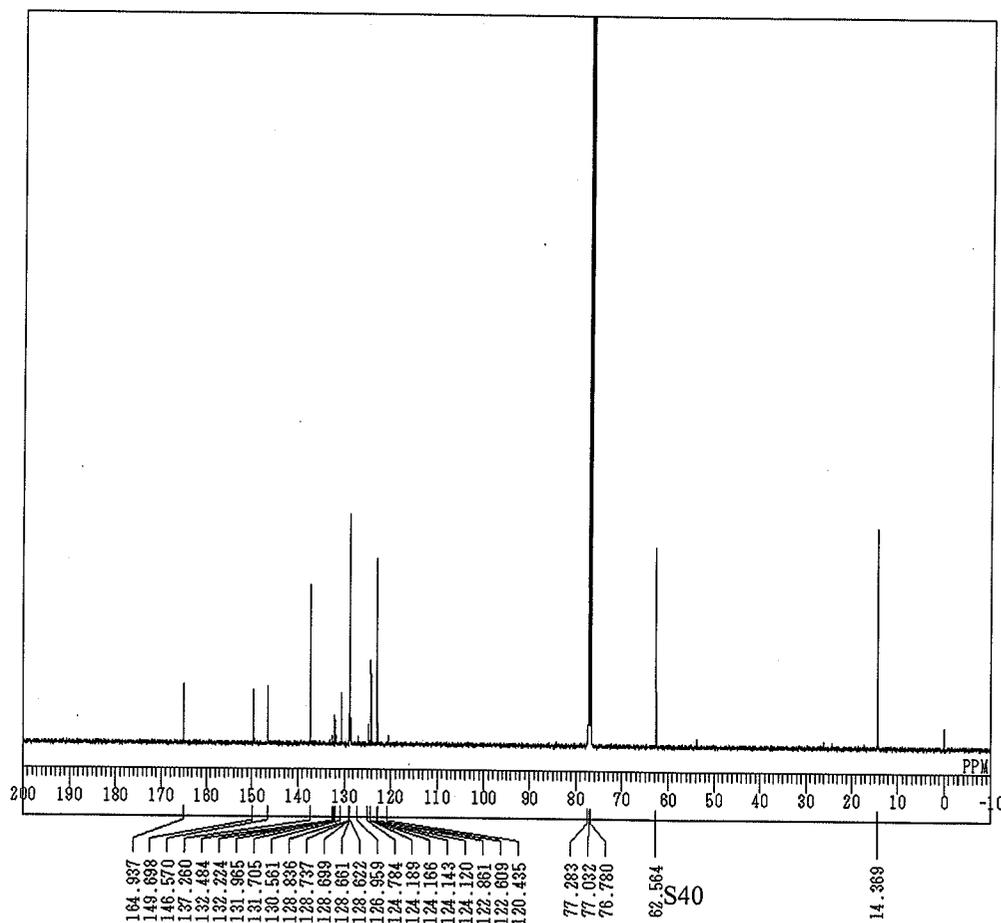
COMNT Single Pulse Experiment
 DATIM 09-08-2011 05:04:04
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFREQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 18384
 FREQ 7507.51 Hz
 SCANS 8
 ACQTM 2.1823 sec
 PD 4.0000 sec
 PW1 7.00 usec
 IRNUC
 CTEMP 22.9 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 14



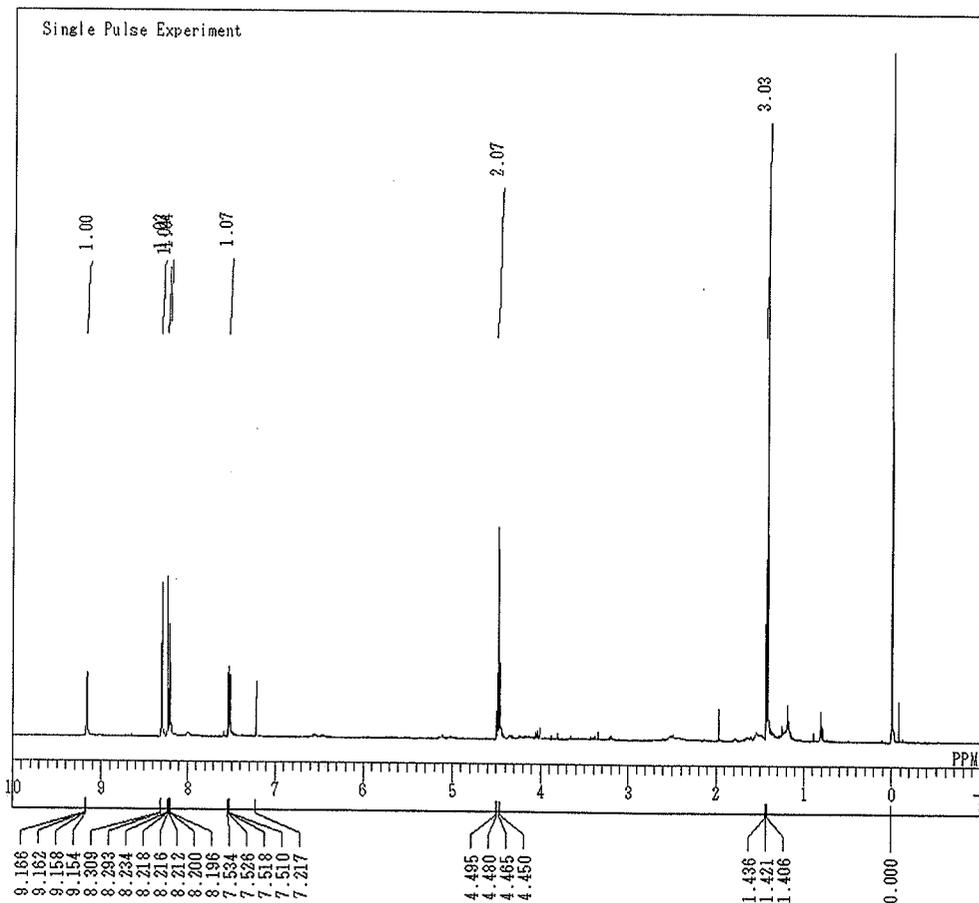
COMNT Single Pulse with Broadband D
 DATIM 09-08-2011 05:09:52
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFREQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQ 31446.54 Hz
 SCANS 71
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 24.2 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.96 Hz
 RGAIN 30



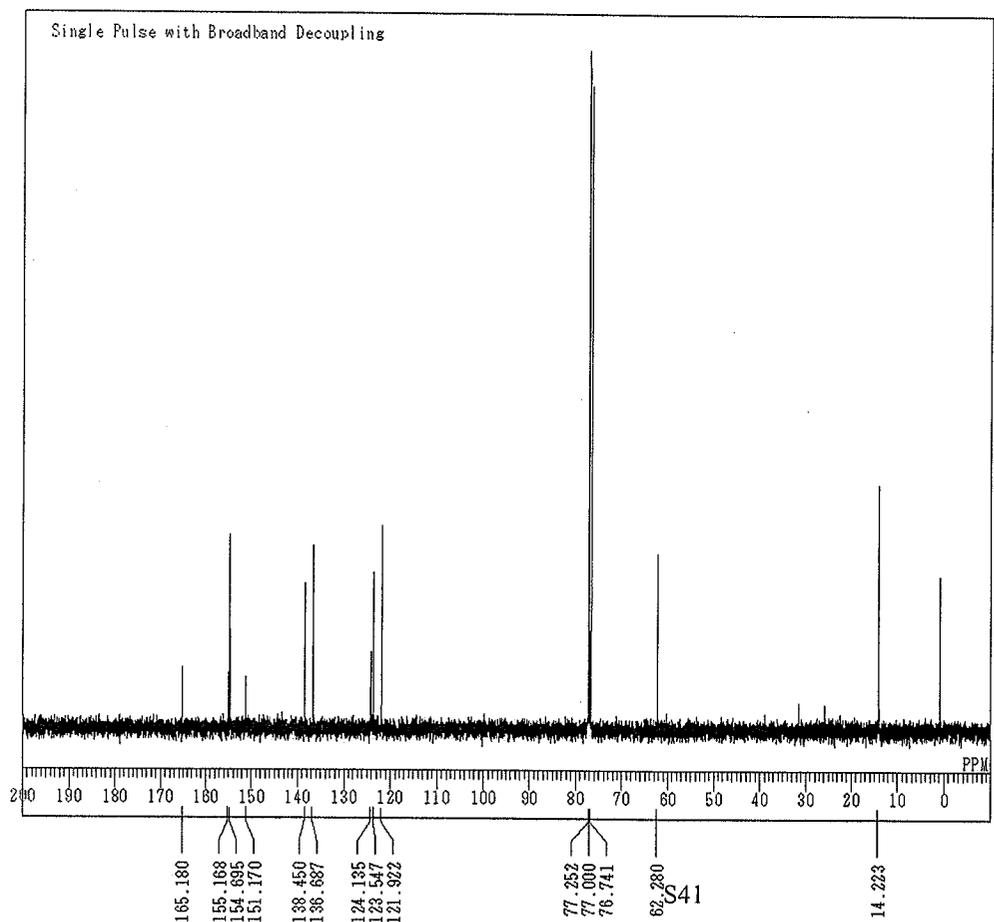
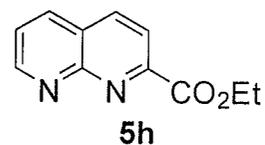
COMNT Single Pulse Experiment
 DATIM 13-12-2011 08:29:09
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16384
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 2.1823 sec
 PD 4.0000 sec
 PW1 7.00 usec
 IRNUC
 CTEMP 19.5 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 19



COMNT Single Pulse with Broadband D
 DATIM 13-12-2011 18:51:58
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 31446.54 Hz
 SCANS 18219
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 22.0 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.96 Hz
 RGAIN 30



COMNT Single Pulse Experiment
 DATIM 12-12-2011 08:41:32
 OBNUC 1H
 EXMOD single_pulse.exp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16384
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 2.1823 sec
 PD 4.0000 sec
 PW1 7.00 usec
 IRNUC
 CTEMP 19.1 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.23 Hz
 RGAIN 15



COMNT Single Pulse with Broadband Decoupling
 DATIM 12-12-2011 08:51:16
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 31446.54 Hz
 SCANS 203
 ACQTM 1.0420 sec
 PD 1.0000 sec
 PW1 4.17 usec
 IRNUC 1H
 CTEMP 21.4 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.96 Hz
 RGAIN 30