Supporting Information for

Allenylazide Cycloaddition Chemistry. Synthesis of Annelated Indoles from 2-(Allenyl)phenylazide Substrates.

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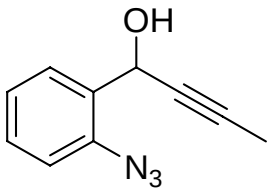
General Experimental

Moisture and oxygen sensitive reactions were carried out in flame-dried glassware under a nitrogen atmosphere. Solvents were dried by passage through an activated alumina column under nitrogen. All organic reagents were used as purchased. Flash chromatography was performed using 32 – 63 μm silica gel with the indicated solvent systems. Melting points are uncorrected. Low and high resolution mass spectra were obtained according to the specified technique and were performed at the Proteomics and Mass Spectrometry Core Facility at the Pennsylvania State University. Copies of ^{1}H and ^{13}C NMR spectra are supplied in the Supporting Information as criteria of purity.

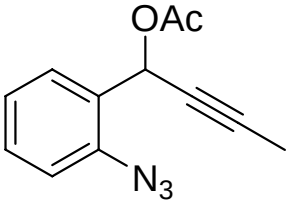
1-(2-Azidophenyl)but-2-yn-1-ol.



To a solution of 2-azidobenzaldehyde (**8**) (2.0 g, 14 mmol) in 20 mL of THF at -20 °C was added 1-propynylmagnesium bromide (0.50 M, 30 mL, 15 mmol) and the mixture was stirred for 2 h. The reaction mixture was warmed to room temperature over an additional hour and poured into 20 mL of saturated NH_4Cl solution. The organic layer was extracted with 3x30 mL of Et_2O and the combined organic extracts were washed with water and brine, dried over Na_2SO_4 and the solvent was evaporated to yield a brown oil. The crude material was carried over to the next step. A small batch was purified by column chromatography (40% Et_2O in hexanes) to give a white solid: mp 49 - 50 °C; IR (neat): 3400, 2125 cm^{-1}; ^{1}H NMR (300 MHz, C_6D_6) δ 7.69 (m, 1H), 6.86 (m, 2H), 6.61 (m, 1H), 5.67 (d, J = 2.1 Hz, 1H), 3.07 (br s, 1H), 1.47 (d, J = 2.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.0, 131.9, 129.4, 128.1, 124.9, 118.0, 82.9, 78.2, 60.3, 3.6; TOFESMS *m/z* relative intensity 210.1 (MNa^+ 100%); HRMS (+ES) Calcd for $C_{10}H_9N_3ONa$: 210.0643, Found: 210.0639.

Representative Procedure 1. Azidophenyl Alkynyl Acetate Synthesis.

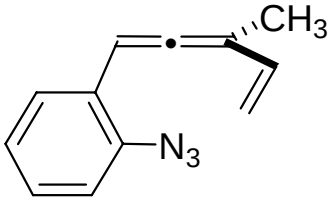
1-Acetoxy-1(2-azidophenyl)but-2-yne (9a).



Acetic anhydride (1.2 mL, 13 mmol) and DMAP (1.6 g, 13 mmol) were added to an ice-cold solution of 1-(2-azidophenyl)but-2-yn-1-ol (2.0 g, 11 mmol) in 40 mL of CH_2Cl_2, and the mixture was stirred for 24 h with warming to room temperature. The reaction mixture was poured into 30 mL of saturated $NaHCO_3$ solution. The mixture was then extracted with 3x30 mL of CH_2Cl_2 and the combined organic layers were washed with water and brine, dried over Na_2SO_4 and concentrated *in vacuo*. The crude material was purified by column chromatography (25% Et_2O in hexanes) to give the acetate **9a** as a pale yellow solid (1.5 g, 61%): mp 49 – 50 °C; IR (neat): 2140, 1736 cm^{-1}; ^1H NMR (360 MHz, C_6D_6) δ 7.75 (dd, J = 7.5, 1.8 Hz, 1H), 6.91 (m, 3H), 6.64 (dd, J = 7.6, 1.4 Hz, 1H), 1.66 (s, 3H), 1.45 (d, J = 2.3 Hz, 3H); ^{13}C NMR (90 MHz, C_6D_6) δ 168.9, 138.1, 130.2, 129.5, 129.3, 124.9, 118.4, 83.6, 76.4, 61.3, 20.4, 3.3; TOFESMS *m/z* (relative intensity) 252.0 (MNa^+, 100%), 284.0 ($MMeOH^+$,70%); HRMS Calcd for $C_{12}H_{11}N_3O_2Na$: 252.0749, Found: 252.0762.

Representative Procedure 2. Allenylazide Synthesis.

1-(2-Azidophenyl)3-methylpent-1,2,4-triene (6a).

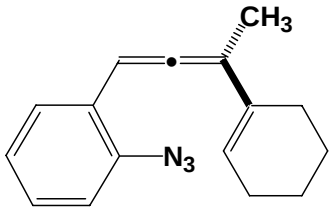


To a solution of $ZnCl_2$ (0.34 g, 2.5 mmol) in 10 mL of THF was added vinylMgBr (1.0 M in THF, 2.5 mL, 2.5 mmol) and the mixture was stirred at room temperature for 1 h. $Pd(PPh_3)_4$ (58 mg, 5.0 mol%) in 2mL of THF and the propargylic acetate **9a** (0.23 g, 1.0 mmol) in 5 mL of THF were added sequentially. The reaction mixture was stirred at

room temperature for 20 min. After addition of 10 mL of saturated NH_4Cl solution, the reaction mixture was extracted with 2x20 mL of Et_2O and the combined organic layers were washed with water and brine. Drying over Na_2SO_4 and removal of solvent under reduced pressure resulted in a brown oil. The crude product was purified by flash chromatography using pure hexanes as the eluent to give **6a** as a pale yellow oil (114 mg, 58%). IR (neat): 2120, 1930 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 7.4 Hz, 1H), 7.27 (d, *J* = 7.4, 1H), 7.16 (d, *J* = 7.3 Hz, 1H), 7.10 (t, *J* = 7.3 Hz, 1H), 6.54 (m, 1H), 6.42 (dd, *J* = 17.4, 10.5 Hz, 1H), 5.25 (d, *J* = 17.4 Hz, 1H), 5.15 (d, *J* = 10.5 Hz, 1H), 1.97 (d, *J* = 2.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 210.3, 136.8, 135.0, 128.9, 128.4, 126.3, 125.3, 118.9, 114.0, 104.6, 89.1, 14.9; TOFESMS *m/z* relative intensity 170 (MH^+-N_2 100); HRMS (+ES) Calcd for $C_{12}H_{12}N$: 170.0970, Found: 170.0977.

Representative Procedure 3. Allenylazide Synthesis.

1-(2-Azidophenyl)3-cyclohex-1-enylbut-1,2-diene (6h).



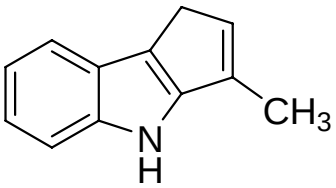
To a solution of CuBr•Me_2S (2.5 g, 12 mmol) in 20 mL of THF at -40 °C was added MeMgBr (3.0 M in THF, 4.0 mL, 12 mmol) and the mixture was stirred for 1 h, after which acetate **9h** (0.30 g, 1.3 mmol) in 5 mL of THF was cannulated into the reaction mixture at -40 °C. The reaction mixture was warmed to room temperature over a period of 8 h to allow complete consumption of starting material. The excess cuprate was then quenched with drop-wise addition of saturated NH_4Cl solution. The organic layer was extracted with 3x50 mL of Et_2O and washed with water and brine. Drying the combined extracts over Na_2SO_4 and removal of solvent under reduced pressure resulted in a brown oil. The crude product was purified by column chromatography using pure hexanes as the eluent to give **6h** as a pale yellow oil (120 mg, 37%). IR (neat): 2123, 1928 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, *J* = 7.5 Hz, 1H), 7.24 (d, *J* = 7.3 Hz, 1H), 7.16 (d, *J* = 7.0, 1H), 7.09 (t, *J* = 7.5 Hz, 1H), 6.57 (m, 1H), 5.81 (m, 1H), 2.2 (m, 3H), 2.07 (m, 1H), 1.97 (d, *J* = 2.7 Hz, 3H), 1.63-1.68 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 207.6, 136.5,

133.4, 128.4, 128.2, 127.2, 125.2, 123.9, 118.8, 107.1, 90.5, 27.4, 26.4, 23.2, 22.7, 16.3 ; TOFESMS *m/z* relative intensity 224 (MH^+-N_2 100); HRMS (+ES) Calcd for $C_{16}H_{18}N$: 224.1439, Found: 224.1459

Representative Procedure 4. Cyclization.

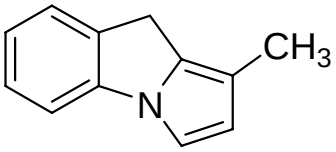
A deoxygenated solution of allenylazide **6a** (35 mg, 0.18 mmol) in toluene-d_8, (1.8 mL) was heated at 110 ^oC in a clean, sealed tube for 15 min, after which the reaction mixture was cooled to room temperature. Evaporation of the solvent *in vacuo* gave a brown oil. The ratio of the products was determined at this stage by ^1H NMR analysis. Purification of this crude oil using an alumina column resulted in two products; **7a** (80% Et_2O in hexanes, 12 mg, 40%) and **11a** (10% Et_2O in hexanes 17 mg, 56%).

3-Methyl-1,4-dihydrocyclopenta[b]indole (7a).



IR (neat): 3406 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.03 (bs, 1H), 7.58 (d, *J* = 7.43 Hz, 1H), 7.41 (d, *J* = 7.8 Hz, 1H), 7.09-7.24 (m, 2H), 6.22 (m, 1H), 3.25 (s, 2H), 2.22 (d, *J* = 1.6 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.7, 140.5, 132.7, 131.6, 125.2, 121.0, 120.6, 120.3, 118.5, 112.3, 31.9, 13.5; TOFESMS *m/z* relative intensity 170 (MH^+ 35); HRMS (+ES) Calcd for $C_{12}H_{12}N$:170.0970, Found: 170.0967.

1-Methyl-9H-pyrrolo[1,2-a]indole (11a).

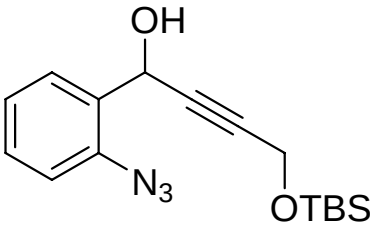


Crystals suitable for X-ray crystallographic analysis were obtained by slow evaporation of an Et_2O solution of **11a** over a period of 48 h at 25 °C. ^1H NMR (300 MHz, $CDCl_3$) δ 7.42 (d, *J* = 7.4 Hz, 1H), 7.24-7.33 (m, 2H), 7.10 (t, *J* = 7.3 Hz, 1H), 7.05 (d, *J* = 2.6 Hz, 1H), 6.25 (d, *J* = 2.6 Hz, 1H), 3.77 (s, 2H), 2.18 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.8, 135.3, 132.7, 127.7, 126.3, 123.0, 114.8, 111.9, 109.8, 109.6, 28.4, 11.7;

TOFESMS *m/z* relative intensity 170 (MH^+ 35); HRMS (+ES) Calcd for $C_{12}H_{12}N$:170.0970, Found: 170.0967.

Representative Procedure 5. Azidophenyl Alkynyl Alcohol Synthesis.

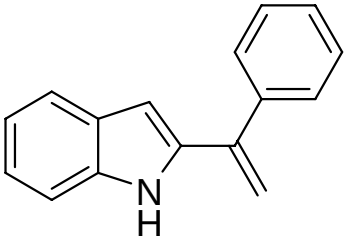
1-*t*-Butyldimethylsilyloxy-4-(2-azidophenyl)but-2-yn-4-ol.



A solution of 1-*t*-butyldimethylsilyloxybut-2-yne (0.68 g, 4.0 mmol) in THF (20 mL) was cooled to -78 °C and *n*-butyllithium (2.3 M in hexanes, 1.8 mL, 4.0 mmol) was added. The reaction mixture was stirred at -78 °C for 30 min. 2-Azidobenzaldehyde (**8**) (0.59 g, 4.0 mmol) in 5 mL of THF was cannulated into the reaction mixture and the reaction was warmed to room temperature over a period of 2 h. The reaction mixture was poured into 10 mL of saturated NH_4Cl solution and extracted with 3x30 mL of Et_2O. The combined organic layers were washed with water and brine, dried over Na_2SO_4 and the solvent was evaporated. The crude alcohol was purified by flash chromatography using 40% Et_2O in hexanes to give 1.2 g (96%) of the alcohol as yellow oil. IR (neat): 3404, 2127 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 7.8 Hz, 1H), 7.39 (t, *J* = 7.6 Hz, 1H), 7.19 (d, *J* = 7.3 Hz, 2H), 5.68 (m, 1H), 4.44 (s, 2H), 0.91 (s, 9H), 0.12 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.7, 131.5, 130.1, 128.8, 125.5, 118.7, 85.7, 84.0, 61.1, 52.2, 26.2, 18.7, -4.8; TOFESMS *m/z* relative intensity 318 (MH^+ 30); HRMS (+ES) Calcd for $C_{16}H_{24}N_3O_2Si$: 318.1638, Found: 318.1636.

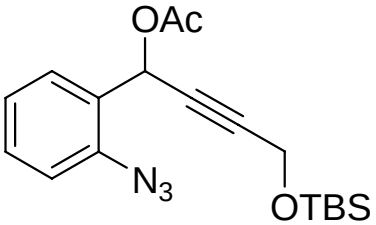
Representitive Procedure 6. Thermolysis of Crude Phenyl-substituted Allenylazides.

2-(1-Phenyl-vinyl)-1H-indole (14j)



To a solution of propargyl acetate **9a** (77 mg, 0.34 mmol) and $Pd(PPh_3)_4$ (35 mg, 0.03 mmol) in 3.4 mL of THF was added drop-wise phenylzinc bromide (0.50 M in THF, 1.0 mL, 0.50 mmol) and the reaction mixture was stirred at room temperature for 1 h, and then brought to reflux and held there for 14 h. The reaction mixture was allowed to cool to room temperature and added to 30 mL of ice-cold saturated NH_4Cl solution. The mixture was extracted with Et_2O (3x30 mL), and the combined organic layers were washed with water and brine, then dried over $MgSO_4$ and concentrated *in vacuo*. This crude material was purified by column chromatography (5% EtOAc in hexanes) to give the product **14j** as a yellow oil (25 mg, 34%). IR (neat): 3416 cm^{-1}; ^1H NMR (400 MHz, $(CD_3)_2CO$) δ 10.30 (br s, 1H), 7.51-7.48 (m, 3H), 7.42-7.36 (m, 4H), 7.10 (m, 1H), 6.99 (m, 1H), 6.30 (dd, *J* = 2.2, 0.8 Hz, 1H), 5.76 (s, 1H), 5.33 (d, *J* = 0.6 Hz, 1H); ^{13}C NMR (100 MHz, $(CD_3)_2CO$) δ 143.0, 141.5, 138.8, 138.3, 129.5, 129.3, 129.1, 128.9, 123.1, 121.3, 120.3, 112.7, 111.9, 104.0; APCI *m/z* (relative intensity) 220.1 (MH^+, 100%); HRMS Calcd for $C_{16}H_{14}N$: 220.1126, Found: 220.1121.

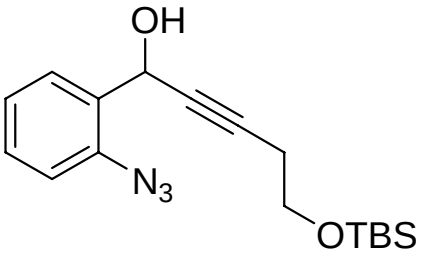
1-*t*-Butyldimethylsilyloxy-4-acetoxy-4-(2-azidophenyl)but-2-yne (9b).



Following representative procedure (**1**) for acetate synthesis, 1-*t*-butyldimethylsilyloxy-4-(2-azidophenyl)but-2-yn-4-ol (1.3 g, 3.9 mmol) was converted to acetate **9b** (1.3 g, 92%). IR (neat): 2127, 1745 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.6 Hz, 1H), 7.40 (t, *J* = 7.6 Hz, 1H), 7.18 (d, *J* = 7.8 Hz, 2H), 6.68 (m,1H), 4.44 (d, *J* = 1.8 Hz, 2H), 2.11 (s, 3H), 0.91 (s, 9H), 0.11 (s, 3H), 0.10 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.8,

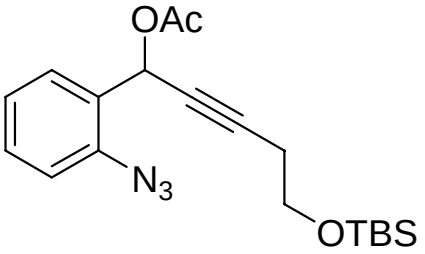
138.2, 130.7, 129.7, 128.3, 125.3, 118.6, 86.3, 81.2, 61.2, 52.2, 26.1, 21.3, 18.6, -4.8; TOFESMS *m/z* relative intensity 360 (MH^+ 80); HRMS (+ES) Calcd for $C_{18}H_{26}N_3O_3Si$: 360.1743, Found: 360.1731.

1-*t*-butyldimethylsilyloxy-5-(2-azidophenyl)pent-3-yn-5-ol.



Following the representative procedure (**5**) for alcohol synthesis, 2-azidobenzaldehyde (**8**) (0.66 g, 4.4 mmol) was treated with 1-*t*-butyldimethylsilyloxypent-3-ynyllithium (1.0 equiv, 4.4 mmol) to give 1-*t*-butyldimethylsilyloxy-5-(2-azidophenyl)pent-3-yn-5-ol as a yellow oil (1.3 g, 96%). IR (neat): 3423, 2110 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 7.5 Hz, 1H), 7.37 (t, *J* = 7.2 Hz, 1H), 7.18 (t, *J* = 7.3 Hz, 2H), 5.64 (m,1H), 3.76 (t, *J* = 7.1 Hz, 2H), 2.50 (dt, *J* = 7.0, 1.8 Hz, 2H), 0.91 (s, 9H), 0.08 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.8, 132.3, 130.1, 128.9, 125.5, 118.7, 85.0, 80.4, 62.1, 61.2, 26.3, 23.7, 18.7, -4.9 (2C); TOFESMS *m/z* relative intensity 332 (MH^+ 40); HRMS (+ES) Calcd for $C_{17}H_{26}N_3O_2Si$: 332.1794, Found: 332.1780.

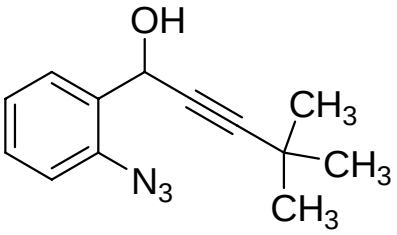
1-*t*-Butyldimethylsilyloxy-5-acetoxy-5-(2-azidophenyl)pent-3-yne (9c).



Following representative procedure (**1**) for acetate synthesis, 1-*t*-butyldimethylsilyloxy-5-(2-azidophenyl)pent-3-yn-5-ol (1.3g, 3.9 mmol) was converted to acetate **9c** (1.2 g, 82%). IR (neat): 2129, 1747 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, *J* = 7.7 Hz, 1H), 7.41 (dt, *J* = 7.7, 1.5 Hz, 1H), 7.19 (t, *J* = 7.7 Hz, 2H), 6.64 (t, *J* = 2.0 Hz, 1H), 3.75 (t, *J* = 6.9 Hz, 2H), 2.49 (dt, *J* = 6.9, 2.1 Hz, 1H), 2.11 (s, 3H), 0.89 (s, 9H), 0.07 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.9, 138.2, 130.6, 129.9, 128.9, 125.4, 118.7, 85.7.3, 82.2,

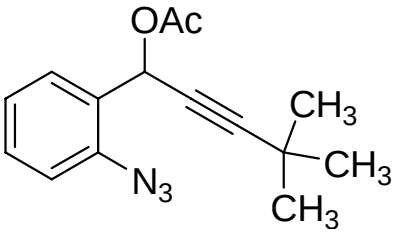
61.9 61.5, 26.3, 23.7, 21.4, 18.7, -4.9 (2C); TOFESMS *m/z* relative intensity 396 (MNa^+ 65); HRMS (+ES) Calcd for $C_{19}H_{27}N_3O_3SiNa$: 396.1719, Found: 396.1716.

1-(2-Azidophenyl)4,4-dimethylpent-2-yn-1-ol.



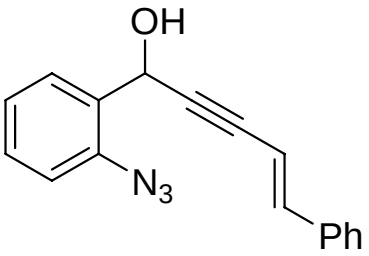
Following the representative procedure (**5**) for alcohol synthesis, 2-azidobenzaldehyde (**8**) (0.60 g, 4.0 mmol) was treated with 3,3-dimethylbutynyllithium (1.0 equiv, 4.0 mmol) to give 1-(2-azidophenyl)4,4-dimethylpent-2-yn-1-ol as a yellow oil (800 mg, 87%). IR (neat): 3375, 2128 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.6 Hz, 1H), 7.37 (t, *J* = 7.5 Hz, 1H), 7.17-7.28 (m, 2H), 5.67 (s,1H), 2.53 (bs, 1H) 1.28 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.9, 132.6, 130.0, 128.9, 125.4, 118.7, 96.4, 77.7, 60.1, 31.3 27.9; TOFESMS *m/z* relative intensity 230 (MH^+ 80); HRMS (+ES) Calcd for $C_{12}H_{16}N_3O$: 230.1293, Found: 230.1280.

1-Acetoxy-1-(2-azidophenyl)4,4-dimethylpent-2-yne (9d).



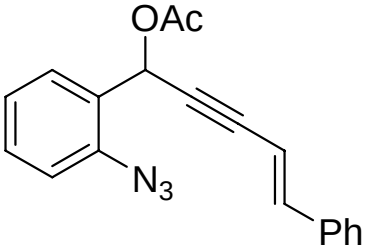
Following representative procedure (**1**) for acetate synthesis, 1-(2-azidophenyl)4,4-dimethylpent-2-yn-1-ol (0.80 g, 3.5 mmol) was converted to acetate **9d** (850 mg, 90%). IR (neat): 2127, 1745 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 7.8 Hz, 1H), 7.39 (t, *J* = 7.7 Hz, 1H), 7.20 (t, *J* = 8.3 Hz, 2H), 6.64 (s,1H), 2.10 (s, 3H), 1.26 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.9, 138.4, 130.5, 130.0, 129.1, 125.3, 118.7, 96.7, 74.9 61.6, 31.2, 27.9, 21.5; TOFESMS *m/z* relative intensity 272 (MH^+ 32); HRMS (+MSES) Calcd for $C_{15}H_{18}N_3O_2$: 272.1399, Found: 272.1392.

1-(2-Azidophenyl)5-phenylpent-4-en-2-yn-ol.



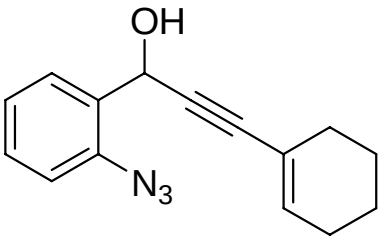
Following the representative procedure (**5**) for alcohol synthesis, 2-azidobenzaldehyde (**8**) (0.46 g, 3.1 mmol) was treated with 4-phenylbut-3-en-1-ynyllithium (1.0 equiv, 3.1 mmol) to give 1-(2-azidophenyl)5-phenylpent-4-en-2-yn-ol as a bright yellow oil (460 mg, 54%). IR (neat): 3396, 2126 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.71 (dd, *J* = 8.1, 1.3 Hz, 1H), 7.44-7.23 (m, 8H), 7.02 (d, *J* = 16.3 Hz, 1H), 6.24 (dd, *J* = 16.3, 1.9 Hz, 1H), 5.84 (m, 1H), 2.75 (bs, 1H) ; ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.6, 137.8, 136.5, 132.2, 130.2,129.3, 129.2, 128.9, 126.8, 125.6, 118.8, 107.8, 90.7, 86.1, 61.5; TOFESMS *m/z* relative intensity 276 (MH^+ 30); HRMS (+ES) Calcd for $C_{13}H_{15}N_3O$: 276.1137, Found: 276.1123.

1-Acetoxy-1-(2-azidophenyl) 5-phenylpent-4en-2-yne (9g).



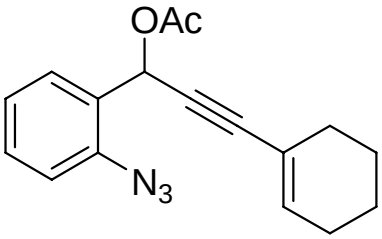
Following representative procedure (**1**) for acetate synthesis, 1-(2-azidophenyl)5-phenylpent-4-en-2-yn-ol (0.40 g, 1.6 mmol) was converted to acetate **9g** (460 mg, 91%). IR (neat): 2127, 1742 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.78 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.44-7.20 (m, 8H), 7.04 (d, *J* = 16.3 Hz, 1H), 6.87 (d, *J* = 1.8 Hz, 1H) 6.22 (dd, *J* = 16.3, 2.0 Hz, 1H), 2.16 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.0, 143.2, 138.3, 136.3, 130.8, 129.8, 129.4, 129.2, 128.6, 126.8, 125.5, 118.8, 107.5, 87.5, 86.7, 61.9, 21.4; TOFESMS *m/z* relative intensity 318 (MH^+ 68); HRMS (+ES) Calcd for $C_{19}H_{16}N_3O_2$: 318.1243, Found: 318.1261.

1-(2-Azidophenyl)3-cyclohex-1-enylprop-2-yn-1-ol.



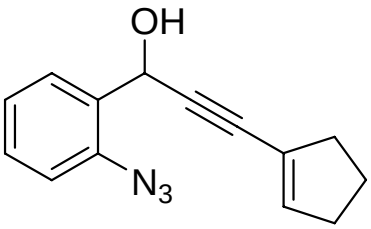
Following the representative procedure (**5**) for alcohol synthesis, 2-azidobenzaldehyde (**8**) (0.50 g, 3.4 mmol) was treated with 1-cyclohexenylethynyllithium (0.85 equiv, 2.9 mmol) to give 1-(2-azidophenyl)3-cyclohex-1-enylprop-2-yn-1-ol as a yellow oil (280 mg, 38%). IR (neat): 3390, 2126 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.67 (d, J = 7.7 Hz, 1H), 7.36 (m, 1H), 7.19-7.11 (m, 2H), 6.16 (m,1H), 5.76 (d, J = 4.6 Hz, 1H), 3.23 (d, J = 5.3 Hz, 1H), 2.15-2.09 (m, 4H), 1.64-1.58 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.7, 136.1, 132.5, 130.0, 128.9,125.5, 120.5, 118.7, 88.7, 85.9, 61.1 29.5, 26.1, 22.7, 21.9; TOFESMS *m/z* relative intensity 254 (MH^+ 75); HRMS (+ES) Calcd for $C_{15}H_{16}N_3O$: 254.1293, Found: 254.1302.

1-Acetoxy-1-(2-azidophenyl) 3-cyclohex-1-enylprop-2-yne (9h).



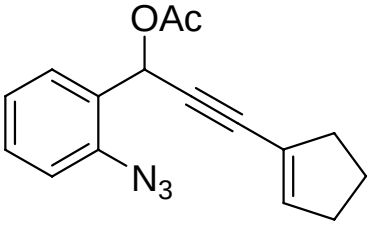
Following representative procedure (**1**) for acetate synthesis, 1-(2-azidophenyl)3-cyclohex-1-enylprop-2-yn-1-ol (0.28 g, 1.1 mmol) was converted to acetate **9h** (260 mg, 80%). IR (neat): 2127, 1742 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.72 (dd, J = 7.5, 1.3 Hz, 1H), 7.42 (dd, J = 7.2, 1.5 Hz, 1H), 7.18-7.22 (m, 2H), 6.76 (m, 1H) 6.20 (m, 1H), 2.11 (s, 3H), 2.16-2.10 (m, 4H), 1.67-1.55 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.9, 138.2, 137.0, 130.6, 129.8, 128.9, 125.4, 120.2, 118.7, 89.3, 82.7, 61.7, 29.3, 26.0, 22.6, 21.8, 21.5; TOFESMS *m/z* relative intensity 318 (MNa^+ 100); HRMS (+ES) Calcd for $C_{17}H_{17}N_3O_2Na$: 318.1203, Found: 318.1218.

1-(2-Azidophenyl)3-cyclopent-1-enylprop-2-yn-1-ol.



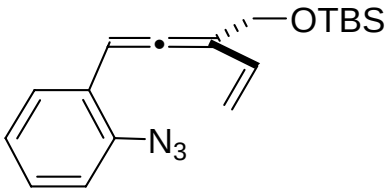
Following the representative procedure (**5**) for alcohol synthesis, 2-azidobenzaldehyde (**8**) (0.35 g, 2.4 mmol) was treated with 1-cyclopentenylethynyllithium (1.3 equiv, 3.2 mmol) to give 1-(2-Azidophenyl)3-cyclopent-1-enylprop-2-yn-1-ol as a yellow oil (330 mg, 54%). IR (neat): 3354, 2126 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.67 (dd, *J* = 8.2, 1.6 Hz, 1H), 7.43 (m, 1H), 7.22-7.15 (m, 2H), 6.12 (t, *J* = 2.4 Hz, 1H), 5.80 (d, *J* = 6.2 Hz 1H), 2.61 (d, *J* = 6.1 Hz, 1H), 2.51-2.42 (m, 4H), 1.96-1.89 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.0, 137.4, 131.7, 129.8, 128.5, 125.1, 123.8, 118.3, 88.9, 84.1, 61.2 36.3, 33.3, 23.3; TOFESMS *m/z* relative intensity 240 (MH^+ 30); HRMS (+ES) Calcd for $C_{14}H_{14}N_3O$: 240.1127, Found: 240.1137.

1-Acetoxy-1(2-azidophenyl) 3-cyclopent-1-enylprop-2-yne (9i).



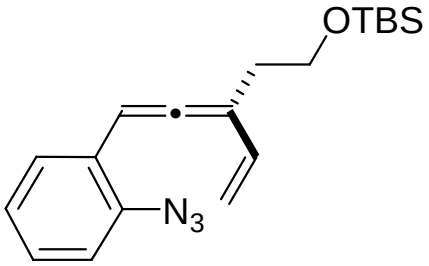
Following representative procedure (**1**) for acetate synthesis, 1-(2-azidophenyl)3-cyclopent-1-enylprop-2-yn-1-ol (0.33 g, 1.3 mmol) was converted to acetate **9i** (330 mg, 88%). IR (neat): 2126, 1746 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, *J* = 8.6 Hz, 1H), 7.42 (d, *J* = 7.7 Hz, 1H), 7.25-7.18 (m, 2H), 6.79 (m, 1H), 6.22 (m, 1H), 2.62-2.45 (m, 4H), 2.12 (s, 3H), 1.96-1.88 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.9, 140.1, 138.3, 130.6, 129.8, 128.7, 125.4, 124.0, 118.7, 86.4, 84.8, 61.9, 36.6, 33.7, 23.7, 21.5; TOFESMS *m/z* relative intensity 282 (MH^+ 30); HRMS (+ES) Calcd for $C_{16}H_{16}N_3O_2$: 282.1243, Found: 282.1229.

1-*t*-Butyldimethylsilyloxy-2-vinyl-4-(2-azidophenyl)but-2,3-diene (6b).



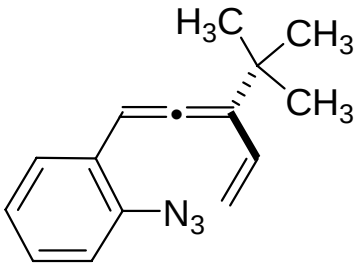
Following representative procedure (**2**) for allenylazide synthesis, acetate **9b** (0.50 g, 1.4 mmol) was converted to **6b** (250 mg, 55%). IR (neat): 2122, 1934 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.39 (dd, *J* = 7.7, 1.5 Hz, 1H), 7.29 (m, 1H), 7.18-7.09 (m, 2H), 6.10 (m, 1H), 6.38 (dd, *J* = 17.7, 10.1 Hz, 1H), 5.38 (dd, *J* = 17.4, 1.1 Hz, 1H), 5.16 (d, *J* = 10.5, 1.2 Hz, 1H), 4.48 (d, *J* = 2.5 Hz, 2H), 0.9 (s, 9H), 0.09 (s, 3H), 0.08 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 209.0, 136.8, 132.0, 129.0, 128.7, 125.9, 125.2, 118.8, 115.1, 109.8, 91.9, 61.9, 26.2, 18.7, -4.8, -4.9; TOFESMS *m/z* relative intensity 300 (MH^+-N_2 100); HRMS (+ES) Calcd for $C_{18}H_{26}NOSi$: 300.1784, Found: 300.1812.

1-*t*-Butyldimethylsilyloxy-3-vinyl-5-(2-azidophenyl)but-3,4-diene (6c).



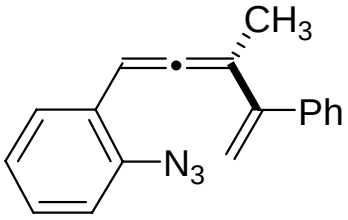
Following representative procedure (**2**) for allenylazide synthesis, acetate **9c** (0.50 g, 1.3 mmol) was converted to **6c** (259 mg, 57%). IR (neat): 2122, 1930 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 7.9 Hz, 1H), 7.25 (d, *J* = 7.3 Hz, 1H), 7.15 (d, *J* = 8.0 Hz, 1H), 7.08 (t, *J* = 7.5 Hz, 1H), 6.59 (m, 1H), 6.32 (dd, *J* = 17.5, 10.7 Hz, 1H), 5.29 (d, *J* = 17.6 Hz, 1H), 5.13 (d, *J* = 10.7 Hz, 1H), 3.81 (t, *J* = 5.8 Hz, 2H), 2.54-2.49 (m, 2H), 0.89 (s, 9H), 0.06 (s, 3H), 0.04 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 209.8, 136.7, 134.3, 128.8, 128.6, 126.1, 125.2, 118.9, 114.0, 106.2, 90.6, 62.1, 32.1, 26.3, 18.8, -4.8 (2C); TOFESMS *m/z* relative intensity 314 (MH^+-N_2 100); HRMS (+ES) Calcd for $C_{19}H_{28}NOSi$: 314.1940, Found: 314.1919.

1-(2-Azidophenyl)3-*t*-butylpent-1,2,4-triene (6d).



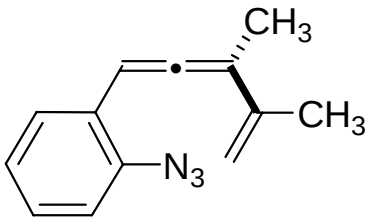
Following representative procedure (**2**) for allenylazide synthesis, acetate **9d** (0.27 g, 1.0 mmol) was converted to **6d** (150 mg, 63%). IR (neat): 2124, 1930 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.43 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.27 (dt, *J* = 8.1, 1.5 Hz, 1H), 7.19 (d, *J* = 7.0 Hz, 1H), 7.12 (t, *J* = 7.6 Hz, 1H), 6.73 (m, 1H), 6.28 (ddd, *J* = 17.0, 10.4, 1.3 Hz, 1H), 5.51 (dd, *J* = 17.1, 0.9 Hz, 1H), 5.26 (dd, *J* = 10.4, 0.9 Hz, 1H), 1.24 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.7, 136.6, 130.9, 128.3, 128.0, 126.9, 125.3, 119.0,118.6, 117.2, 92.3, 34.3, 29.9; TOFESMS *m/z* relative intensity 210 (MH^+-N_2 100); HRMS (-ES) Calcd for $C_{15}H_{16}N$: 210.1283, Found: 210.1277.

1-(2-Azidophenyl)3-methyl-5phenylpent-1,2,4-triene (6e).



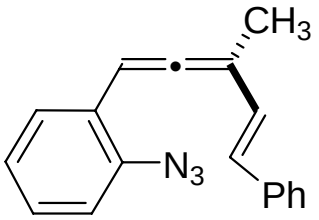
Following representative procedure (**2**) for allenylazide synthesis, acetate **9a** (0.20 g, 0.88 mmol) was converted to **6e** (160 mg, 67%). IR (neat): 2124, 1932 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.25-7.55 (m, 7H), 7.16 (d, *J* = 7.6 Hz, 2H), 6.48 (m, 1H), 5.41(s, 1H), 5.32 (s, 1H), 2.19 (d, *J* = 2.7 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 209.8, 147.2, 141.7, 136.7, 128.6, 128.5, 128.4, 128.1, 128.0, 127.9, 126.4, 118.9, 114.4, 106.1, 90.4, 18.0; TOFESMS *m/z* relative intensity 246 (MH^+-N_2 100); HRMS (+ES) Calcd for $C_{18}H_{16}N$: 246.1283, Found: 246.1275.

1-(2-Azidophenyl)3-methyl-4-phenylpent-1,2,4-triene (6f).



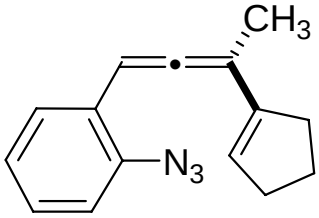
Following representative procedure (**2**) for allenylazide synthesis, acetate **9a** (0.23 g, 1.0 mmol) was converted to **6e** (125 mg, 59%). IR (neat): 2124, 1930 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.41 (d, *J* = 7.7 Hz, 1H) 7.28 (t, *J* = 7.9 Hz, 1H), 7.16-7.10 (m, 2H), 6.6 (m, 1H), 5.08 (s, 1H), 5.04 (s, 1H), 2.05 (s, 3H), 2.04 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 208.7, 140.8, 136.7, 128.5, 128.4, 126.7, 125.3, 118.9, 112.0, 107.1, 90.3, 22.0, 16.7; TOFESMS *m/z* relative intensity 184 (MH^+-N_2 100); HRMS (+ES) Calcd for $C_{13}H_{14}N$: 184.1121, Found: 184.1126.

1-(2-Azidophenyl)3-methyl-4-phenylpent-1,2,4-triene (6g).



Following representative procedure (**3**) for allenylazide synthesis, acetate **9g** (0.21 g, 0.65 mmol) was converted to **6e** (60 mg, 34%). IR (neat): 2122, 1927 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.55-7.10 (m, 9H), 6.82 (d, *J* = 16.1 Hz, 1H), 6.63 (m, 1H), 6.56 (d, *J* = 16.2 Hz, 1H), 2.09 (d, *J* = 2.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 211.6, 137.7, 136.7, 130.5, 129.0, 128.9, 128.8, 128.6, 127.8, 126.9, 126.7, 125.3, 118.9, 104.8, 89.3, 15.7; TOFESMS *m/z* relative intensity 246 (MH^+-N_2 100); HRMS (+ES) Calcd for $C_{18}H_{16}N$: 246.1283, Found: 246.1289.

1-(2-Azidophenyl)3-cyclopent-1-enylbut-1,2-diene (6i).

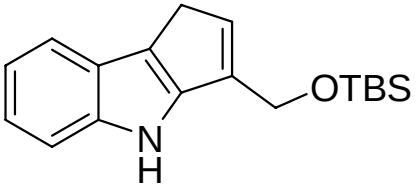


Following representative procedure (**3**) for allenylazide synthesis, acetate **9i** (0.30 g, 1.1 mmol) was converted to **6e** (75 mg, 30%). IR (neat): 2112, 1930 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.38 (dd, *J* = 7.7, 1.5 Hz, 1H), 7.25 (dt, *J* = 7.0, 1.6 Hz, 1H), 7.16 (dd, *J* = 7.9, 1.1 1H), 7.09 (dt, *J* = 7.3, 1.8 Hz, 1H), 6.55 (m, 1H), 5.75 (d, *J* = 1.4 Hz, 1H), 2.50-2.35 (m, 4H), 2.02 (d, *J* = 2.7 Hz, 3H), 1.93 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.8, 140.6, 136.6, 128.6, 128.2, 127.1, 127.0, 125.2, 118.8, 103.0, 89.5, 34.1, 33.8, 23.7, 17.0; APCIMS *m/z* relative intensity 210 (MH^+-N_2 100); HRMS (+APMS) Calcd for $C_{15}H_{16}N$: 210.1283, Found: 210.1302.

Cyclization Studies

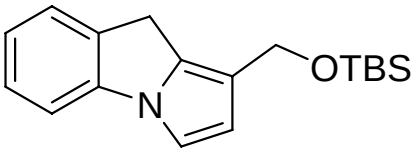
Following the representative procedure (**4**) for cyclization, allenylazide **6b** (49 mg, 0.15 mmol) was converted to compounds **7b** (10 mg, 40%) and **11b** (13 mg, 56%).

3-(*t*-Butyldimethylsilyloxymethyl)-1,4-dihydrocyclopenta[b]indole (7b).



IR (neat): 3395 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.23 (bs, 1H), 7.58 (m, 1H), 7.41 (m, 1H), 7.14-7.06 (m, 2H), 6.29 (m, 1H), 4.76 (d, *J* = 1.7 Hz, 2H), 3.29 (d, *J* = 1.6 Hz, 2H), 0.97 (s, 1H), 0.14 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.1, 140.4, 137.8, 129.7, 124.8, 121.2, 120.7, 120.2, 118.5, 112.4, 61.1, 32.0, 29.4, 18.8, -4.8 (2C); TOFESMS *m/z* relative intensity 300 (MH^+ 10); HRMS (+ES) Calcd for $C_{36}H_{51}N_2O_2Si_2$: 599.3489, Found: 599.3466.

1-(*t*-Butyldimethylsilyloxymethyl)-9H-pyrrolo[1,2-a]indole (11b).

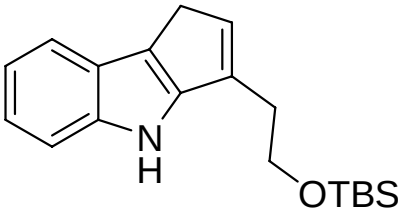


^1H NMR (300 MHz, $CDCl_3$) δ 7.41 (d, *J* = 7.5 Hz, 1H), 7.30-7.24 (m, 2H), 7.09 (t, *J* = 7.3 Hz, 1H), 7.06 (d, *J* = 2.1 Hz, 1H), 6.33 (d, *J* = 2.6 Hz, 1H), 4.73 (s, 2H), 3.85 (s, 2H), 0.96 (s, 9H) 0.13 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.5, 135.2, 133.0, 127.7, 126.3, 123.3, 117.1, 112.8, 109.94, 109.92, 59.6, 29.1, 26.5, 18.9, -4.7 (2C); TOFESMS

m/z relative intensity 300 (MH^+ 10); HRMS (+ES) Calcd for $C_{36}H_{51}N_2O_2Si_2$: 599.3489, Found: 599.3466.

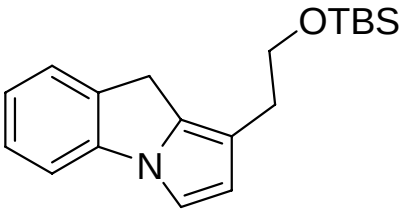
Following the representative procedure (**4**) for cyclization, allenylazide **6c** (25 mg, 0.07 mmol) was converted to compounds **7c** (12 mg, 52%) and **11c** (10 mg, 43%).

3-(*t*-Butyldimethylsilyloxyethyl)-1,4-dihydrocyclopenta[b]indole (7c).



IR (neat): 3372 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.8 (bs, 1H), 7.57 (m, 1H), 7.38 (m, 1H), 7.13-7.07 (m, 2H), 6.22 (m, 1H), 3.95 (t, *J* = 5.7 Hz, 2H), 3.25 (s, 2H), 2.82 (t, *J* = 5.6 Hz, 1H), 0.99 (s, 9H), 0.14 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.5, 140.1, 136.1 131.9, 125.2, 120.6, 120.4, 120.0, 118.4, 112.2, 63.9, 32.7, 31.8, 26.5, 18.9, -4.9; TOFMSES *m/z* relative intensity 314 (MH^+ 25); HRMS (+MSES) Calcd for $C_{19}H_{28}NOSi$: 314.1940, Found: 314.1929.

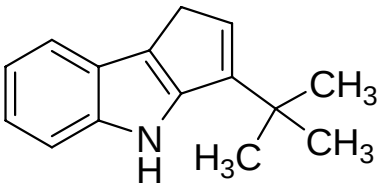
1-(*t*-Butyldimethylsilyloxyethyl)-9H-pyrrolo[1,2-a]indole (11c).



^1H NMR (300 MHz, $CDCl_3$) δ 7.40 (d, *J* = 7.6 Hz, 1H), 7.30 (d, *J* = 7.8 Hz, 1H), 7.24 (d, *J* = 7.7 Hz, 1H), 7.07 (t, *J* = 7.4 Hz, 1H) 7.04 (d, *J* = 2.4 Hz, 1H), 6.25 (d, *J* = 2.6 Hz, 1H), 3.84 (t, *J* = 7.7 Hz, 2H), 3.79 (s, 2H), 2.78 (t, *J* = 7.5 Hz, 2H), 0.93 (s, 9H) 0.07 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.7, 135.1, 133.0, 127.8, 126.3, 123.3, 114.2, 113.6, 109.9, 109.7, 64.5, 31.0, 28.7, 26.4, 18.9, -4.8; TOFESMS *m/z* relative intensity 314 (MH^+ 25); HRMS (+ES) Calcd for $C_{19}H_{28}NOSi$: 314.1940, Found: 314.1929.

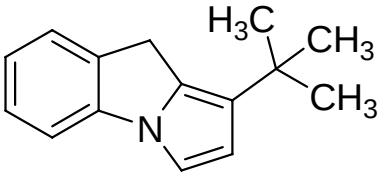
Following the representative procedure (**4**) for cyclization, allenylazide **6d** (60 mg, 0.25 mmol) was converted to compounds **7d** (30 mg, 57%) and **11d** (11 mg, 20%).

3-*t*-Butyl-1,4-dihydrocyclopenta[b]indole (7d).



IR (neat): 3414 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.09 (bs, 1H), 7.60 (m, 1H), 7.43 (m, 1H), 7.29-7.13 (m, 2H), 6.19 (t, *J* = 1.6 Hz, 1H), 3.24 (d, *J* = 1.6 Hz, 2H), 1.4 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.5, 147.0, 140.5, 127.6, 124.8, 122.1, 120.7, 120.3, 118.5, 112.2, 32.8, 31.2, 30.2; TOFESMS *m/z* relative intensity 212 (MH^+ 100); HRMS (+ES) Calcd for $C_{15}H_{18}N$: 212.1439, Found: 212.1436.

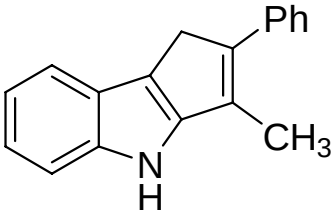
1-*t*-Butyl-9H-pyrrolo[1,2-a]indole (11d).



^1H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, *J* = 7.4 Hz, 1H), 7.28-7.22 (m, 2H), 7.07 (dd, *J* = 7.4, 1.2 Hz, 1H), 7.04 (d, *J* = 2.9 Hz, 1H), 6.32 (d, *J* = 2.8 Hz, 1H), 3.92 (s, 2H), 1.36 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.8, 135.2, 130.1, 127.8, 127.7, 126.0, 123.0, 111.7, 109.7, 109.2, 31.7, 31.3, 30.8; TOFESMS *m/z* relative intensity 212 (MH^+ 100); HRMS (+ES) Calcd for $C_{15}H_{18}N$: 212.1439, Found: 212.1436.

Following the representative procedure (**4**) for cyclization allenylazide **6e** (45 mg, 0.16 mmol) was converted to compounds **7e** (16 mg, 40%) and **11e** (12 mg, 30%).

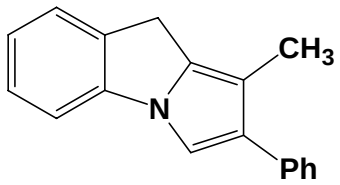
3-Methyl-2-phenyl-1,4-dihydrocyclopenta[b]indole (7e).



IR (neat): 3414 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.07 (bs, 1H), 7.60 (d, *J* = 7.4 Hz, 1H), 7.52 (m, 2H), 7.45-7.41 (m, 3H), 7.25 (m, 1H), 7.18-7.11 (m, 2H), 3.69 (q, *J* = 1.8 Hz, 2H), 2.39 (t, *J* = 1.7 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.0, 143.5, 140.2,

138.3, 128.9, 128.1, 127.6, 126.6, 125.1, 120.8, 120.6, 118.7, 118.6, 112.3 34.7, 12.9; TOFESMS *m/z* relative intensity 246 (MH^+ 100); HRMS (+ES) Calcd for $C_{18}H_{16}N$: 246.1283, Found: 246.1262.

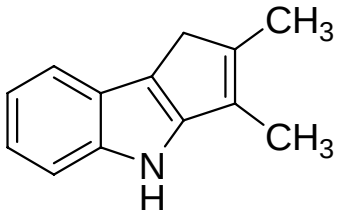
1-Methyl-2-phenyl-9H-pyrrolo[1,2-a]indole (11e).



^1H NMR (300 MHz, $CDCl_3$) δ 7.54-7.52 (m, 2H), 7.45-7.40 (m, 3H), 7.34-7.28 (m, 3H), 7.24 (m, 1H), 7.11 (dt, *J* = 7.2, 1.5 Hz, 1H), 3.85 (s, 2H), 2.3 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.6, 137.0, 134.8, 134.0, 130.0, 128.8, 128.2, 127.9, 126.3, 126.1, 123.2, 110.3, 109.9, 107.7, 28.6, 11.4; TOFESMS *m/z* relative intensity 246 (MH^+ 100); HRMS (+ES) Calcd for $C_{18}H_{16}N$: 246.1283, Found: 246.1262.

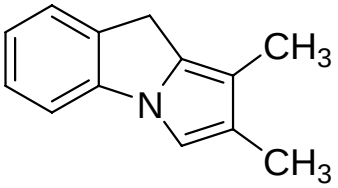
Following the representative procedure (**4**) for cyclization, allenylazide **6f** (35 mg, 0.17 mmol) was converted to compounds **7f** (11 mg, 36%) and **11f** (11 mg, 36%).

2,3-Dimethyl -1,4-dihydrocyclopenta[b]indole (7f).



IR (neat): 3406 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.94 (bs, 1H), 7.51 (d, *J* = 7.7 Hz, 1H), 7.38 (d, *J* = 7.8 Hz, 1H), 7.13-7.04 (m, 2H), 3.19 (s, 2H), 2.13 (s, 3H), 2.08 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.9, 141.6, 139.7, 125.4, 125.3, 120.3, 119.7, 117.9, 116.9, 112.1, 36.5, 15.0, 10.8; TOFESMS *m/z* relative intensity 183 (MH^+ 100); HRMS (+ES) Calcd for $C_{13}H_{14}N$: 184.1126, Found: 184.1095.

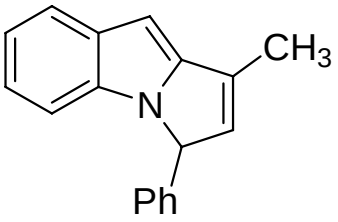
1,1-Dimethyl-9H-pyrrolo[1,2-a]indole (11f).



^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37 (d, *J* = 7.3 Hz, 1H), 7.27 (t, *J* = 7.6 Hz, 1H), 7.16 (d, *J* = 7.7 Hz, 1H), 7.02 (t, *J* = 7.2 Hz, 1H), 6.88 (s, 1H), 3.75 (s, 2H), 2.14 (s, 3H), 2.08 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.9, 134.7, 132.8, 127.7, 126.1, 124.0, 122.4, 111.6, 109.3, 107.6, 28.5, 11.3, 9.8; TOFESMS *m/z* relative intensity 183 (MH^+ 100); HRMS (+ES) Calcd for $C_{13}H_{14}N$: 184.1126, Found: 184.1095.

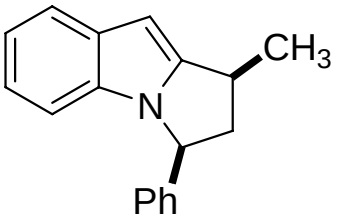
Following the representative procedure (**4**) for cyclization, allenylazide **6g** (35 mg, 0.13 mmol) was converted to compound **10g** (9 mg, 36%).

1-Methyl-3-phenyl-3H-pyrrolo[1,2-a]indole (10g).



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (dd, *J* = 6.6, 1.7 Hz, 1H), 7.36-7.30 (m, 3H), 7.21-7.11 (m, 2H), 7.05-7.01 (m, 2H), 6.94 (dd, *J* = 7.7, 1.5 Hz, 1H), 6.34 (s, 1H), 6.17 (m, 1H), 5.70 (s, 1H), 2.21 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.0, 138.2, 134.5, 133.9, 133.3, 132.1, 129.3, 128.4, 127.3, 122.0, 121.5, 119.5, 109.8, 90.6, 65.7, 12.9; TOFESMS *m/z* relative intensity 246 (MH^+ 20); HRMS (+ES) Calcd for $C_{18}H_{16}N$: 246.1283, Found: 246.1259.

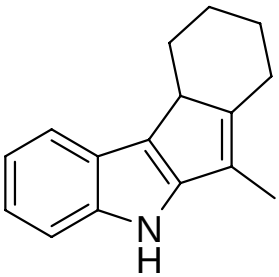
1-Methyl-3-phenyl-2,3-dihydro-1H-pyrrolo[1,2-a]indole (12g).



A deoxygenated solution of compound **10g** (9.0 mg, 0.04 mmol) and 5 mg of 10% Pd on carbon in 5 mL of THF was stirred at room temperature under H_2 at 1 atm for 2 h. The solution was then filtered through Celite and concentrated *in vacuo* to afford a yellow oil. The crude compound was purified over an alumina column using 20% Et_2O in hexanes to give **12g** as a yellow film (6 mg, 54%). Crystals suitable for X-ray crystallographic analysis were obtained by slow evaporation of an Et_2O solution of **12g** over a period of 48 h at 25 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (d, *J* = 7.9 Hz, 1H), 7.38-7.35 (m, 3H), 7.28-7.24 (m, 2H), 7.03 (dt, *J* = 7.1, 0.8 Hz, 1H), 6.90 (dt, *J* = 7.1, 1.0 Hz, 1H), 6.59 (d, *J* = 8.1 Hz, 1H), 6.24 (s, 1H), 5.35 (t, *J* = 8.2 Hz, 1H), 3.45 (m, 1H), 3.18 (m, 1H), 2.13 (m, 1H), 1.49 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.6, 141.4, 133.8, 132.6, 129.2, 128.3, 127.1, 120.8, 120.4, 119.6, 110.8, 92.1, 61.9, 49.1, 32.7, 20.1; TOFESMS *m/z* relative intensity 248 (MH^+ 45); HRMS (+ES) Calcd for $C_{18}H_{18}N$: 248.1439, Found: 248.1445.

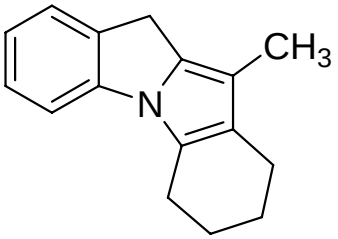
Following the representative procedure (**4**) for cyclization, allenylazide **6h** (31 mg, 0.12 mmol) was converted to compounds **7h** (10 mg, 36%) and **11h** (14 mg, 51%).

6-Methyl -5,7,8,9-hexahydroindeno[2,1-b]indole (7h).



IR (neat): 3406 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (bs, 1H), 7.52 (d, *J* = 7.8 Hz, 1H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.10 (t, *J* = 7.1 Hz, 1H), 7.03 (t, *J* = 7.2 Hz, 1H) 3.05 (dd, *J* = 12.5, 5.5 Hz, 1H), 2.8 (m, 1H), 2.68 (m, 1H), 2.27 (m, 1H), 2.08 (s, 3H), 2.6 (m, 1H), 1.85 (m, 1H), 1.28-1.21 (m, 2H), 1.00 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.7, 148.8, 136.0, 139.7, 122.3, 121.2, 120.2, 119.6, 117.9, 112.2, 45.2, 33.6, 28.9, 27.3, 26.0, 10.4; TOFESMS *m/z* relative intensity 224 (MH^+ 10); HRMS (+ES) Calcd for $C_{16}H_{18}N$: 214.1422, Found: 224.1439.

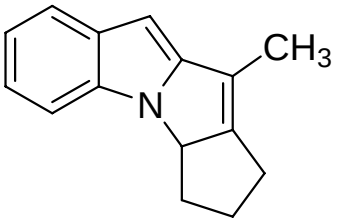
11-Methyl-2,3,4,10-tetrahydro-1H-indolo[1,2-a]indole (11h).



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, *J* = 7.4 Hz, 1H), 7.28-7.24 (m, 2H), 7.05 (m, 1H), 3.74 (s, 2H), 2.96(t, *J* = 5.6 Hz, 2H), 2.51(t, *J* = 6.0 Hz, 2H), 2.04 (s, 3H), 1.95-1.85 (m, 2H), 1.85-1.80 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.5, 135.2, 130.6, 127.6, 126.2, 122.5, 122.0, 121.6, 110.2, 109.5, 28.1, 23.8, 23.7, 23.2, 22.4, 9.6; TOFESMS *m/z* relative intensity 224 (MH^+ 10); HRMS (+ES) Calcd for $C_{16}H_{18}N$: 214.1422, Found: 224.1439.

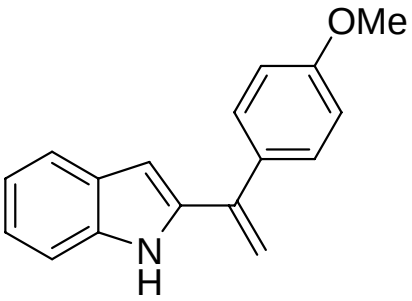
Following the representative procedure (**4**) for cyclization, allenylazide **6i** (40 mg, 0.17 mmol) was converted to compound **10i** (14 mg, 40%),

Pyrrolo-indole (10i)



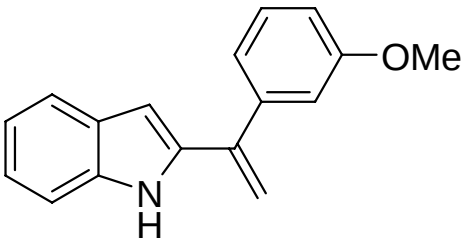
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 8.2 Hz, 1H), 7.25 (s, 1H), 7.12 (t, *J* = 7.3 Hz, 1H), 7.03 (t, *J* = 7.1 Hz, 1H), 6.11 (s, 1H), 4.8 (t, *J* = 8.5 Hz, 1H), 2.46-2.27 (m, 6H), 2.04 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 154.1, 152.2, 135.2, 132.8, 123.4, 121.7, 121.0, 119.1, 108.9, 89.1, 66.3, 29.7, 27.8, 21.2, 11.2; TOFMSES *m/z* relative intensity 210 (MH^+ 10); HRMS (+MSES) Calcd for $C_{15}H_{16}N$: 210.1283, Found: 210.1274.

2-[1-(4-Methoxy-phenyl)-vinyl]-1H-indole (14k)



Following representative procedure (**6**) for crude thermolysis, propargyl acetate **9a** (78 mg, 0.34 mmol) was treated with 4-methoxyphenylzinc iodide (0.50M in THF, 1.0 mL, 0.51 mmol) to give compound **14k** (30 mg, 35%) as a yellow solid: mp 145 - 146 °C; IR (neat): 3429 cm^{-1}; ^1H NMR (300 MHz, $(CD_3)_2CO$) δ 10.35 (br s, 1H), 7.49 (dd, *J* = 6.8, 1.0 Hz, 1H), 7.42 (dd, *J* = 6.7, 2.2 Hz, 2H), 7.37 (dq, *J* = 8.1, 0.9 Hz, 1H), 7.10 (td, *J* = 7.1, 1.2 Hz, 1H), 7.01-6.94 (m, 3H), 6.32 (dd, *J* = 2.2, 0.8 Hz, 1H), 5.66 (d, *J* = 0.7, 1H), 5.27 (d, *J* = 0.8 Hz, 1H), 3.84 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.7, 141.0, 138.1, 136.3, 132.4, 129.6, 128.6, 122.6, 120.7, 120.0, 113.7, 111.7, 110.7, 103.1, 55.3; APCI *m/z* (relative intensity) 250.1 (MH^+: 100%); HRMS Calcd for $C_{17}H_{16}NO$: 250.1232, Found: 250.1226.

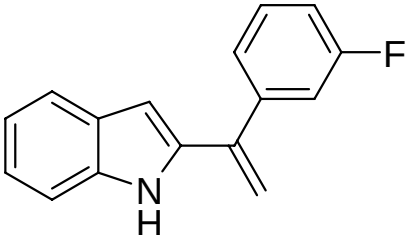
2-[1-(3-Methoxy-phenyl)-vinyl]-1H-indole (14l)



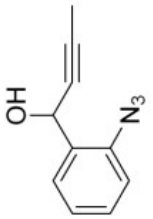
Following representative procedure (**6**) for crude thermolysis using a reflux time of 4 h and 6% Et_2O in hexanes as chromatography eleuent, propargyl acetate **9a** (81 mg, 0.35 mmol) was treated with 3-methoxyphenylzinc iodide (0.50 M in THF, 1.8 mL, 0.88 mmol) to give the product **14l** as a yellow oil (20 mg, 23%). IR (neat): 3413 cm^{-1}; ^1H NMR (400 MHz, $(CD_3)_2CO$) δ 10.40 (br s, 1H), 7.49 (dd, *J* = 7.9, 0.8 Hz, 1H), 7.37 (dq, *J* = 8.2, 0.8 Hz, 1H), 7.32 (t, *J* = 7.9 Hz, 1H), 7.10 (m, 1H), 7.07-7.03 (m, 2H), 7.00-6.96 (m, 2H), 6.35 (dd, *J* = 5.1, 0.8 Hz, 1H), 5.76 (s, 1H), 5.34 (s, 1H), 3.82 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.5, 141.5, 141.4, 137.6, 136.4, 129.4, 128.6, 122.7, 121.0, 120.8,

120.1, 114.0, 113.9, 112.8, 110.8, 103.2, 55.3; ESI *m/z* (relative intensity) 250.1 (MH^+, 100%); HRMS Calcd for $C_{17}H_{16}NO$: 250.1232, Found: 250.1230.

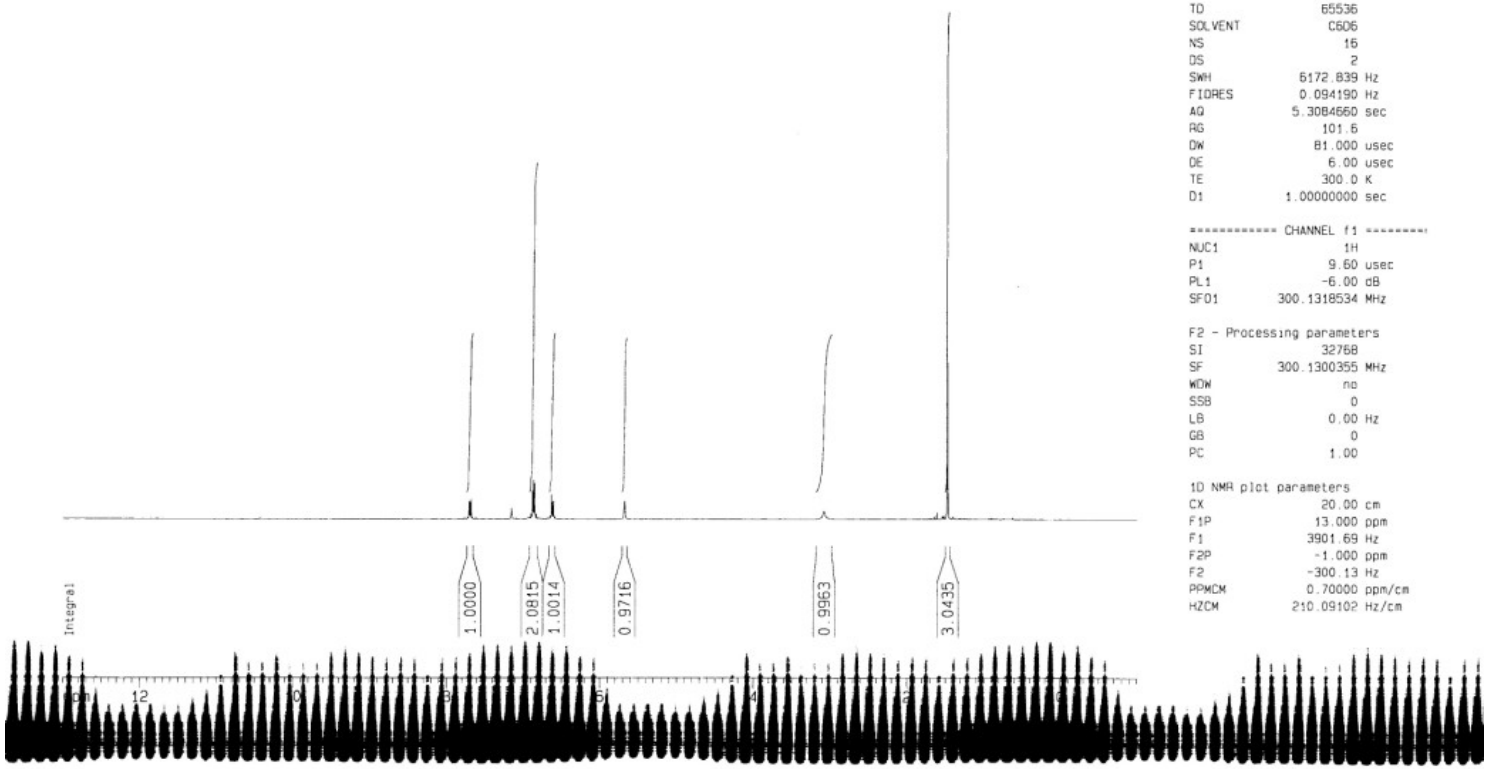
2-[1-(3-Fluoro-phenyl)-vinyl]-1H-indole (14m)



Following representative procedure (**6**) for crude thermolysis, propargyl acetate **9a** (82 mg, 0.36 mmol) was treated with 3-fluorophenylzinc iodide (0.50 M in THF, 1.1 mL, 0.54 mmol) to give compound **14m** (45 mg, 53%) as an orange solid: mp 74 - 75 °C; IR (neat): 3415 cm^{-1}; ^1H NMR (300 MHz, $(CD_3)_2CO$) δ 10.48 (br s, 1H), 7.52 (d, J = 7.9 Hz, 1H), 7.48-7.33 (m, 3H), 7.26 (d, J = 10.2 Hz, 1H), 7.21-7.11 (m, 2H), 7.01 (t, J = 7.5 Hz, 1H), 6.35 (s, 1H), 5.82 (s, 1H), 5.41 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.7 (d, J_{CF} = 244.7 Hz), 142.2 (d, J_{CF} = 7.6 Hz), 140.6 (d, J_{CF} = 2.2 Hz), 137.1, 136.5, 129.8 (d, J_{CF} = 8.2 Hz), 128.5, 124.2 (d, J_{CF} = 2.9 Hz), 122.9, 120.9, 120.2, 115.4 (d, J_{CF} = 25.6 Hz), 115.1 (d, J_{CF} = 24.7 Hz), 113.2, 110.8, 103.7; CI *m/z* (relative intensity) 238.0 (MH^+ 100%); HRMS Calcd for $C_{16}H_{13}NF$: 238.1032, Found: 238.1027.



DKHII-43 Gringard Product (lower Rf than S.M., yellow oil)

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PROCNO                  1

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FIDRES           0.094190 Hz
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DE                  6.00 usec
TE                 300.0 K
D1              1.00000000 sec

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PL1                 -6.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
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1D NMR plot parameters
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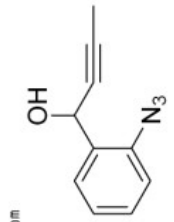
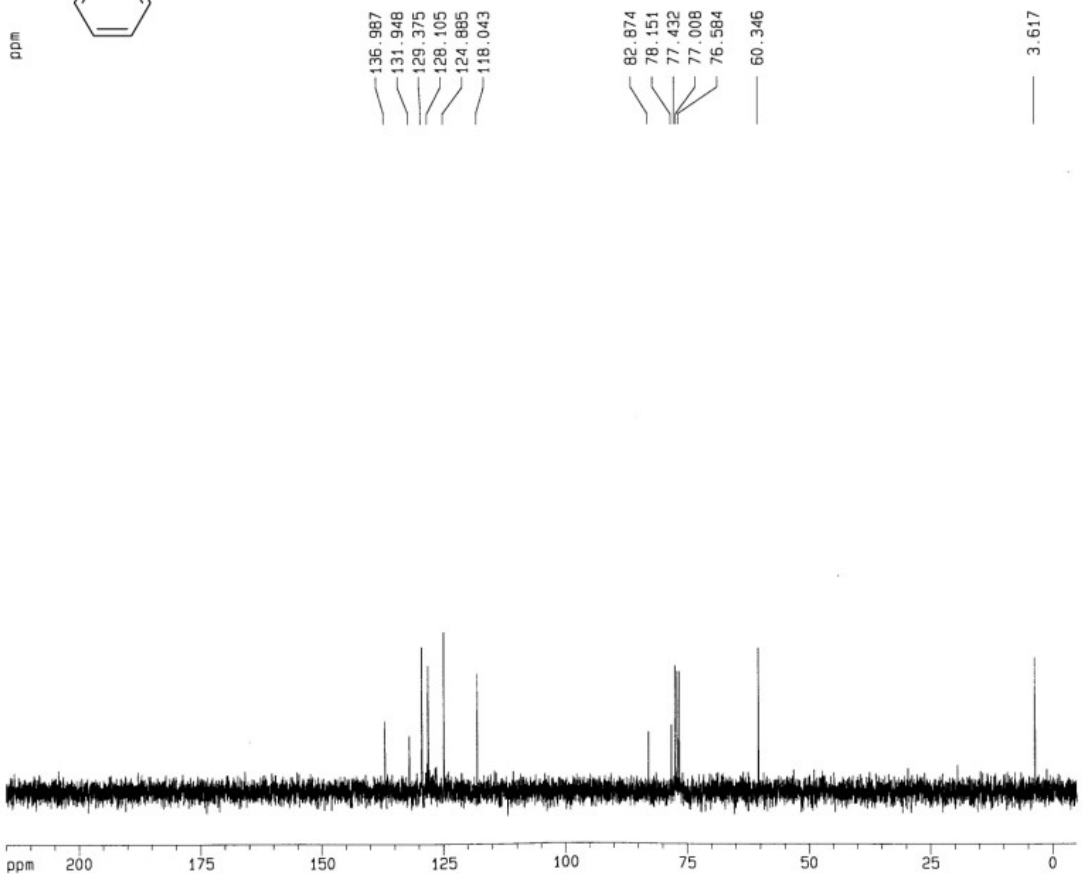
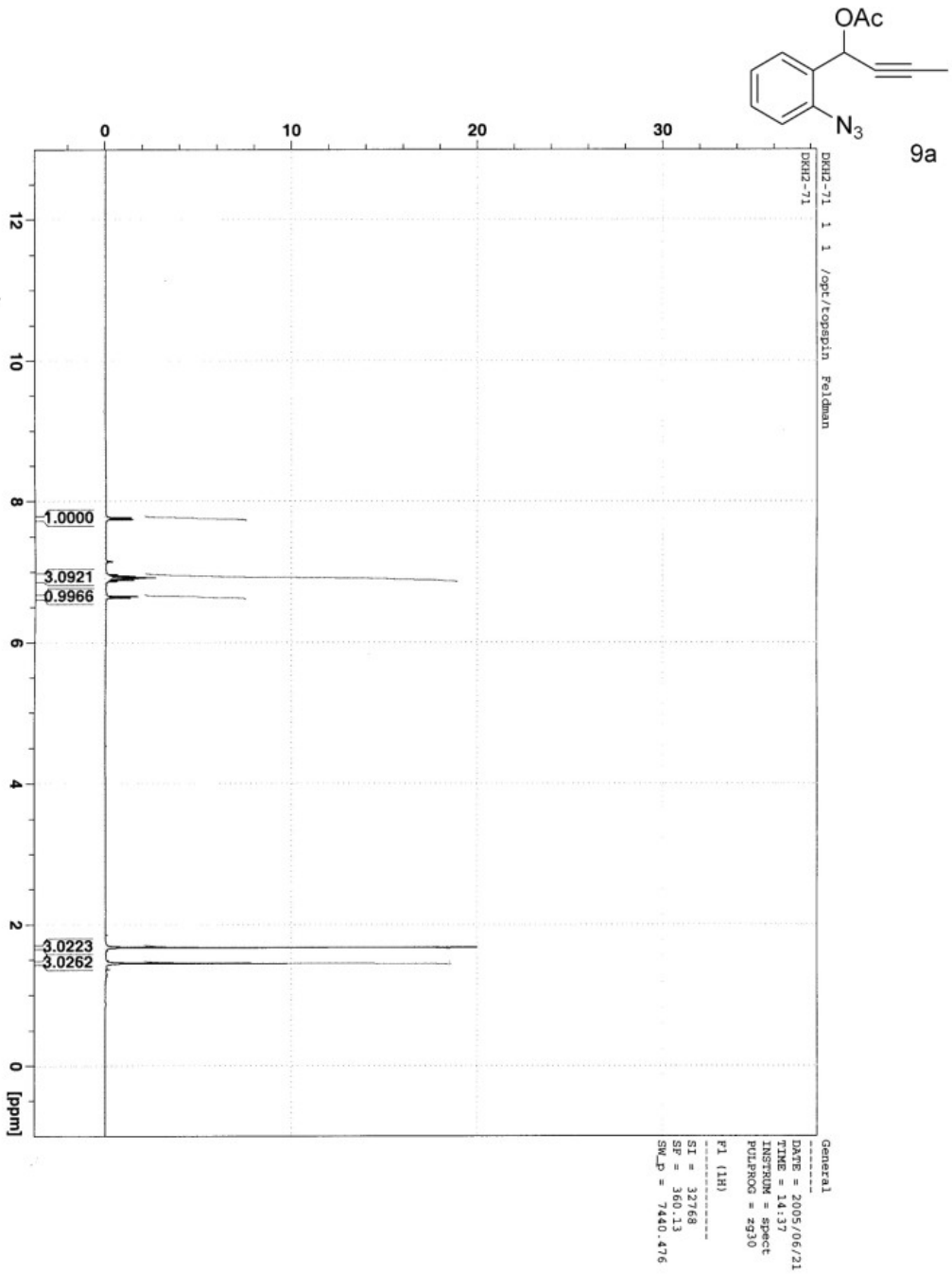
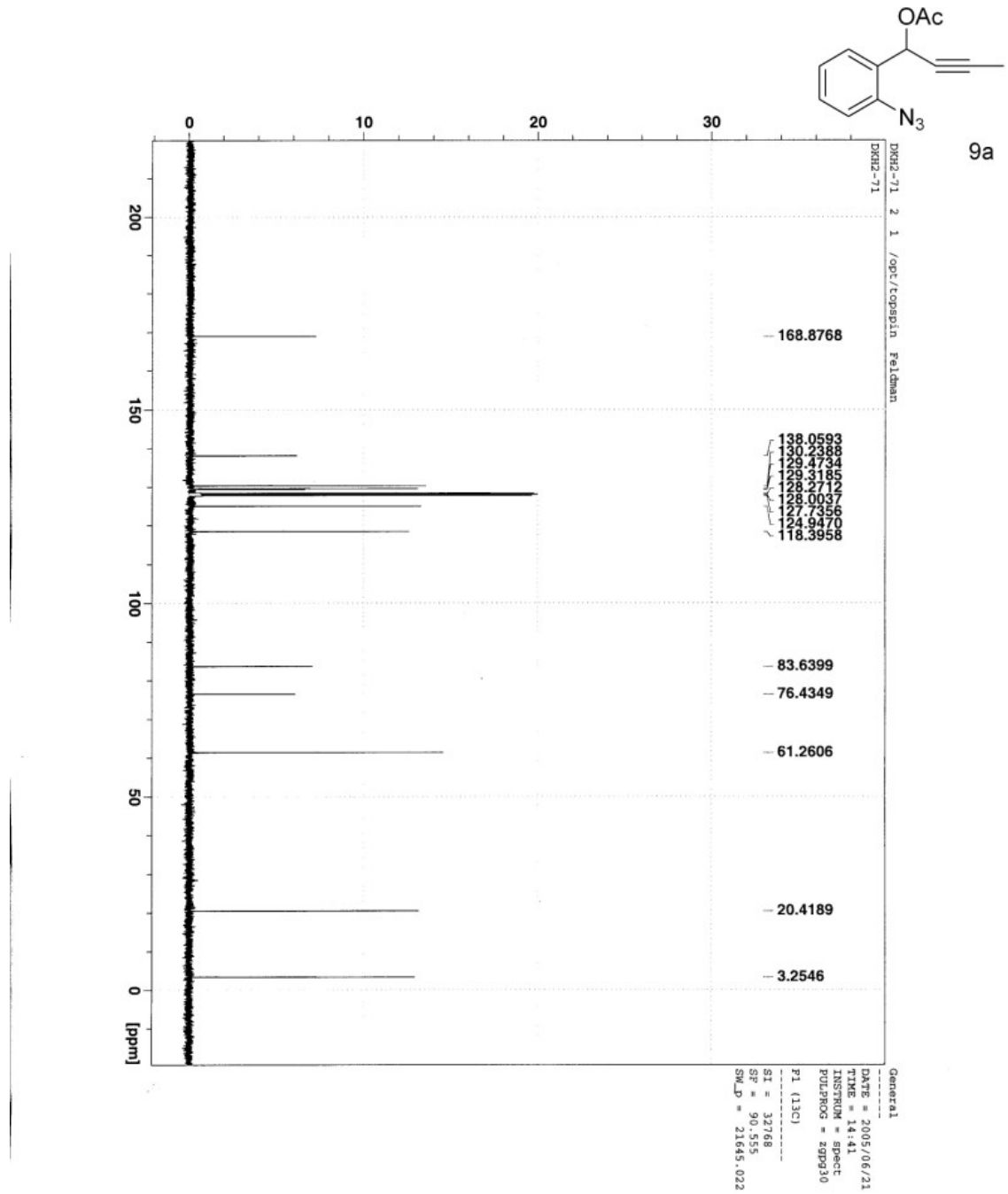


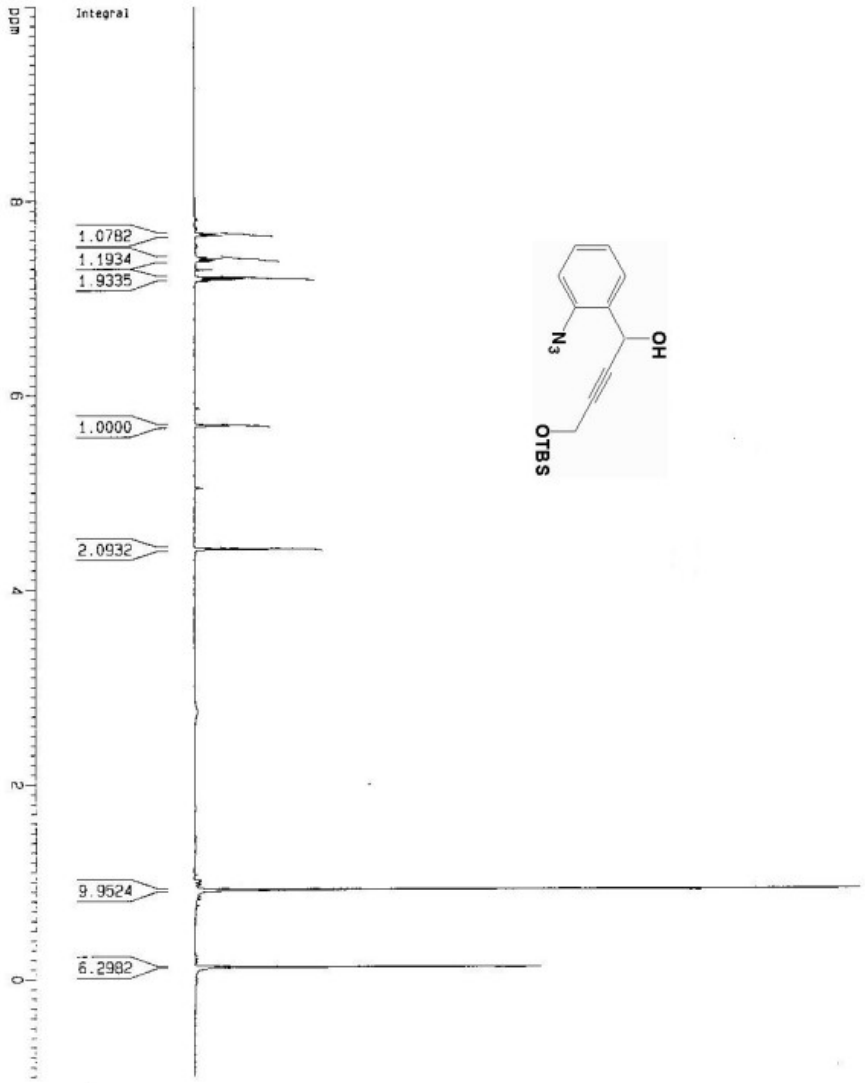
DKH2-43 Yellow Oil



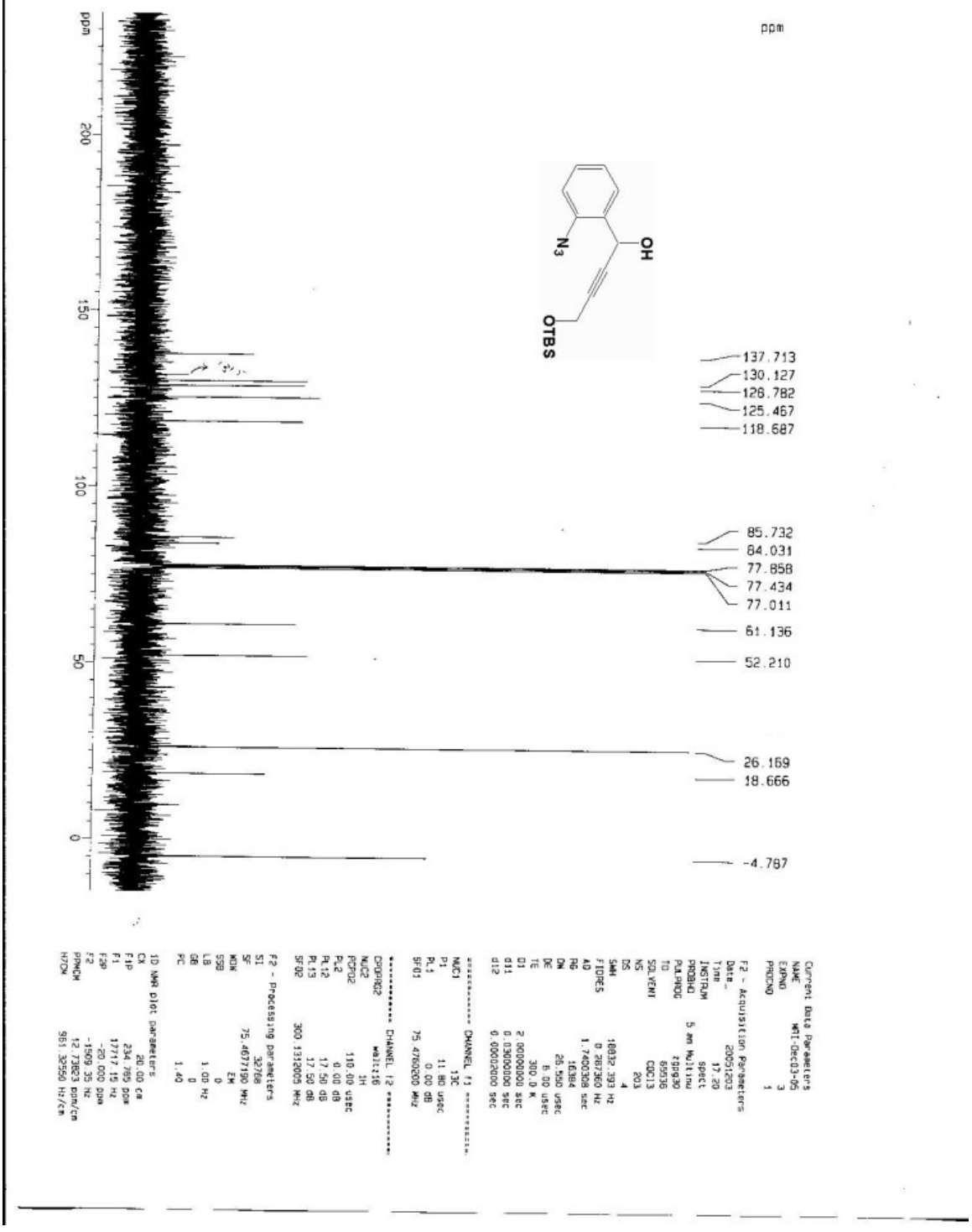

```
Current Data Parameters
NAME        DKH2-43Carbon
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20050527
Time              10.21
INSTRUM           spect
PROBHD     5 mm Multinu
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                   14
DS                    4
SWH           18832.393 Hz
FIDRES        0.287360 Hz
AQ            1.7400308 sec
RG                 8192
DW               26.550 usec
DE                6.00 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
d12           0.00002000 sec

============ CHANNEL f1 ============
NUC1                13C
P1                11.80 usec
PL1               0.00 dB
SF01         75.4760200 MHz

============ CHANNEL f2 ============
CPDPRG2          waltz16
NUC2                1H
PCPD2            110.00 usec
PL2               0.00 dB
PL12             17.50 dB
PL13             17.50 dB
SF02        300.1312005 MHz

F2 - Processing parameters
SI                 32768
SF           75.4677619 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
F1P              215.000 ppm
F1             16225.57 Hz
F2P               -5.000 ppm
F2              -377.34 Hz
PPMCM          11.00000 ppm/cm
HZCM          830.14532 Hz/cm
```

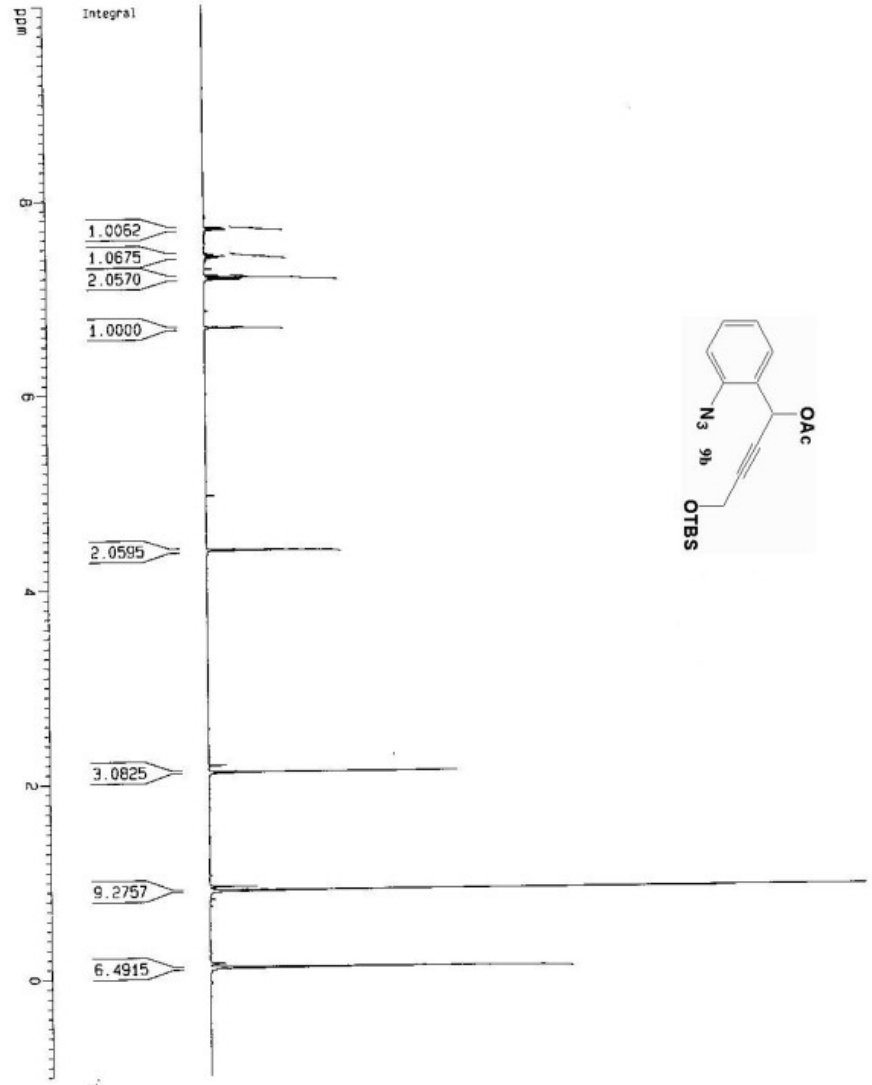

OAc
N3
9a
0
10
20
30
12
10
8
6
4
2
0
[ppm]
1.0000
3.0921
0.9966
3.0223
3.0262
DRH2-71 1 1 /opt/topspin Feldman
DRH2-71
General
DATE = 2005/06/21
TIME = 14:37
INSTRUM = spect
PULPROG = zg30
F1 (1H)
SI = 32768
SF = 360.13
SW_p = 7440.476


OAc
N3
9a
0
10
20
30
200
150
100
50
0
[ppm]
168.8768
138.0593
130.2388
129.4734
129.3185
128.2712
128.0037
127.7356
124.9470
118.3958
83.6399
76.4349
61.2606
20.4189
3.2546
DKH2-71
DKH2-71 2 1 /opt/topspin Feldman
General
DATE = 2005/06/21
TIME = 14:41
INSTRUM = spect
PULPROG = zgpg30
F1 (13C)
SI = 32768
SF = 90.555
SW_p = 21645.022

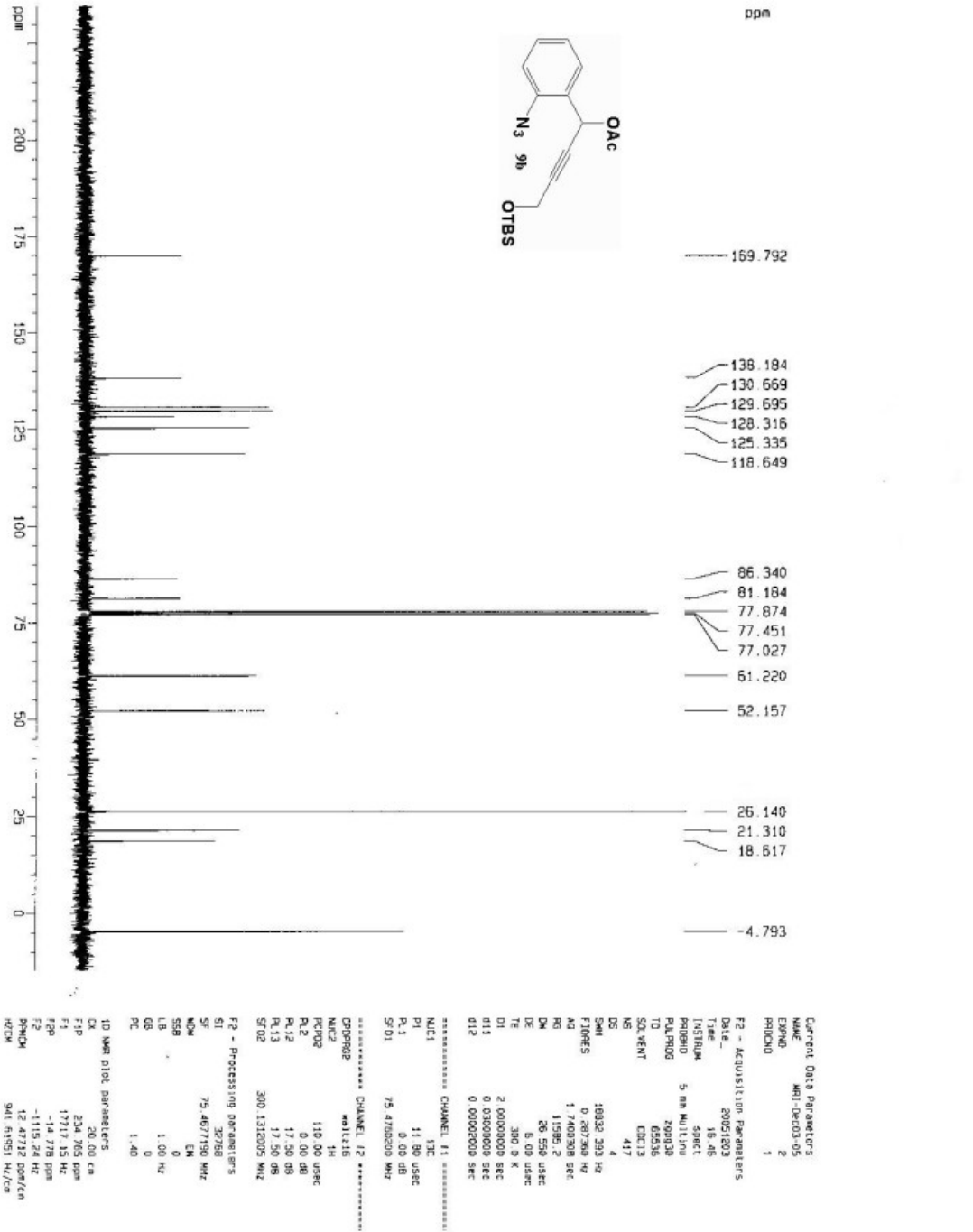


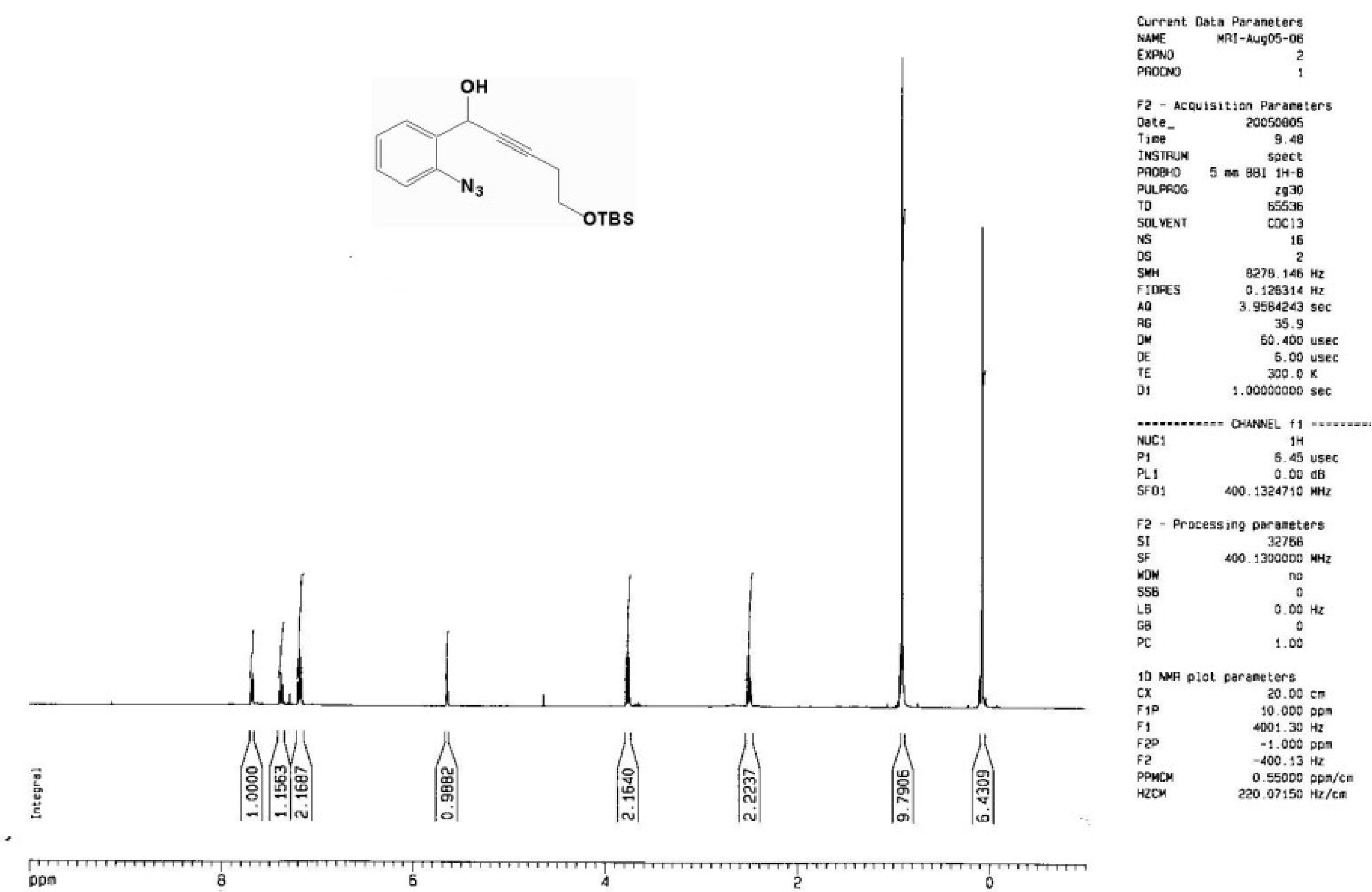

```
Current Data Parameters
NAME                  MA1-Dec03-05
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_               20051203
Time                      16.45
INSTRUM                   spect
PROBHD     5 mm DBI 1H-B
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                 8278.146 Hz
FIDRES              0.126314 Hz
AQ                  3.9584243 sec
RG                         512
DW                      60.400 usec
DE                       6.00 usec
TE                      300.0 K
D1                  1.00000000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                       6.45 usec
PL1                      0.00 dB
SFO1             400.1324710 MHz

F2 - Processing parameters
SI                       32768
SF               400.1300000 MHz
WDW                         no
SSB                          0
LB                        0.00 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       20.00 cm
F1P                     10.000 ppm
F1                    4001.30 Hz
F2P                     -1.000 ppm
F2                    -400.13 Hz
PPMCM                 0.55000 ppm/cm
HZCM                220.07150 Hz/cm
```

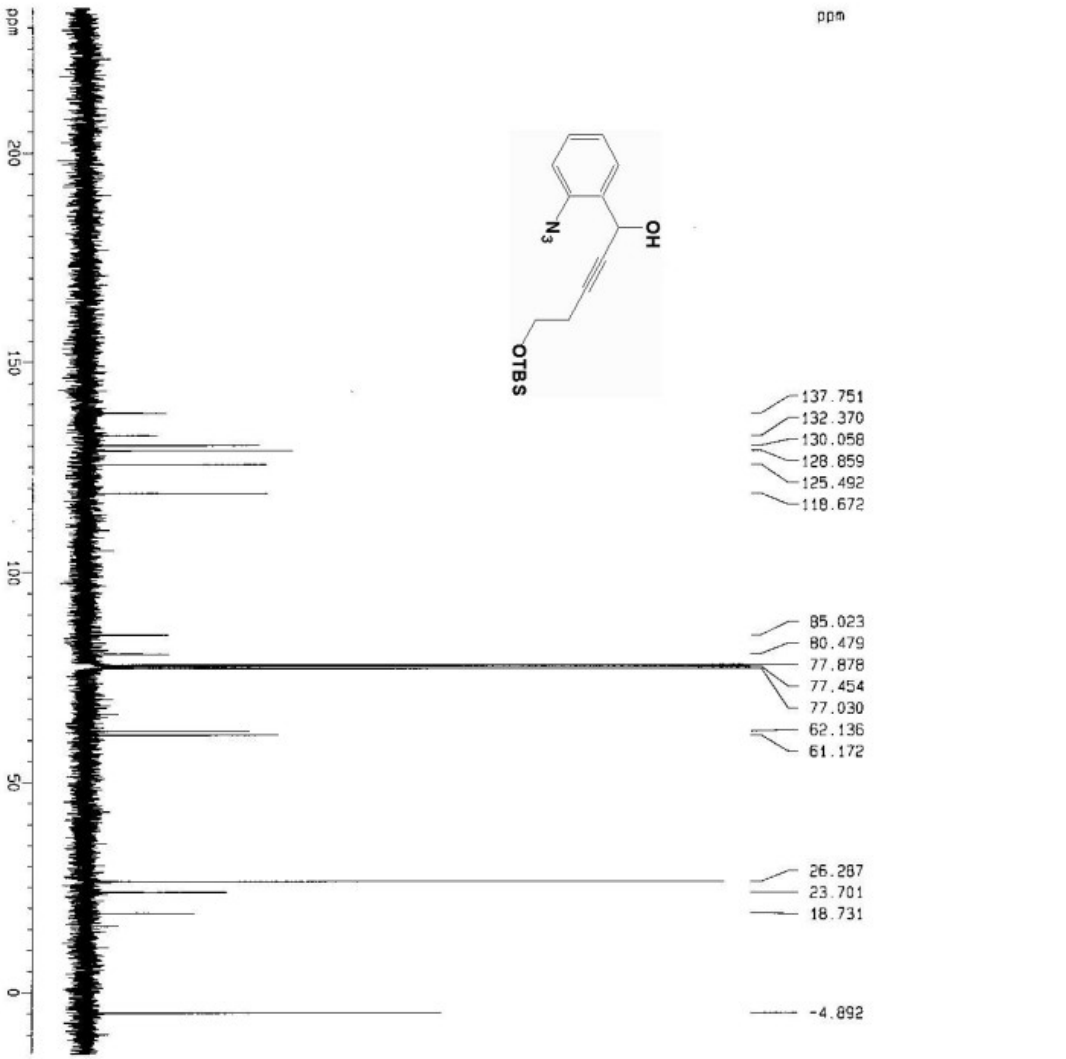

ppm
200
150
100
50
0
N3
OH
OTBS
137.713
130.127
128.782
125.467
118.687
85.732
84.031
77.858
77.434
77.011
61.136
52.210
26.169
18.666
-4.787




```
Current Data Parameters
NAME                  HRT-Dec05-05
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_               20051205
Time                      11.46
INSTRUM                    spect
PROBHD     5 mm BBI 1H-B
PULPROG                   zg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                 8278.146 Hz
FIDRES                 0.126314 Hz
AQ                  3.9584243 sec
RG                          256
DW                       60.400 usec
DE                        6.00 usec
TE                       300.0 K
D1                  1.00000000 sec
======== CHANNEL f1 ========
NUC1                        1H
P1                       6.45 usec
PL1                      0.00 dB
SFO1               400.1324710 MHz

F2 - Processing parameters
SI                        32768
SF                 400.1300000 MHz
WDW                          no
SSB                           0
LB                         0.00 Hz
GB                            0
PC                         1.00

1D NMR plot parameters
CX                       20.00 cm
F1P                     10.000 ppm
F1                    4001.30 Hz
F2P                     -1.000 ppm
F2                    -400.13 Hz
PPMCM                  0.55000 ppm/cm
HZCM                 220.07150 Hz/cm
```


ppm
OAc
N3
9b
OTBS
169.792
138.184
130.669
129.695
128.316
125.335
118.649
86.340
81.184
77.874
77.451
77.027
61.220
52.157
26.140
21.310
18.617
-4.793
Current Data Parameters
NAME MRI-Dec03-05
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051203
Time 19.46
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 417
DS 4
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7403308 sec
RG 11585.2
DW 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 11.80 usec
PL1 0.00 dB
SFO1 75.4750200 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 110.00 usec
PL2 0.00 dB
PL12 17.50 dB
PL13 17.50 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 234.765 ppm
F1 17717.15 Hz
F2P -14.778 ppm
F2 -1115.24 Hz
PPMCM 12.47712 ppm/cm
HZCM 941.51951 Hz/cm


OH
N_3
OTBS
Integral
1.0000
1.1563
2.1687
0.9882
2.1640
2.2237
9.7906
6.4309
ppm
8
6
4
2
0
Current Data Parameters
NAME MRI-Aug05-06
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050805
Time 9.48
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 35.9
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
============ CHANNEL f1 ========
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm



Current Data Parameters
NAME MTI-Dec03-05
EXPNO 1
PROCNO 1

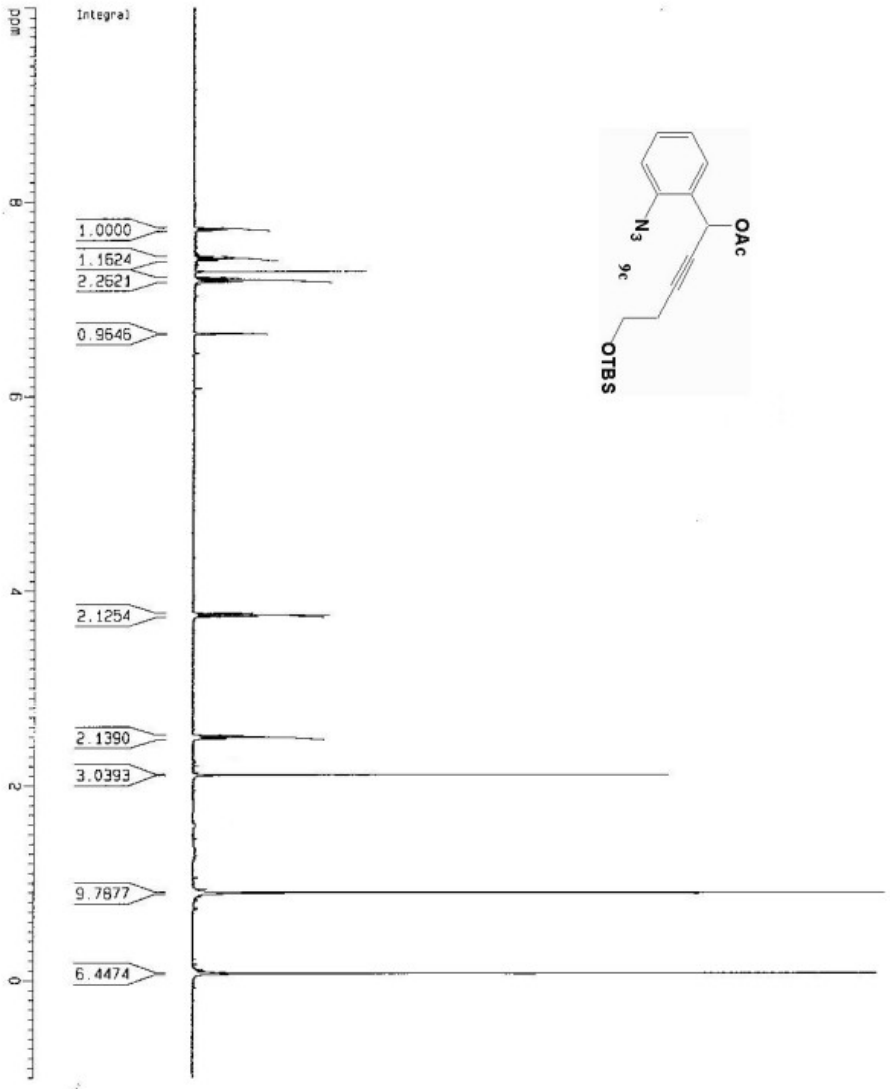
F2 - Acquisition Parameters
Date_ 20051203
Time 17.44
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 147
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AQ 1.7433075 sec
RG 256
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00005000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SFO1 75.4103397 MHz

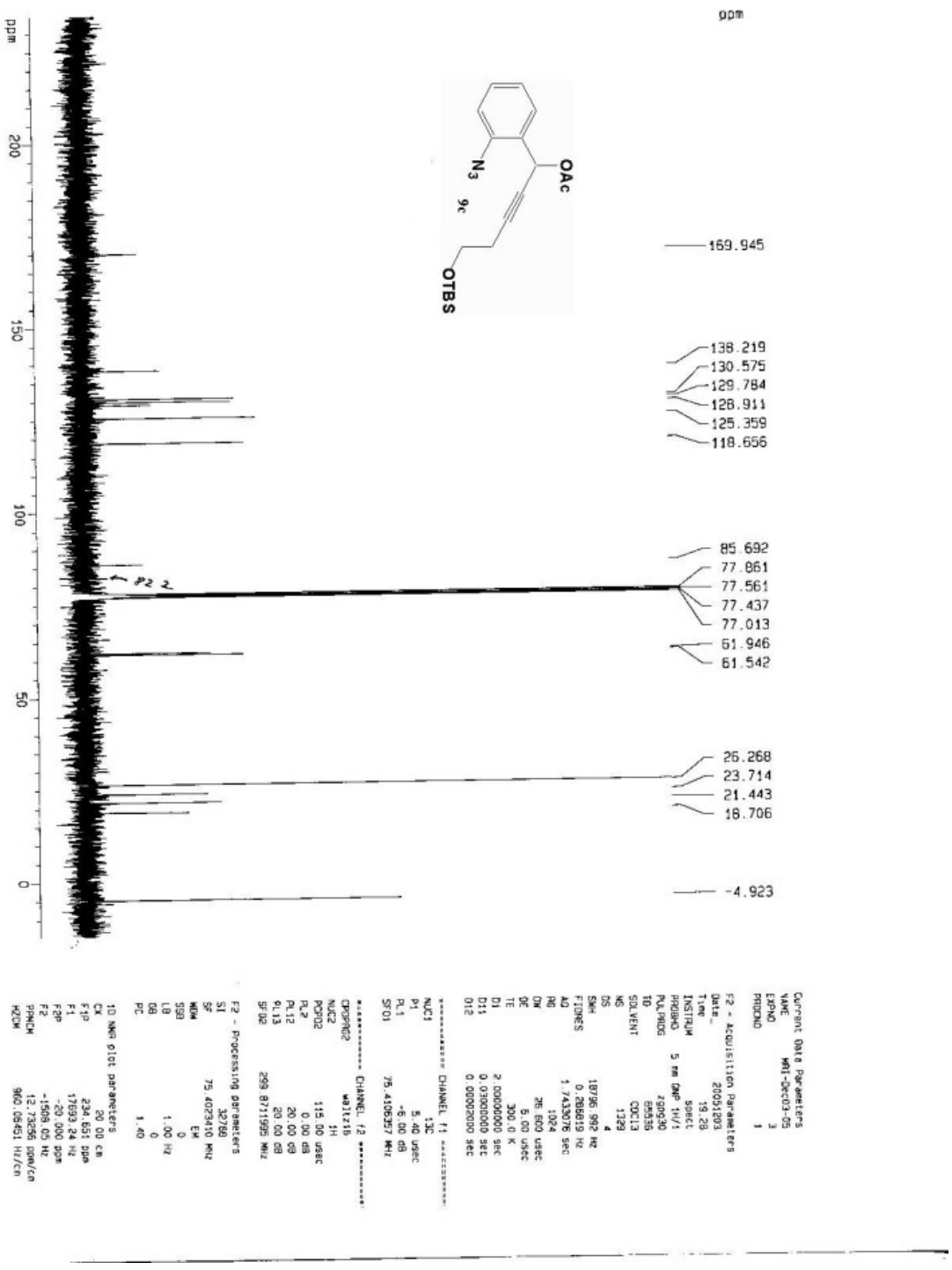
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711995 MHz

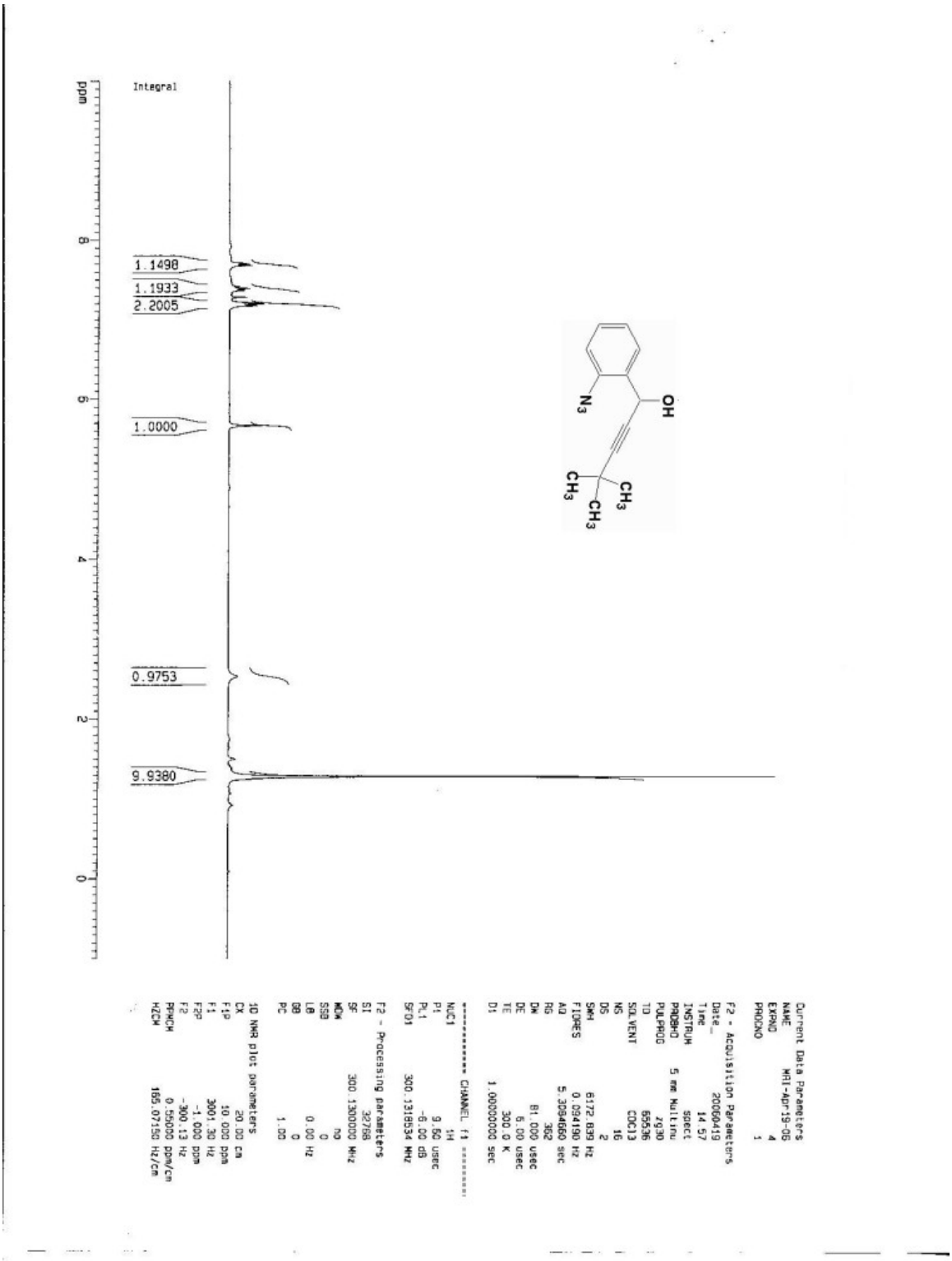
F2 - Processing parameters
SI 32768
SF 75.4623410 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 234.551 ppm
F1 17693.24 Hz
F2P -20.000 ppm
F2 -1508.05 Hz
PPMCM 12.73255 ppm/cm
HZCM 960.06451 Hz/cm




```
Current Data Parameters
NAME                  MRI-Dec03-05
EXPNO                           2
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20051203
Time                        16.51
INSTRUM                      spect
PROBHD     5 mm BBI 1H-D
PULPROG                     z930
TD                         65536
SOLVENT                    CDCl3
NS                              9
DS                              2
SWH                     8278.146 Hz
FIDRES                  0.126314 Hz
AQ                      3.9584243 sec
RG                         1624.6
DW                         60.400 usec
DE                          6.00 usec
TE                          300.0 K
D1                      1.00000000 sec

======== CHANNEL f1 ========
NUC1                          1H
P1                          6.45 usec
PL1                         0.00 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                         32768
SF                400.1300000 MHz
WDW                           no
SSB                            0
LB                          0.00 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                         20.00 cm
F1P                     10.000 ppm
F1                      4001.30 Hz
F2P                     -1.000 ppm
F2                      -400.13 Hz
PPMCM                  0.55000 ppm/cm
HZCM                   220.07150 Hz/cm
```


ppm
200
150
100
50
0
OAc
N3
9c
OTBS
169.945
138.219
130.575
129.784
128.911
125.359
118.656
85.692
77.861
77.561
77.437
77.013
61.946
61.542
26.268
23.714
21.443
18.706
-4.923



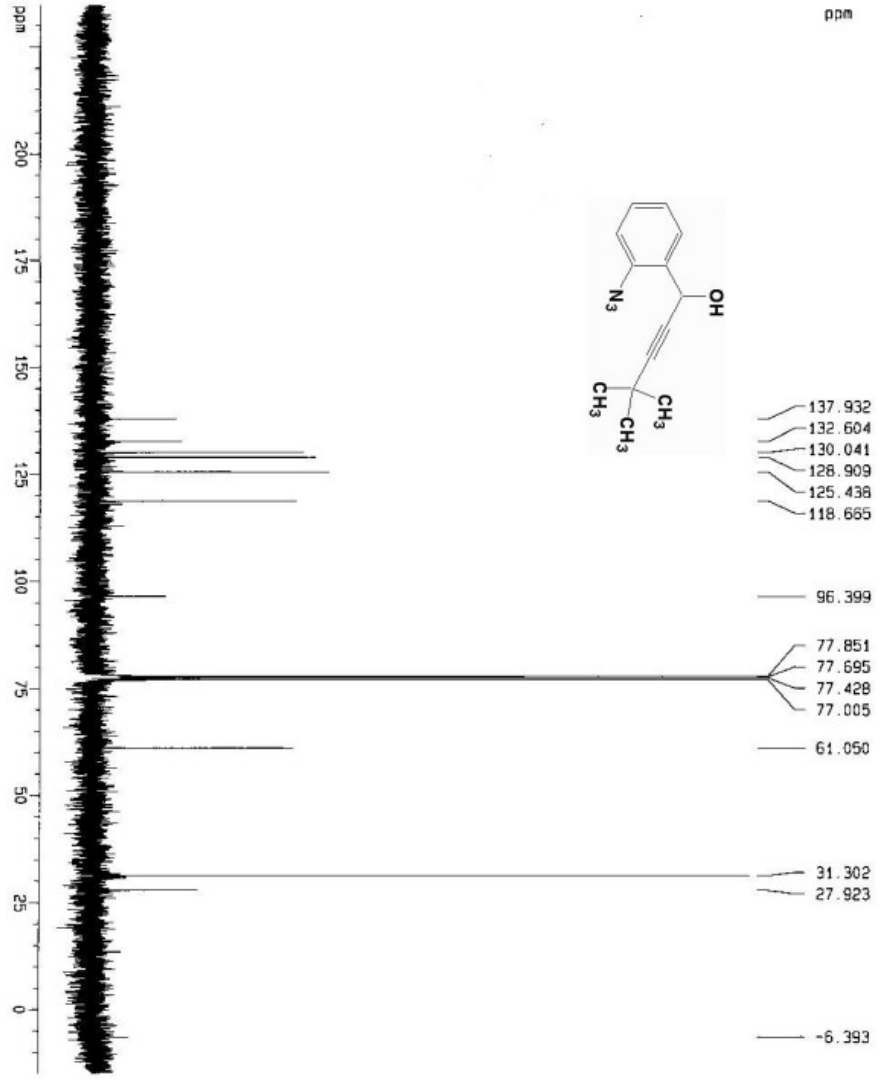
Current Data Parameters
NAME MRI-Apr19-05
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060419
Time 14.57
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 362
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

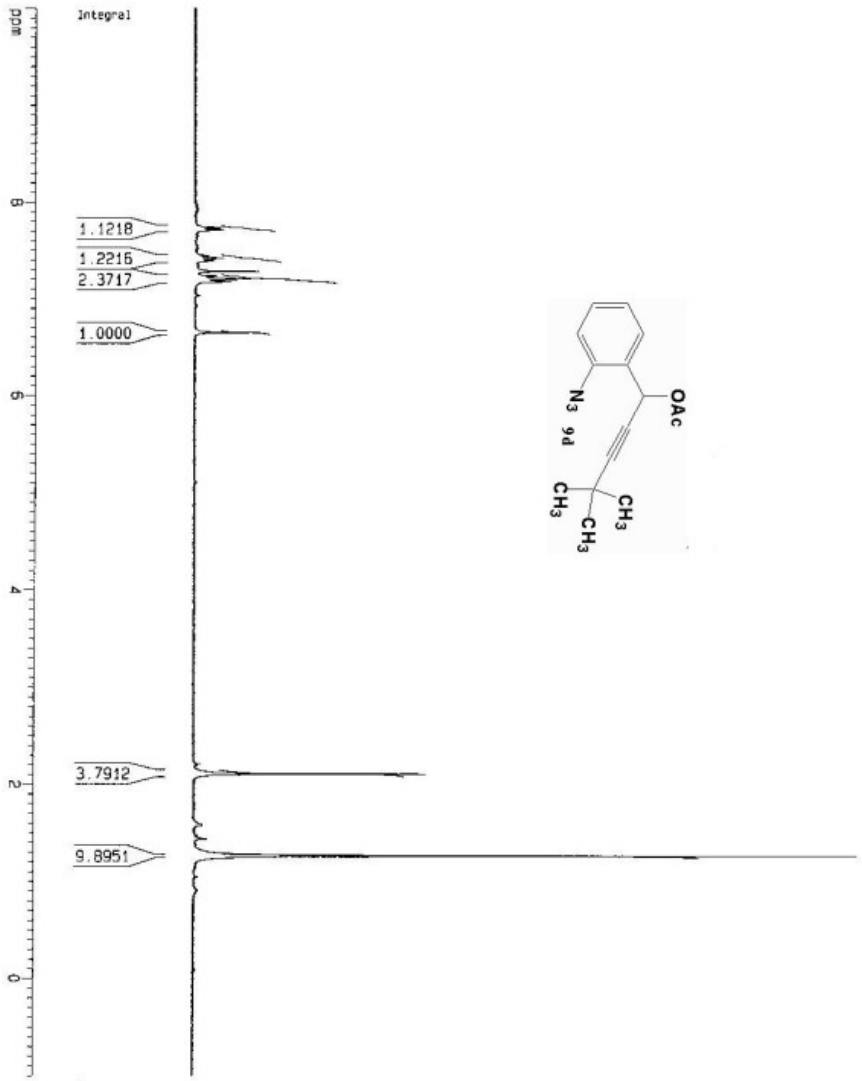
======== CHANNEL f1 ========
NUC1 1H
P1 9.60 usec
PL1 -6.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

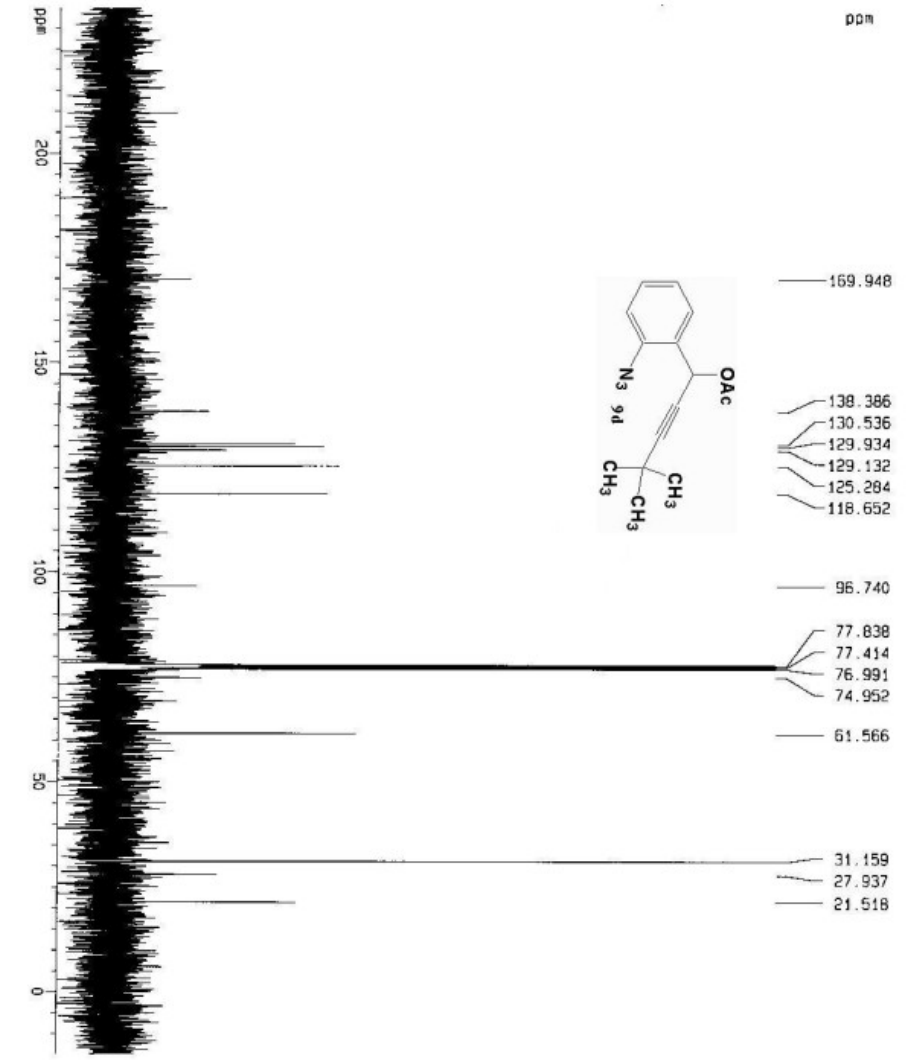
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 165.07150 Hz/cm

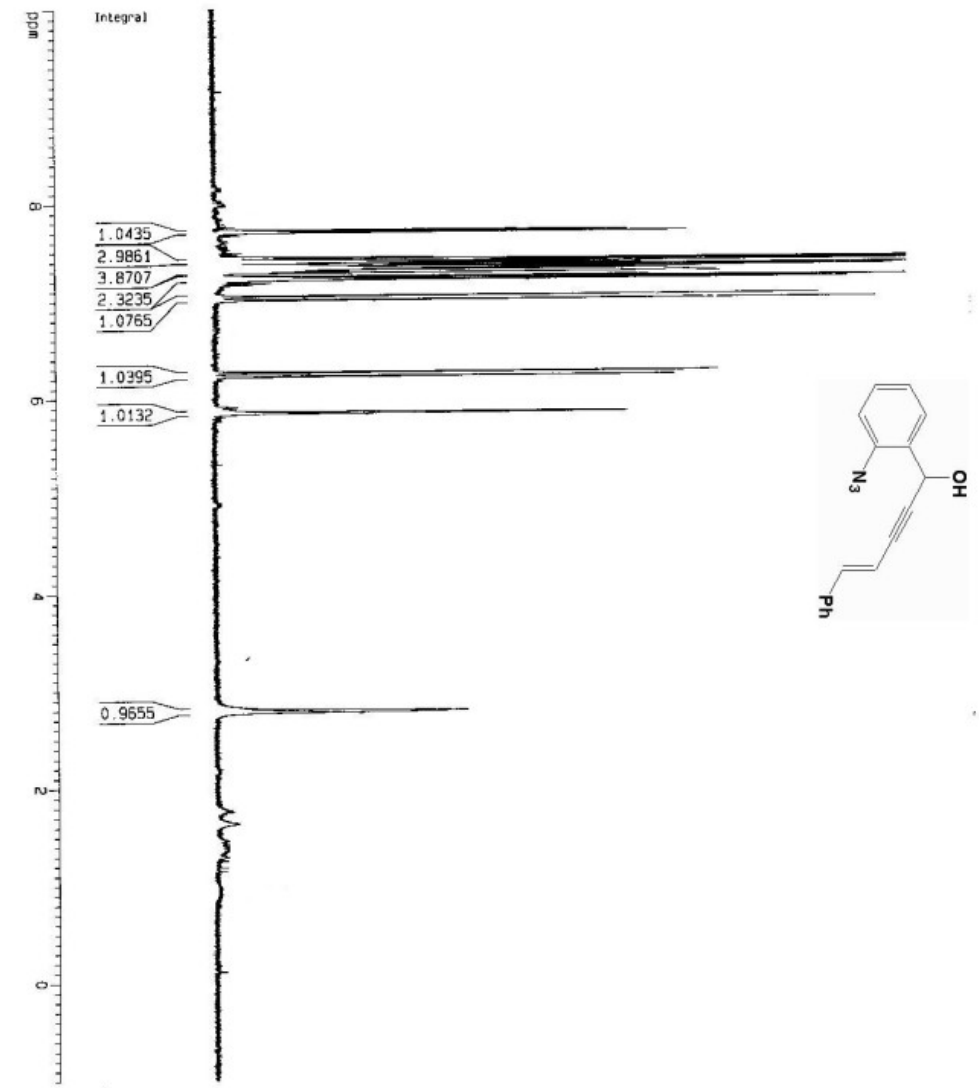



```
Current Data Parameters
NAME                  MR1-Apr19-06
EXPNO                          5
PROCNO                         1

F2 - Acquisition Parameters
Date_              20060419
Time                      15.04
INSTRUM                   spect
PROBHD     5 mm Multinu
PULPROG                   zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                          311
DS                            4
SWH               18832.393 Hz
FIDRES             0.287360 Hz
AQ                 1.7400308 sec
RG                      1586.2
DW                   26.550 usec
DE                    6.00 usec
TE                   300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
D12                0.00002000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                      11.80 usec
PL1                     0.00 dB
SFO1              75.4760200 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                      1H
PCPD2                110.00 usec
PL2                     0.00 dB
PL12                   17.50 dB
PL13                   17.50 dB
SFO2              300.1312005 MHz

F2 - Processing parameters
SI                        32768
SF                75.4677190 MHz
WDW                          EM
SSB                           0
LB                     1.00 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                       20.00 cm
F1P                     234.765 ppm
F1                    17717.16 Hz
F2P                    -14.778 ppm
F2                    -1115.23 Hz
PPMCM                 12.47712 ppm/cm
HZCM                  941.61951 Hz/cm
```

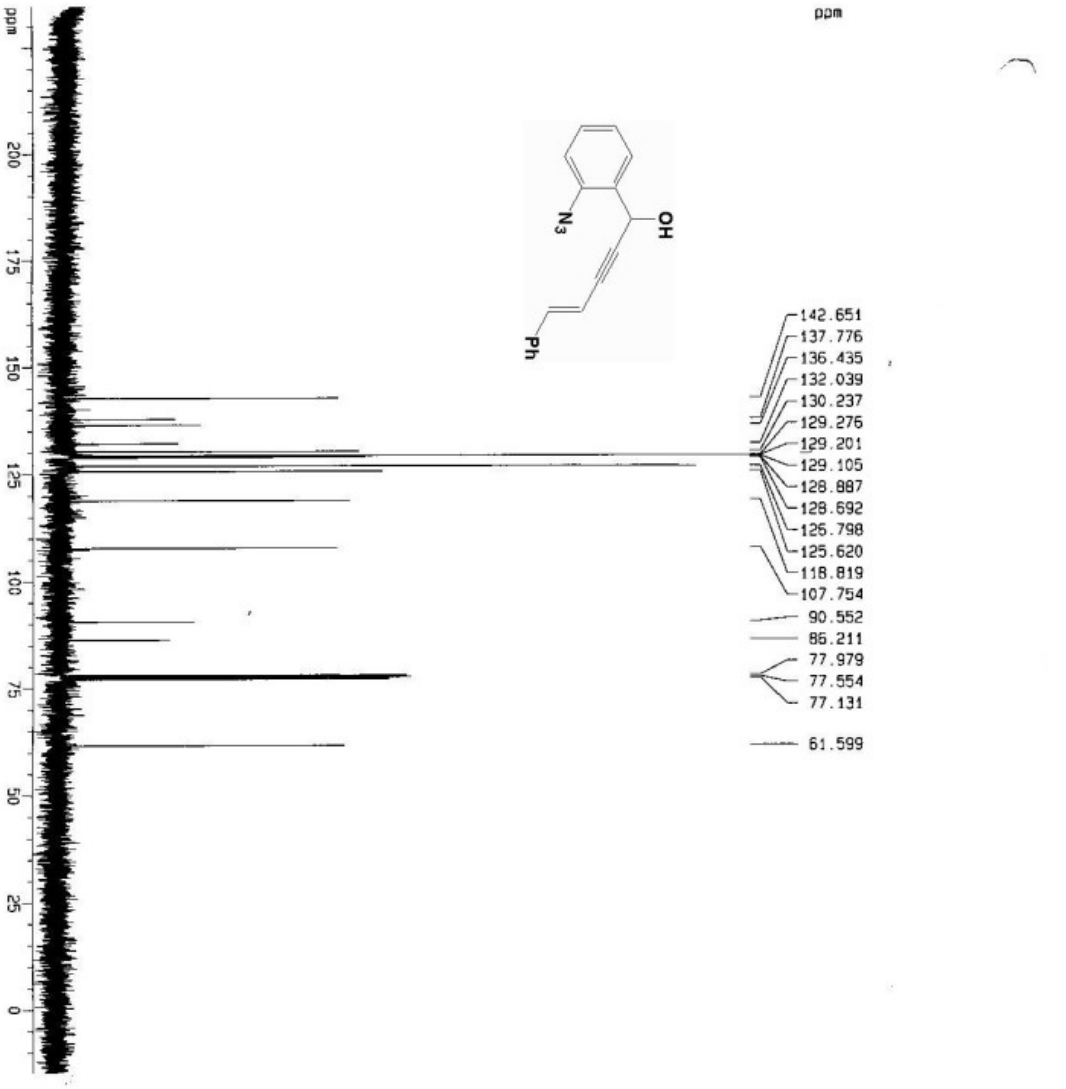



```
Current Data Parameters
NAME                  MRT-Apr20-06
EXPNO                           2
PROCNO                          1

F2 - Acquisition Parameters
Date_              20050420
Time                      16.09
INSTRUM                   spect
PROBHD     5 mm Multinu  z030
PULPROG                   zg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                  6172.839 Hz
FIDRES               0.094190 Hz
AQ                   5.3084660 sec
RG                          512
DW                   81.000 usec
DE                    6.00 usec
TE                      300.0 K
D1                   1.00000000 sec

======== CHANNEL f1 ========
NUC1                        1H
P1                        9.50 usec
PL1                     -6.00 dB
SFO1          300.1318534 MHz

F2 - Processing parameters
SI                        32768
SF            300.1300000 MHz
WDW                        no
SSB                         0
LB                       0.00 Hz
GB                          0
PC                        1.00

1D NMR plot parameters
CX                       20.00 cm
F1P                   10.000 ppm
F1                   3001.30 Hz
F2P                   -1.000 ppm
F2                    -300.13 Hz
PPMCM                 0.55000 ppm/cm
HZCM                165.07150 Hz/cm
```


ppm
200
150
100
50
0
169.948
138.386
130.536
129.934
129.132
125.284
118.652
96.740
77.838
77.414
76.991
74.952
61.566
31.159
27.937
21.518
OAc
N3
9d
CH3
CH3
CH3




```
Current Data Parameters
NAME                  MEI-Dec05-05
EXPNO                           3
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20051205
Time                        12.01
INSTRUM                      spect
PROBHD     5 mm BBI 1H-B
PULPROG                      zg30
TD                          65536
SOLVENT                     CDCl3
NS                             12
DS                              2
SWH                    8278.146 Hz
FIDRES                 0.126314 Hz
AQ                     3.9584243 sec
RG                         812.7
DW                        60.400 usec
DE                         6.00 usec
TE                         300.0 K
D1                     1.00000000 sec

======== CHANNEL f1 ========
NUC1                         1H
P1                         6.45 usec
PL1                        0.00 dB
SF01           400.1324710 MHz

F2 - Processing parameters
SI                         32768
SF             400.1300000 MHz
WDW                           no
SSB                            0
LB                      0.00 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                        20.00 cm
FIP                   10.000 ppm
F1                    4001.30 Hz
F2P                   -1.000 ppm
F2                    -400.13 Hz
PPMCM                 0.55000 ppm/cm
HZCM                  220.07150 Hz/cm
```



Current Data Parameters
NAME MR1-Sept19-05
EXPNO 4
PROCNO 1

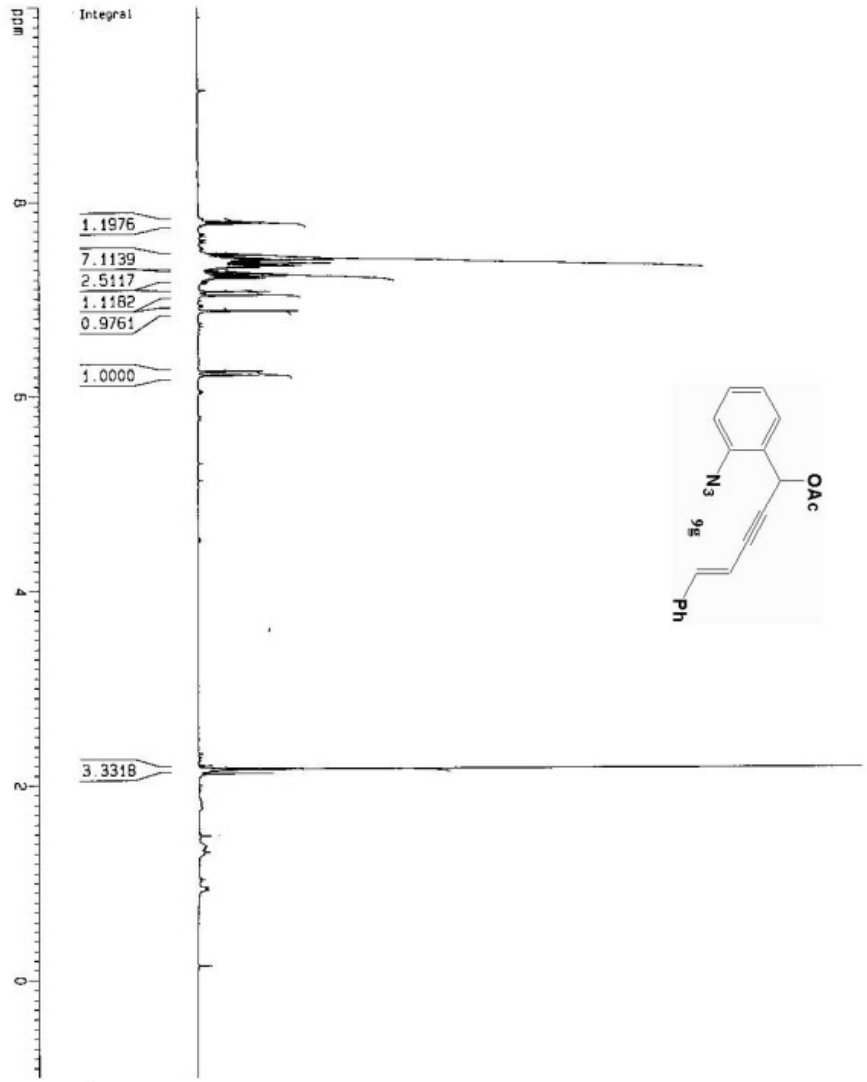
F2 - Acquisition Parameters
Date_ 20050919
Time 17.13
INSTRUM spect
PROBHD 5 mm DNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 37
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AQ 1.7433076 sec
RG 1024
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SFO1 75.4105957 MHz

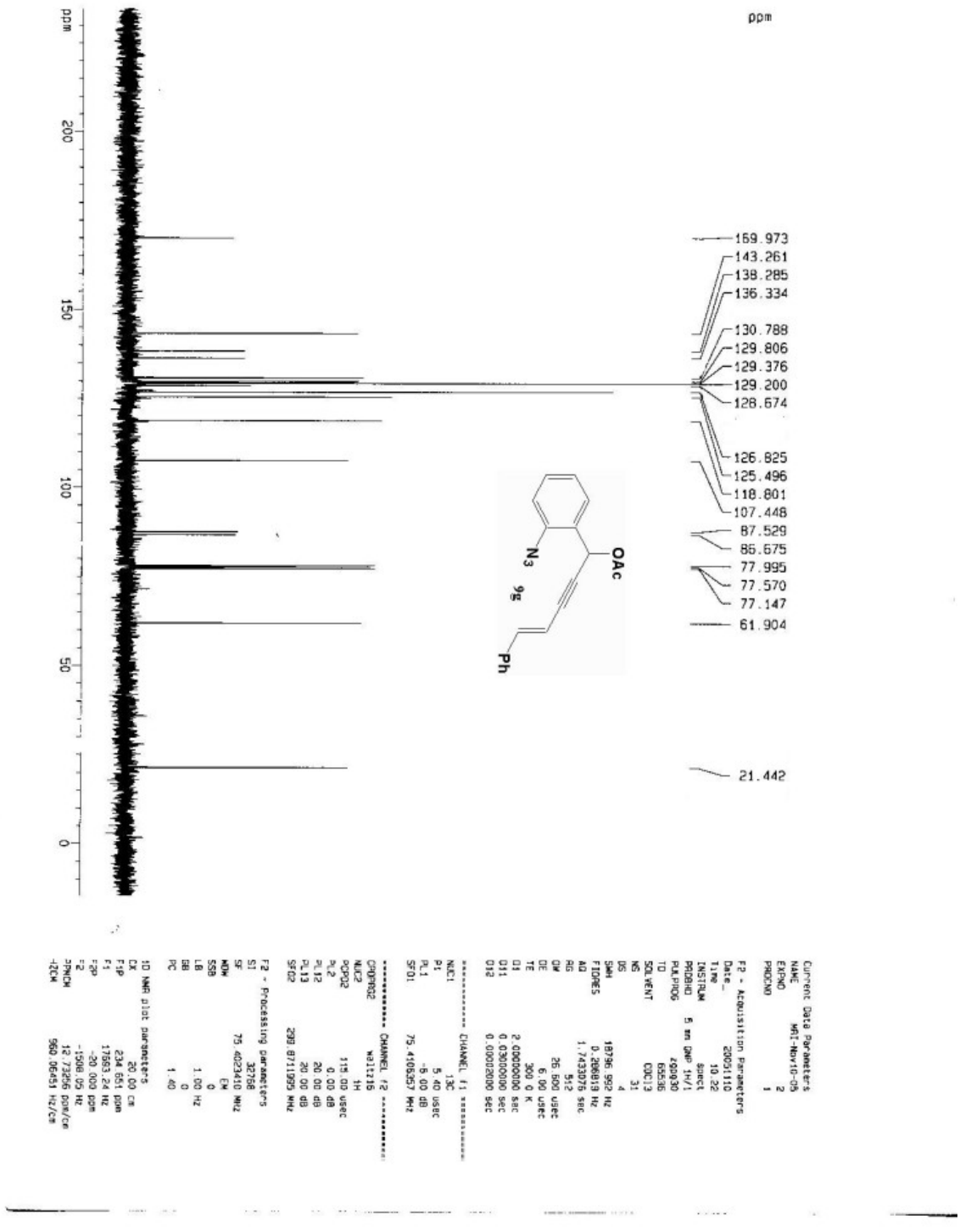
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.6711955 MHz

F2 - Processing parameters
SI 32768
SF 75.4623410 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 234.651 ppm
F1 17693.24 Hz
F2P -154.038 ppm
F2 -11613.75 Hz
PPMCM 12.45446 ppm/cm
HZCM 939.64967 Hz/cm




```
Current Data Parameters
NAME                  WAT-Nov10-05
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_              20051110
Time                   10.10
INSTRUM                spect
PROBHD     5 mm BBI 1H-B
PULPROG                zg30
TD                     65536
SOLVENT                CDCl3
NS                         7
DS                         2
SWH                8278.146 Hz
FIDRES             0.126314 Hz
AQ                 3.9584243 sec
RG                       181
DW                  60.400 usec
DE                   6.00 usec
TE                    300.0 K
D1                 1.00000000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                       6.45 usec
PL1                      0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                       32768
SF            400.1300000 MHz
WDW                       no
SSB                        0
LB                      0.00 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                     20.00 cm
F1P                10.000 ppm
F1                 4001.30 Hz
F2P                -1.000 ppm
F2                 -400.13 Hz
PPMCM              0.55000 ppm/cm
HZCM               220.07150 Hz/cm
```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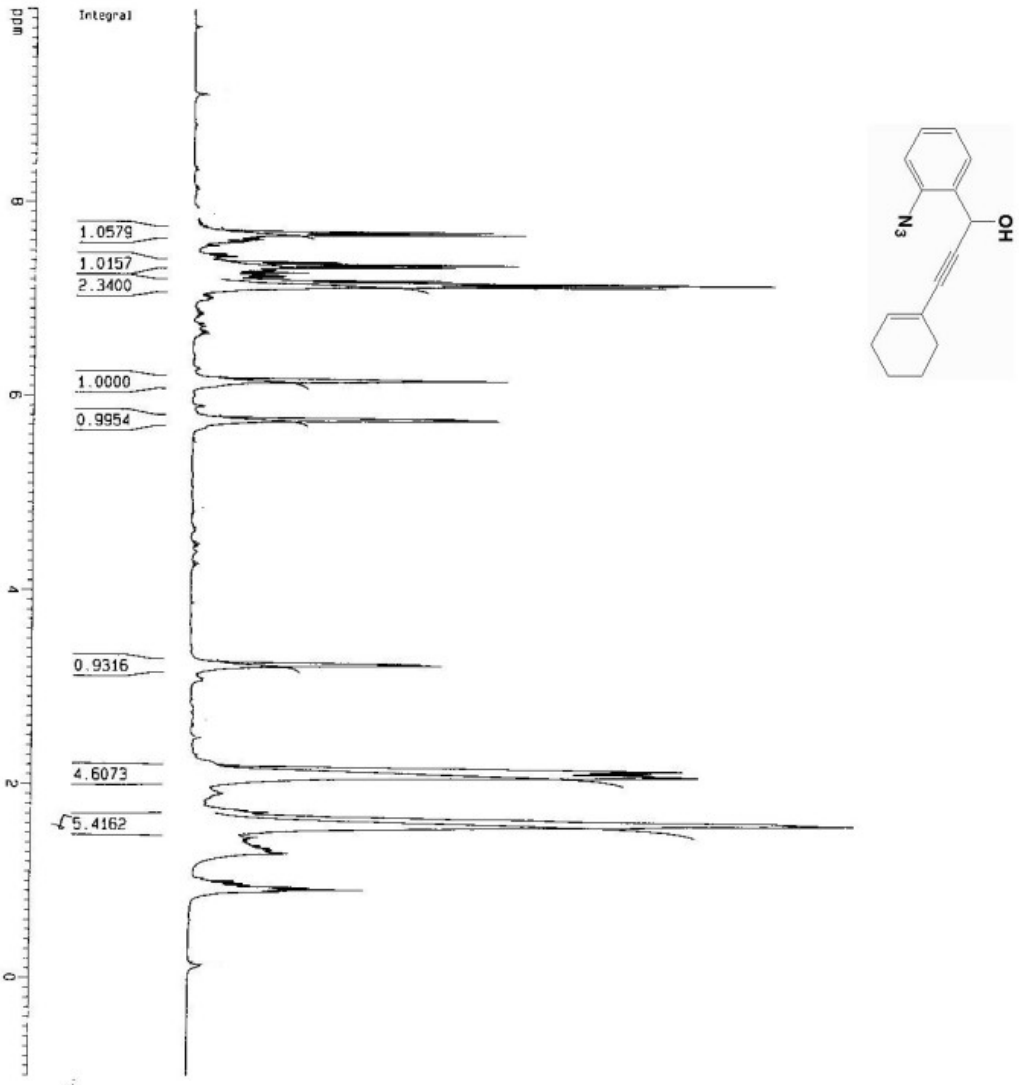
143.261
138.285
136.334
130.788
129.806
129.376
129.200
128.674
126.825
125.496
118.801
107.448
87.529
86.675
77.995
77.570
77.147
61.904
21.442
N3
OAc
Ph
9g



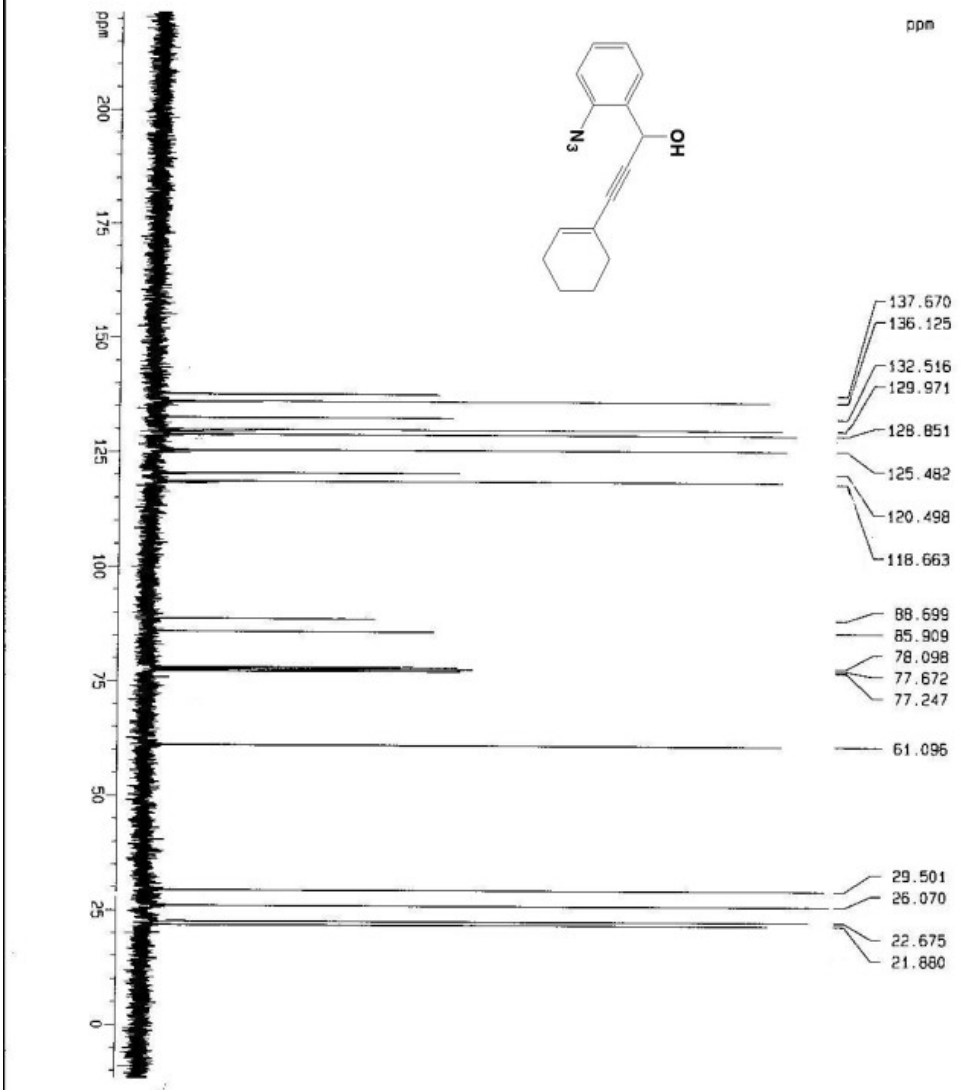
Current Data Parameters
NAME MR1-Sept19-05
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080919
Time 16.51
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 5172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3094660 sec
RG 35.9
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SFO1 299.5718518 MHz

F2 - Processing parameters
SI 32768
SF 299.8700000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 2998.70 Hz
F2P -1.000 ppm
F2 -299.87 Hz
PPMCM 0.50000 ppm/cm
HZCM 164.92850 Hz/cm



Current Data Parameters
NAME MEI-Sept19-05
EXPNO 2
PROCNO 1

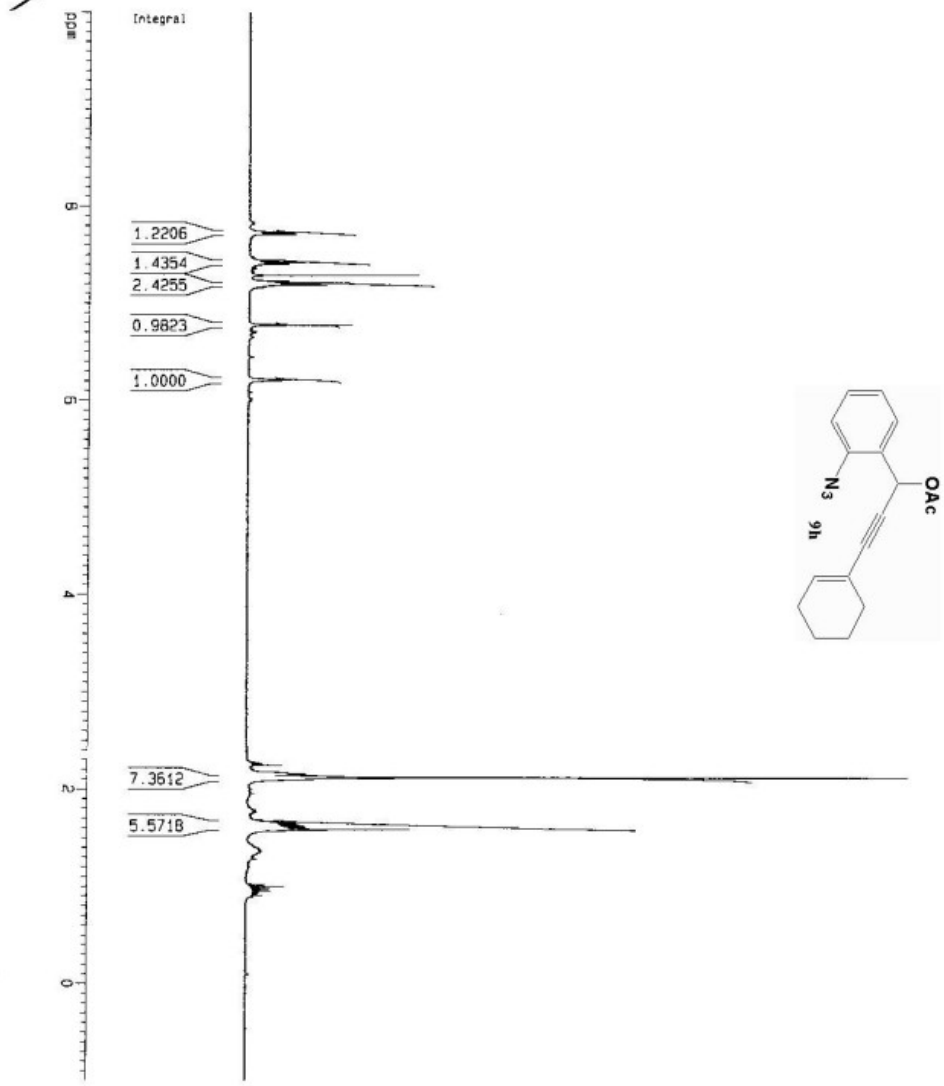
F2 - Acquisition Parameters
Date_ 20050919
Time 16.59
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgdp30
TD 65536
SOLVENT CDCl3
NS 43
DS 4
SWH 18796.982 Hz
FIDRES 0.286819 Hz
AQ 1.7423078 sec
RG 1024
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

====== CHANNEL f1 ======
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SFO1 75.4106357 MHz

====== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711995 MHz

F2 - Processing parameters
SI 32768
SF 75.4023410 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
FIXP 221.444 ppm
F1 16697.38 Hz
F2P -11.611 ppm
F2 -875.53 Hz
PPMCM 11.85276 ppm/cm
HZCM 878.64539 Hz/cm




```
Current Data Parameters
NAME                  MRI-Apr21-05
EXPNO                           4
PROCNO                          1

F2 - Acquisition Parameters
Date_              20060421
Time                    10.30
INSTRUM                 spect
PROBHD     5 mm BBI 1H-D
PULPROG                 zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                 8278.146 Hz
FIDRES              0.126314 Hz
AQ                  3.9584243 sec
RG                     1149.4
DW                    60.400 usec
DE                     6.00 usec
TE                     300.0 K
D1                  1.00000000 sec

========== CHANNEL f1 ==========
NUC1                       1H
P1                      6.45 usec
PL1                     0.00 dB
SF01        400.1324710 MHz

F2 - Processing parameters
SI                        32768
SF          400.1300000 MHz
WDW                       no
SSB                        0
LB                      0.00 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                     20.00 cm
F1P                  10.000 ppm
F1                 4001.30 Hz
F2P                  -1.000 ppm
F2                 -400.13 Hz
PPMCM              0.55000 ppm/cm
HZCM             220.07149 Hz/cm
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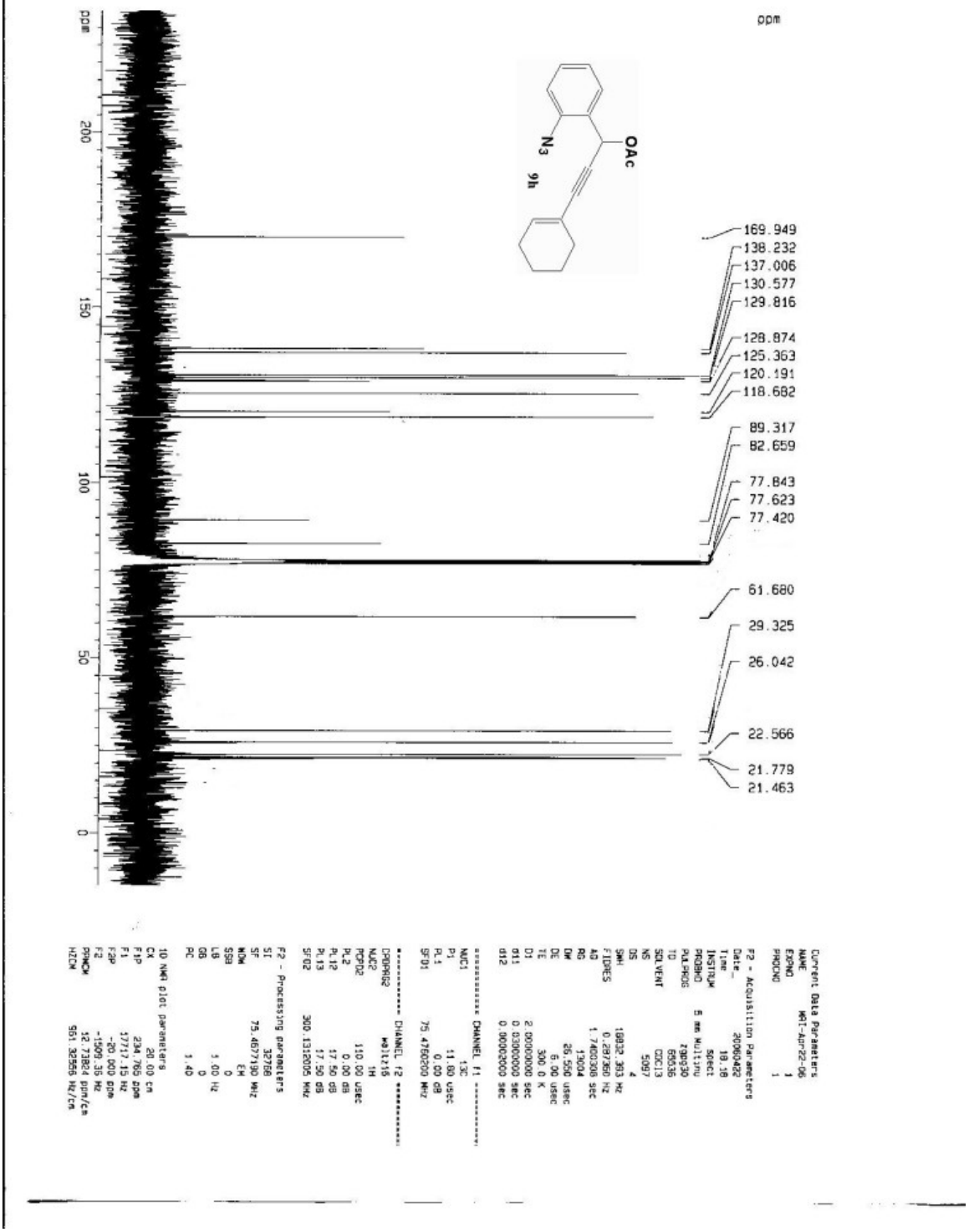


Current Data Parameters
NAME MOI-Apr22-06
EXPNO 1
PROCNO 1

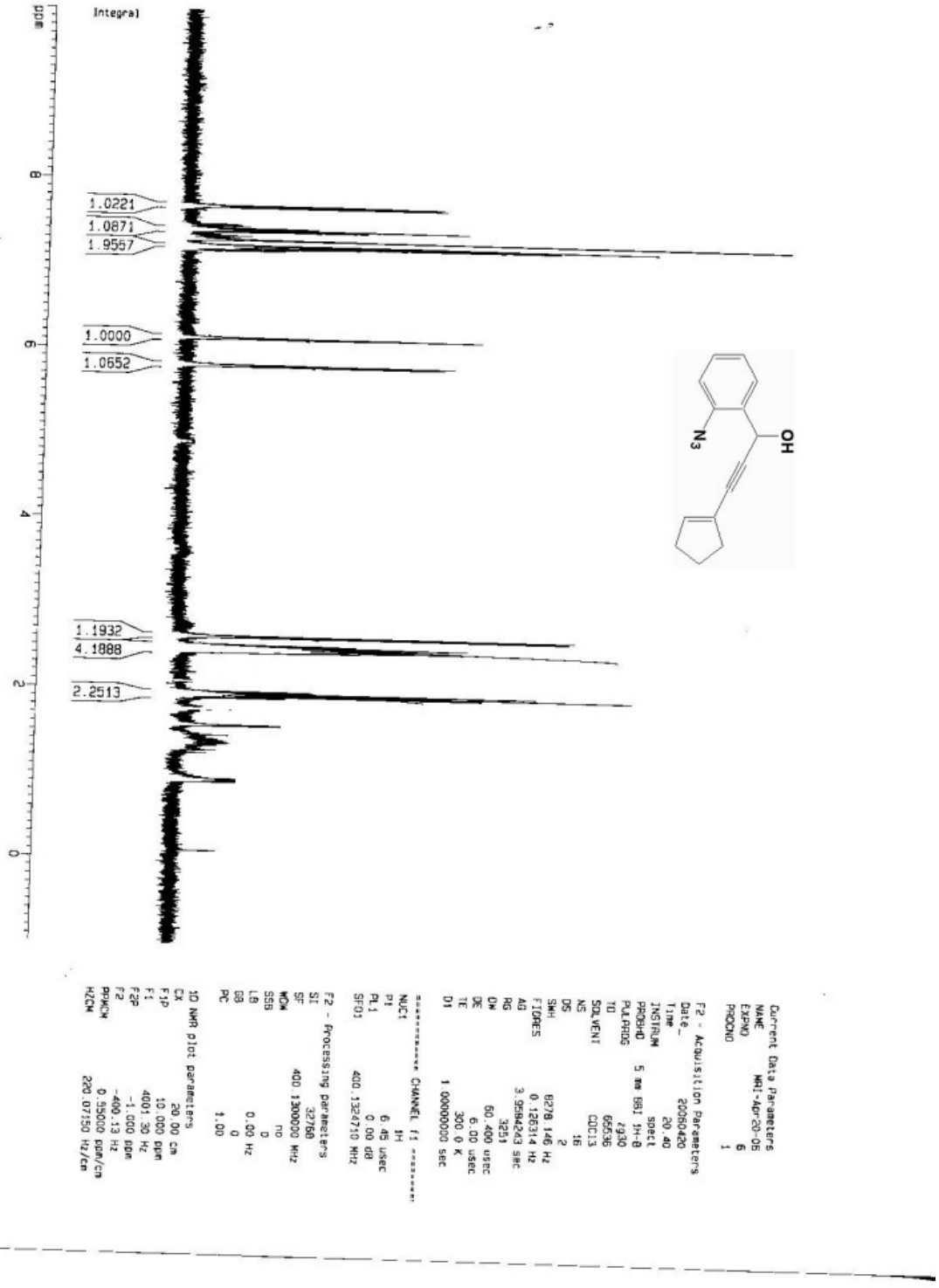
F2 - Acquisition Parameters
Date_ 20060422
Time 18.16
INSTRM spect
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 5097
DS 4
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 13004
DW 26.556 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

====== CHANNEL f1 ======
NUC1 13C
P1 11.00 usec
PL1 0.00 dB
SF01 75.4760200 MHz

====== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 110.00 usec
PL2 0.00 dB
PL12 17.50 dB
PL13 17.50 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4577190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
F1P 234.765 ppm
F1 17717.15 Hz
F2P -20.000 ppm
F2 -1509.35 Hz
PPMCM 12.73824 ppm/cm
HZCM 961.32556 Hz/cm



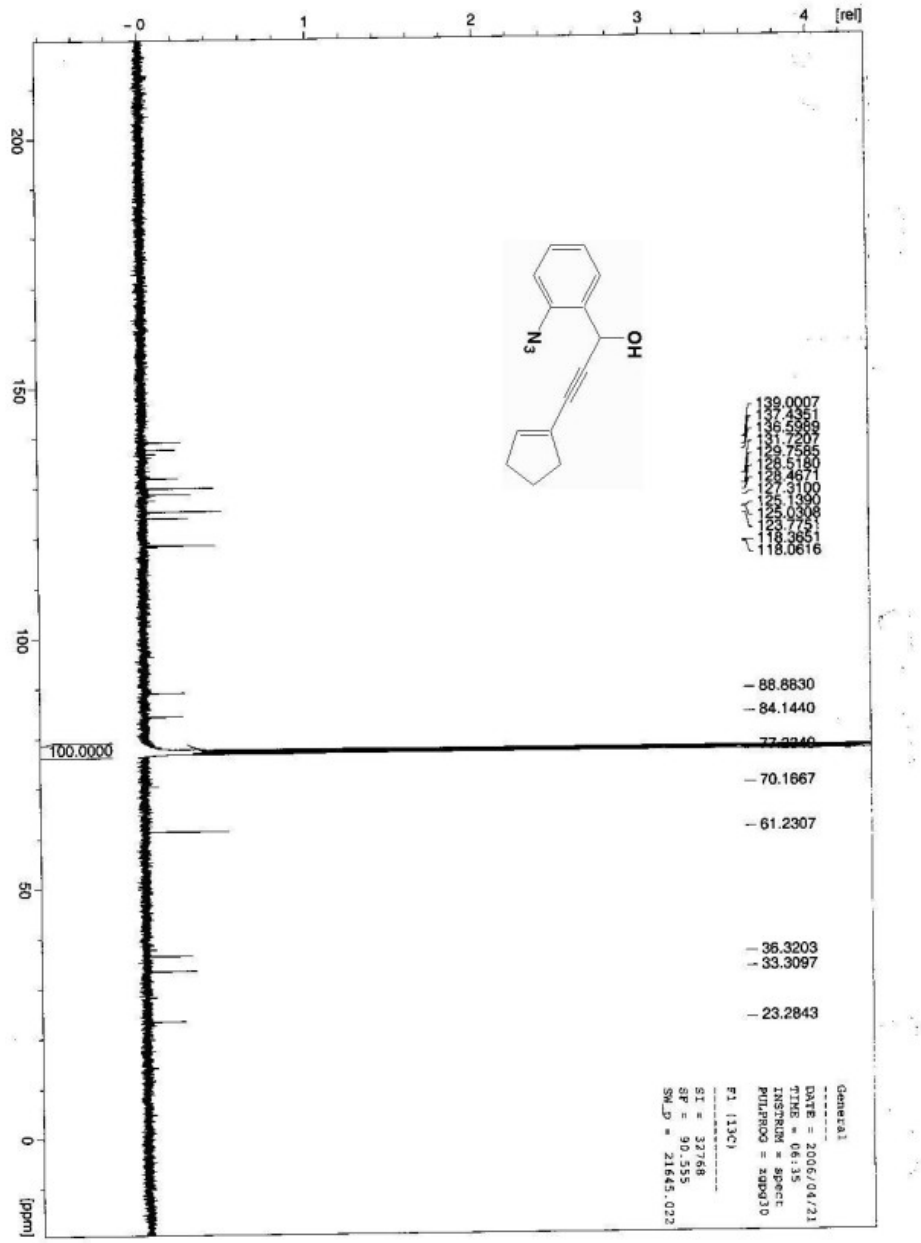
Current Data Parameters
NAME MRI-Apr20-05
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060420
Time 20.40
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 3251
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SF01 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm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[ppm]
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136.5989
131.7207
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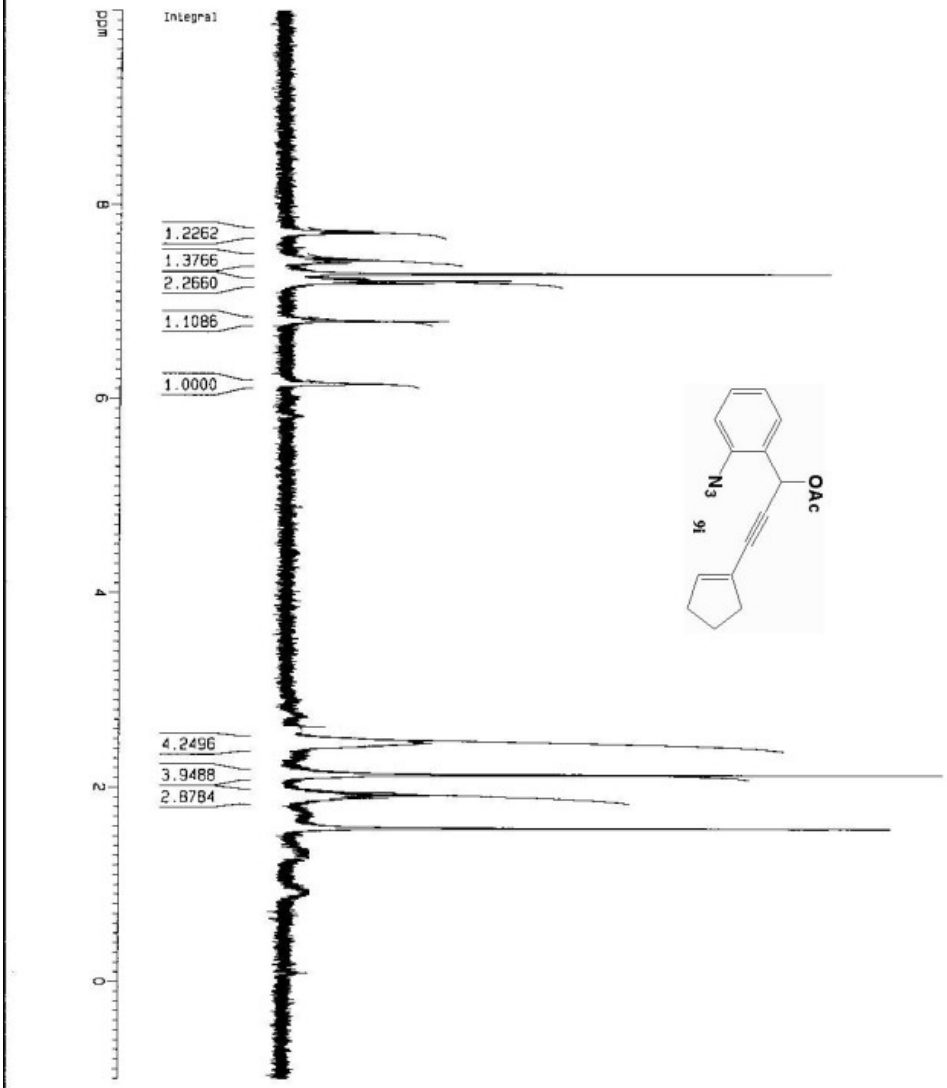
128.5180
128.4671
127.3100
125.1390
125.0308
123.7751
118.3651
118.0616
88.8830
84.1440
77.2340
70.1667
61.2307
36.3203
33.3097
23.2843
100.0000
General
DATE = 2006/04/21
TIME = 06:35
INSTRUM = spect
PULPROG = zgpg30
F1 [13C]
SI = 32768
SF = 90.555
SW_p = 21645.022



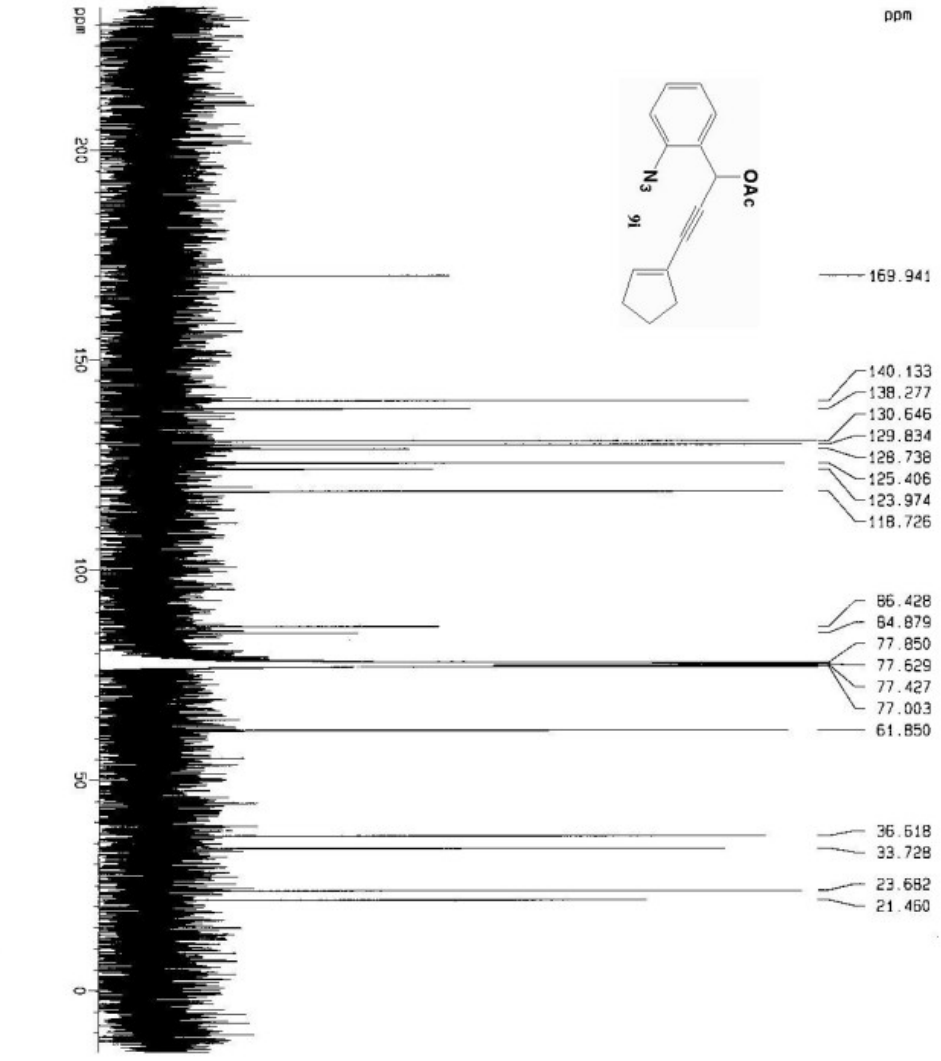

```
Current Data Parameters
NAME                  MRI-Apr21-06
EXPNO                            1
PROCNO                           1

F2 - Acquisition Parameters
Date_                     20060421
Time                         15.00
INSTRUM                       spect
PROBHD     5 mm Multinu
PULPROG                      zg30
TD                          65536
SOLVENT                     CDCl3
NS                             16
DS                              2
SWH                      6172.839 Hz
FIDRES                   0.094190 Hz
AQ                       5.3084660 sec
RG                          645.1
DW                         81.000 usec
DE                          6.00 usec
TE                         300.0 K
D1                       1.00000000 sec

======== CHANNEL f1 ========
NUC1                          1H
P1                          9.50 usec
PL1                        -6.00 dB
SFO1                300.1318534 MHz

F2 - Processing parameters
SI                          32768
SF                  300.1300000 MHz
WDW                            no
SSB                             0
LB                           0.00 Hz
GB                              0
PC                           1.00

1D NMR plot parameters
CX                          20.00 cm
F1P                        10.000 ppm
F1                       3001.30 Hz
F2P                        -1.000 ppm
F2                         -300.13 Hz
PPMCM                    0.55000 ppm/cm
HZCM                   165.07150 Hz/cm
```



Current Data Parameters
NAME
EXPNO
PROCNO

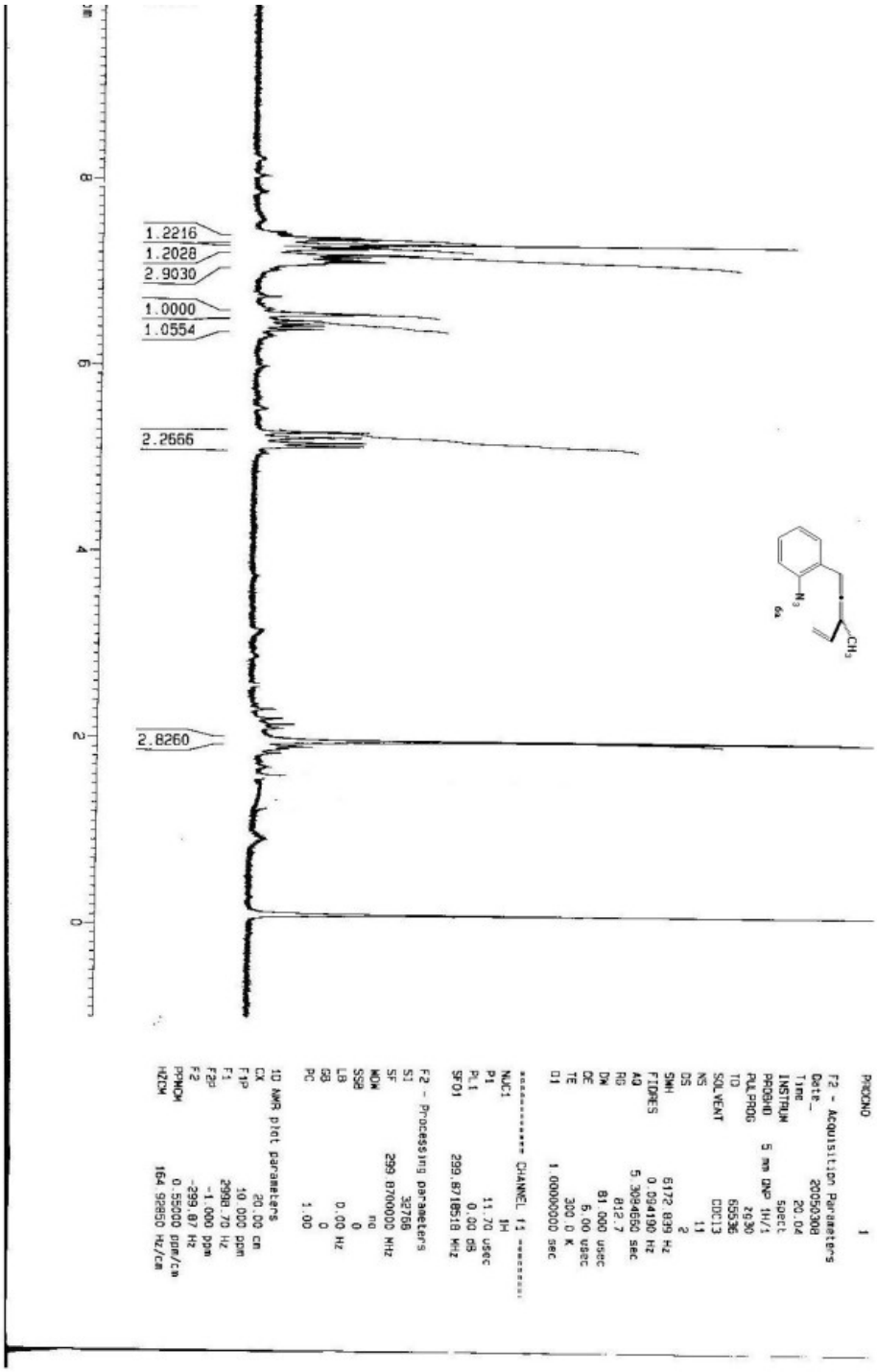
F2 - Acquisition Parameters
Date_
Time
INSTRUM
PROBHD
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
D11
D12

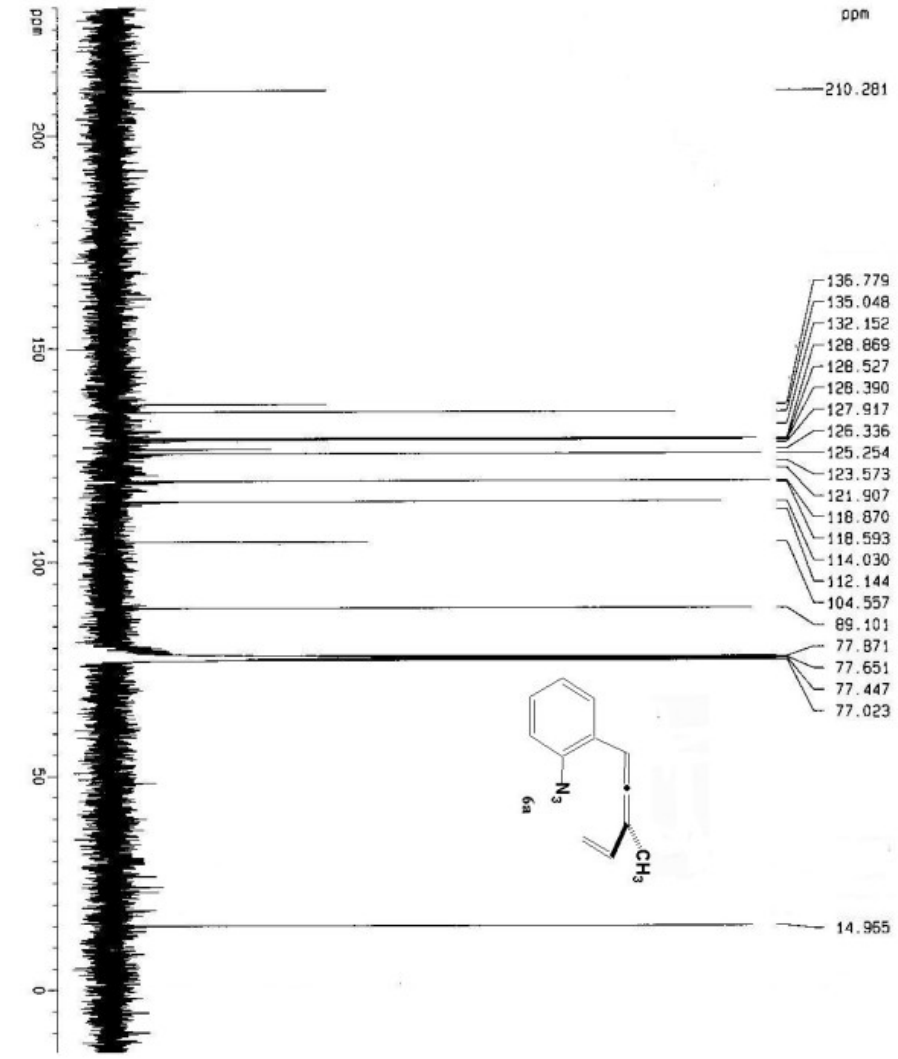
CHANNEL f1
NUC1
P1
PL1
SF01

CHANNEL f2
CPDPRG2
NUC2
PCPD2
PL2
PL12
PL13
SF02

F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC

1D NMR plot parameters
CX
F1P
F1
F2P
F2
PPMCM
HZCM


1.2216
1.2028
2.9030
1.0000
1.0554
2.2666
2.8260
8
6
4
2
0
N3
6a
CH3



Current Data Parameters
NAME Mei-Mar08-06
EXPNO 7
PROCNO 1

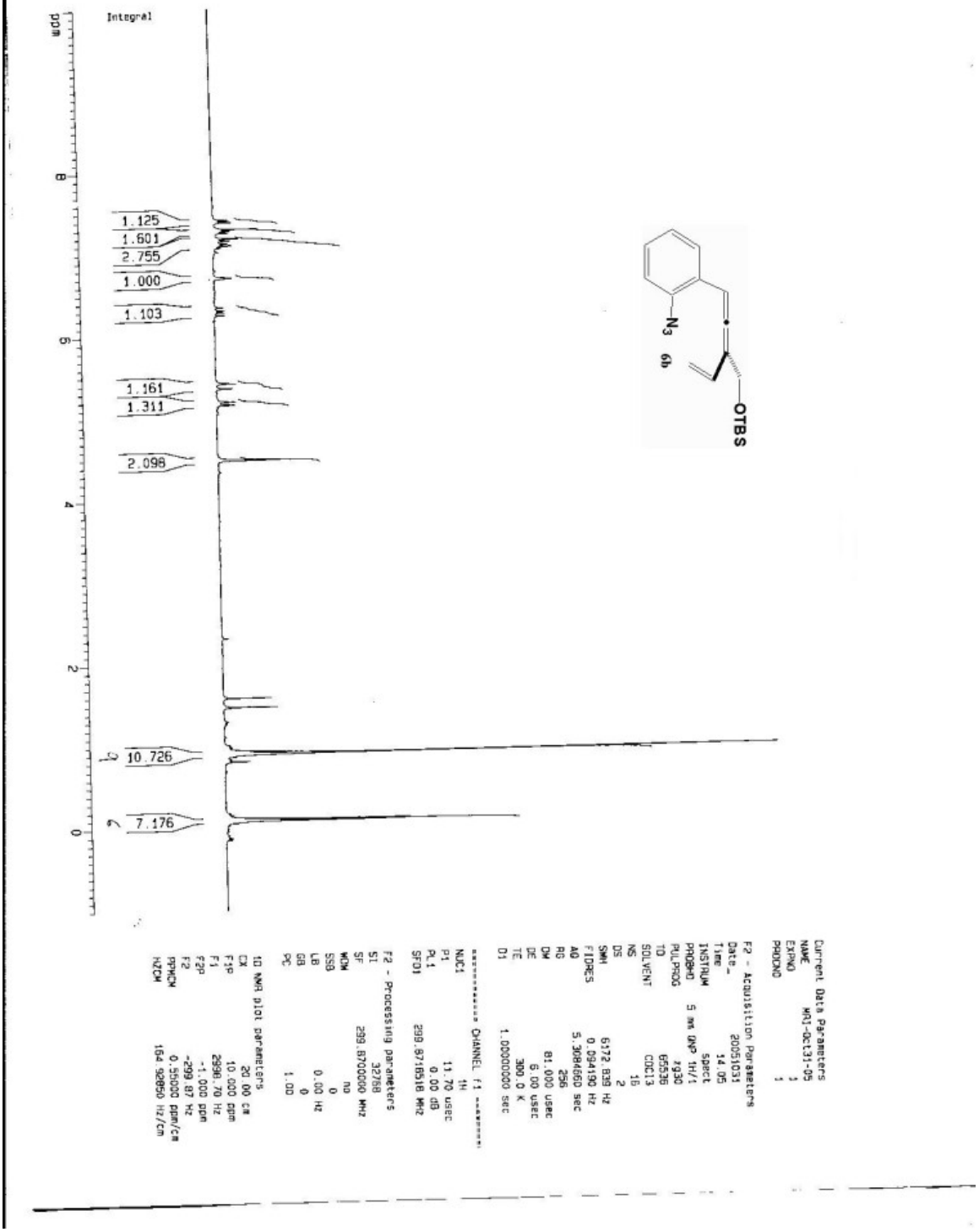
F2 - Acquisition Parameters
Date_ 20050309
Time 8.07
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1500
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AQ 1.7433076 sec
RG 1024
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

========== CHANNEL f1 ==========
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SFO1 75.4100357 MHz

========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.9715383 MHz

F2 - Processing parameters
SI 32768
SF 75.4023410 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 230.000 ppm
F1 17342.54 Hz
F2P -20.000 ppm
F2 -1508.05 Hz
PPMCM 12.50000 ppm/cm
HZCM 942.52930 Hz/cm



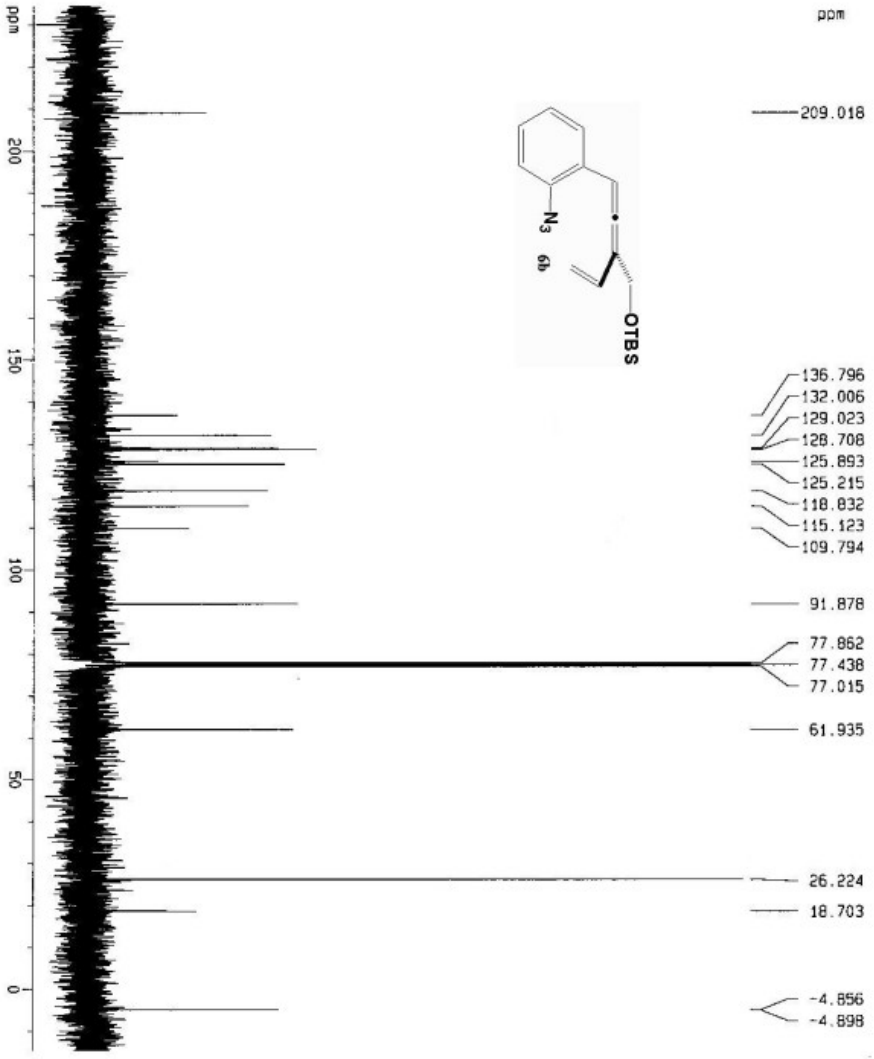
Current Data Parameters
NAME MOJ-Oct31-05
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20051031
Time 14.05
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084860 sec
RG 256
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

========== CHANNEL f1 ==========
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SFO1 299.9716518 MHz

F2 - Processing parameters
SI 32768
SF 299.8700000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 2998.70 Hz
F2P -1.000 ppm
F2 -299.87 Hz
PPMCM 0.55000 ppm/cm
HZCM 164.92850 Hz/cm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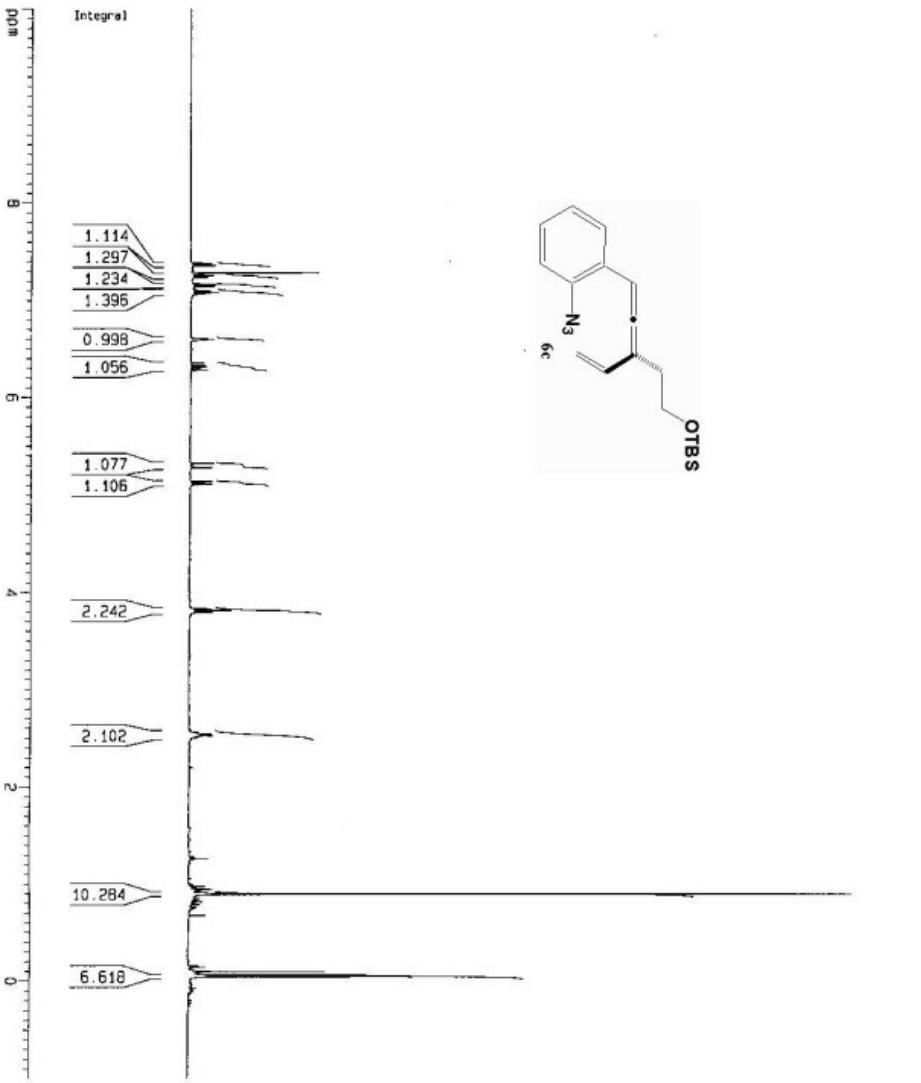
129.023
128.708
125.893
125.215
118.832
115.123
109.794
91.878
77.862
77.438
77.015
61.935
26.224
18.703
-4.856
-4.898
N3
6b
OTBS



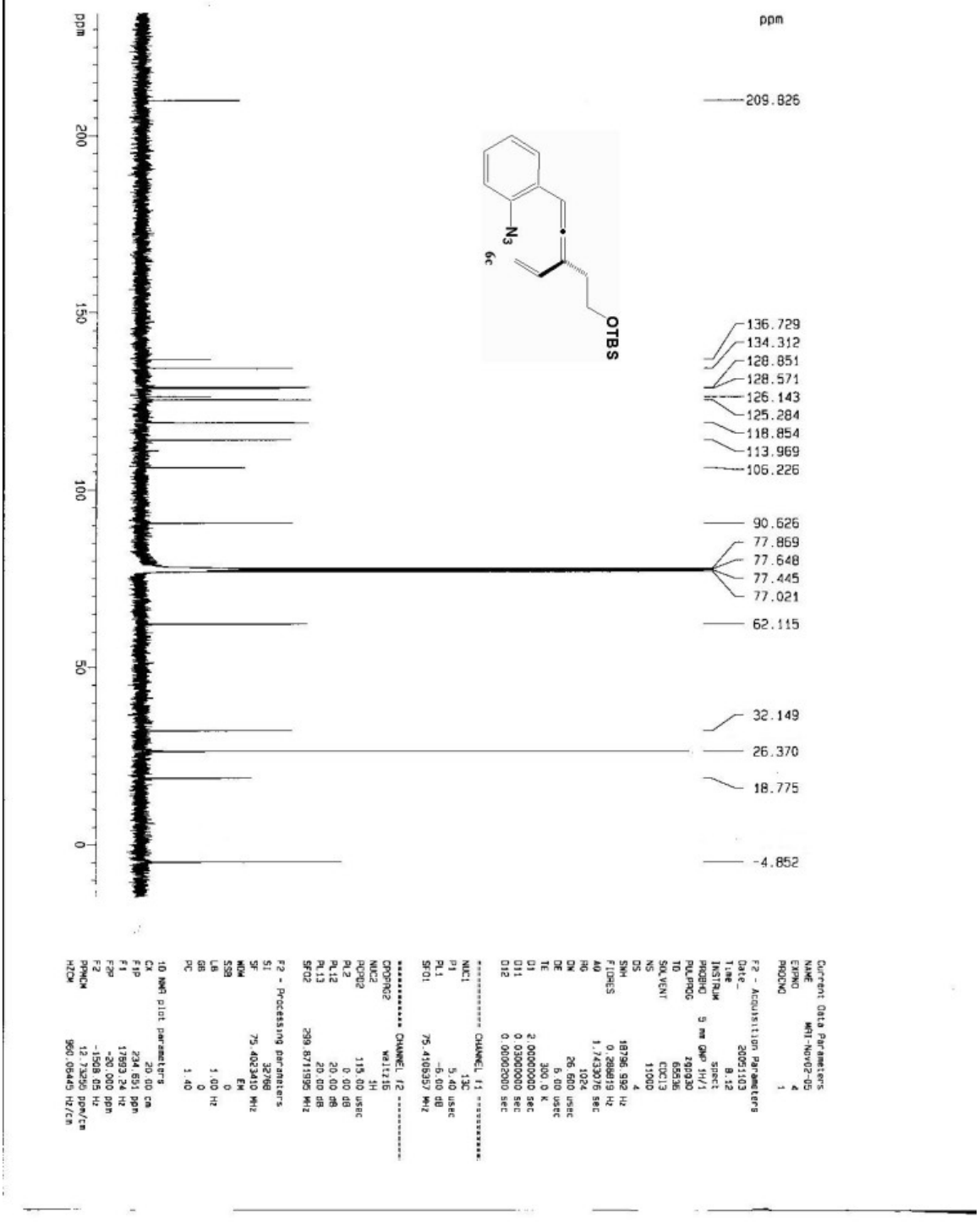

```
Current Data Parameters
NAME                  MRI-Oct27-05
EXPNO                          3
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20051027
Time                        9.43
INSTRUM                     spect
PROBHD     5 mm BBI 1H-B
PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                            16
DS                             2
SWH                    8278.146 Hz
FIDRES                 0.126314 Hz
AQ                     3.9558243 sec
RG                         574.7
DW                        60.400 usec
DE                         6.00 usec
TE                        300.0 K
D1                     1.00000000 sec

======== CHANNEL f1 ========
NUC1                         1H
P1                         6.45 usec
PL1                        0.00 dB
SFO1            400.1324710 MHz

F2 - Processing parameters
SI                         32768
SF                400.1300000 MHz
WDW                           no
SSB                            0
LB                          0.00 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                         20.00 cm
F1P                       10.000 ppm
F1                      4001.30 Hz
F2P                       -1.000 ppm
F2                      -400.13 Hz
PPMCM                  0.55000 ppm/cm
HZCM                 220.07150 Hz/cm
```


ppm
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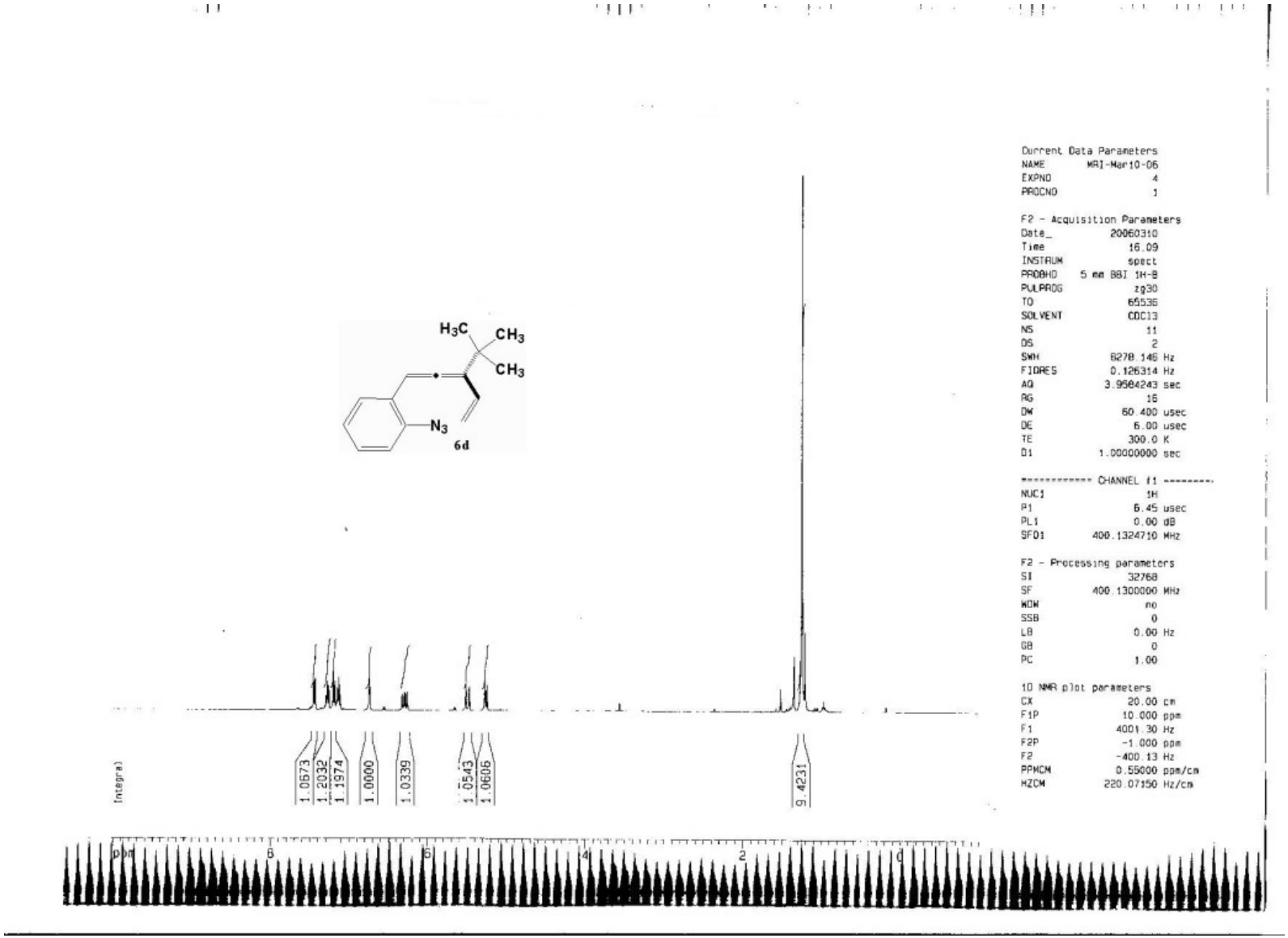
136.729
134.312
128.851
128.571
126.143
125.284
118.854
113.969
106.226
90.626
77.869
77.648
77.445
77.021
62.115
32.149
26.370
18.775
-4.852
N3
6c
OTBS
Current Data Parameters
NAME MRT-Nov02-05
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051103
Time 8.12
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1100
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AQ 1.7433076 sec
RG 1024
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 5.40 usec
PL1 -5.00 dB
SFO1 75.4106957 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711996 MHz
F2 - Processing parameters
SI 32768
SF 75.4623410 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 234.691 ppm
F1 17693.24 Hz
F2P -30.000 ppm
F2 -1508.05 Hz
PPMCM 12.73296 ppm/cm
HZCM 960.06445 Hz/cm



Current Data Parameters
NAME MRI-Mar10-06
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060310
Time 16.09
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 11
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 16
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

========== CHANNEL f1 =========
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm


ppm
204.749
136.556
130.952
128.280
127.964
126.914
125.321
118.982
118.639
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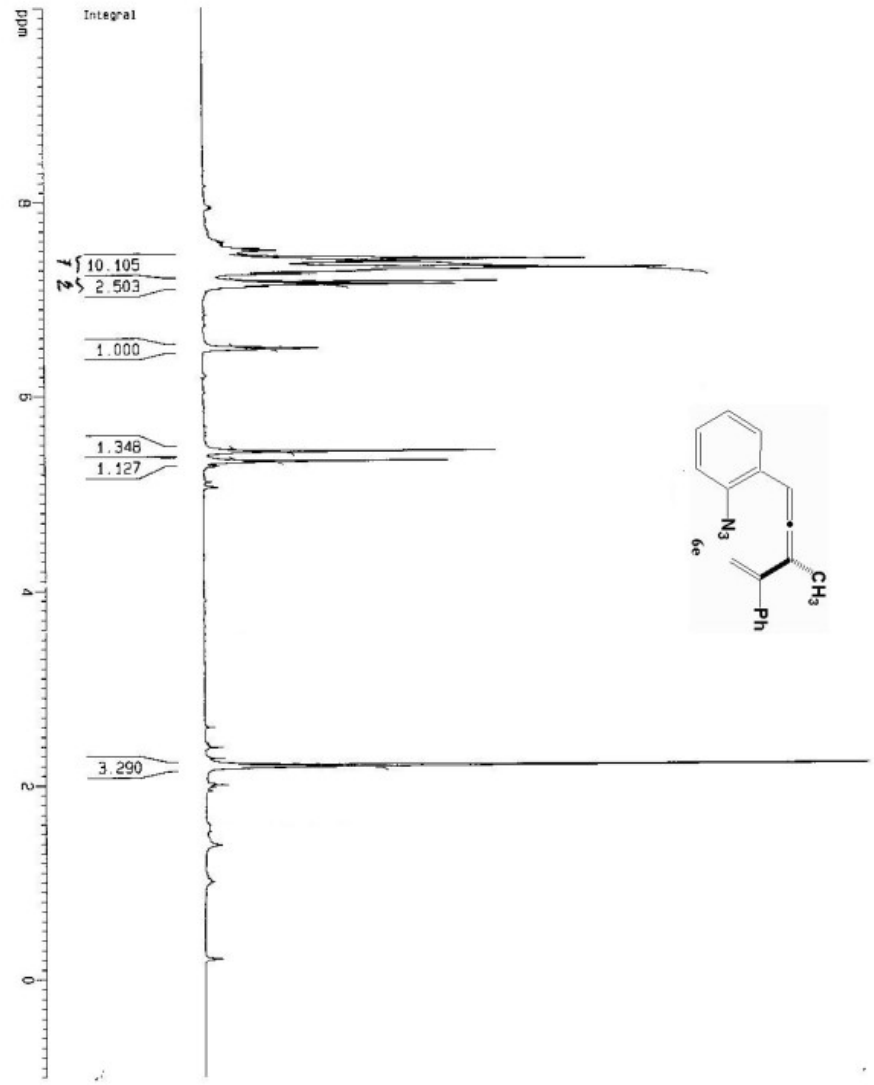
29.873
N3
H3C
CH3
CH3
6d

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Current Data Parameters
NAME                  MEI-Dec30-05
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_              20051230
Time               17.46
INSTRUM            spect
PROBHD     5 mm QNP 1H/1
PULPROG            zg30
TD                 65536
SOLVENT            CDCl3
NS                 9
DS                 2
SWH           6172.839 Hz
FIDRES        0.094190 Hz
AQ            5.3084660 sec
RG                 90.5
DW            81.000 usec
DE             6.00 usec
TE             300.0 K
D1            1.00000000 sec

======== CHANNEL f1 ========
NUC1               1H
P1            11.70 usec
PL1            0.00 dB
SFO1      299.8718518 MHz

F2 - Processing parameters
SI                32768
SF          299.8700000 MHz
WDW                no
SSB                 0
LB             0.00 Hz
GB                  0
PC             1.00

1D NMR plot parameters
CX             20.00 cm
F1P            10.000 ppm
F1           2998.70 Hz
F2P            -1.000 ppm
F2           -299.87 Hz
PPMCM          0.55000 ppm/cm
HZCM         164.92850 Hz/cm
```
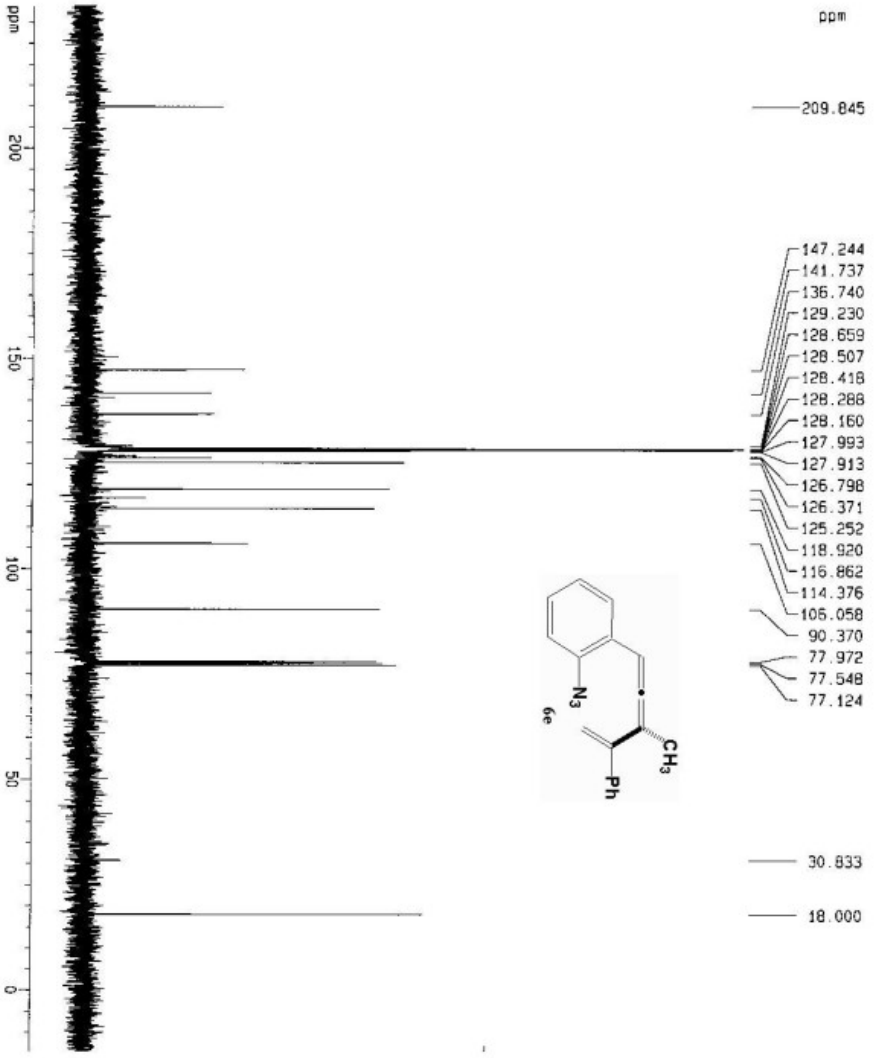
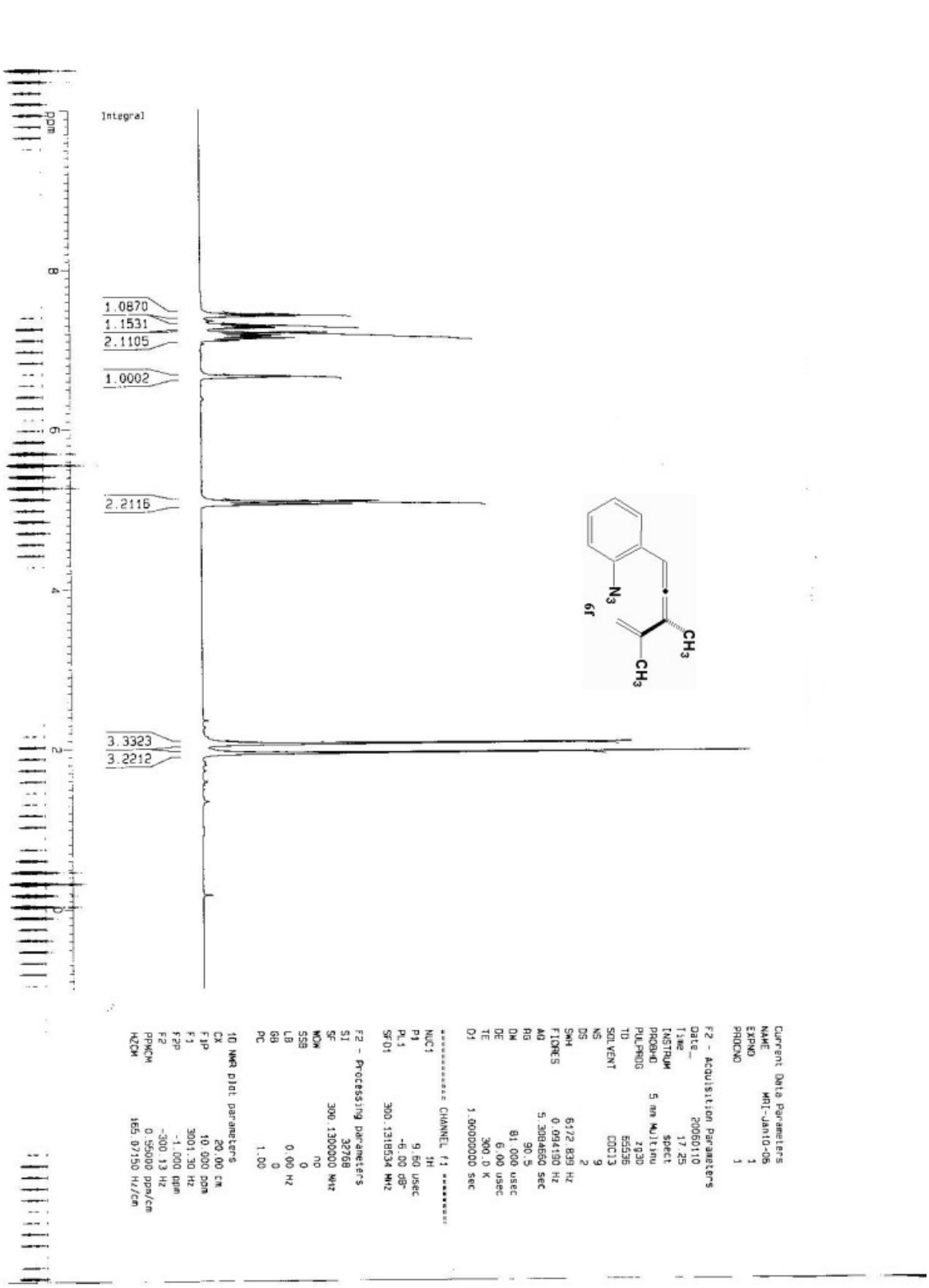
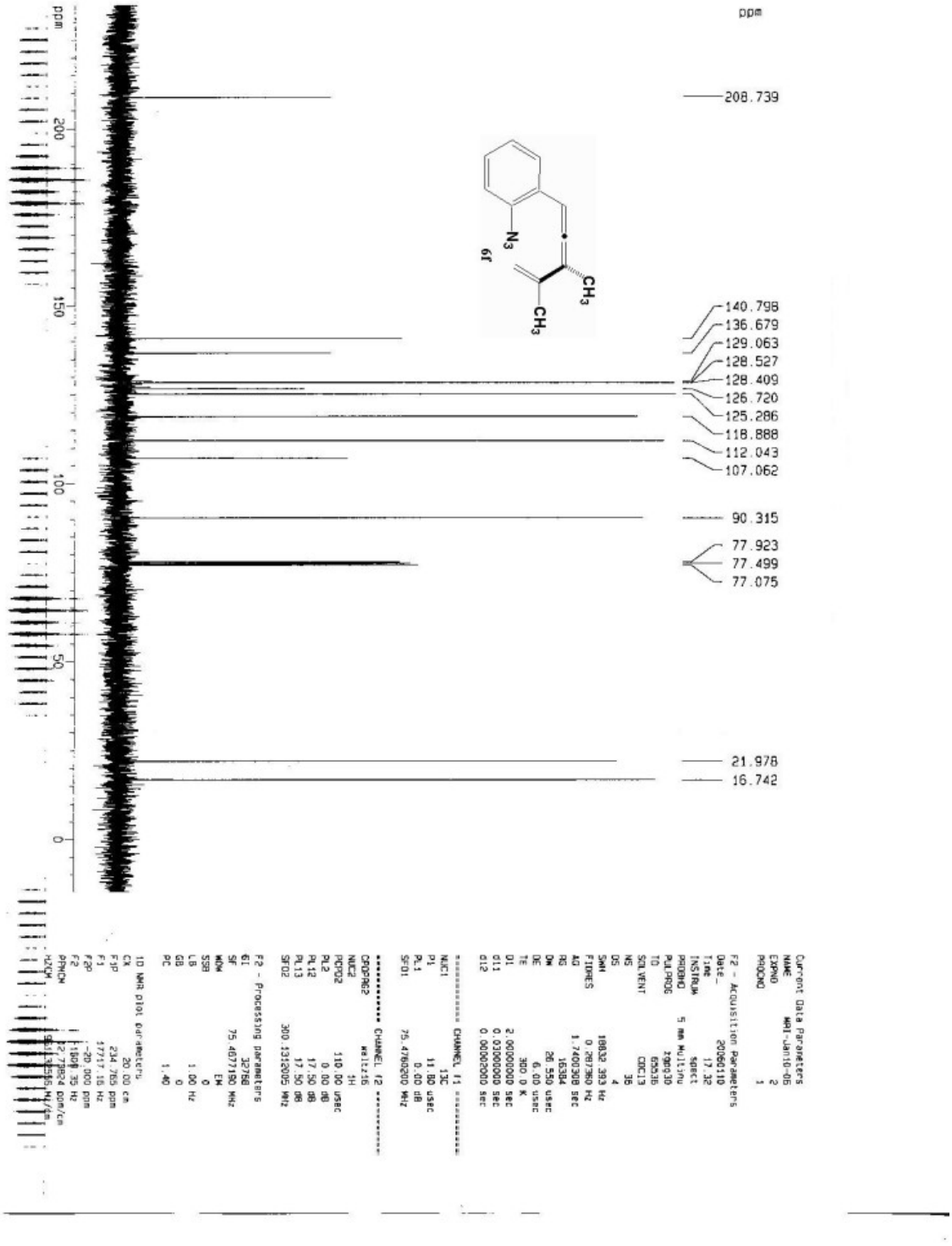
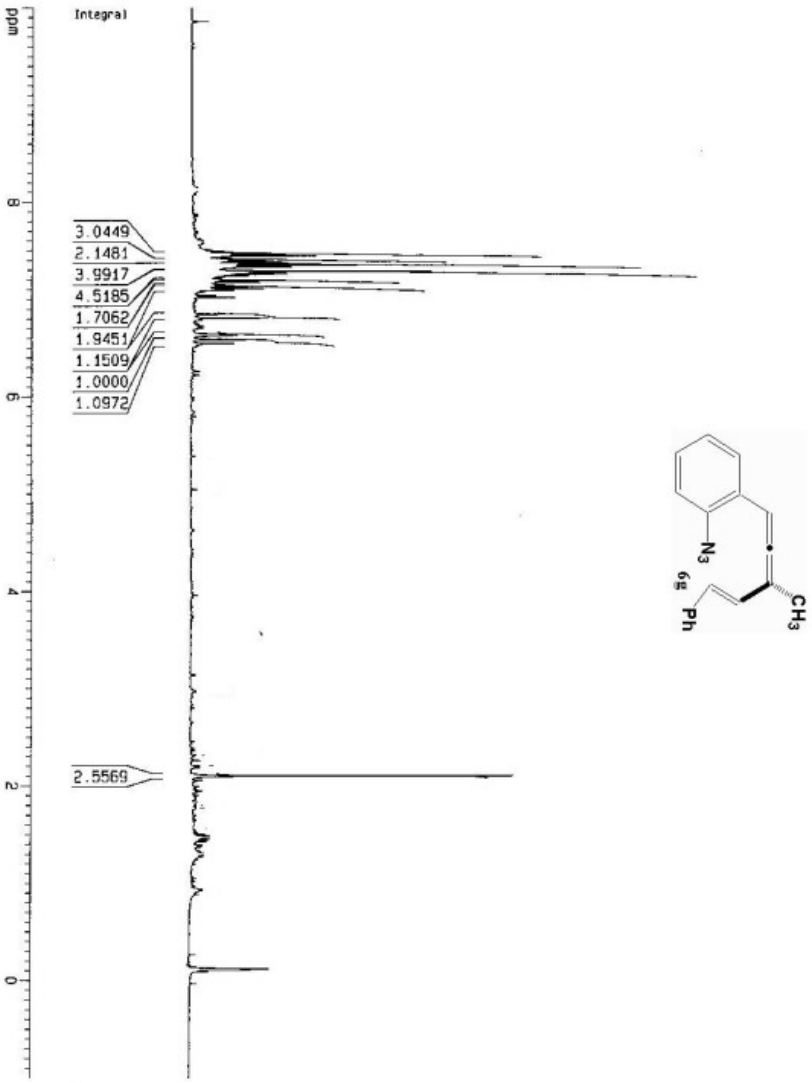



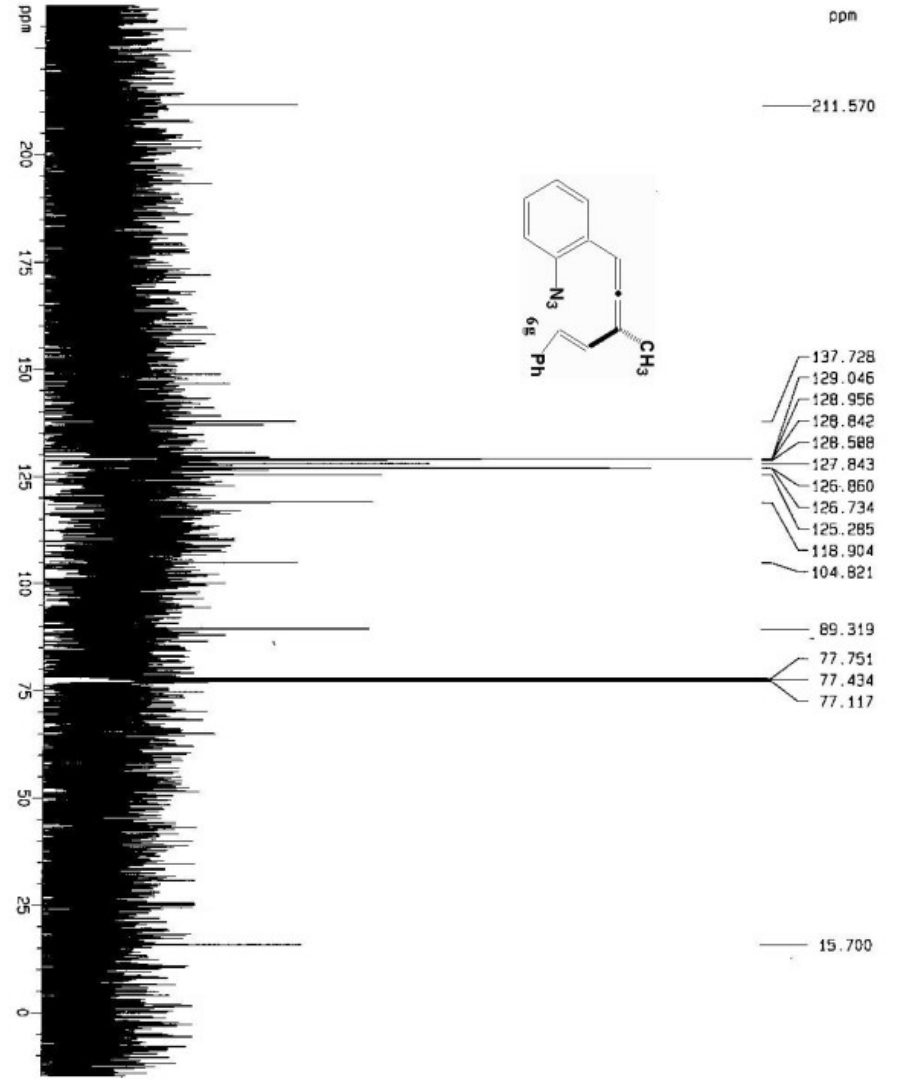

```
Current Data Parameters
NAME                  NRI-DEC30-05
EXPNO                           3
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20051230
Time                        17.52
INSTRUM                      spect
PROBHD     5 mm QNP 1H/1
PULPROG                  zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                            28
DS                             4
SWH                    18796.992 Hz
FIDRES                  0.286819 Hz
AQ                     1.743076 sec
RG                           1024
DW                         26.600 usec
DE                          6.00 usec
TE                         300.0 K
D1                     2.00000000 sec
D11                    0.03000000 sec
D12                    0.00002000 sec

============ CHANNEL f1 ============
NUC1                          13C
P1                           5.40 usec
PL1                        -6.00 dB
SFO1                   75.4196367 MHz

============ CHANNEL f2 ============
CPDPRG2                    waltz16
NUC2                          1H
PCPD2                      115.00 usec
PL2                         0.00 dB
PL12                       20.00 dB
PL13                       20.00 dB
SFO2                   299.8711595 MHz

F2 - Processing parameters
SI                            32768
SF                     75.4823410 MHz
WDW                              EM
SSB                               0
LB                             1.00 Hz
GB                                0
PC                             1.40

1D NMR plot parameters
CX                           20.00 cm
F1P                         234.000 ppm
F1                        17644.15 Hz
F2P                        -20.000 ppm
F2                        -1508.05 Hz
PPMCM                      12.70000 ppm/cm
HZCM                     957.60974 Hz/cm
```

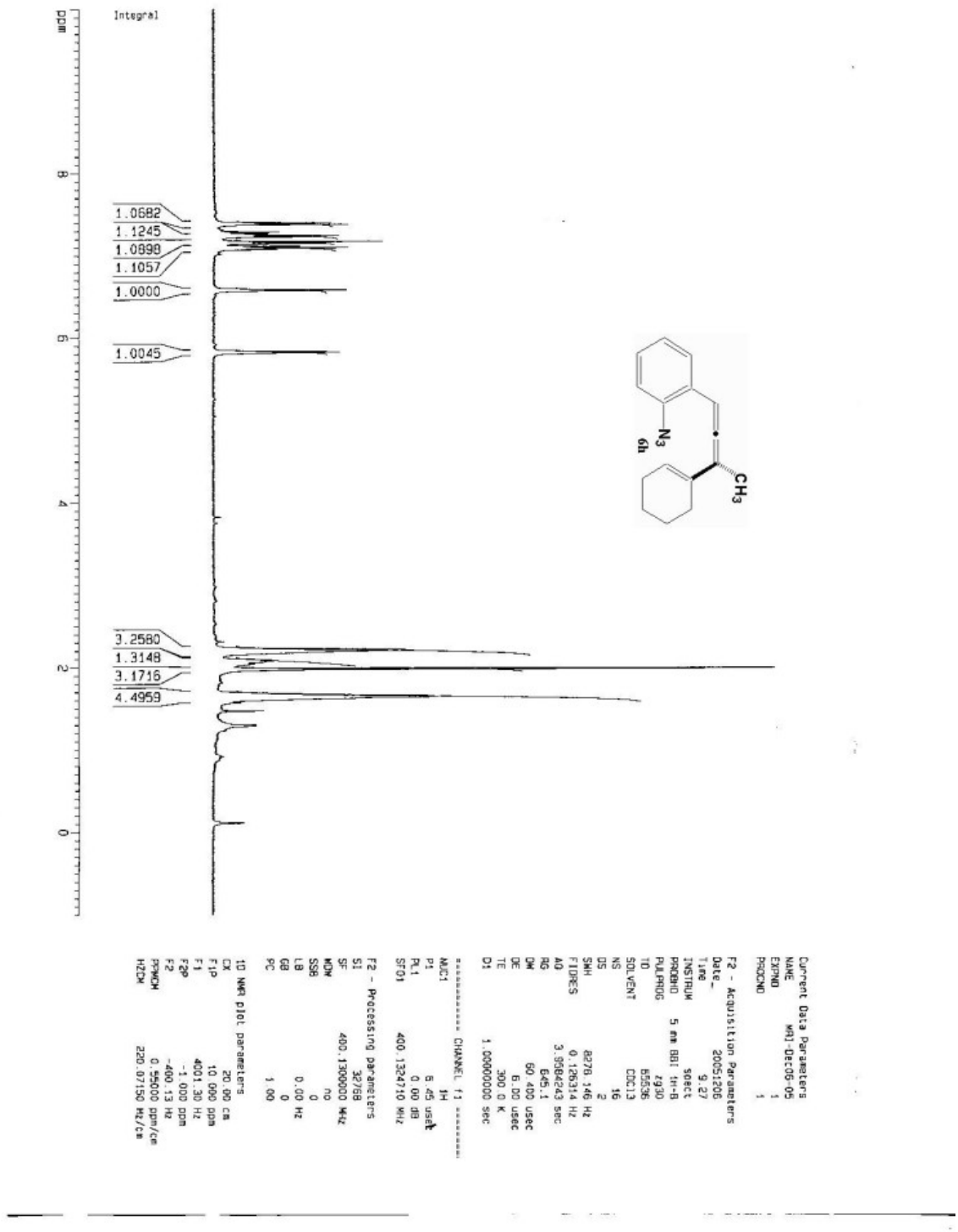

Integral
1.0870
1.1531
2.1105
1.0002
2.2116
3.3323
3.2212
ppm
8
6
4
2
0
N3
6f
CH3
CH3
Current Data Parameters
NAME MRI-JAN10-05
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060110
Time 17.25
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084650 sec
RG 90.5
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 9.60 usec
PL1 -6.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 165.07150 Hz/cm


ppm
208.739
140.798
136.679
129.063
128.527
128.409
126.720
125.286
118.888
112.043
107.062
90.315
77.923
77.499
77.075
21.978
16.742
6f
N3
CH3
CH3
Current Data Parameters
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_
Time
INSTRUM
PROBHD
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
D11
D12
CHANNEL f1
NUC1
P1
PL1
SFO1
CHANNEL f2
CPDPRG2
NUC2
PCPD2
PL2
PL12
PL13
SFO2
F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC
1D NMR plot parameters
CX
F1P
F1
F2P
F2
PPMCM
HZCM

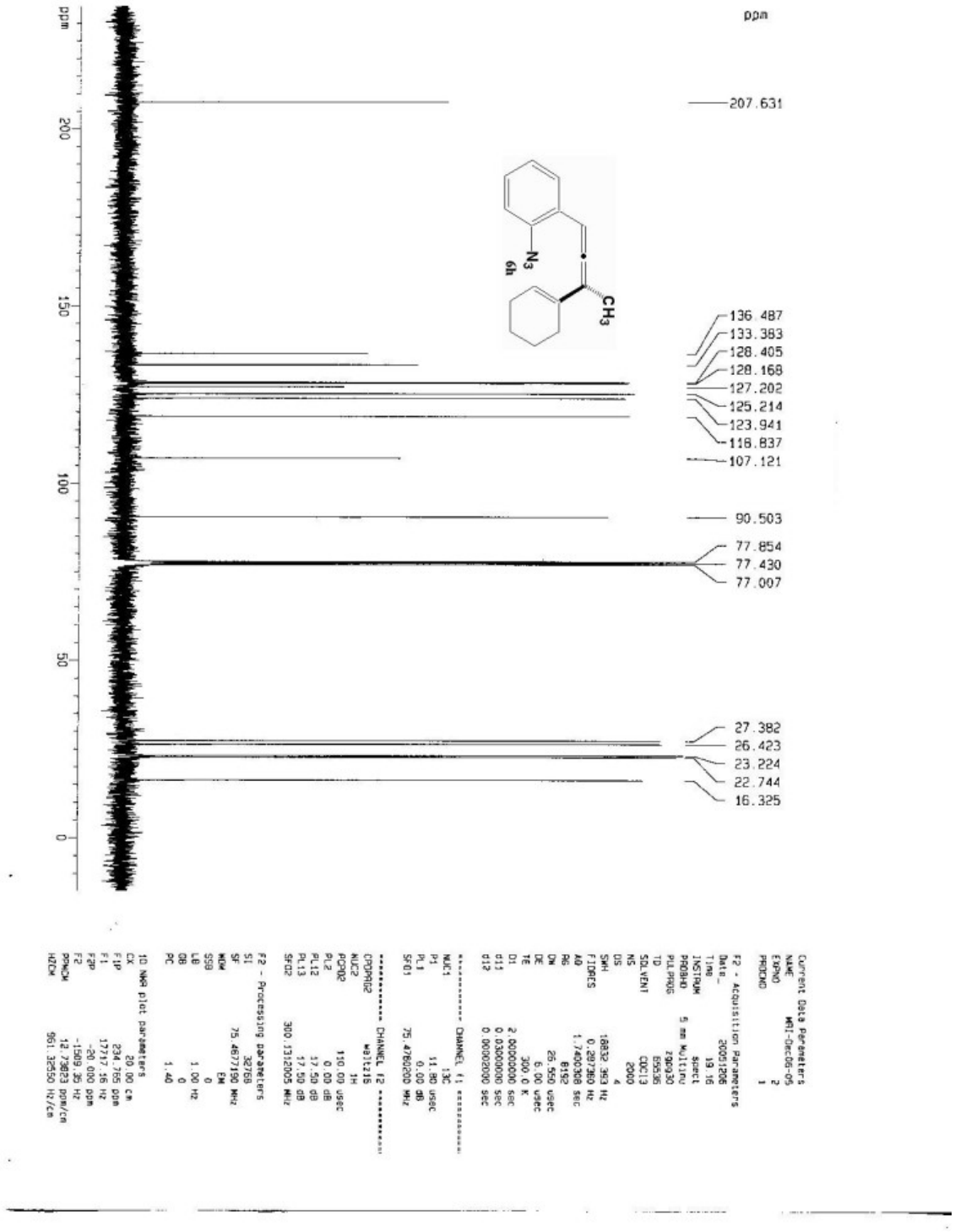



```
Current Data Parameters
NAME                  MRI-Oct27-05
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_               20051027
Time                      9.12
INSTRUM                   spect
PROBHD     5 mm BBI 1H-B
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                           2
DS                           2
SWH                  8278.146 Hz
FIDRES               0.126314 Hz
AQ                   3.9584243 sec
RG                       228.1
DW                      60.400 usec
DE                       6.00 usec
TE                      300.0 K
D1                   1.00000000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                       6.45 usec
PL1                      0.00 dB
SFO1              400.1324710 MHz

F2 - Processing parameters
SI                       32768
SF                400.1300000 MHz
WDW                         no
SSB                          0
LB                        0.00 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       20.00 cm
F1P                     10.000 ppm
F1                    4001.30 Hz
F2P                    -1.000 ppm
F2                     -400.13 Hz
PPMCM                  0.95000 ppm/cm
HZCM                 220.07150 Hz/cm
```


ppm
211.570
137.728
129.046
128.956
128.842
128.588
127.843
126.860
126.734
125.285
118.904
104.821
89.319
77.751
77.434
77.117
15.700
N3
6g
Ph
CH3




```
Current Data Parameters
NAME                  MRJ-Dec06-05
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_              20051206
Time                      9.27
INSTRUM                   spect
PROBHD     5 mm BBI 1H-B
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 8278.146 Hz
FIDRES              0.126314 Hz
AQ                  3.9584243 sec
RG                     645.1
DW                  60.400 usec
DE                   6.00 usec
TE                   300.0 K
D1                  1.00000000 sec
======== CHANNEL f1 ========
NUC1                       1H
P1                       6.45 usec
PL1                      0.00 dB
SF01          400.1324710 MHz

F2 - Processing parameters
SI                        32768
SF                400.1300000 MHz
WDW                          no
SSB                           0
LB                         0.00 Hz
GB                            0
PC                         1.00

1D NMR plot parameters
CX                       20.00 cm
F1P                     10.000 ppm
F1                    4001.30 Hz
F2P                    -1.000 ppm
F2                   -400.13 Hz
PPMCM                 0.55000 ppm/cm
HZCM                220.07150 Hz/cm
```



Current Data Parameters
NAME MRI-DecO6-05
EXPNO 2
PROCNO 1

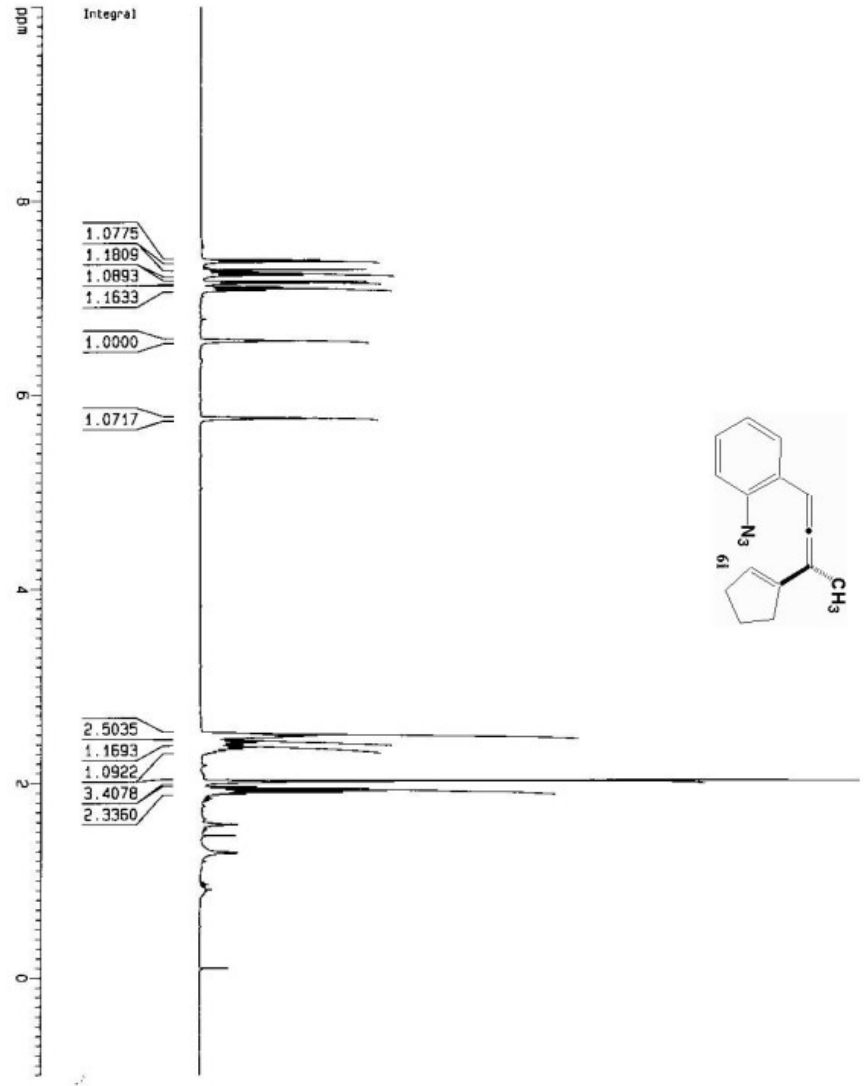
F2 - Acquisition Parameters
Date_ 20051206
Time 19.16
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2000
DS 4
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400368 sec
RG 8192
DW 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 11.80 usec
PL1 0.00 dB
SFO1 75.4760200 MHz

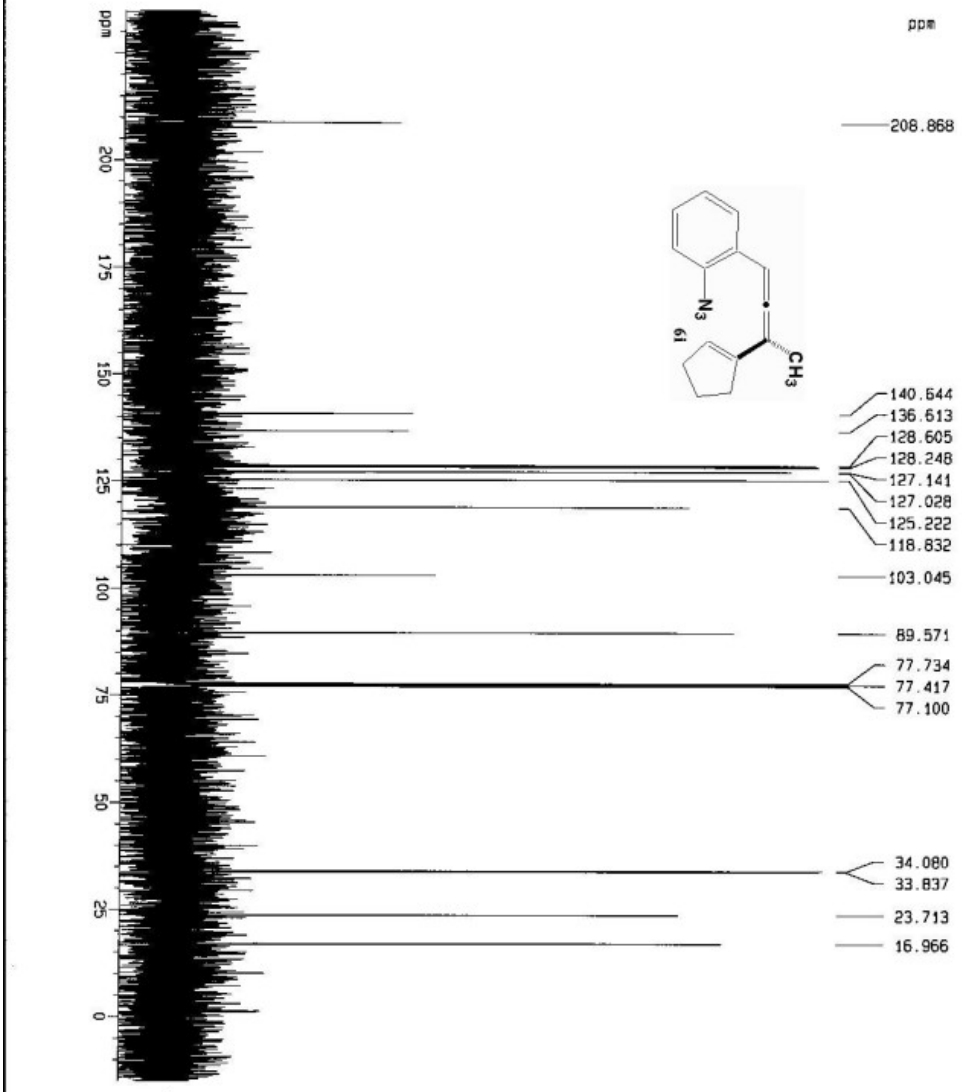
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 110.00 usec
PL2 0.00 dB
PL12 17.50 dB
PL13 17.50 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

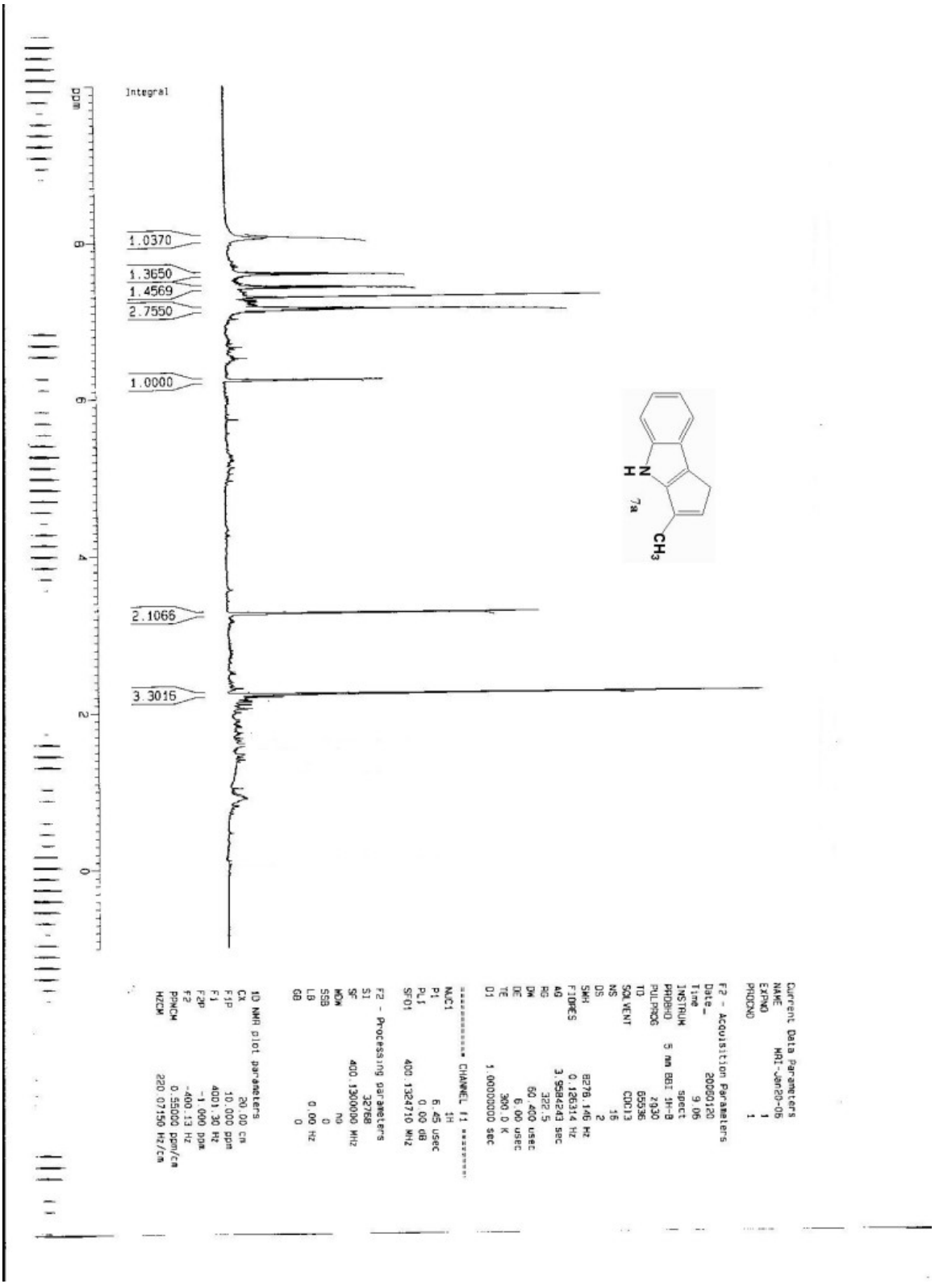
1D NMR plot parameters
CX 20.00 cm
F1P 234.765 ppm
F1 17717.16 Hz
F2P -20.000 ppm
F2 -1509.36 Hz
PPMCM 12.79823 ppm/cm
HZCM 961.12450 Hz/cm

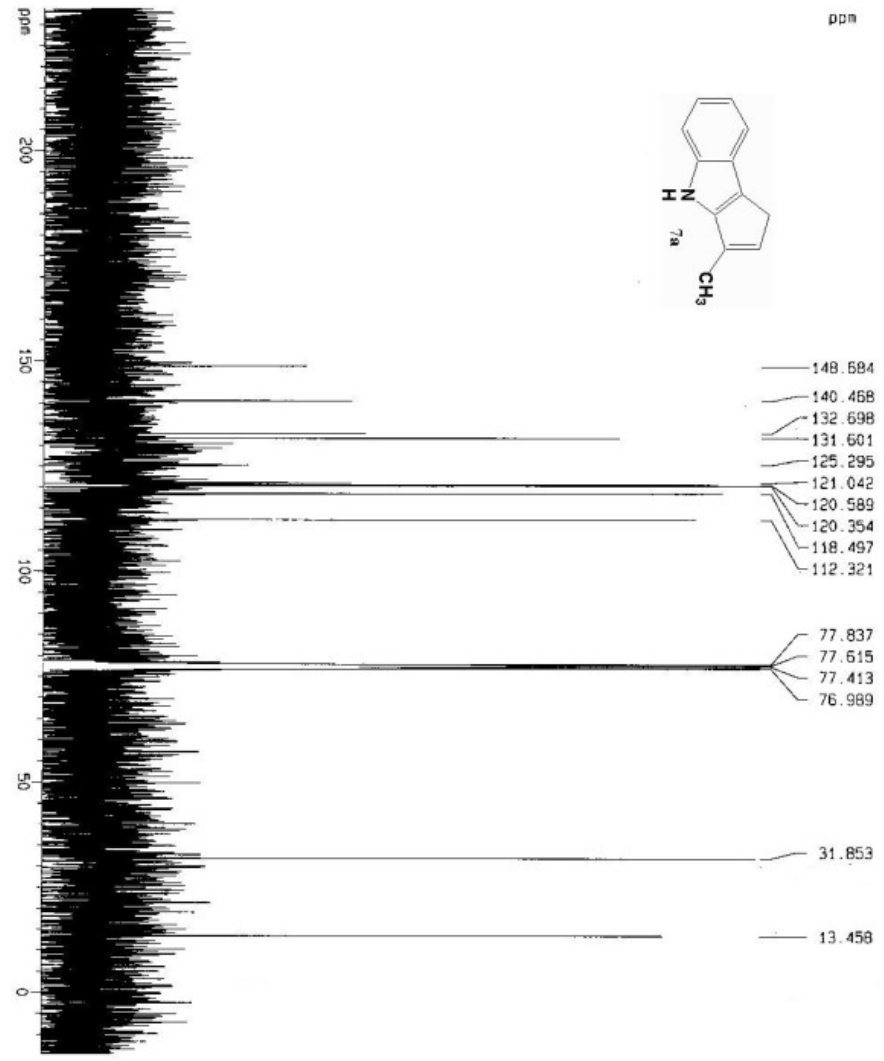



```
Current Data Parameters
NAME                  MIT-Jun26-05
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_              20050628
Time                  13.31
INSTRUM                 spect
PROBHD     5 mm BBI 1H-B
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH               8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                    143.7
DW                   60.400 usec
DE                    6.00 usec
TE                   300.0 K
D1                1.00000000 sec
======== CHANNEL f1 ========
NUC1                      1H
P1                    6.45 usec
PL1                   0.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                    32768
SF            400.1300000 MHz
WDW                      no
SSB                       0
LB                 0.00 Hz
GB                        0
PC                    1.00

1D NMR plot parameters
CX                   20.00 cm
F1P                10.000 ppm
F1                 4001.30 Hz
F2P                -1.000 ppm
F2                 -400.13 Hz
PPMCM              0.55000 ppm/cm
HZCM             220.07150 Hz/cm
```

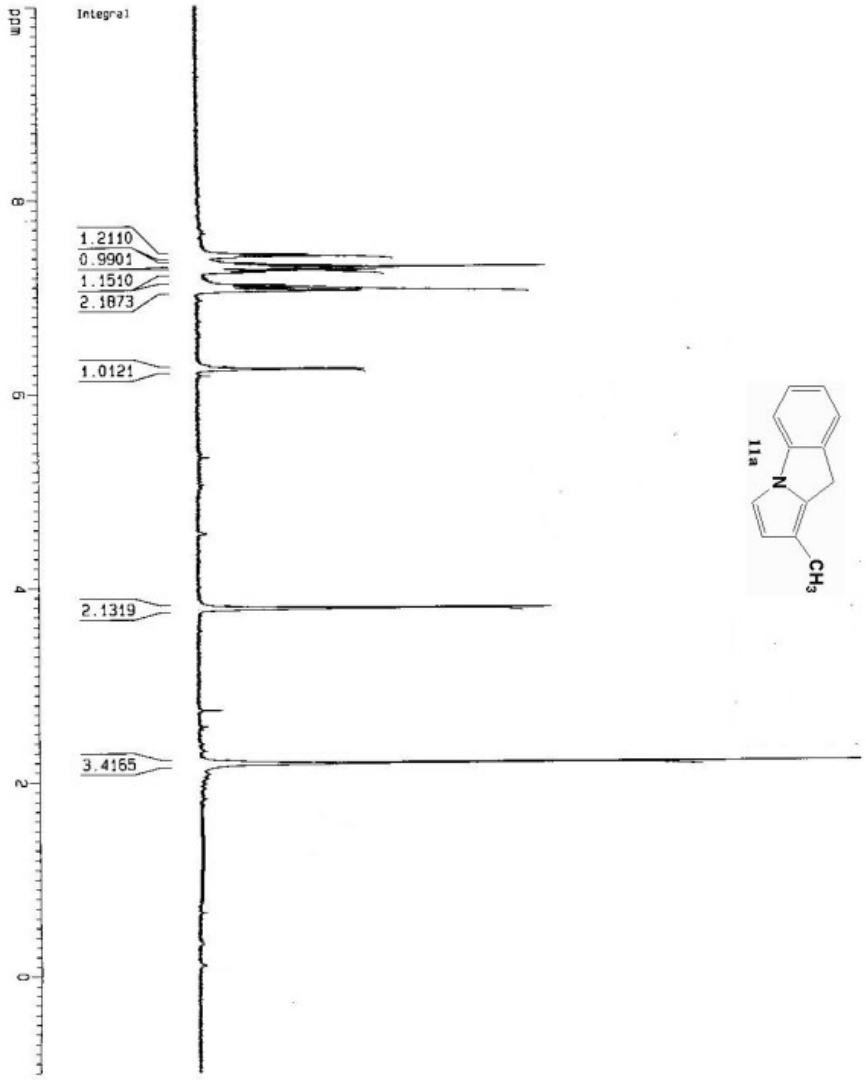



```
Current Data Parameters
NAME                  MRI-Jun28-05
EXPNO                           2
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20050628
Time                        13.45
INSTRUM                      spect
PROBHD     5 mm BBI 1H-B
PULPROG                  zgpg30
TD                           65536
SOLVENT                      CDCl3
NS                             513
DS                               4
SWH                      25125.629 Hz
FIDRES                    0.383387 Hz
AQ                        1.3042164 sec
RG                           16384
DW                       19.900 usec
DE                        6.00 usec
TE                          300.0 K
D1                        2.00000000 sec
D11                       0.03000000 sec
D12                       0.00002000 sec

======== CHANNEL f1 ========
NUC1                          13C
P1                          16.35 usec
PL1                        -6.00 dB
SFO1                  100.6237558 MHz

======== CHANNEL f2 ========
CPDPRG2                   waltz15
NUC2                          1H
PCPD2                     114.00 usec
PL2                         0.00 dB
PL12                       24.00 dB
PL13                       24.00 dB
SFO2                  400.1316005 MHz

F2 - Processing parameters
SI                            32768
SF                    100.6127290 MHz
WDW                             EM
SSB                              0
LB                            1.00 Hz
GB                               0
PC                            1.40

1D NMR plot parameters
CX                           20.00 cm
F1P                         234.959 ppm
F1                        23659.72 Hz
F2P                        -14.958 ppm
F2                        -1495.90 Hz
PPMCM                     12.48831 ppm/cm
HZCM                   1256.28137 Hz/cm
```

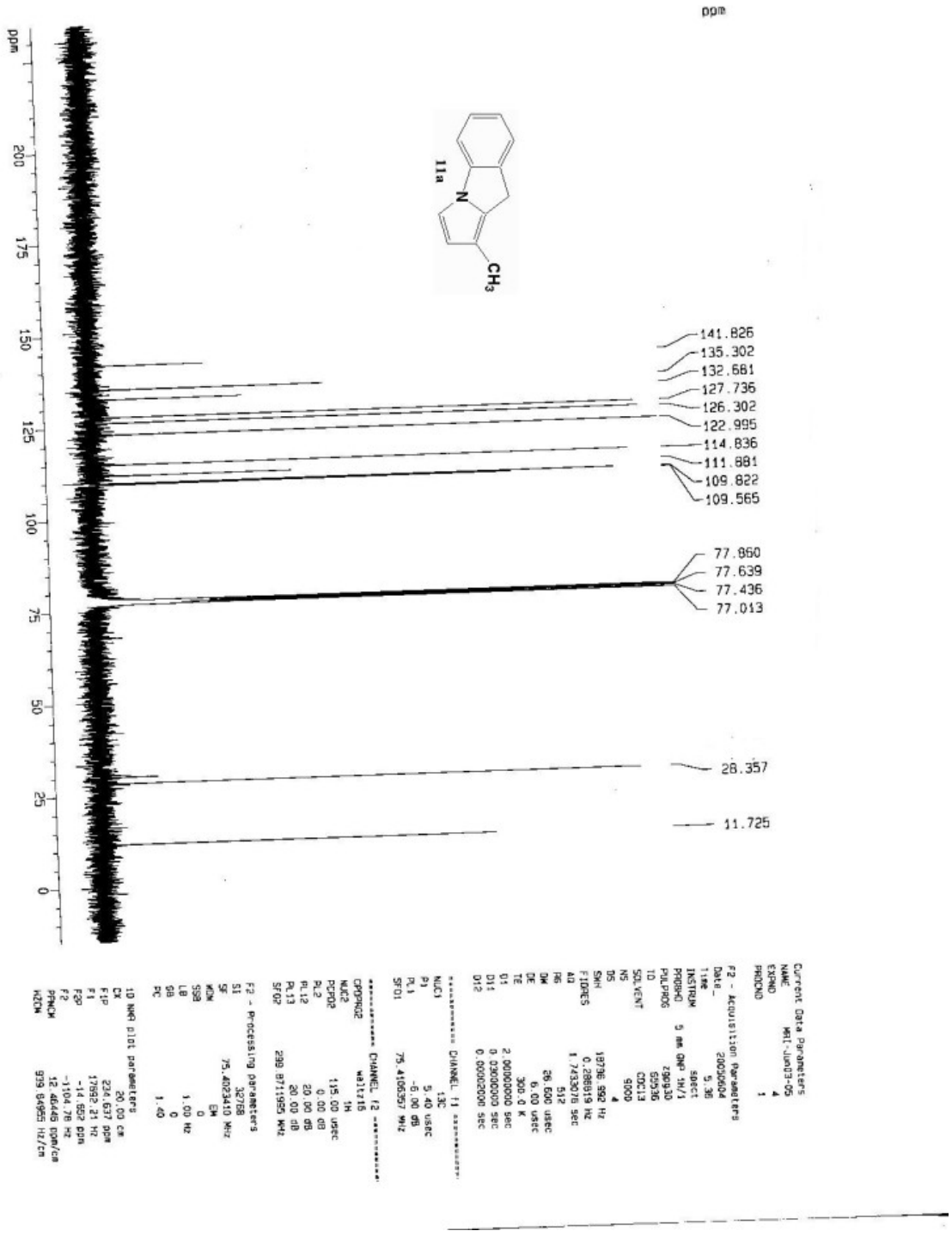

Integral
1.0370
1.3650
1.4569
2.7550
1.0000
2.1066
3.3016
PPM
8
6
4
2
0
N
H
7a
CH3
Current Data Parameters
NAME HRI-Jan20-05
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050120
Time 9.06
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SF01 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

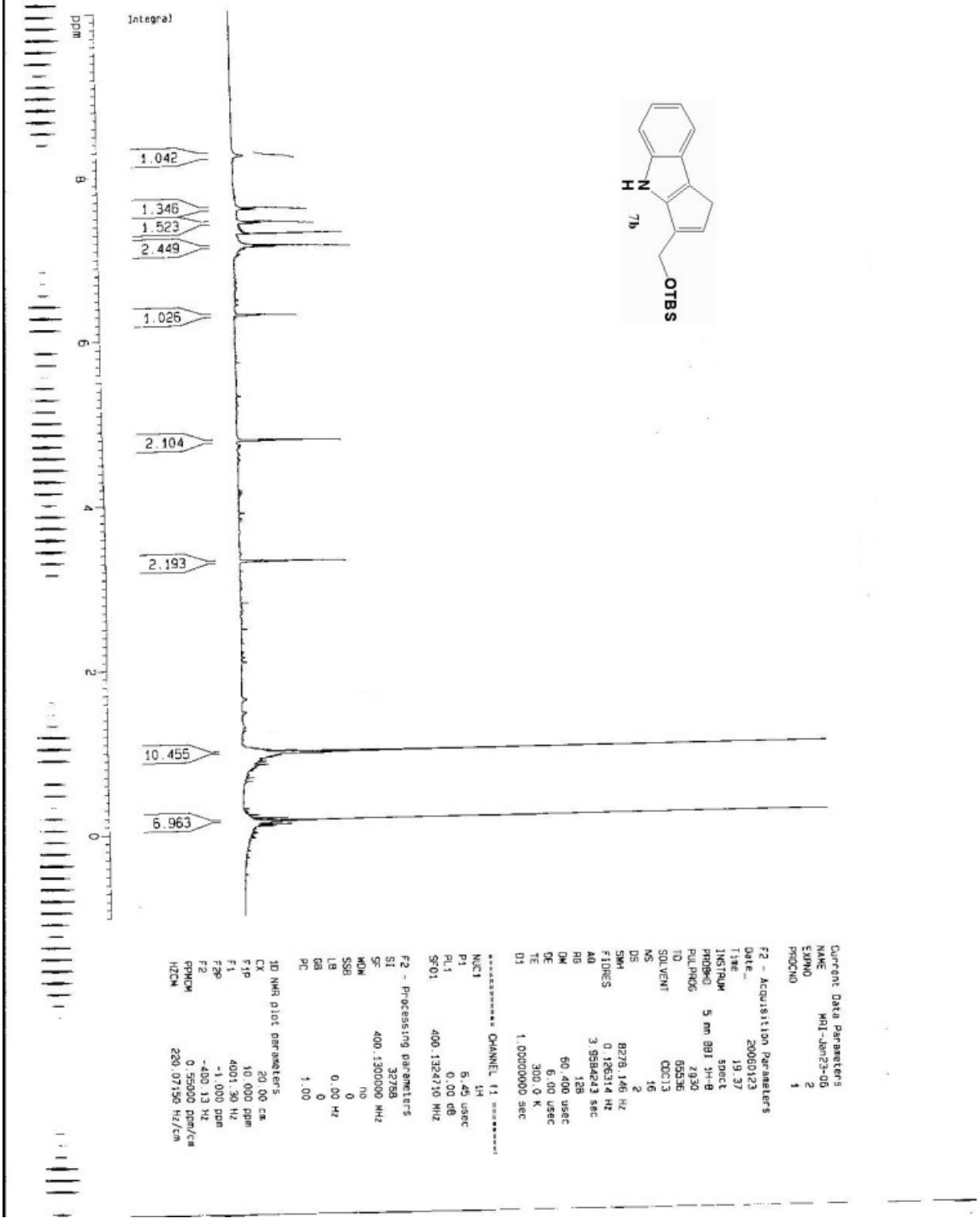


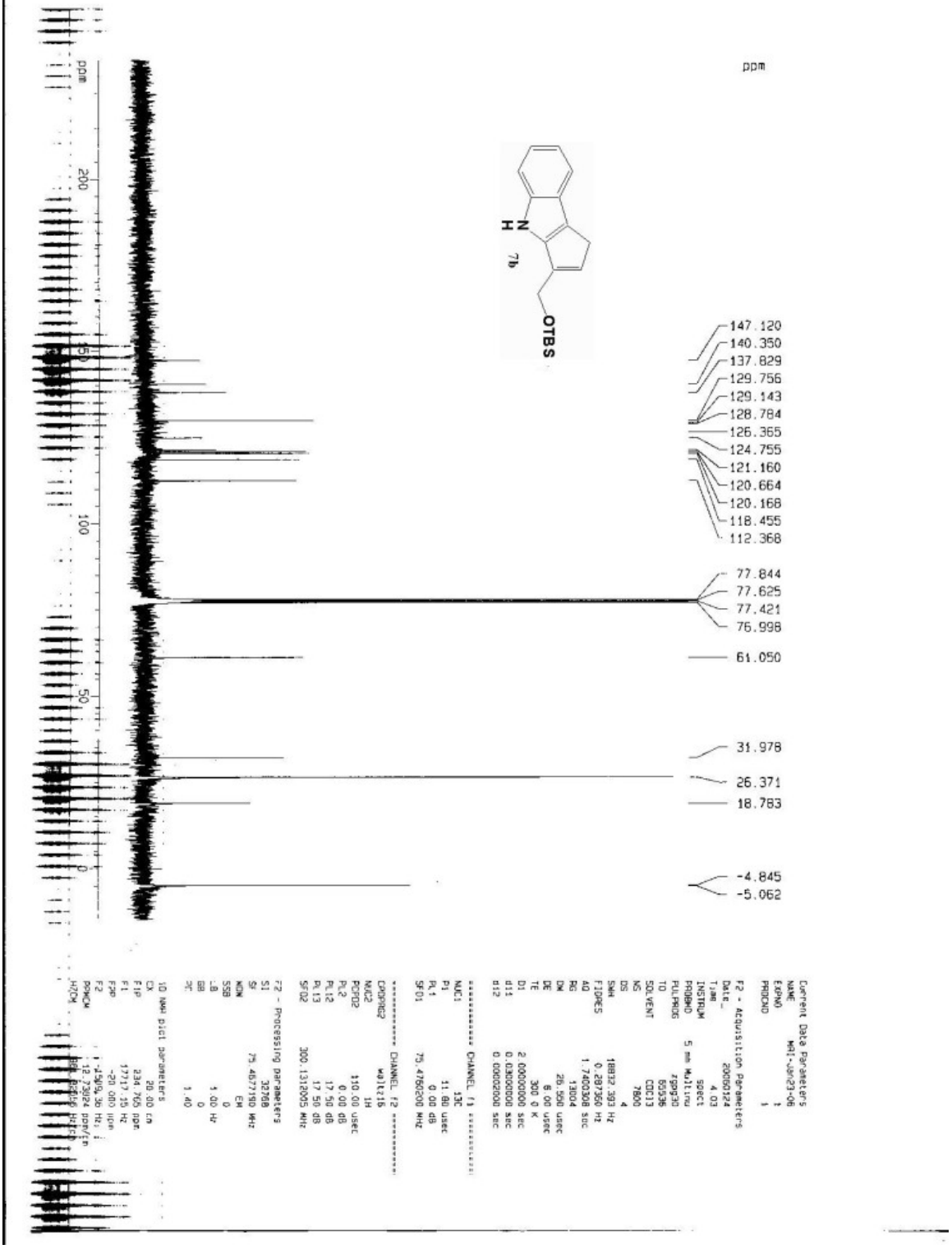

```
Current Data Parameters
NAME                  MRJ-Jan22-06
EXPNO                          3
PROCNO                         1

F2 - Acquisition Parameters
Date_                 20060122
Time                      17.52
INSTRUM                   spect
PROBHD     5 mm QNP 1H/1
PULPROG                   zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                            8
DS                            4
SWH                   18796.992 Hz
FIDRES                0.286819 Hz
AQ                    1.7433076 sec
RG                          512
DW                       26.600 usec
DE                        6.00 usec
TE                       300.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
D12                   0.00002000 sec

============ CHANNEL f1 ============
NUC1                         13C
P1                          5.40 usec
PL1                      -5.00 dB
SFO1                  75.4105357 MHz

============ CHANNEL f2 ============
CPDPRG2                   waltz16
NUC2                         1H
PCPD2                     115.00 usec
PL2                       0.00 dB
PL12                     20.00 dB
PL13                     20.00 dB
SFO2                  299.8711995 MHz

F2 - Processing parameters
SI                        32768
SF                    75.4023410 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                       20.00 cm
F1P                     234.000 ppm
F1                    17644.15 Hz
F2P                    -20.000 ppm
F2                    -1508.05 Hz
PPMCM                 12.70000 ppm/cm
HZCM                  957.60968 Hz/cm
```

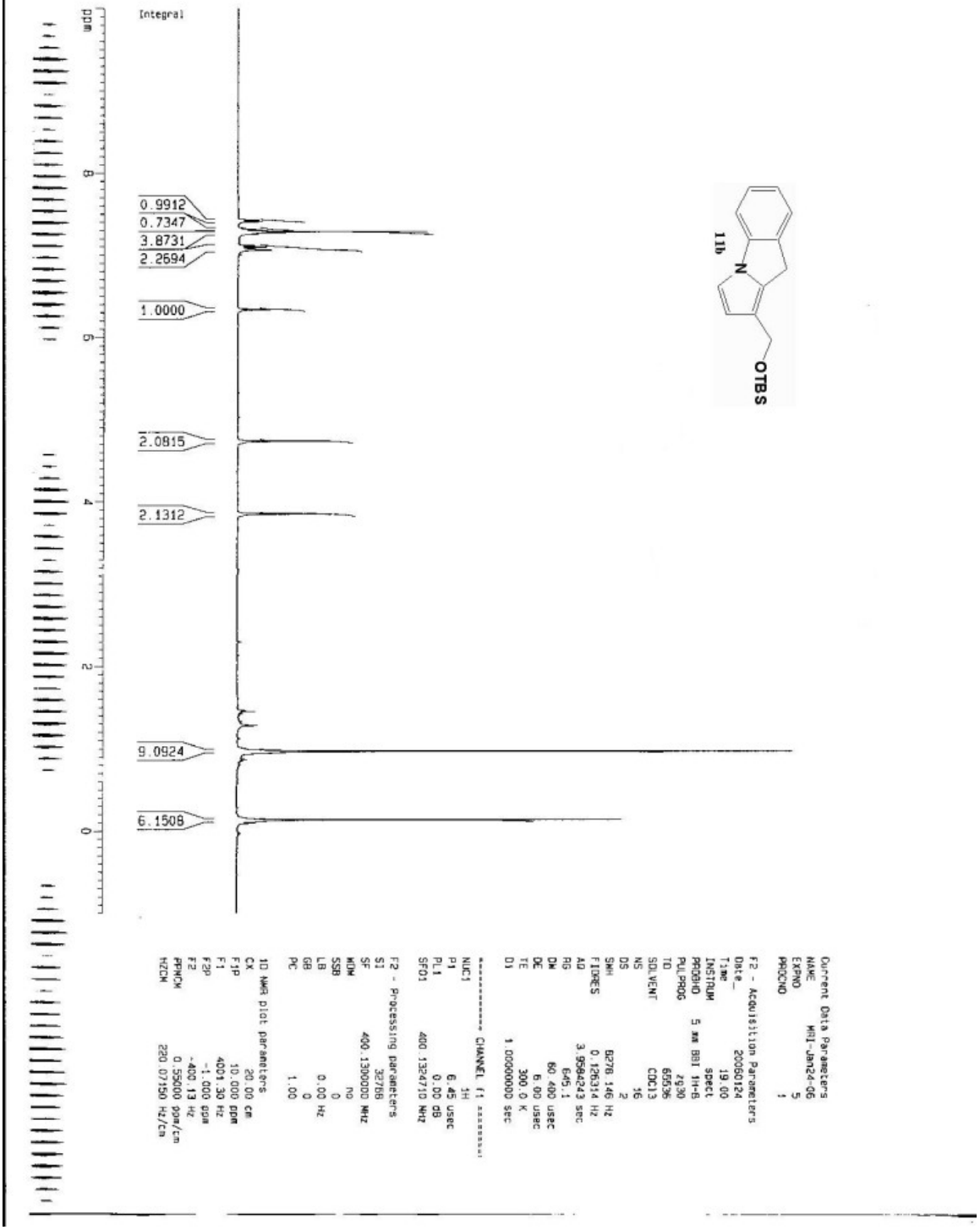


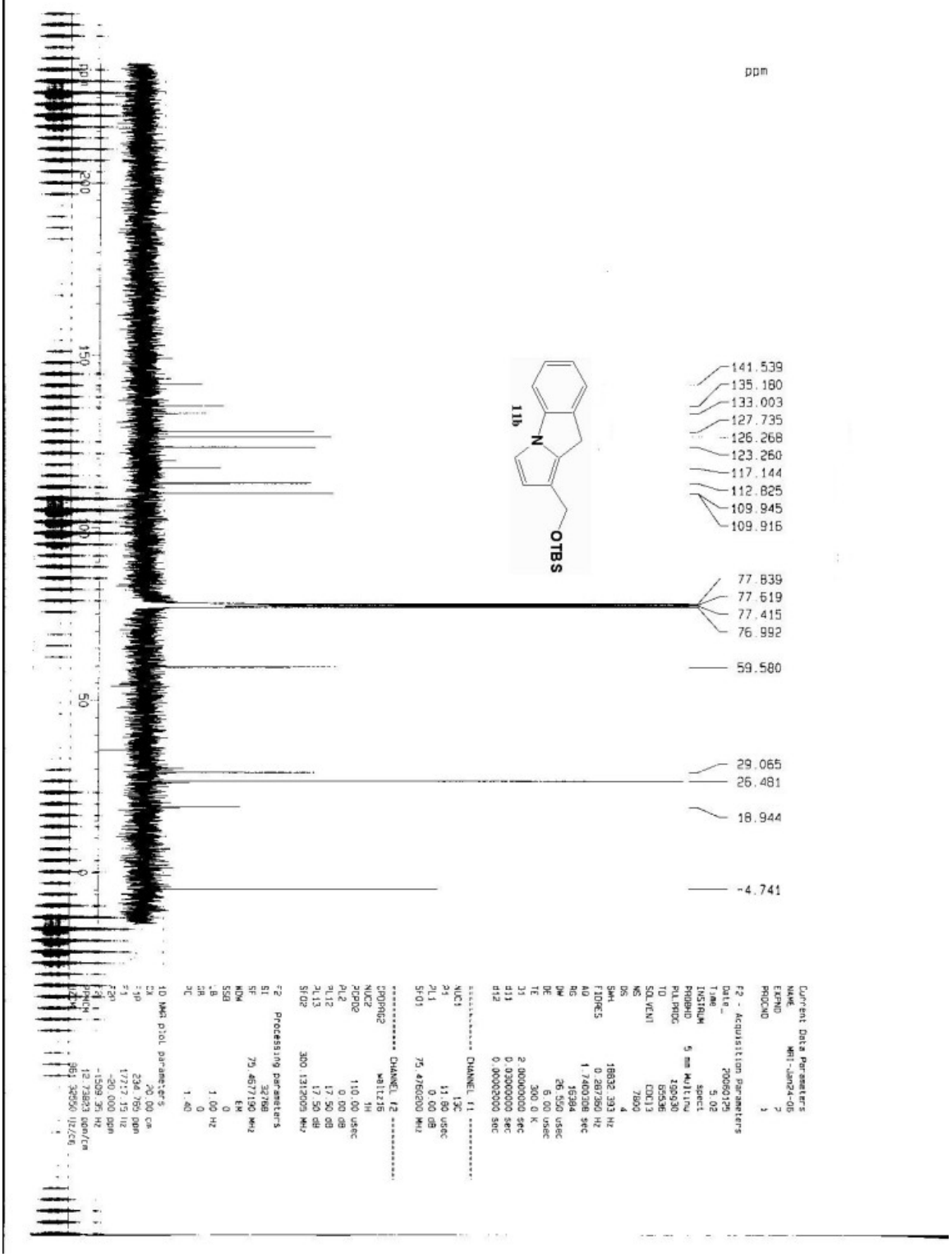

```
Current Data Parameters
NAME          MRI-JUN03-05
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_              20050603
Time                 20.01
INSTRUM               spect
PROBHD   5 mm QNP 1H/1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              6172.839 Hz
FIDRES           0.094190 Hz
AQ               5.3084880 sec
RG                   724.1
DW                61.000 usec
DE                 6.00 usec
TE                 300.0 K
D1               1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  11.70 usec
PL1                  0.00 dB
SFO1         299.8718518 MHz

F2 - Processing parameters
SI                    32768
SF           299.8700000 MHz
WDW                      no
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                   20.00 cm
F1P                 10.000 ppm
F1                2998.70 Hz
F2P                -1.000 ppm
F2                -299.87 Hz
PPMCM             0.55000 ppm/cm
HZCM              164.92850 Hz/cm
```

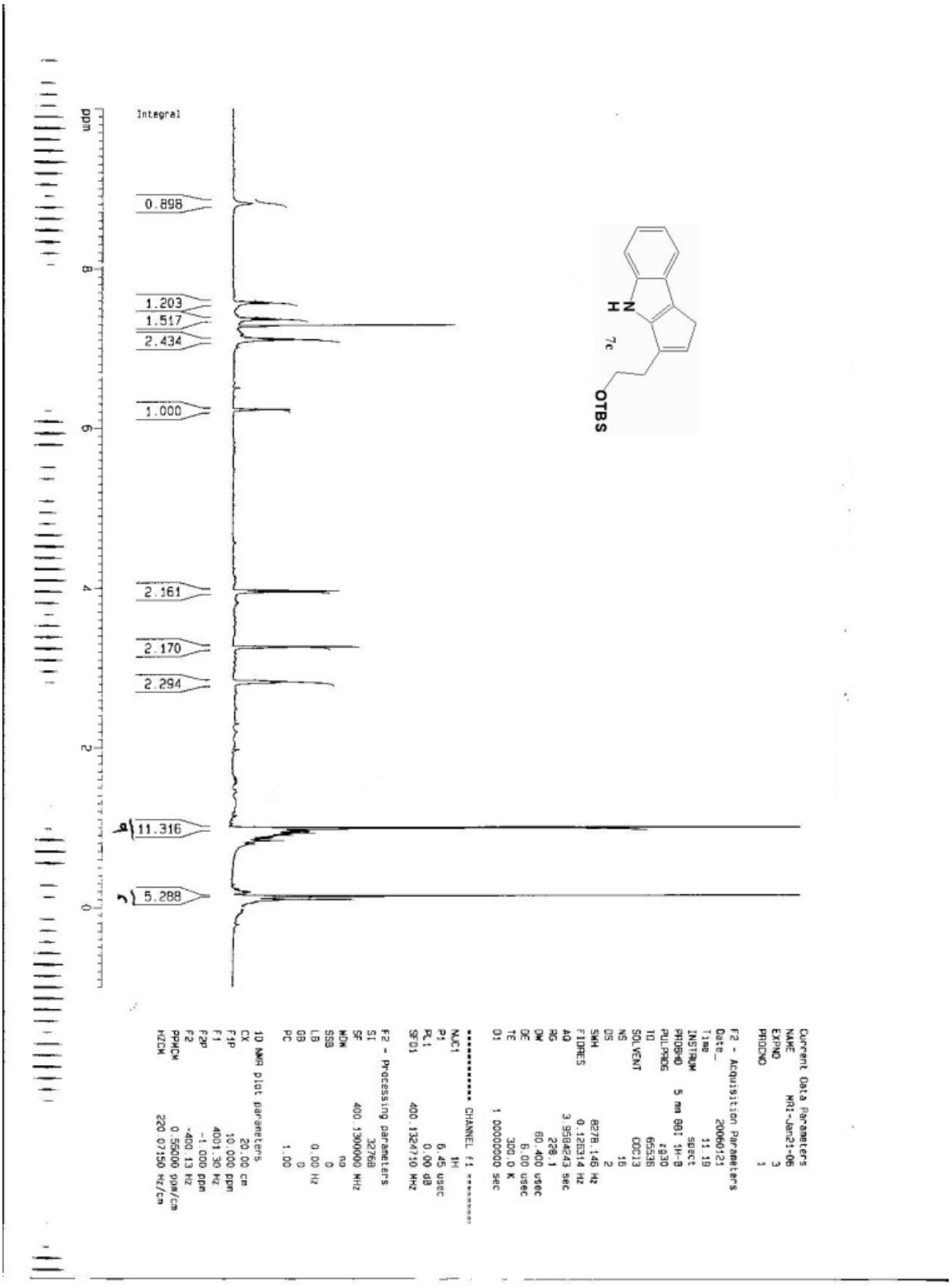

ppm
11a
N
CH3
141.826
135.302
132.681
127.736
126.302
122.995
114.836
111.881
109.822
109.565
77.860
77.639
77.436
77.013
28.357
11.725
Current Data Parameters
NAME MRI-JUN13-05
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050604
Time 5.36
INSTRM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 9000
DS 4
SWH 18796.992 Hz
FIDRES 0.286919 Hz
AQ 1.7433078 sec
RG 512
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 5.40 usec
PL1 -5.00 dB
SFO1 75.4105357 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711955 MHz
F2 - Processing parameters
SI 32768
SF 75.4023410 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
FIP 224.637 ppm
FI 17692.21 Hz
FP -14.552 ppm
F2 -1104.76 Hz
PPMCM 12.46445 ppm/cm
HZCM 939.64955 Hz/cm

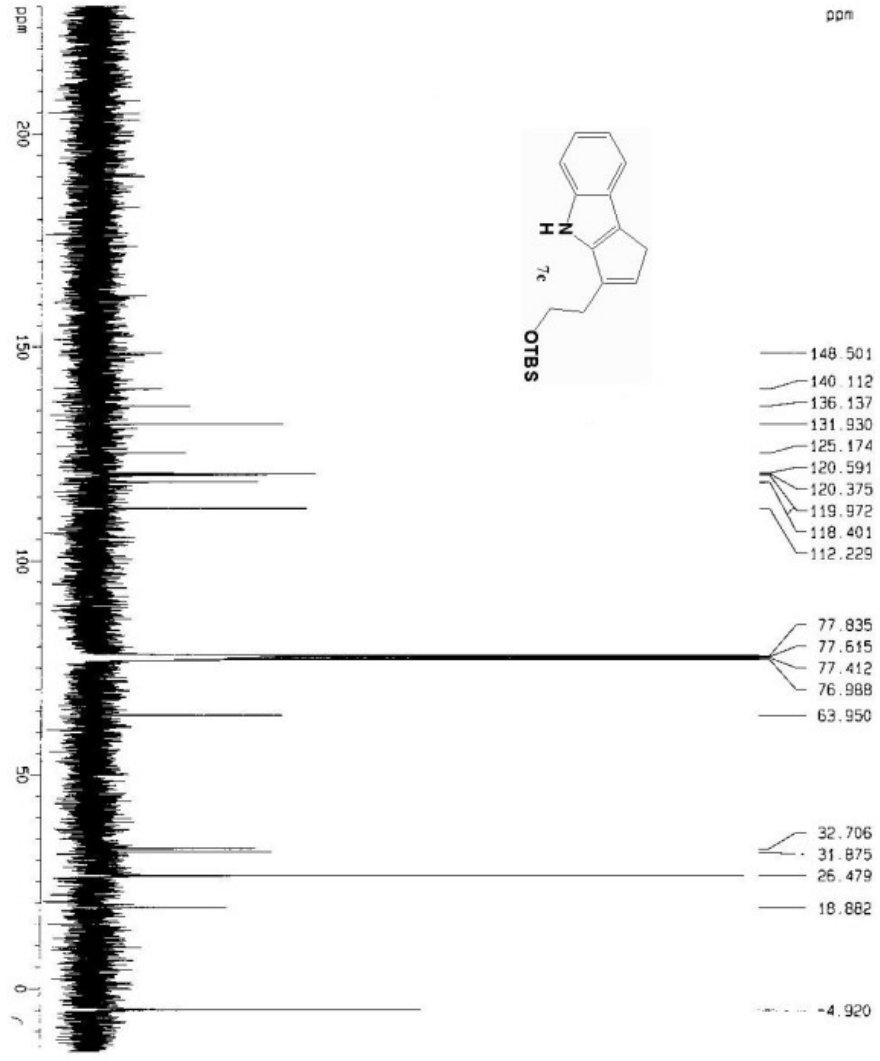

Integral
ppm
1.042
1.346
1.523
2.449
1.026
2.104
2.193
10.455
6.963
N
H
7b
OTBS
Current Data Parameters
NAME MRI-Jan23-06
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060123
Time 19.37
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 128
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm


ppm
147.120
140.350
137.829
129.756
129.143
128.784
126.365
124.755
121.160
120.664
120.168
118.455
112.368
77.844
77.625
77.421
76.998
61.050
31.978
26.371
18.783
-4.845
-5.062
OTBS
7b
N
H

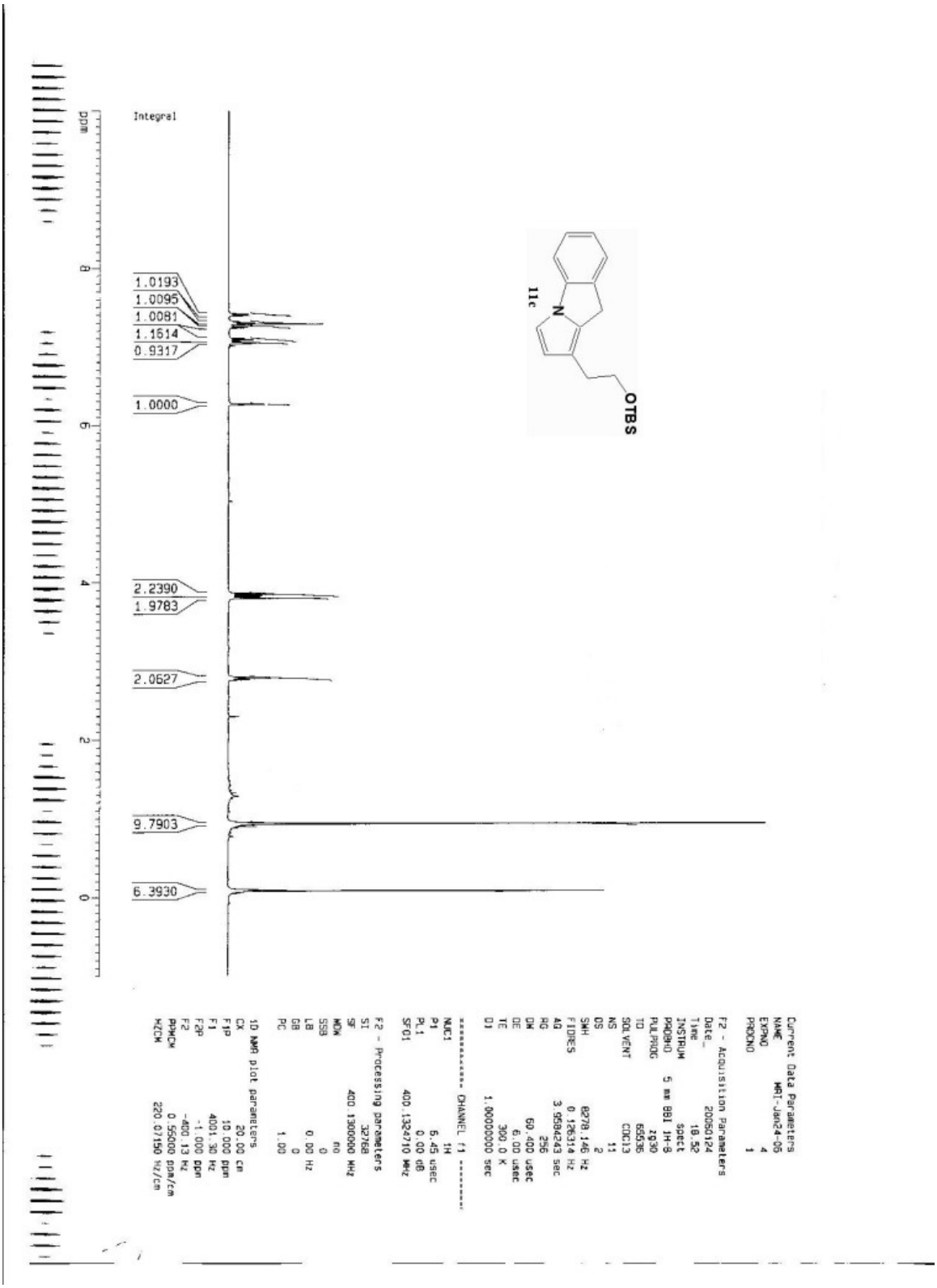

Integral
ppm
0.9912
0.7347
3.8731
2.2694
1.0000
2.0815
2.1312
9.0924
6.1508
11b
N
OTBS
Current Data Parameters
NAME MH1-Jan24-06
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060124
Time 19.00
INSTRUM spect
PROBHD 5 mm BBI 1H-6
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 645.1
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.59500 ppm/cm
HZCM 220.07150 Hz/cm

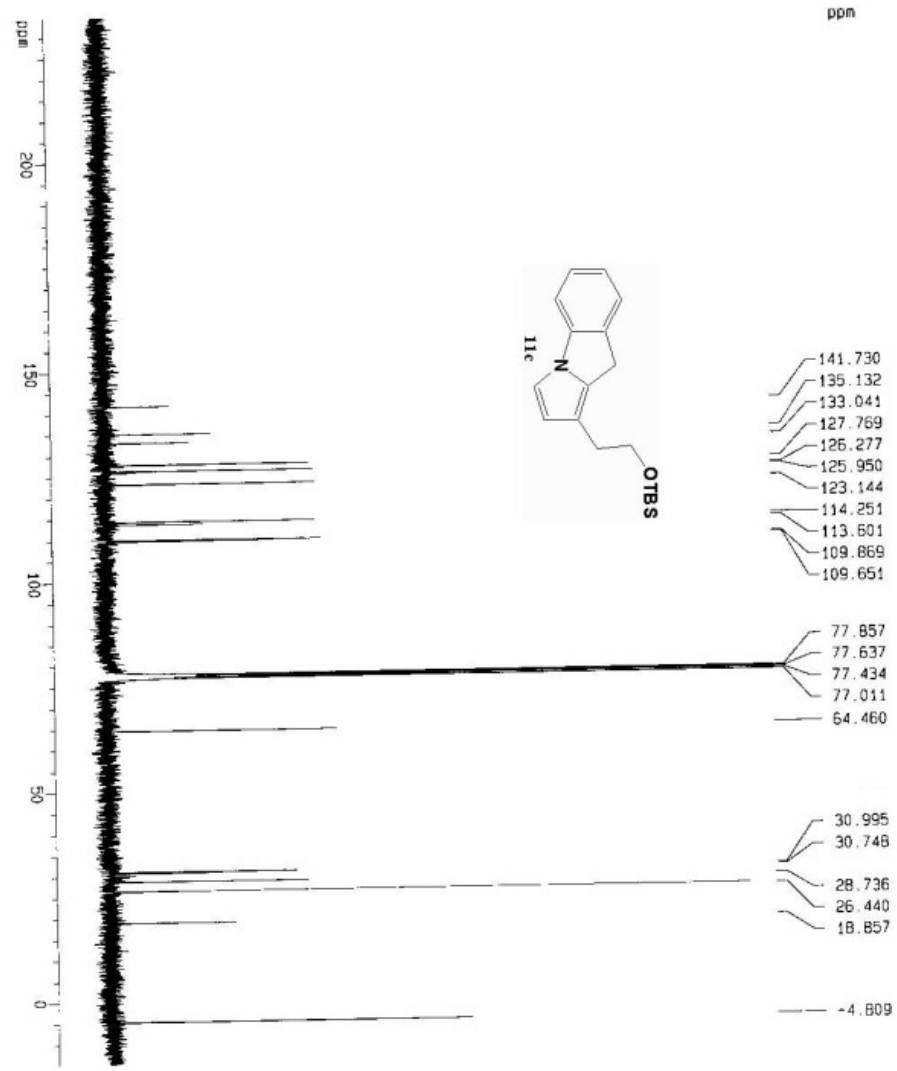

11b
OTBS
141.539
135.180
133.003
127.735
126.268
123.260
117.144
112.825
109.945
109.916
77.839
77.619
77.415
76.992
59.580
29.065
26.481
18.944
-4.741
ppm


Integral
0.898
1.203
1.517
2.434
1.000
2.161
2.170
2.294
11.316
5.288
ppm
H
N
7c
OTBS




```
Current Data Parameters
NAME                  MRI-Jan21-06
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_              20060121
Time                      15.02
INSTRUM                    spect
PROBHD     5 mm QNP 1H/1
PULPROG                 zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       4000
DS                          4
SWH                 18796.992 Hz
FIDRES              0.286819 Hz
AQ                  1.743076 sec
RG                       256
DW                    26.600 usec
DE                     6.00 usec
TE                    300.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
D12                 0.00002000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                       5.40 usec
PL1                    -6.00 dB
SFO1              75.4105357 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                       1H
PCPD2                 115.00 usec
PL2                    0.00 dB
PL12                  20.00 dB
PL13                  20.00 dB
SFO2              299.8711995 MHz

F2 - Processing parameters
SI                        32768
SF                75.4023410 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40

1D NMR plot parameters
CX                       20.00 cm
F1P                     230.000 ppm
F1                    17342.54 Hz
F2P                    -20.000 ppm
F2                    -1506.05 Hz
PPMCM                 12.50000 ppm/cm
HZCM                  942.52930 Hz/cm
```


Integral
1.0193
1.0095
1.0081
1.1614
0.9317
1.0000
2.2390
1.9783
2.0627
9.7903
6.3930
ppm
11c
N
OTBS
Current Data Parameters
NAME MRT-Jan24-05
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050124
Time 18.52
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 11
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.01150 Hz/cm



Current Data Parameters
NAME MRI-Jan24-06
EXPNO 2
PROCNO 1

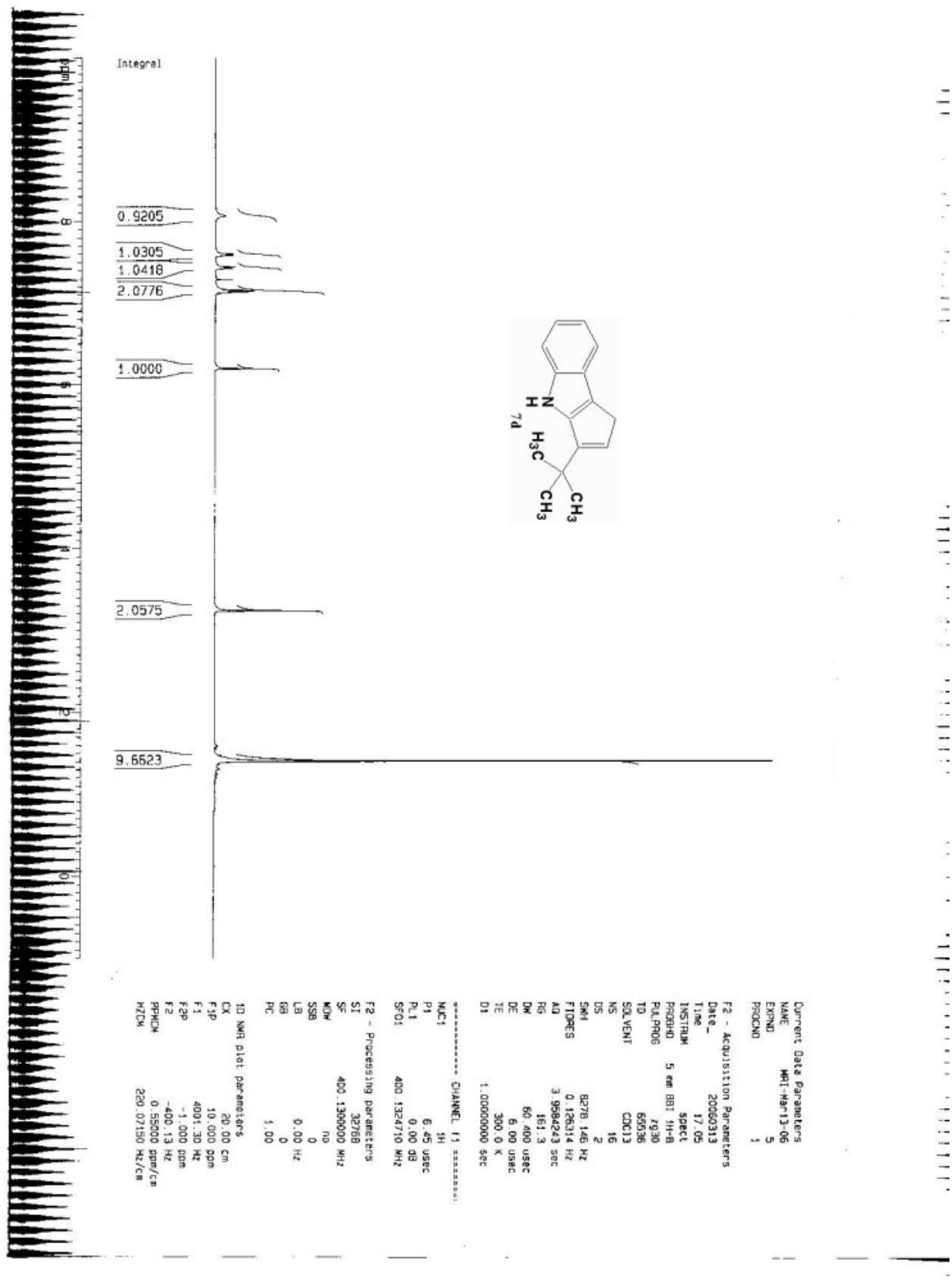
F2 - Acquisition Parameters
Date_ 20060125
Time 5.05
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 7500
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AQ 1.7433076 sec
RG 512
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

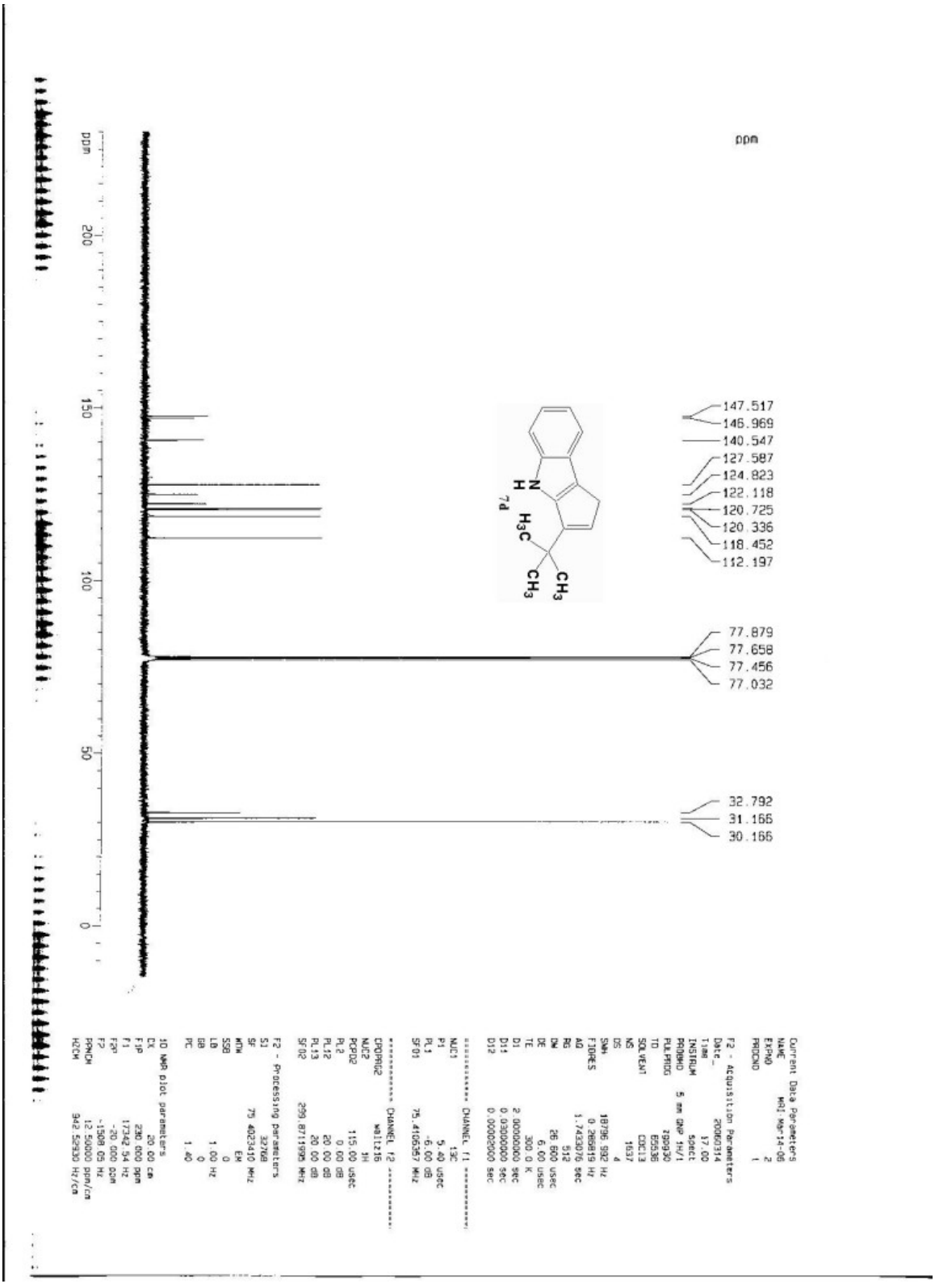
======== CHANNEL f1 ========
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SFO1 75.4106357 MHz

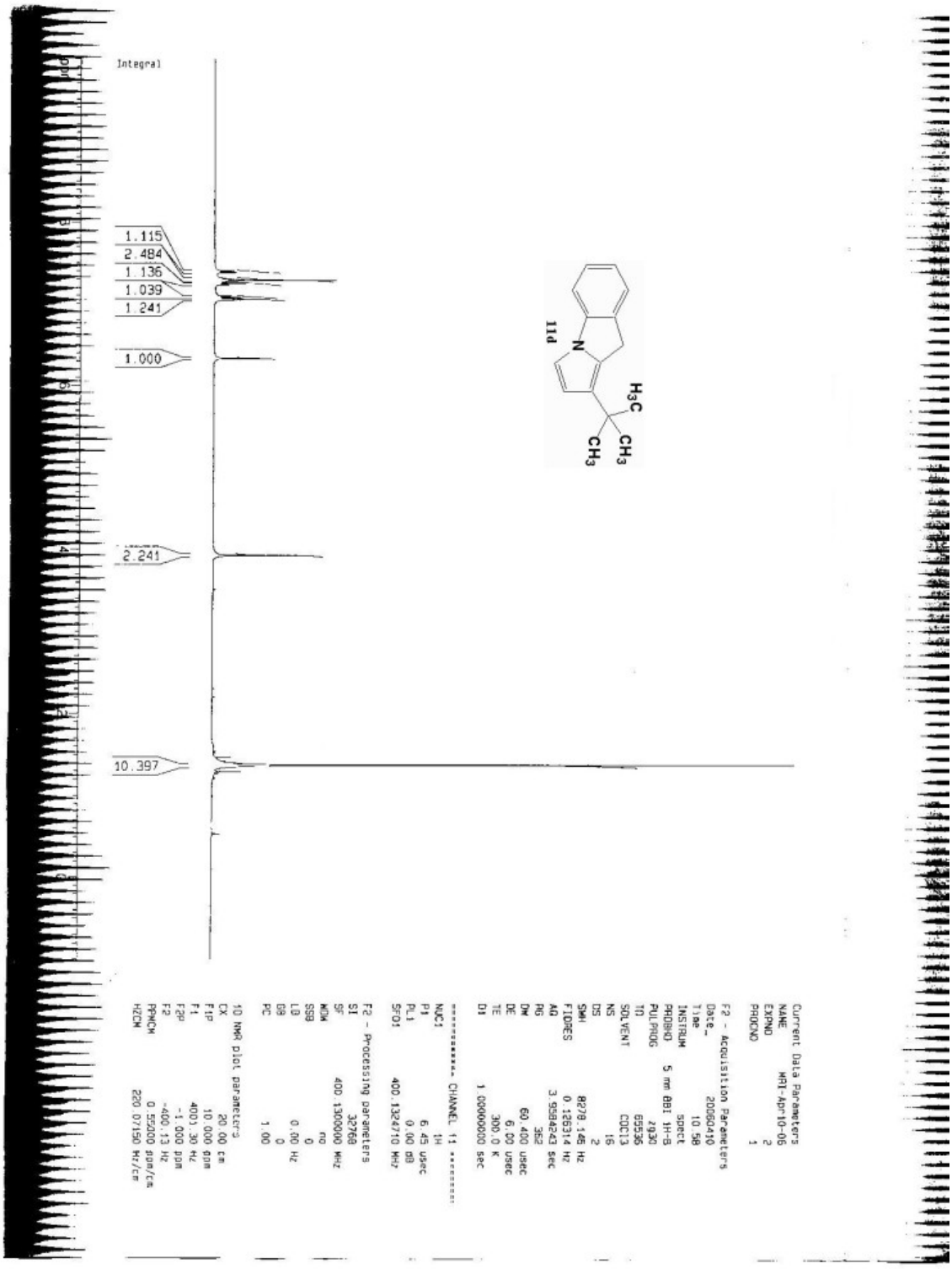
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 289.8711995 MHz

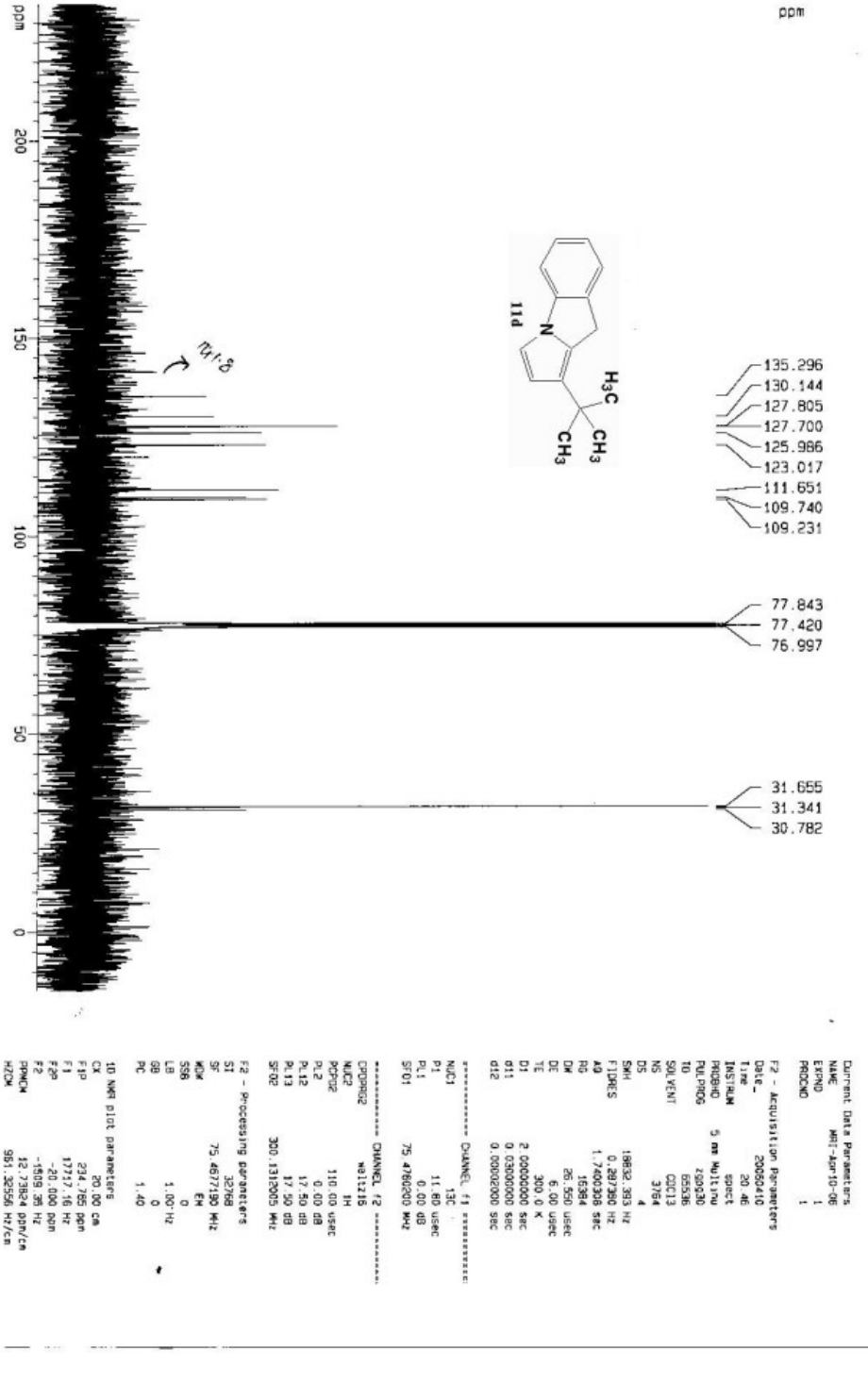
F2 - Processing parameters
SI 32768
SF 75.4023410 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

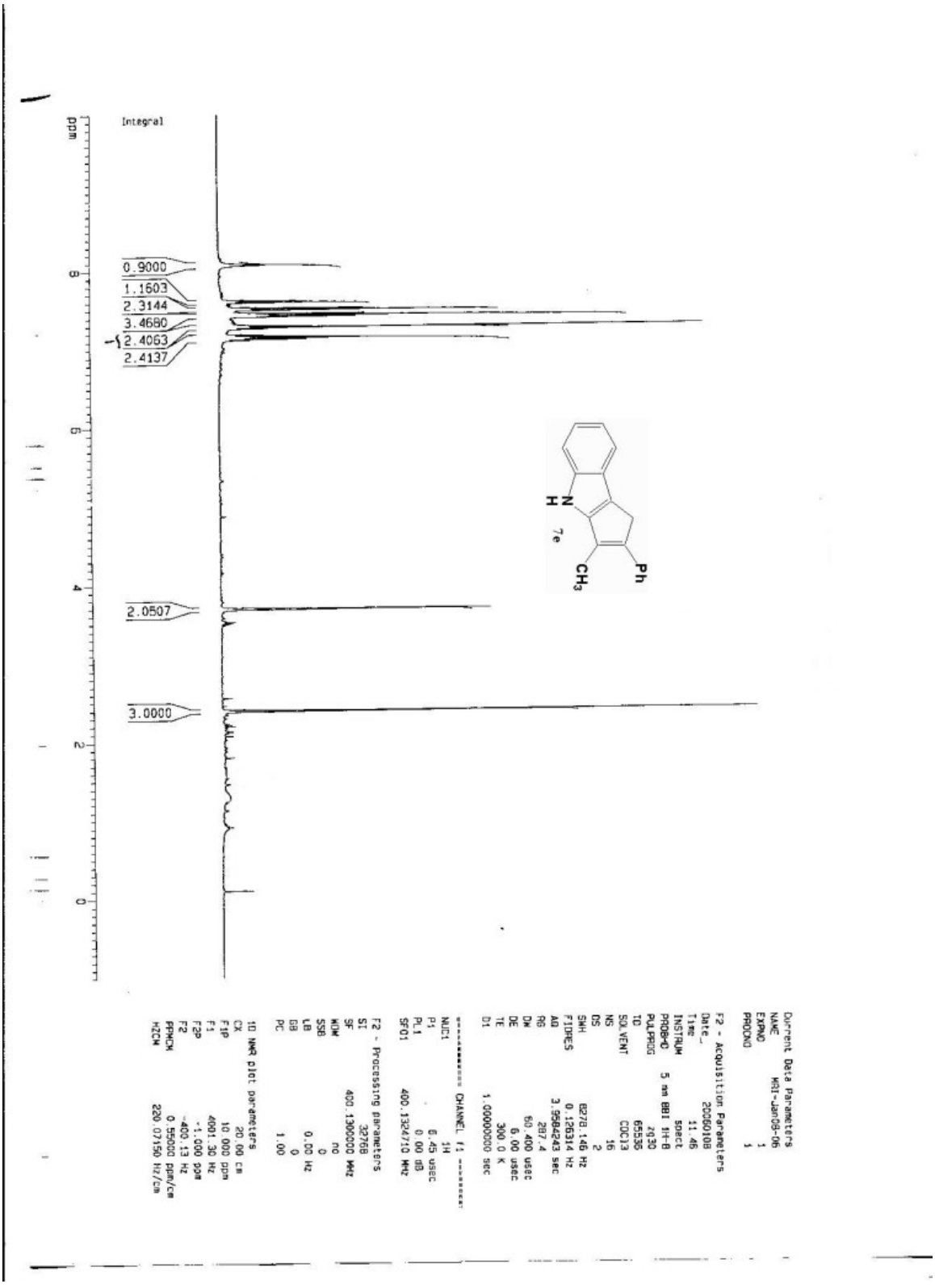
1D NMR plot parameters
CX 20.00 cm
F1P 234.651 ppm
F1 17693.24 Hz
F2P -20.000 ppm
F2 -1508.05 Hz
PPMCM 12.73255 ppm/cm
HZCM 960.08433 Hz/cm


Integral
0.9205
1.0305
1.0418
2.0776
1.0000
2.0575
9.6523
ppm
H
N
7d
H3C
CH3
CH3
Current Data Parameters
NAME MRI-Mar13-06
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060313
Time 17.05
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 161.3
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm


ppm
147.517
146.969
140.547
127.587
124.823
122.118
120.725
120.336
118.452
112.197
77.879
77.658
77.456
77.032
32.792
31.165
30.165
7d
H3C
CH3
CH3
Current Data Parameters
NAME MRI Mar14-08
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080314
Time 17.00
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 1637
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AQ 1.7433076 sec
RG 512
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
========== CHANNEL f1 ==========
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SF01 75.4105357 MHz
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SF02 299.8711908 MHz
F2 - Processing parameters
SI 32768
SF 75.4023410 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 230.000 ppm
F1 17342.54 Hz
F2P -20.000 ppm
F2 -1508.05 Hz
PPMCM 12.50000 ppm/cm
HZCM 942.52930 Hz/cm


Integral
1.115
2.484
1.136
1.039
1.241
1.000
2.241
10.397
11d
N
H3C
CH3
CH3
Current Data Parameters
NAME MRT-Apr10-06
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060410
Time 10.58
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
========== CHANNEL f1 ==========
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm


ppm
11d
N
H3C
CH3
CH3
135.296
130.144
127.805
127.700
125.986
123.017
111.651
109.740
109.231
77.843
77.420
76.997
31.655
31.341
30.782
0
50
100
150
200



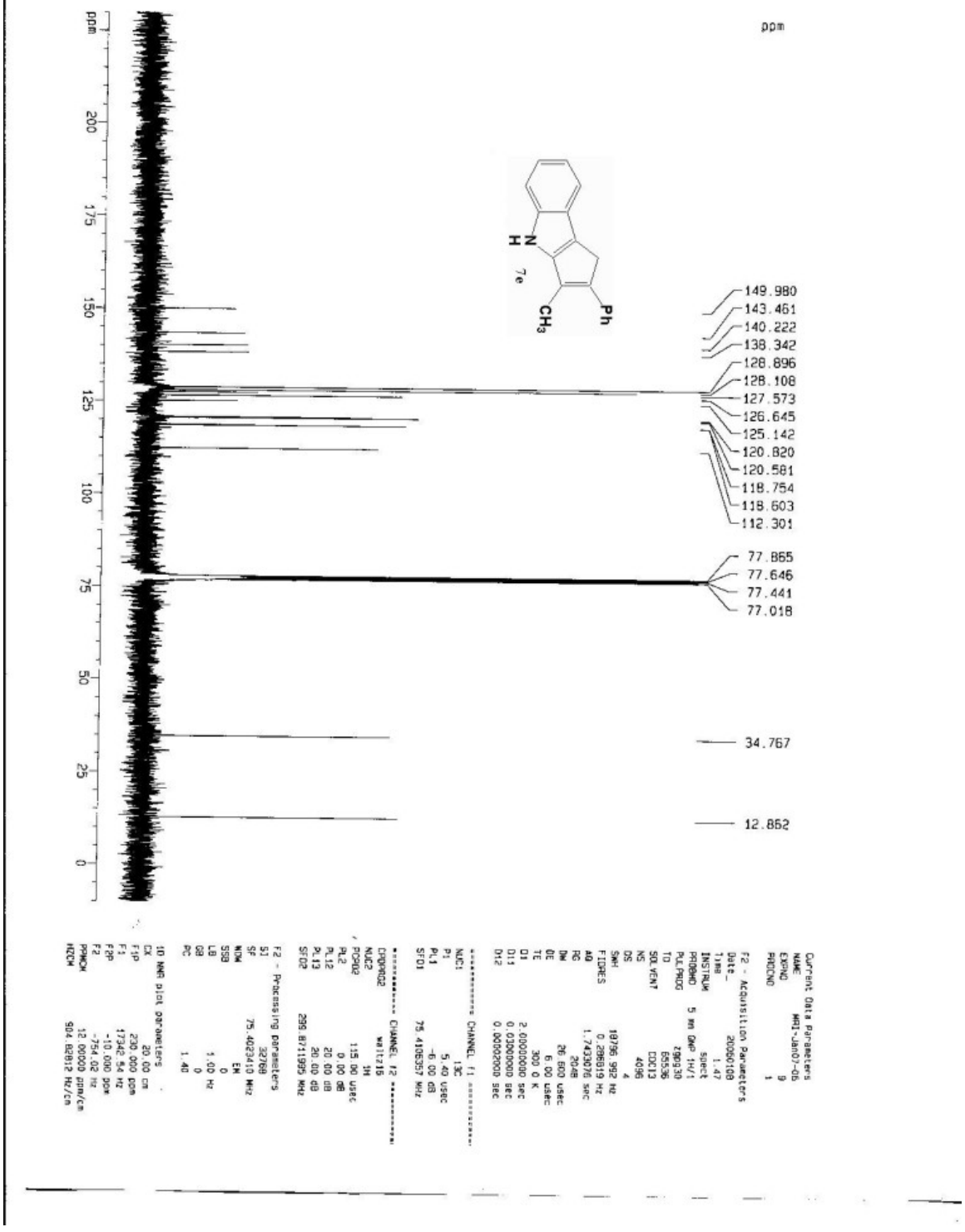
Current Data Parameters
NAME MRI-Jan09-06
EXPNO 1
PROCNO 1

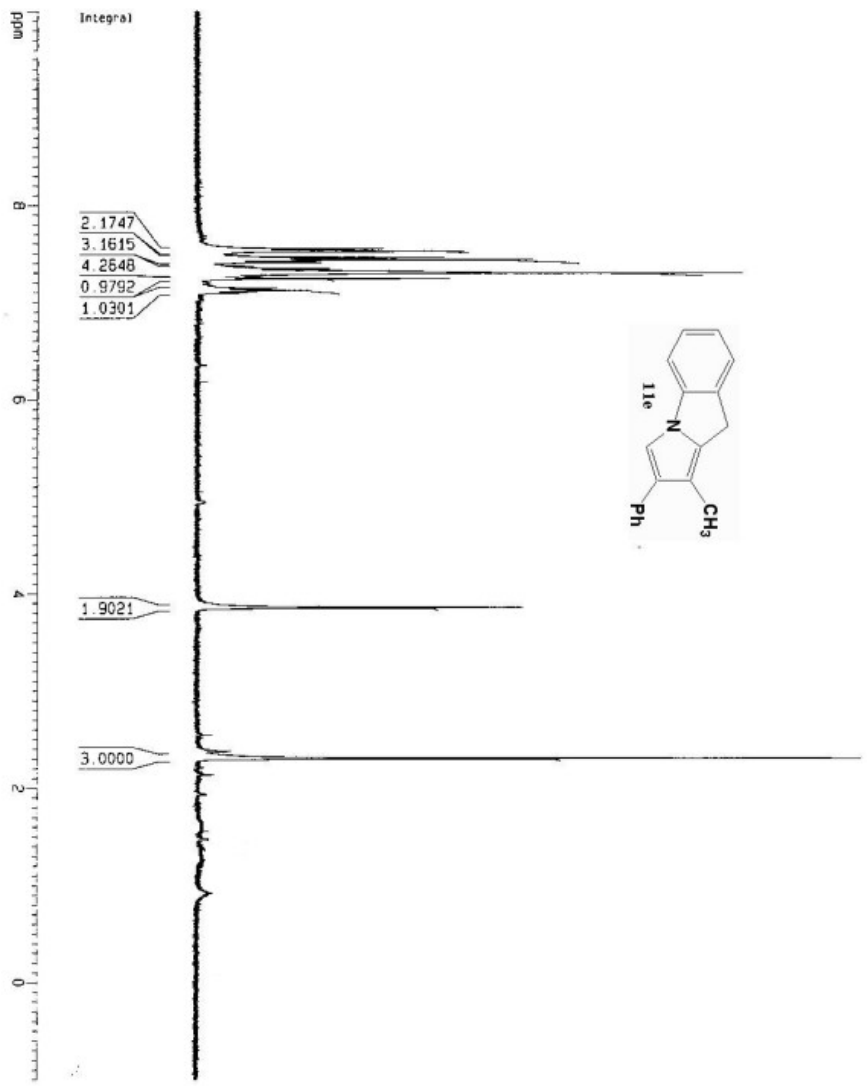
F2 - Acquisition Parameters
Date_ 20060108
Time 11.46
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 287.4
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 8.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

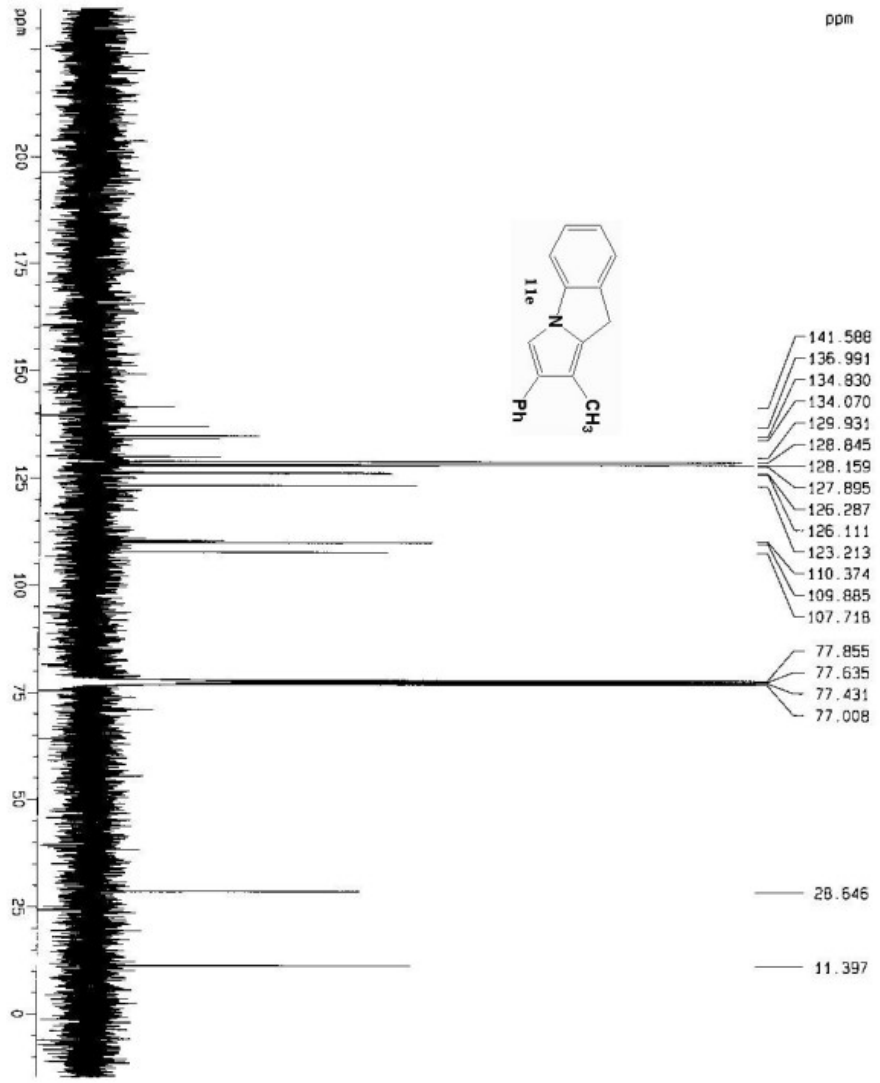
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm


ppm
149.980
143.461
140.222
138.342
128.896
128.108
127.573
126.645
125.142
120.820
120.581
118.754
118.603
112.301
77.865
77.646
77.441
77.018
34.767
12.852
7e
Ph
CH3
N
H




```
Current Data Parameters
NAME                  NHI-Jan07-06
EXPNO                          4
PROCNO                         1

F2 - Acquisition Parameters
Date_              20060107
Time                   14.25
INSTRUM                spect
PROBHD     5 mm QNP 1H/1
PULPROG                zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 6172.839 Hz
FIDRES              0.094190 Hz
AQ                  5.3084560 sec
RG                      1024
DW                   81.000 usec
DE                    6.00 usec
TE                   300.0 K
D1                  1.00000000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                      11.70 usec
PL1                      0.00 dB
SFO1          299.8718518 MHz

F2 - Processing parameters
SI                        32768
SF              299.8700000 MHz
WDW                       no
SSB                        0
LB                      0.00 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                     20.00 cm
F1P                   10.000 ppm
F1                  2998.70 Hz
F2P                   -1.000 ppm
F2                  -299.87 Hz
PPMCM                 0.55000 ppm/cm
HZCM                184.92850 Hz/cm
```



Current Data Parameters
NAME MRJ-Jan07-06
EXPNO 5
PROCNO 1

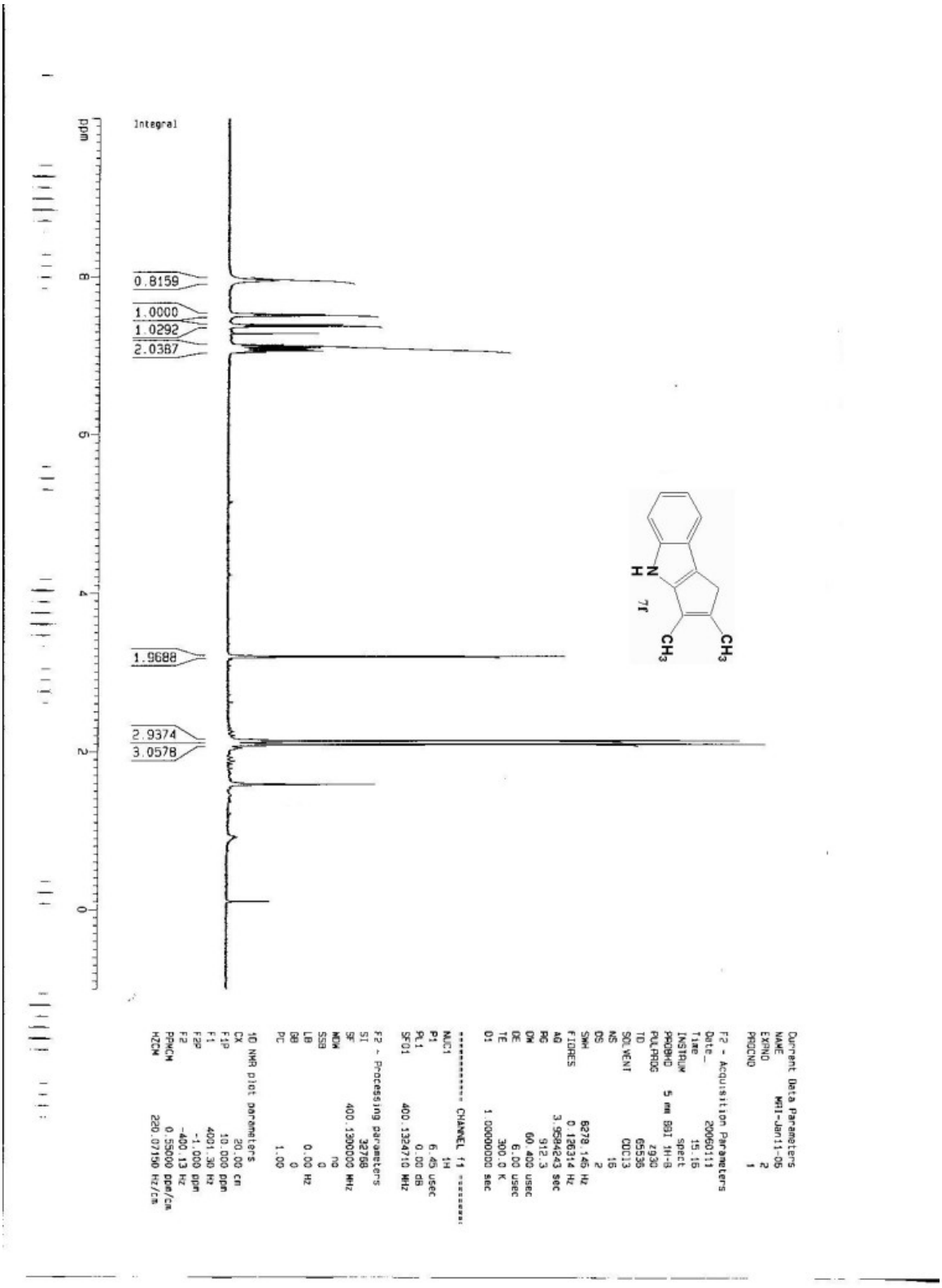
F2 - Acquisition Parameters
Date_ 20060107
Time 18.41
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AQ 1.7431075 sec
RG 512
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

====== CHANNEL f1 ======
NUC1 13C
P1 5.40 usec
PL1 -5.00 dB
SFO1 75.4109357 MHz

====== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711895 MHz

F2 - Processing parameters
SI 32768
SF 75.4023450 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 234.651 ppm
F1 17693.24 Hz
F2P -14.538 ppm
F2 -1103.75 Hz
PPMCM 12.46445 ppm/cm
HZCM 939.84967 Hz/cm



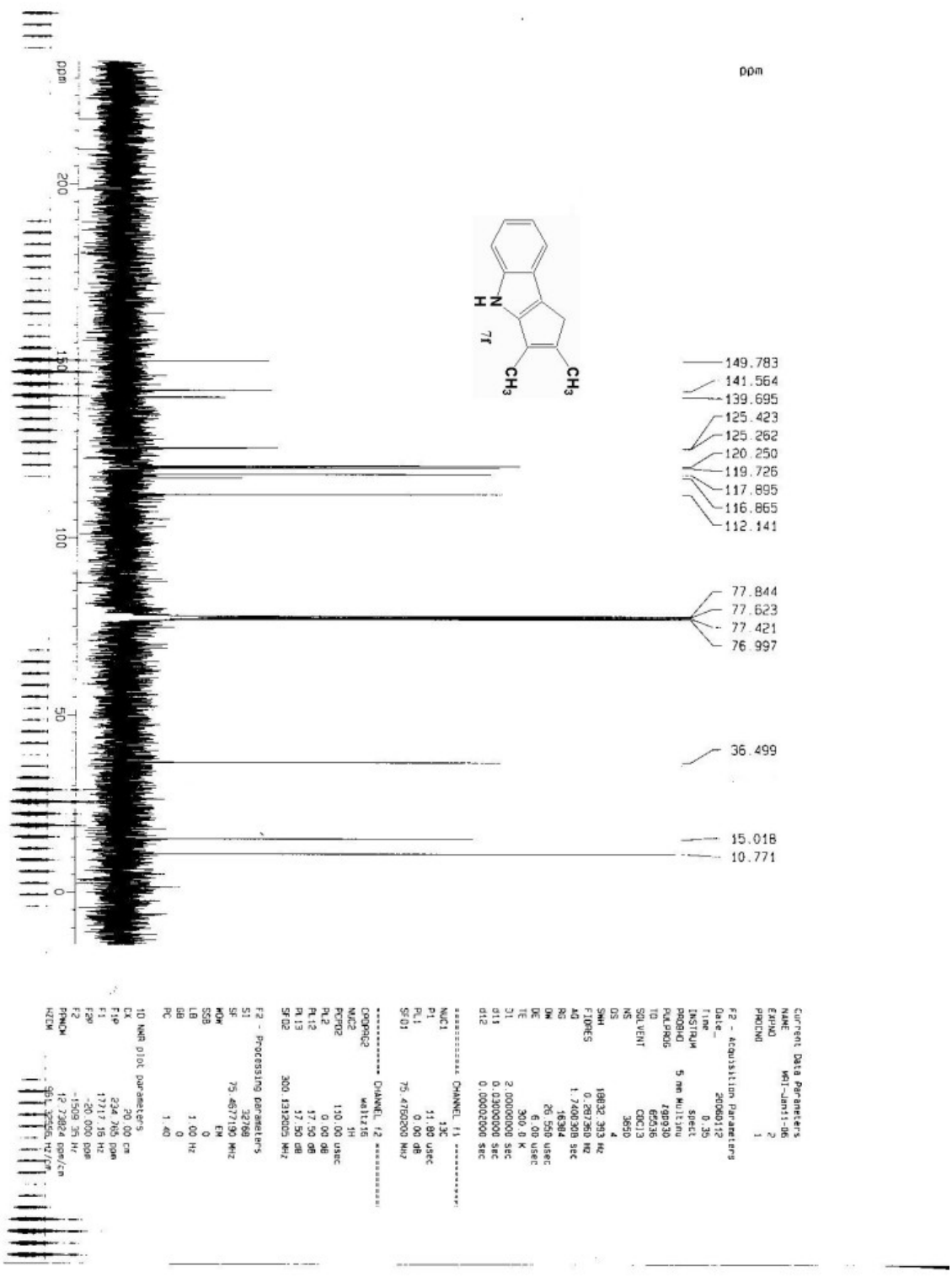
Current Data Parameters
NAME MRI-Jan11-05
EXPNO 2
PROCNO 1

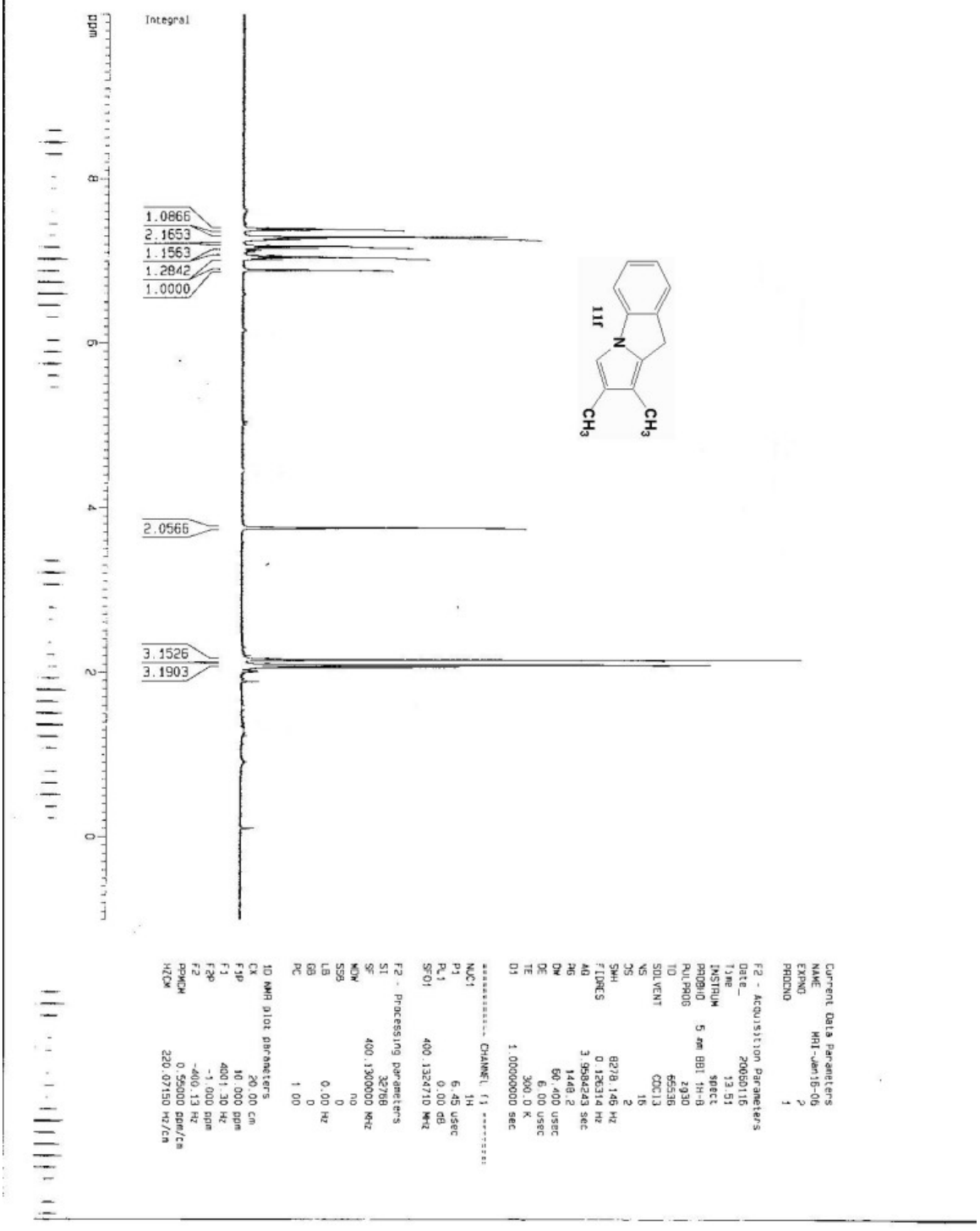
F2 - Acquisition Parameters
Date_ 20060111
Time 15.16
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6278.145 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 912.3
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

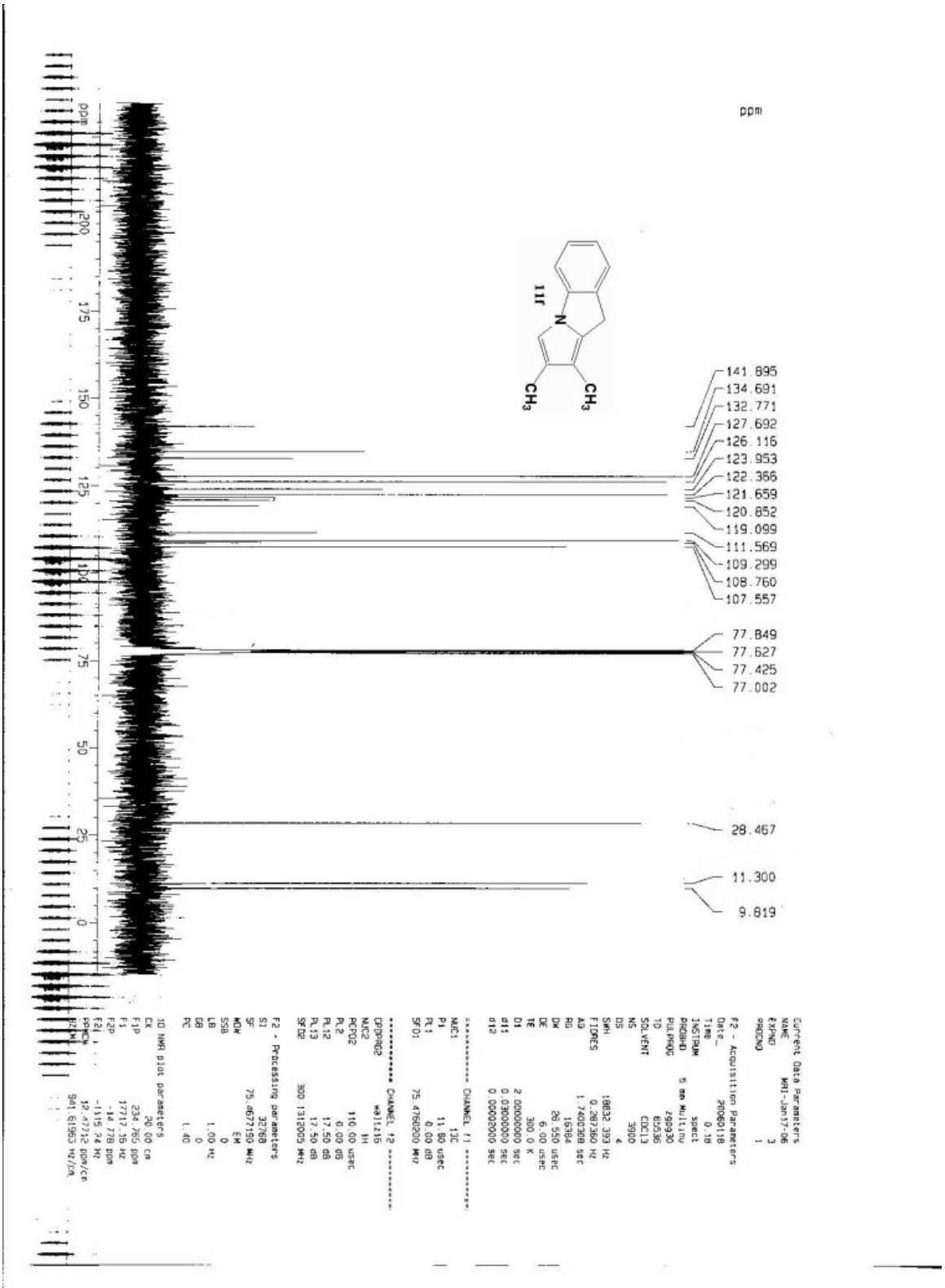
========== CHANNEL f1 ==========
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

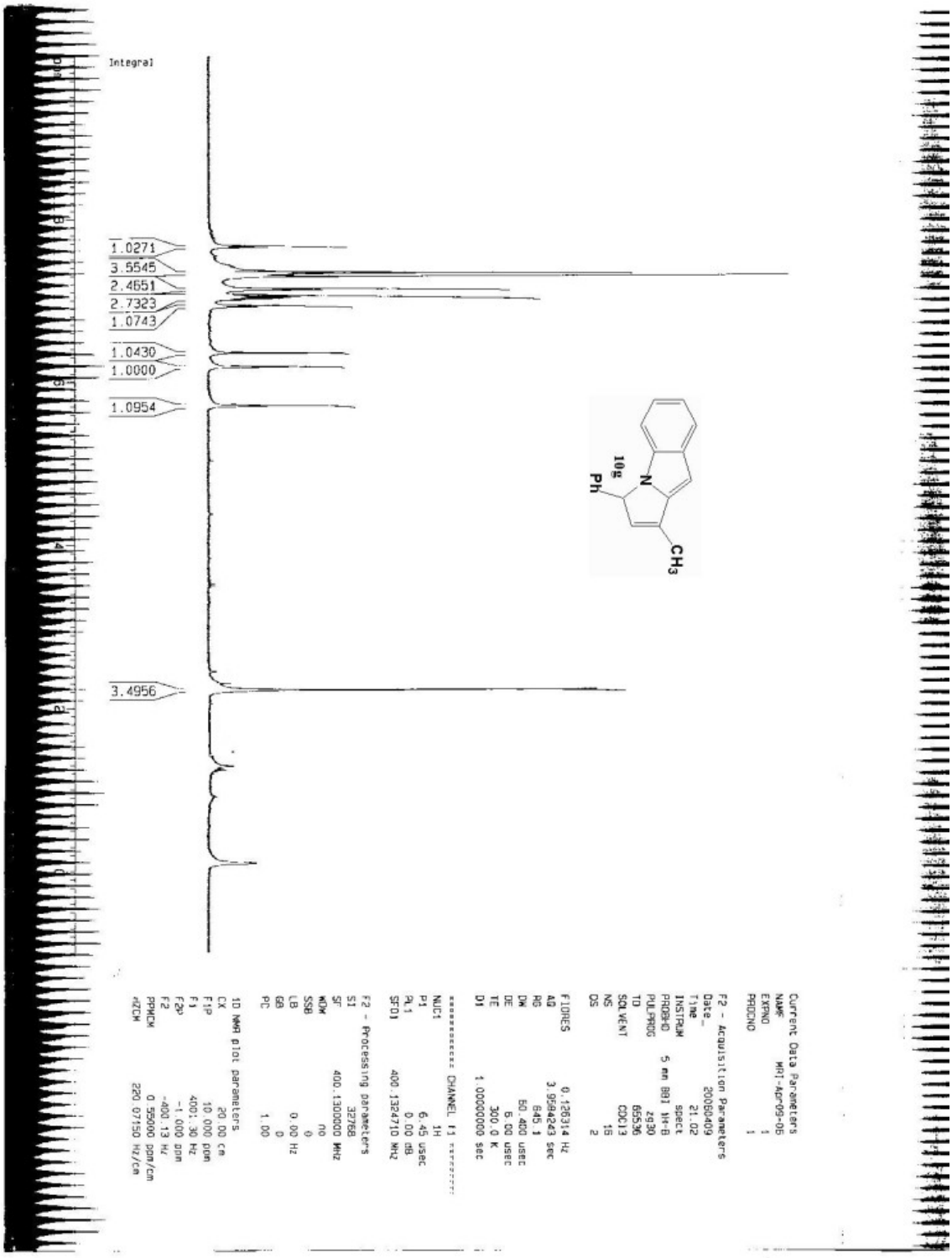
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

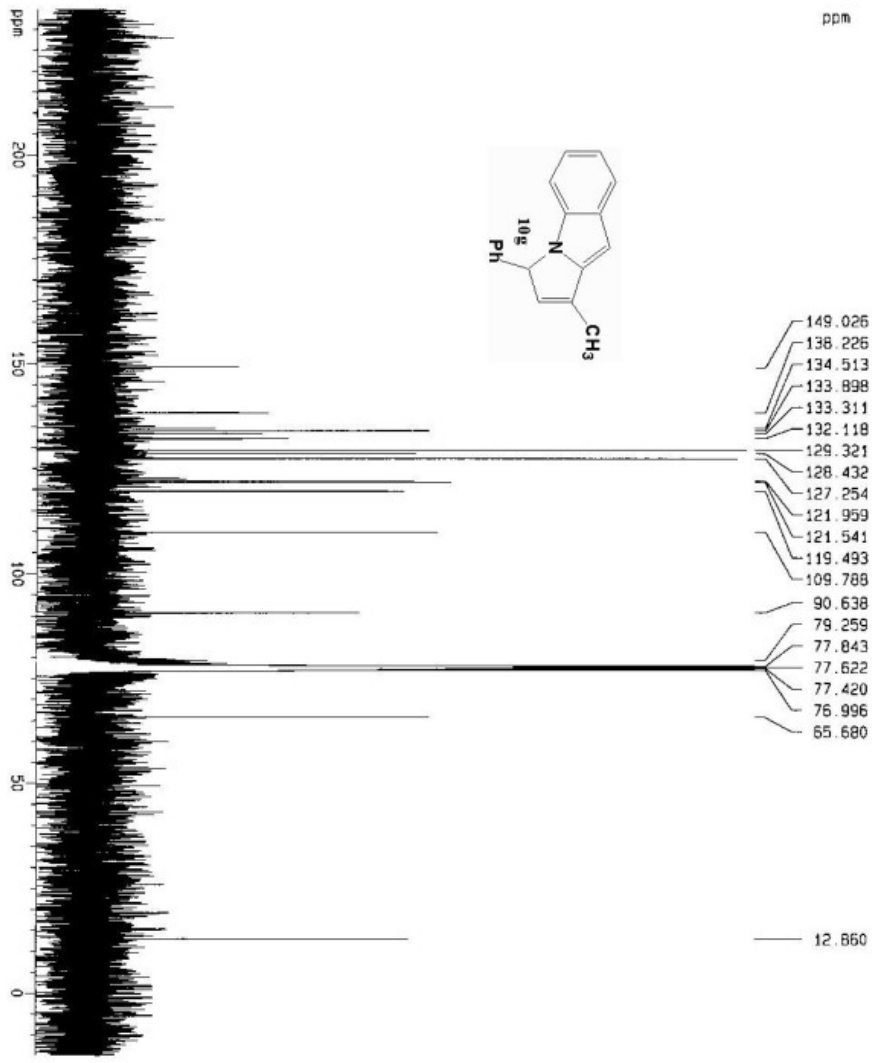
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm


ppm
149.783
141.564
139.695
125.423
125.262
120.250
119.726
117.895
116.865
112.141
77.844
77.623
77.421
76.997
36.499
15.018
10.771
CH3
CH3
N
H


Integral
1.0866
2.1653
1.1563
1.2842
1.0000
2.0566
3.1526
3.1903
ppm
JJ1
N
CH3
CH3


141.895
134.691
132.771
127.692
126.116
123.953
122.366
121.659
120.852
119.099
111.569
109.299
108.760
107.557
77.849
77.627
77.425
77.002
28.467
11.300
9.819
ppm
111
N
CH3
CH3


Integral
1.0271
3.5545
2.4651
2.7323
1.0743
1.0430
1.0000
1.0954
3.4956
10g
Ph
N
CH3
Current Data Parameters
NAME MRI-Apr09-06
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090409
Time 21.02
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
FIDRES 0.128314 Hz
AQ 3.9584243 sec
RG 645.1
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.36 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm



Current Data Parameters
NAME MEI-APR09-06
EXPNO 3
PROCNO 1

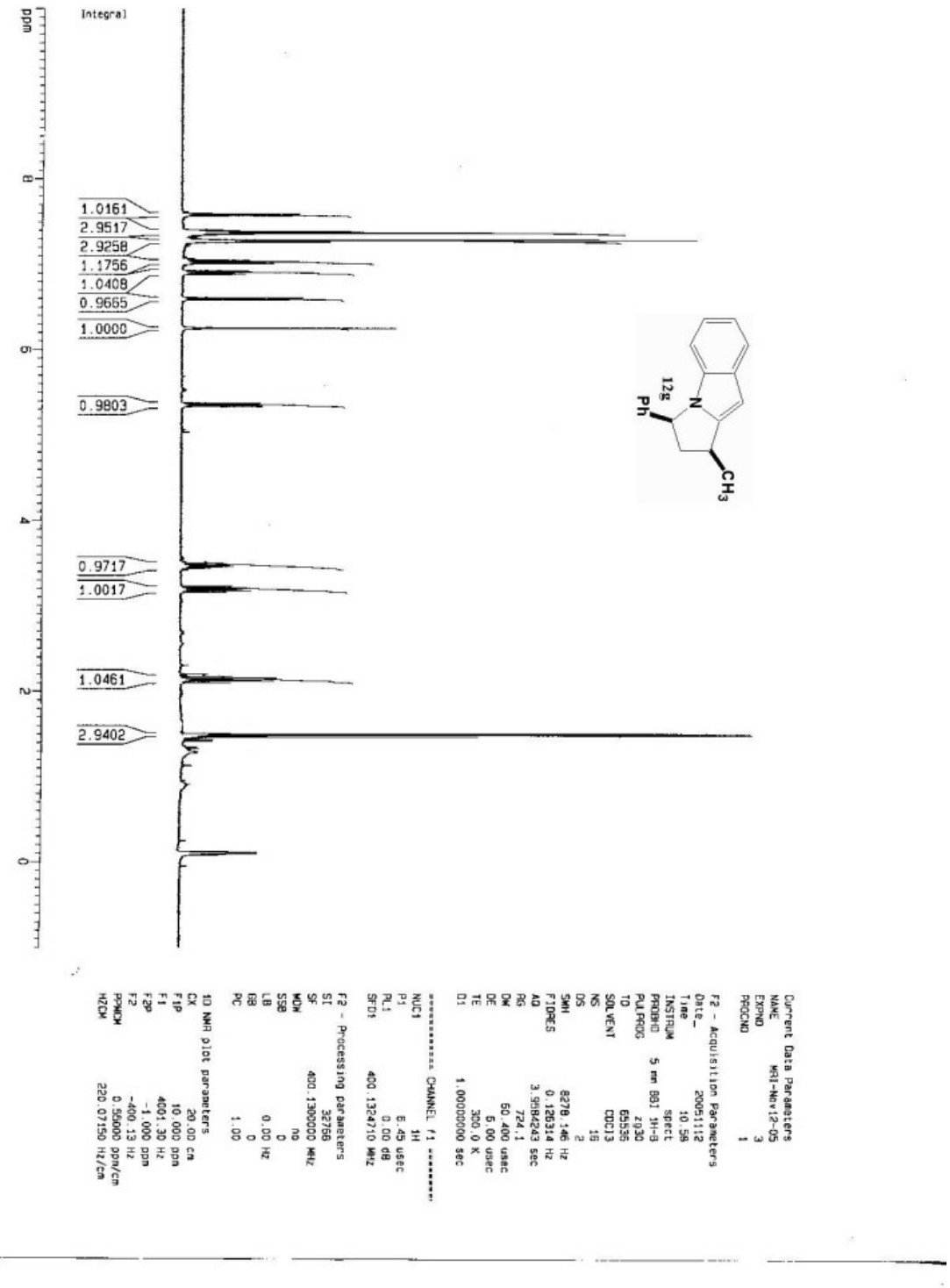
F2 - Acquisition Parameters
Date_ 20060410
Time 8.13
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 10449
DS 4
SWH 16032.393 Hz
FIDRES 0.367360 Hz
AQ 1.7400308 sec
RG 11586.2
DW 26.550 usec
DE 5.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00020000 sec

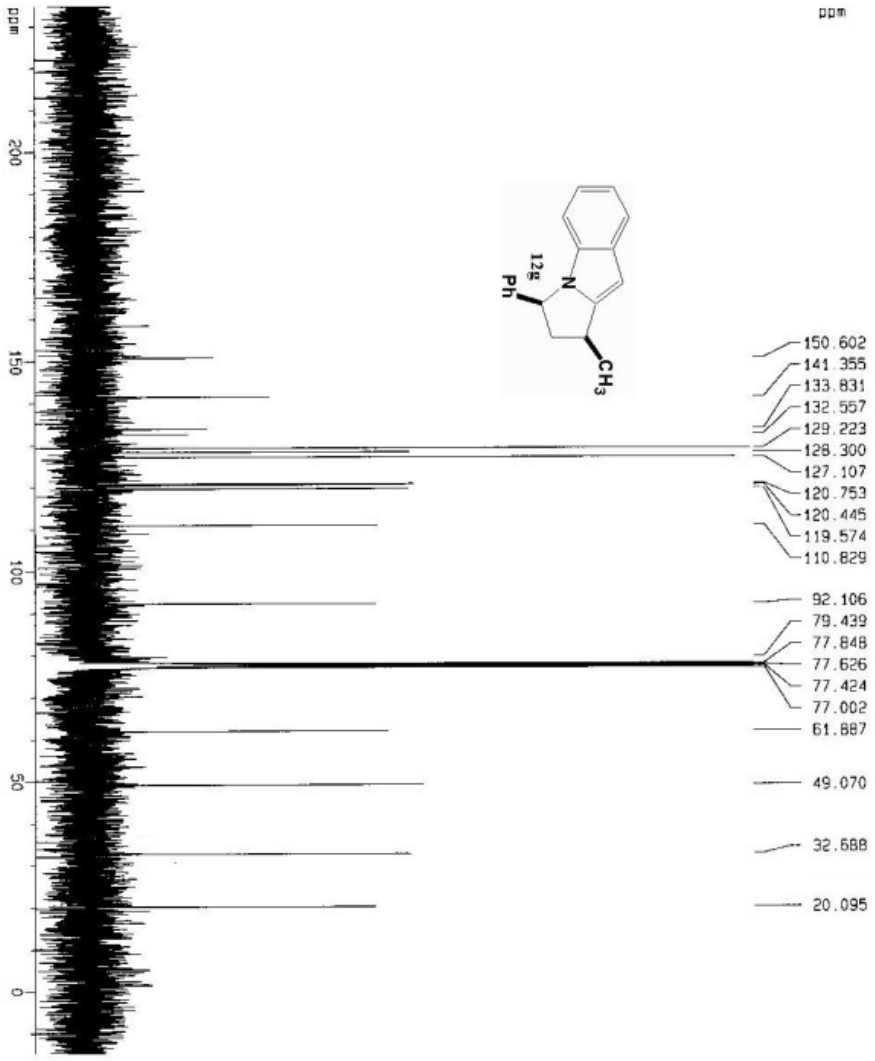
====== CHANNEL f1 ======
NUC1 13C
P1 11.90 usec
PL1 0.00 dB
SFO1 75.4760200 MHz

====== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 110.00 usec
PL2 0.00 dB
PL12 17.50 dB
PL13 17.50 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4577190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 234.765 ppm
F1 17717.16 Hz
F2 -1509.36 Hz
PPMCM 12.73624 ppm/cm
HZCM 961.32596 Hz/cm


Integral
ppm
8
6
4
2
0
1.0161
2.9517
2.9258
1.1756
1.0408
0.9665
1.0000
0.9803
0.9717
1.0017
1.0461
2.9402
12g
N
Ph
CH3
Current Data Parameters
NAME MRI-Nov12-05
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20051112
Time 10.58
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 724.1
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm



Current Data Parameters
NAME MR1-Nov12-05
EXPNO 1
PROCNO 1

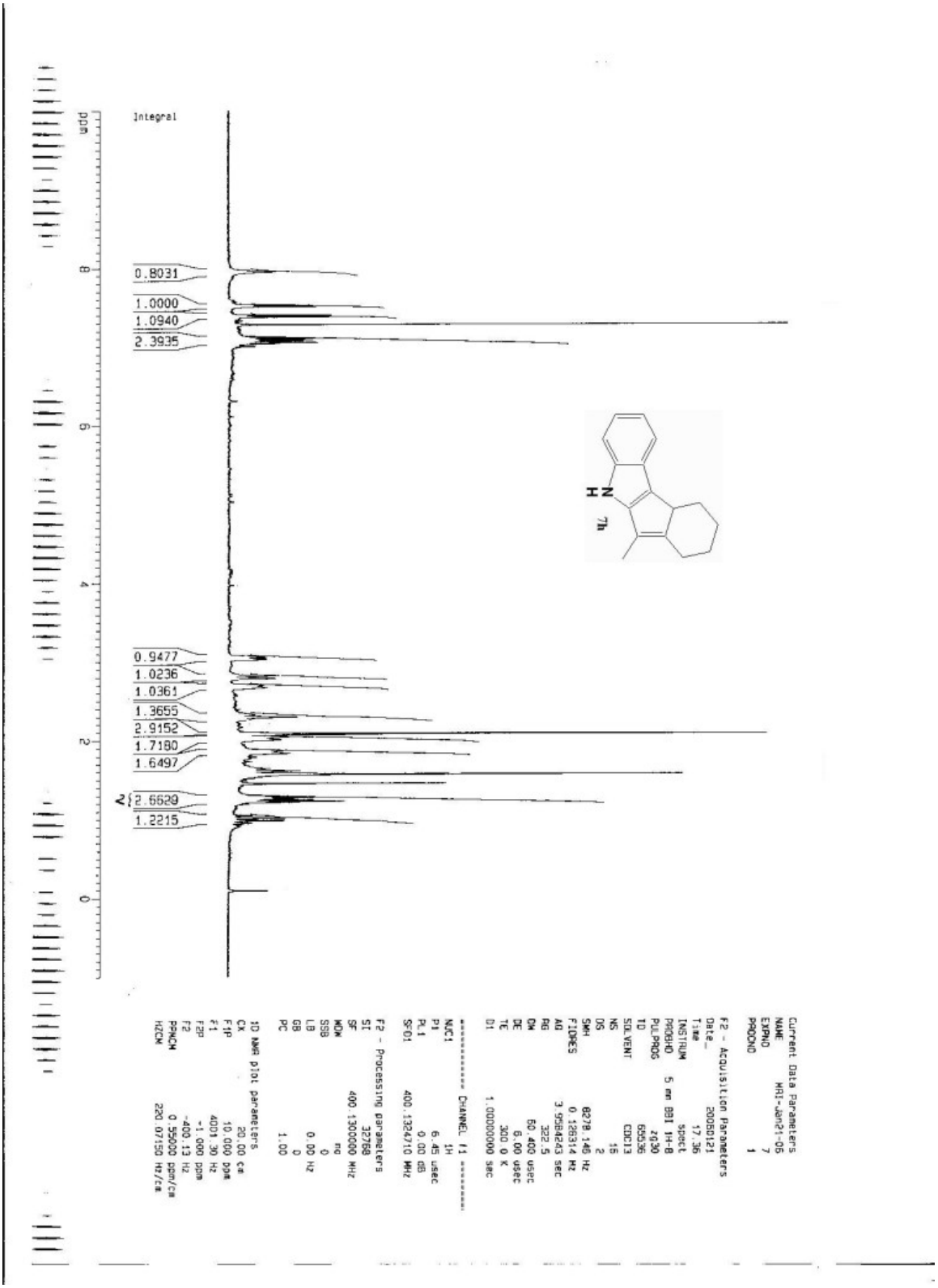
F2 - Acquisition Parameters
Date_ 20051112
Time 13.38
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2233
DS 4
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400309 sec
RG 11585.2
DW 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec

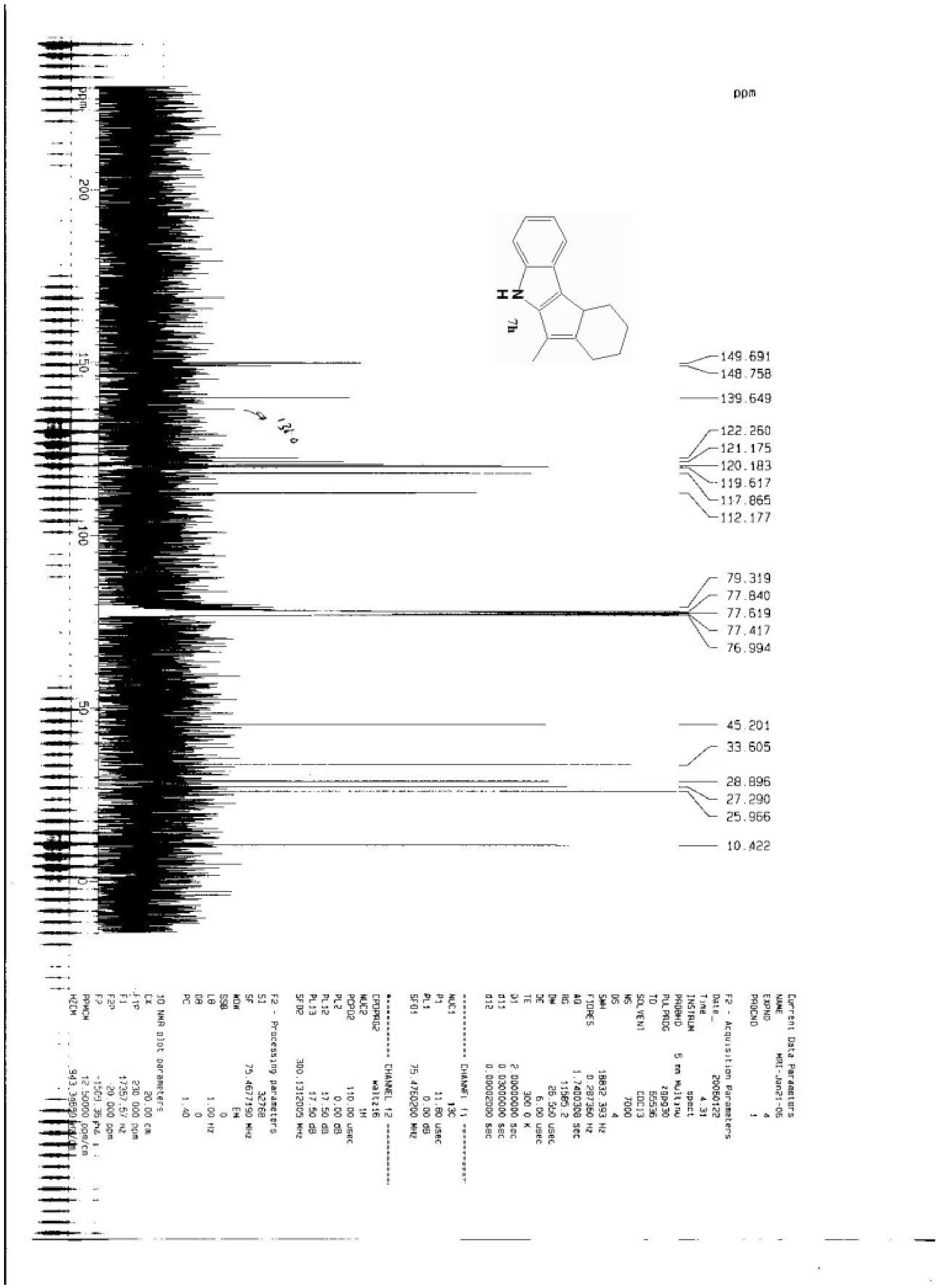
======== CHANNEL f1 ========
NUC1 13C
P1 11.80 usec
PL1 0.00 dB
SFO1 75.4750200 MHz

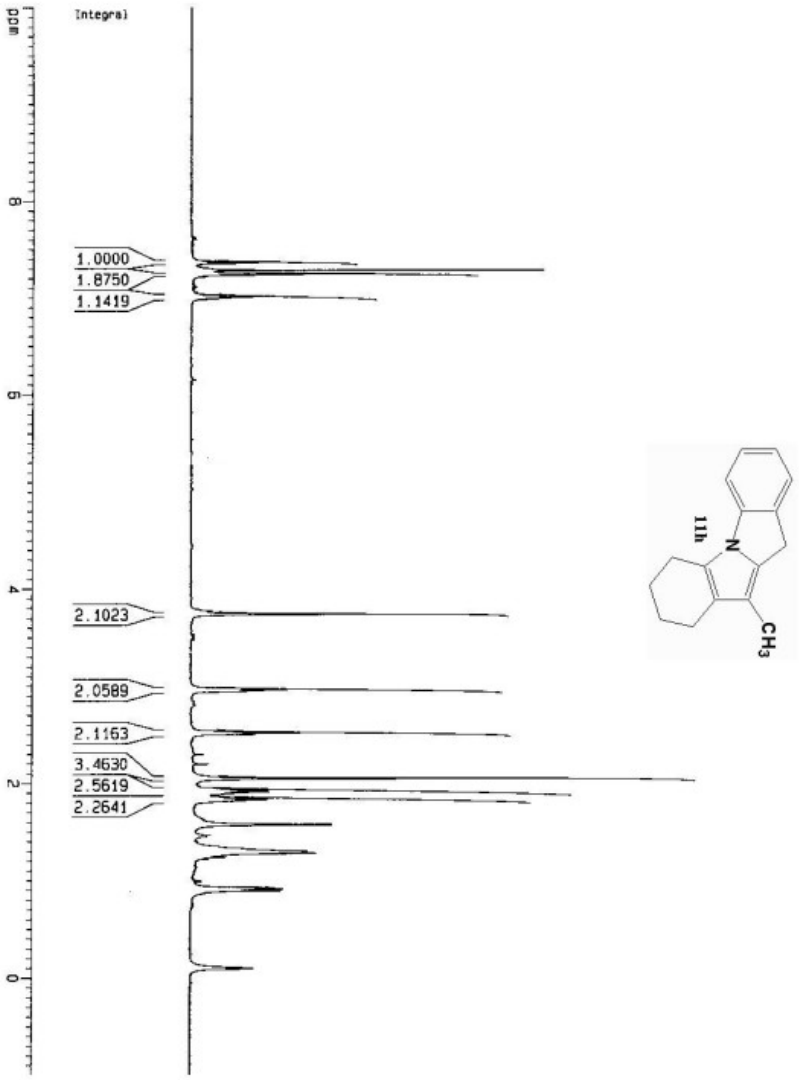
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 110.00 usec
PL2 0.00 dB
PL12 17.50 dB
PL13 17.50 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

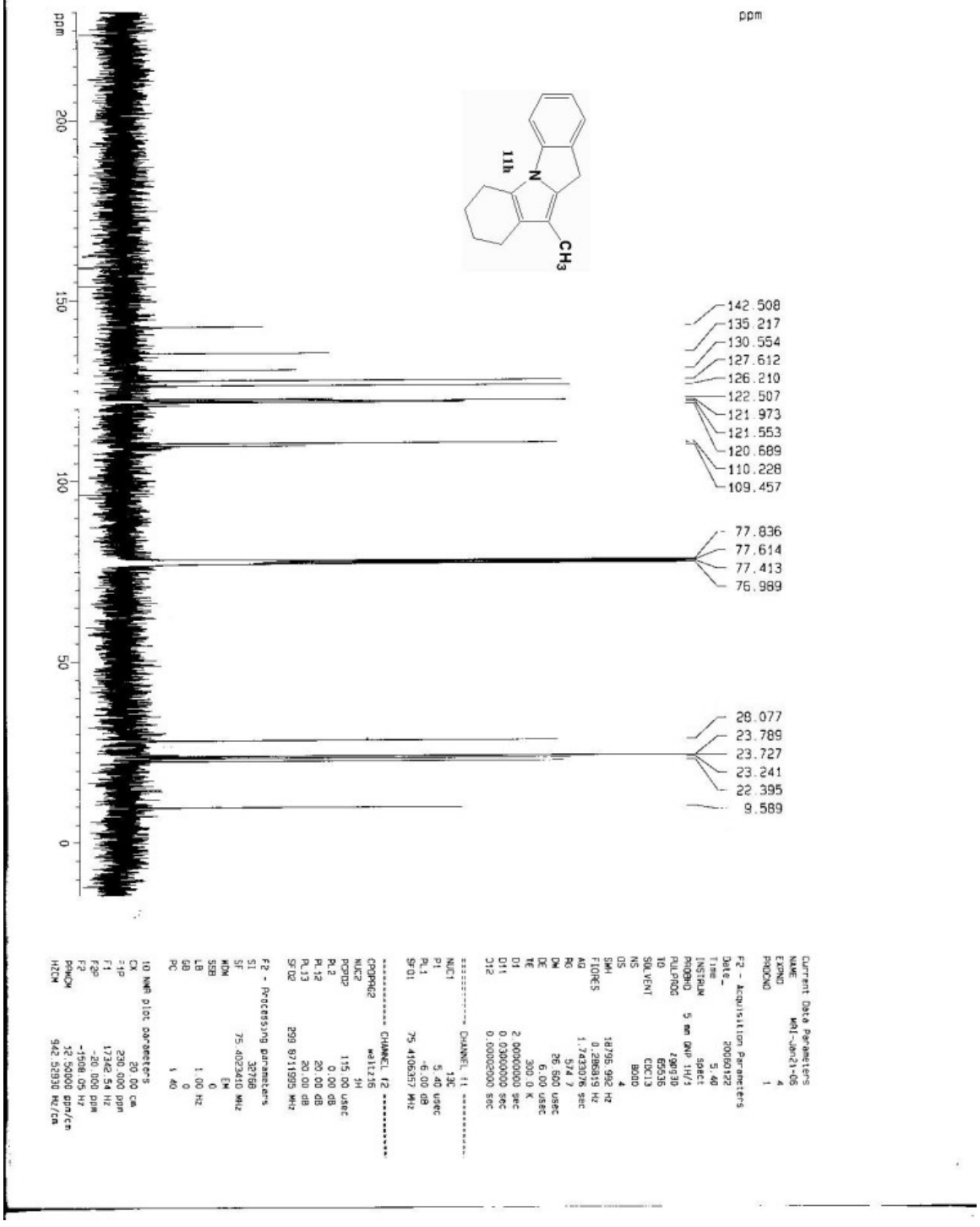
1D NMR plot parameters
CX 20.00 cm
F1P 234.765 ppm
F1 17717.16 Hz
F2P -20.000 ppm
F2 -1509.35 Hz
PPMCM 12.73824 ppm/cm
HZCM 961.32556 Hz/cm

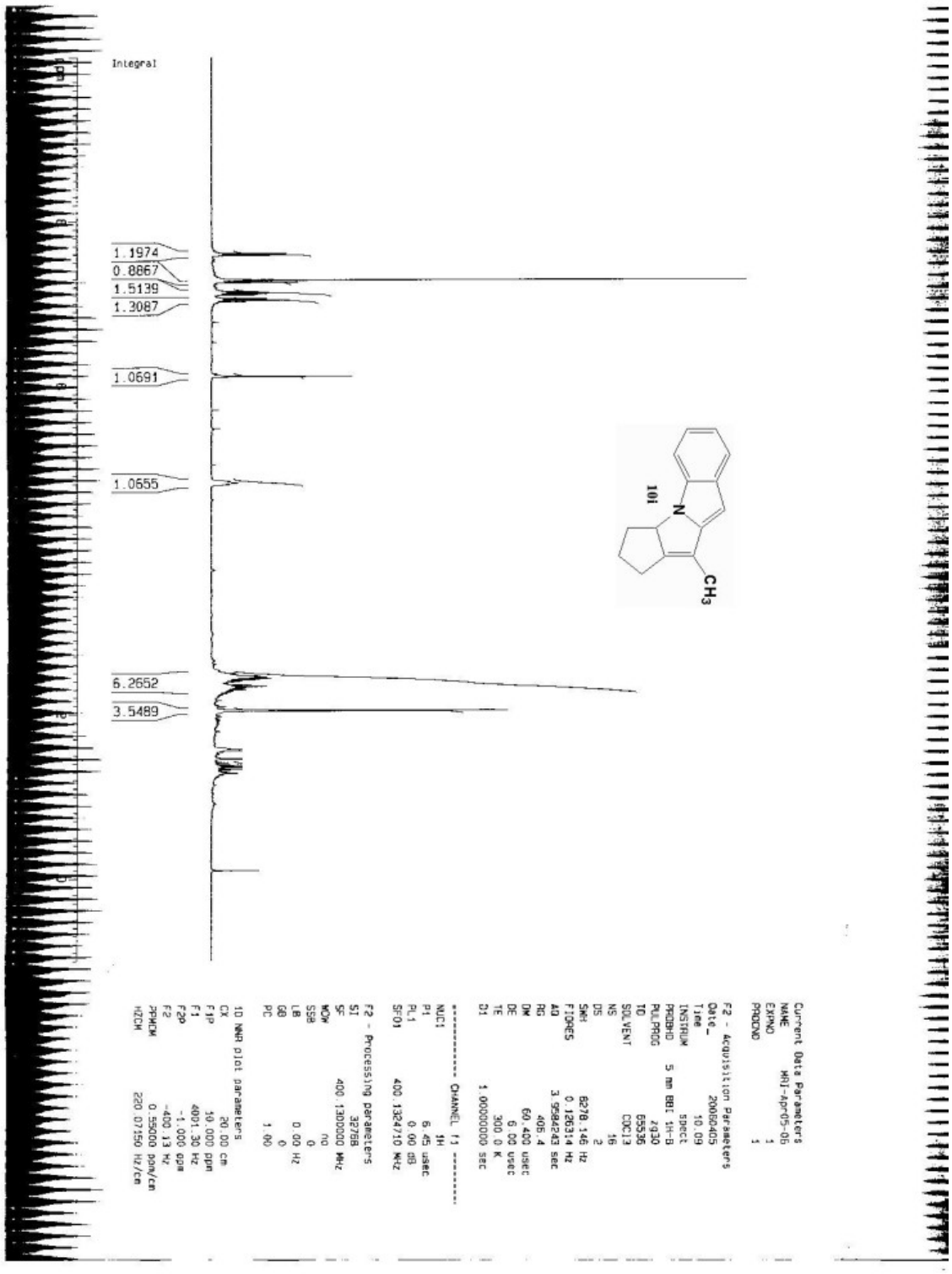

Integral
0.8031
1.0000
1.0940
2.3935
0.9477
1.0236
1.0361
1.3655
2.9152
1.7180
1.6497
2.6629
1.2215
ppm
0
2
4
6
8
7h
Current Data Parameters
NAME HRI-Jan21-06
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060121
Time 17.36
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

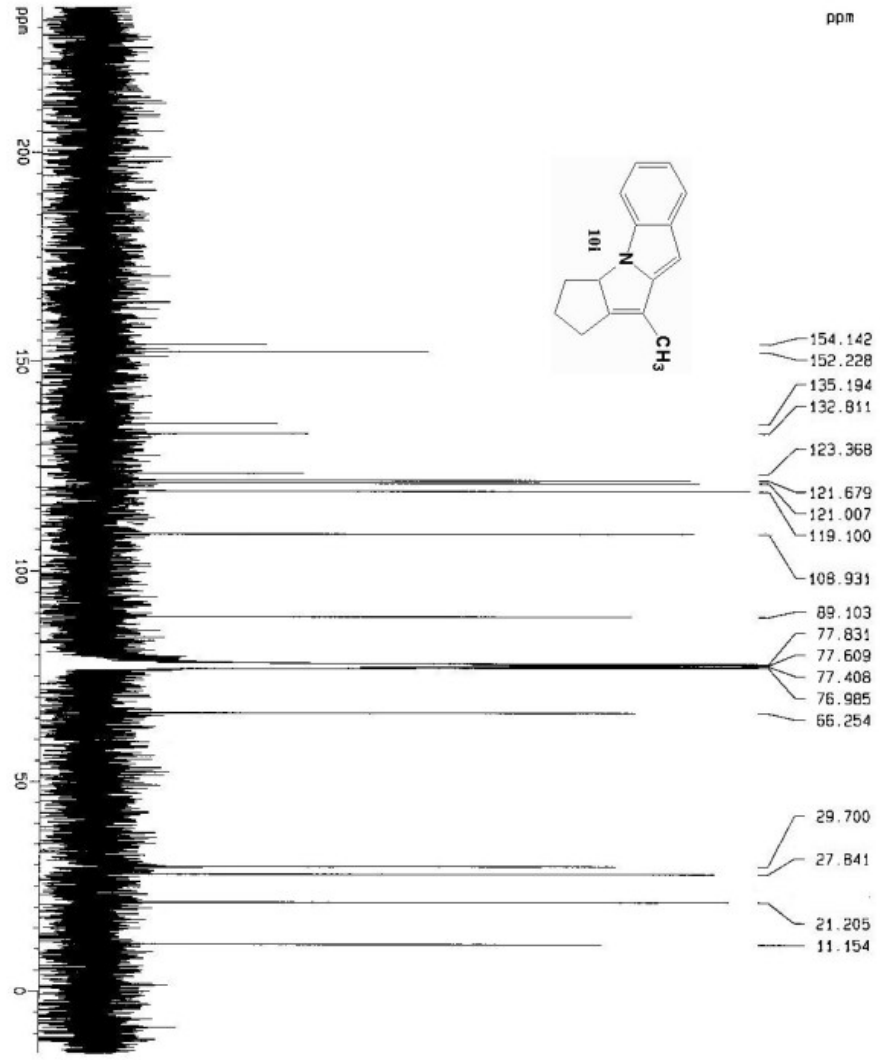

ppm
7h
149.691
148.758
139.649
122.260
121.175
120.183
119.617
117.865
112.177
79.319
77.840
77.619
77.417
76.994
45.201
33.605
28.896
27.290
25.966
10.422



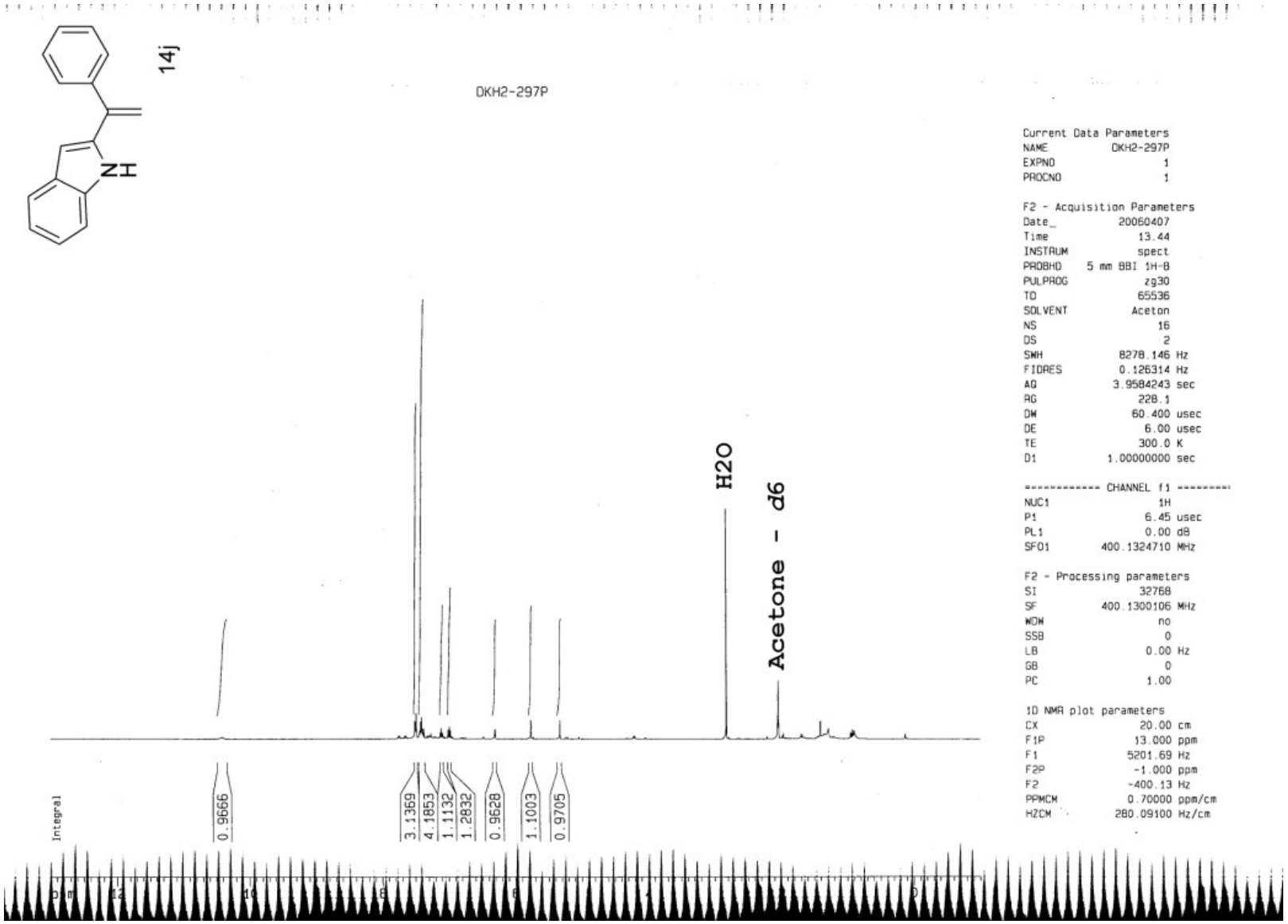

```
Current Data Parameters
NAME          MRI-Jun21-05
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20050621
Time                19.00
INSTRUM              spect
PROBHD     5 mm BBI 1H-B
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.958424 sec
RG                    512
DW                 60.400 usec
DE                  6.00 usec
TE                 300.0 K
D1               1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   6.45 usec
PL1                  0.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                  32768
SF          400.1300000 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  20.00 cm
F1P                10.000 ppm
F1                 4001.30 Hz
F2P                -1.000 ppm
F2                 -400.13 Hz
PPMCM             0.55000 ppm/cm
HZCM              220.07150 Hz/cm
```

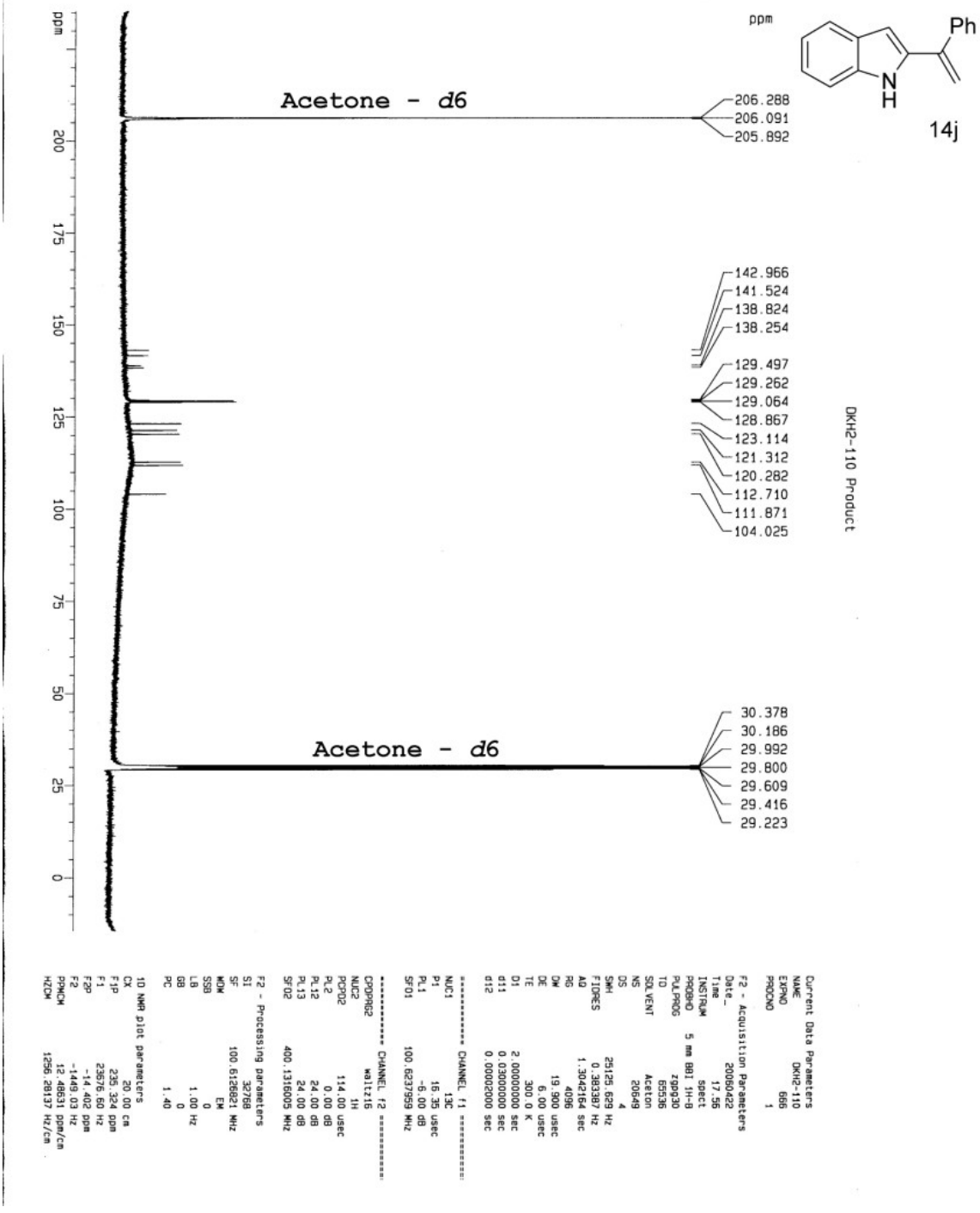

ppm
11h
N
CH3
142.508
135.217
130.554
127.612
126.210
122.507
121.973
121.553
120.689
110.228
109.457
77.836
77.614
77.413
76.989
28.077
23.789
23.727
23.241
22.395
9.589

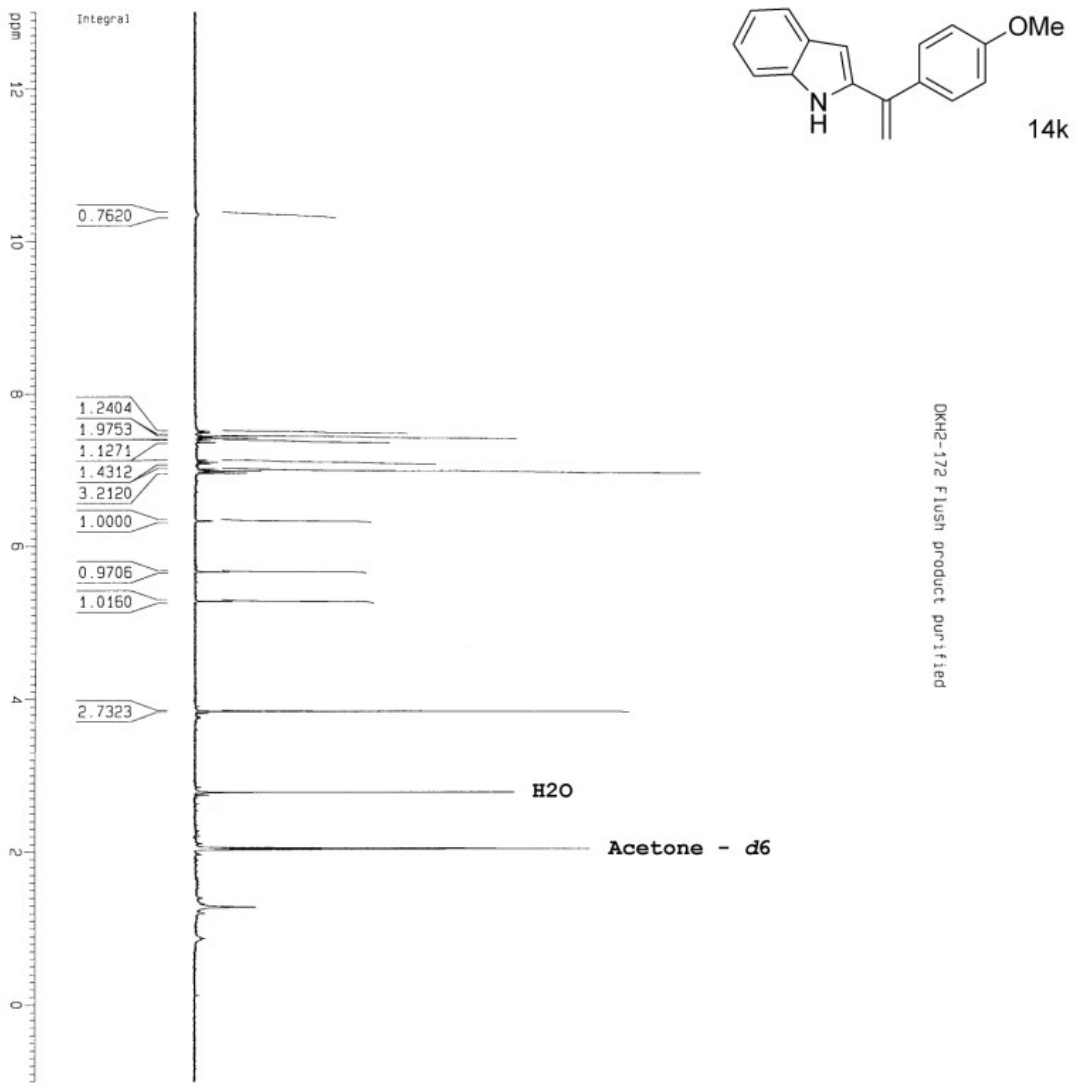

Integral
1.1974
0.8867
1.5139
1.3087
1.0691
1.0655
6.2652
3.5489
10i
N
CH3
Current Data Parameters
NAME HRJ-Apr05-05
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050405
Time 10.09
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 406.4
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm




```
Current Data Parameters
NAME                  mai-Apr21-06
EXPNO                          2
PROCNO                         1

F2 - Acquisition Parameters
Date_              20060421
Time                    20.49
INSTRUM                 spect
PROBHD     5 mm Multinu
PULPROG                 zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                      17000
DS                          4
SWH                 18832.393 Hz
FIDRES              0.287360 Hz
AQ                  1.7408308 sec
RG                      11585.2
DW                      26.550 usec
DE                       6.00 usec
TE                      300.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
D12                 0.00002000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                       11.80 usec
PL1                       0.00 dB
SFO1                75.4760200 MHz

======== CHANNEL f2 ========
DPDPRG2                 waltz16
NUC2                        1H
PCPD2                   110.00 usec
PL2                       0.00 dB
PL12                     17.50 dB
PL13                     17.50 dB
SFO2                300.1312005 MHz

F2 - Processing parameters
SI                         32768
SF                  75.4677190 MHz
WDW                            EM
SSB                             0
LB                       1.00 Hz
GB                              0
PC                       1.40

1D NMR plot parameters
CX                        20.00 cm
F1P                      234.765 ppm
F1                     17717.16 Hz
F2P                     -20.000 ppm
F2                     -1509.35 Hz
PPMCM                  12.73623 ppm/cm
HZCM                   961.32650 Hz/cm
```


14j
DKH2-297P
H2O
Acetone - d6
Integral
0.9666
3.1369
4.1853
1.1132
1.2832
0.9628
1.1003
0.9705
Current Data Parameters
NAME DKH2-297P
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060407
Time 13.44
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 65536
SOLVENT Aceton
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 228.1
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
CHANNEL f1
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300106 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 13.000 ppm
F1 5201.69 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.70000 ppm/cm
HZCM 280.09100 Hz/cm


ppm
Ph
N
H
14j
Acetone - d6
206.288
206.091
205.892
142.966
141.524
138.824
138.254
129.497
129.262
129.064
128.867
123.114
121.312
120.282
112.710
111.871
104.025
DKH2-110 Product
30.378
30.186
29.992
29.800
29.609
29.416
29.223
Acetone - d6
Current Data Parameters
NAME DKH2-110
EXPNO 666
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060422
Time 17.56
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zgpg30
TD 65536
SOLVENT Aceton
NS 2048
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 4096
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 16.35 usec
PL1 -6.00 dB
SFO1 100.6237959 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 114.00 usec
PL2 0.00 dB
PL12 24.00 dB
PL13 24.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6126821 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 235.324 ppm
F1 23676.60 Hz
F2P -14.402 ppm
F2 -1449.03 Hz
PPMCM 12.48631 ppm/cm
HZCM 1256.28137 Hz/cm



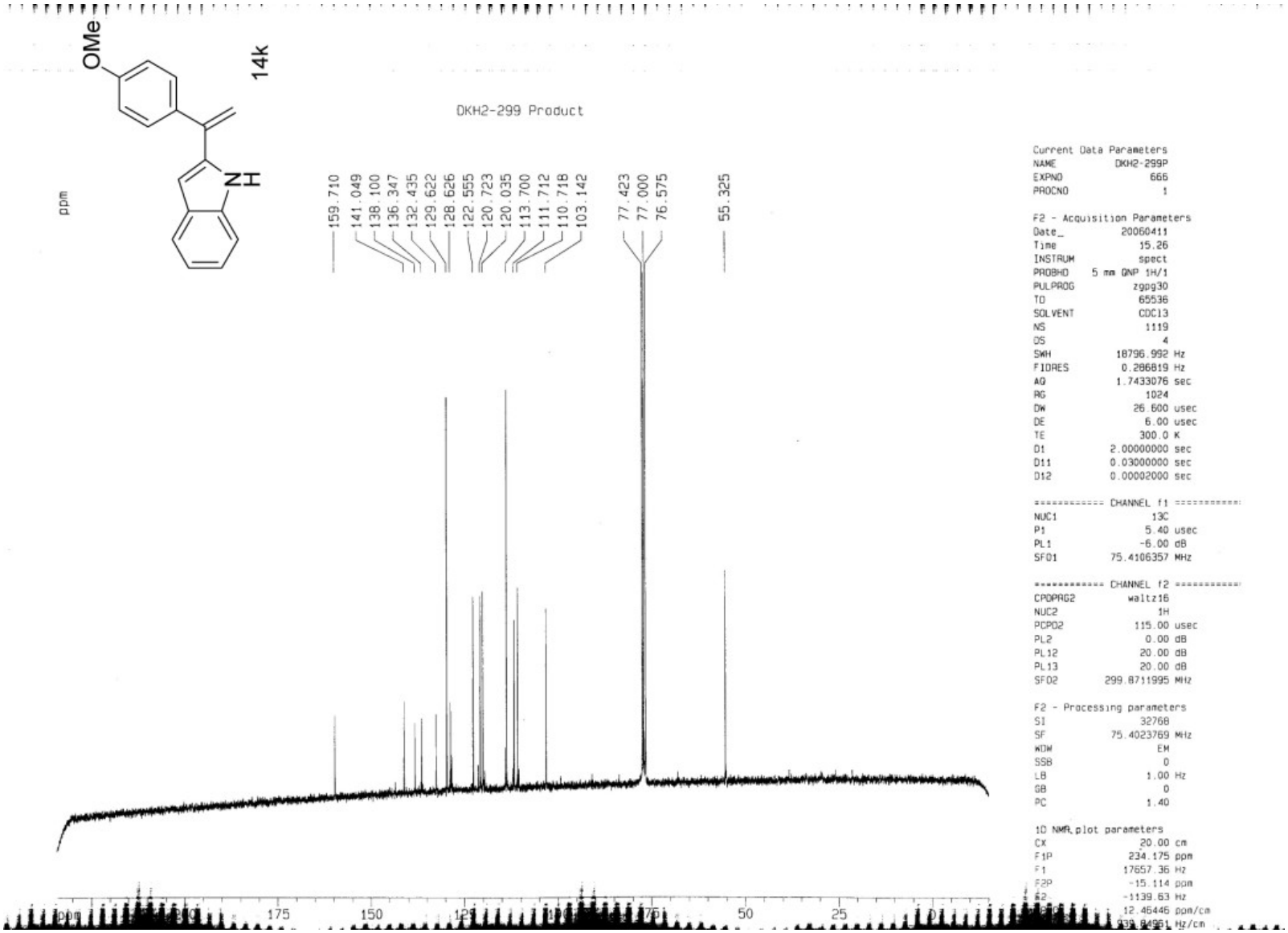
Current Data Parameters
NAME DKH2-172FP
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20051123
Time 16.37
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg30
TD 65536
SOLVENT Aceton
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 724.1
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

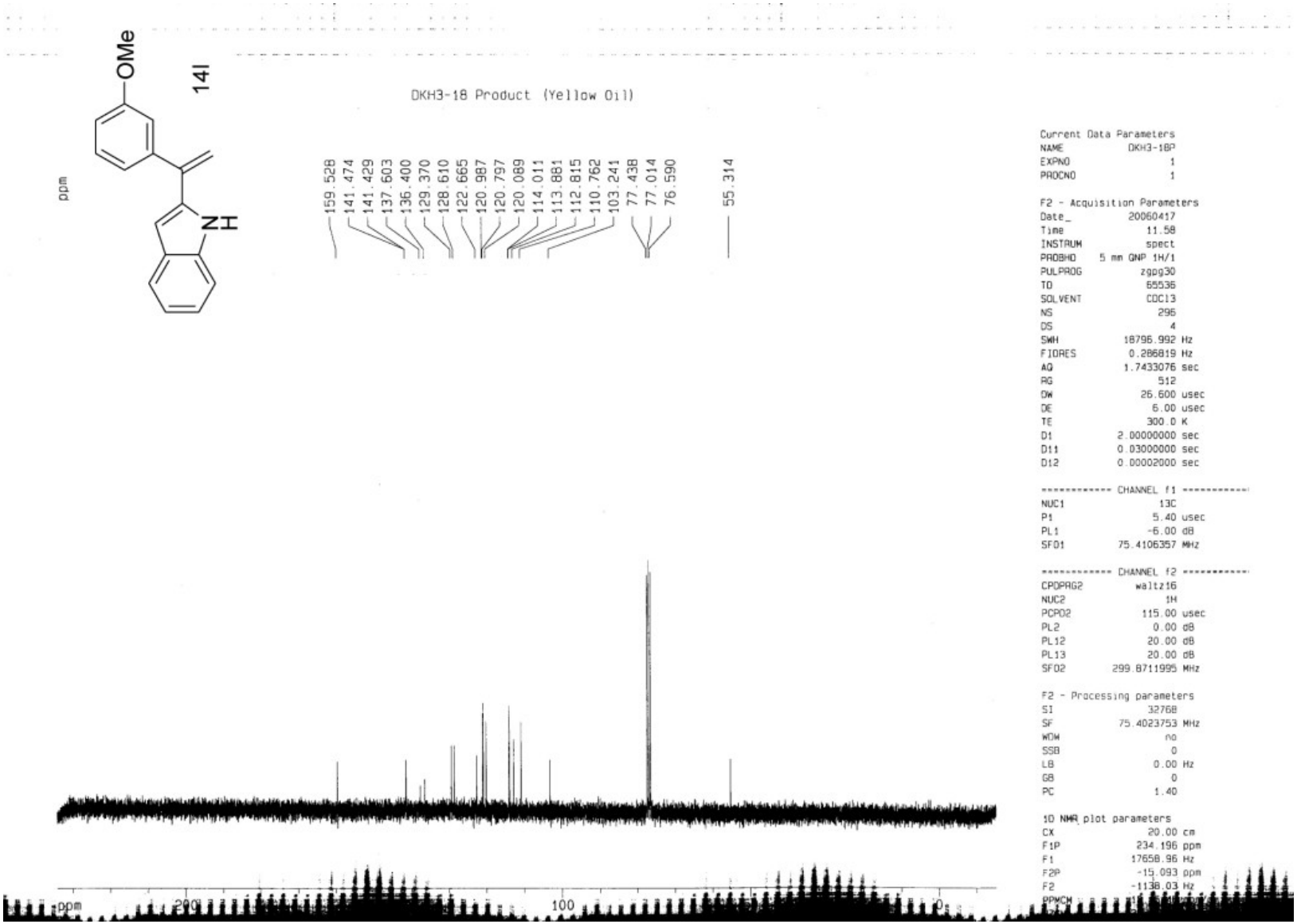
---------- CHANNEL f1 ----------
NUC1 1H
P1 9.60 usec
PL1 -6.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300071 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

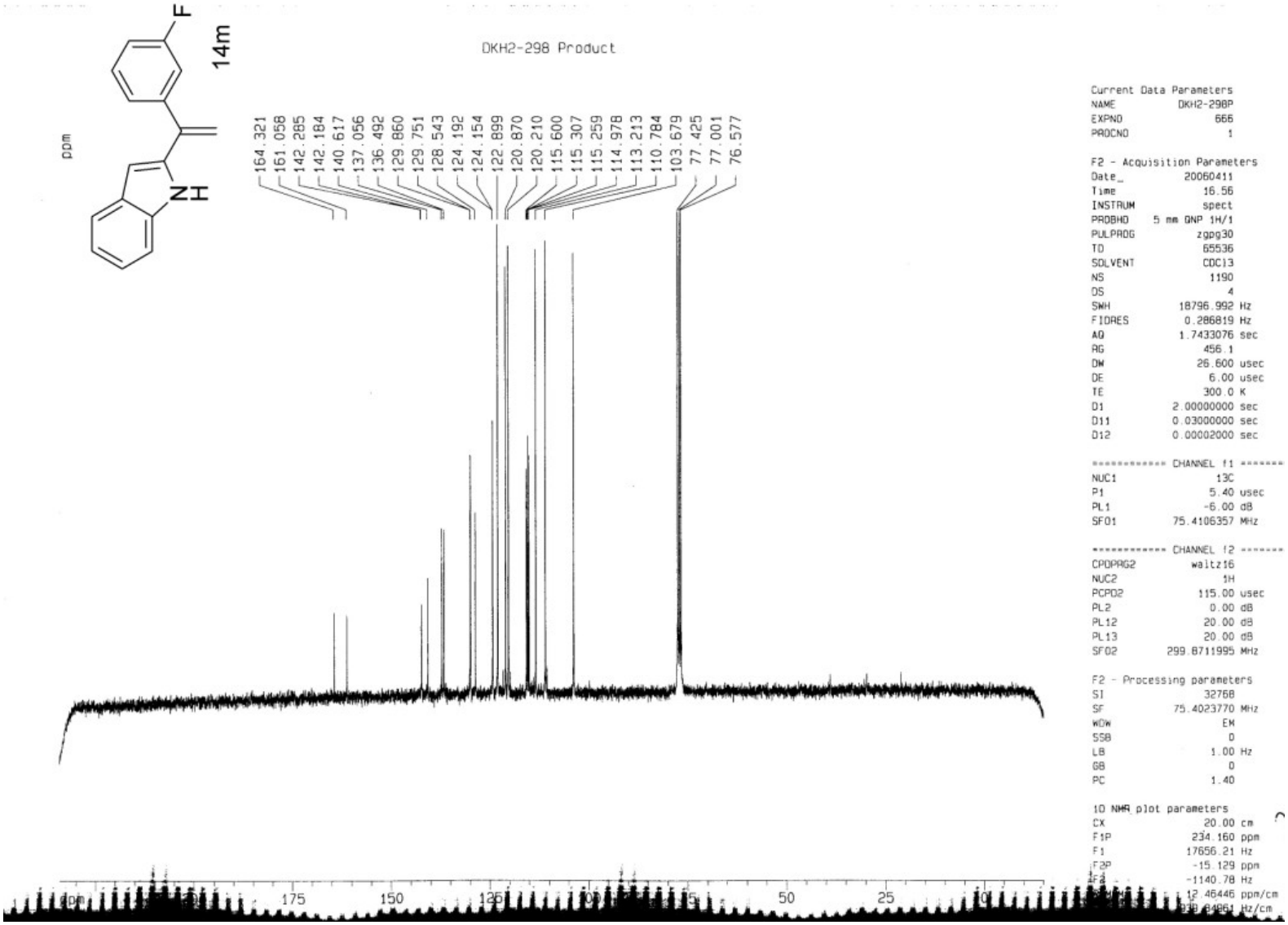
1D NMR plot parameters
CX 20.00 cm
F1P 13.000 ppm
F1 3901.69 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PPMCM 0.70000 ppm/cm
HZCM 210.09100 Hz/cm

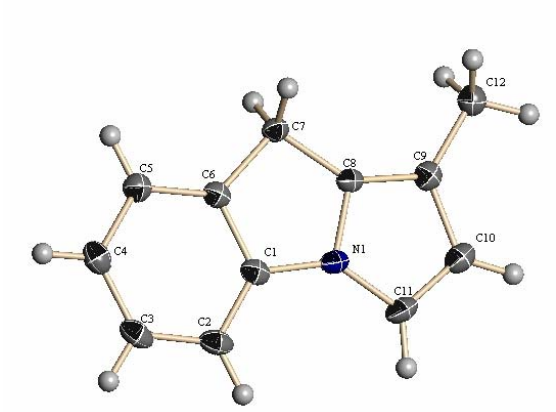

OMe
14k
DKH2-299 Product
ppm
159.710
141.049
138.100
136.347
132.435
129.622
128.626
122.555
120.723
120.035
113.700
111.712
110.718
103.142
77.423
77.000
76.575
55.325
Current Data Parameters
NAME DKH2-299P
EXPNO 666
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060411
Time 15.26
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1119
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AQ 1.7433076 sec
RG 1024
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SFO1 75.4106357 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023769 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 234.175 ppm
F1 17657.36 Hz
F2P -15.114 ppm
F2 -1139.63 Hz
12.46446 ppm/cm
439.84961 Hz/cm
200
175
150
125
100
75
50
25
0

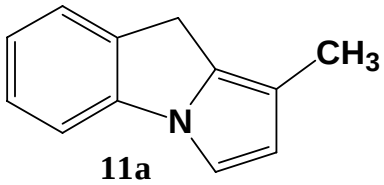
OMe
N
H
14l
DKH2-129 Product
Integral
0.8622
1.0330
1.0571
1.1574
1.3112
2.0950
2.4439
0.9310
1.0000
1.0144
2.9902
H2O
Acetone - d6
Current Data Parameters
NAME DKH2-129P
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060407
Time 13.36
INSTRUM spect
PROBHD 5 mm BBI 1H-B
PULPROG zg30
TD 65536
SOLVENT Aceton
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300106 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 13.000 ppm
F1 5201.69 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCM 0.70000 ppm/cm
HZCM 280.09100 Hz/cm


OMe
14l
NH
DKH3-18 Product (Yellow Oil)
ppm
159.528
141.474
141.429
137.603
136.400
129.370
128.610
122.665
120.987
120.797
120.089
114.011
113.881
112.815
110.762
103.241
77.438
77.014
76.590
55.314
200
100
0
Current Data Parameters
NAME DKH3-18P
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060417
Time 11.58
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 296
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AQ 1.7433076 sec
RG 512
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SFO1 75.4106357 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023753 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 234.196 ppm
F1 17658.96 Hz
F2P -15.093 ppm
F2 -1138.03 Hz
PPMCM

14m
DKH 2-298 Product (Yellow Oil)
Integral
0.8561
1.0433
3.2573
1.2582
2.3079
1.2592
1.0000
0.9638
0.9605
H2O
Acetone - d6
Current Data Parameters
NAME DKH2-298P
EXPNO 101
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060417
Time 10.48
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 228.1
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SFO1 299.8718518 MHz
F2 - Processing parameters
SI 32768
SF 299.8700077 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
F1P 13.000 ppm
F1 3898.31 Hz
F2P -1.000 ppm
F2 -299.87 Hz
PPMCM 0.70000 ppm/cm
HZCM 209.90900 Hz/cm


14m
DKH2-298 Product
ppm
164.321
161.058
142.285
142.184
140.617
137.056
136.492
129.860
129.751
128.543
124.192
124.154
122.899
120.870
120.210
115.600
115.307
115.259
114.978
113.213
110.784
103.679
77.425
77.001
76.577
Current Data Parameters
NAME DKH2-298P
EXPNO 666
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060411
Time 16.56
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1190
DS 4
SWH 18796.992 Hz
FIDRES 0.286819 Hz
AQ 1.7433076 sec
RG 456.1
DW 26.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
CHANNEL f1
NUC1 13C
P1 5.40 usec
PL1 -6.00 dB
SFO1 75.4106357 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 115.00 usec
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 299.8711995 MHz
F2 - Processing parameters
SI 32768
SF 75.4023770 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
F1P 234.160 ppm
F1 17656.21 Hz
F2P -15.129 ppm
F2 -1140.78 Hz
12.46446 ppm/cm
Hz/cm
200
175
150
125
100
75
50
25
0





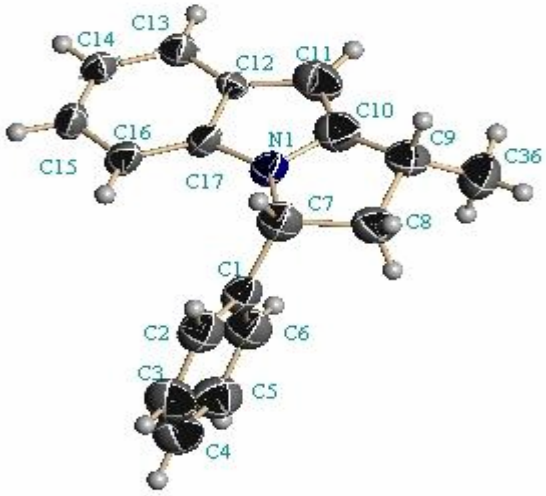
11a

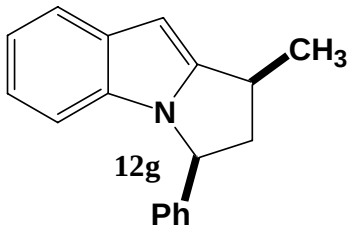
A yellow plate shaped crystal of **11a** (C12 H11 N) with approximate dimensions 0.10 x 0.30 x 0.40 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured at 108(2) K, cooled by Rigaku-MSC X-Stream 2000, on a Bruker SMART APEX CCD area detector system equipped with a graphite monochromator and a MoKα fine-focus sealed tube (λ = 0.71073Å) operated at 1600 watts power (50 kV, 32 mA). The detector was placed at a distance of 5.8 cm from the crystal.

A total of 1850 frames were collected with a scan width of 0.3º in ω and an exposure time of 20 seconds/frame. The total data collection time was about 12 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame integration algorithm. The integration of the data using a Monoclinic unit cell yielded a total of 5358 reflections to a maximum θ angle of 28.28° (0.90 Å resolution), of which 2066 were independent, completeness = 94.9 %, R_{int} = 0.0178, R_{sig} = 0.0245 and 1729 were greater than 2σ(I). The final cell constants: a = 11.907(4)Å, b = 5.6848(16)Å, c = 12.950(4)Å, α = 90°, β = 92.517(5)°, γ = 90°, volume = 875.7(4)Å^3, are based upon the refinement of the XYZ-centroids of 2113 reflections above 20σ(I) with 2.274° <θ <28.242° . Analysis of the data showed negligible decay during data collection. Data were corrected for absorption effects using the multiscan technique (SADABS). The ratio of minimum to maximum apparent transmission was 0.849448.

The structure was solved and refined using the Bruker SHELXTL (Version 6.1) Software Package, using the space group P2(1)/n, with Z = 4 for the formula unit, C12 H11 N . The final anisotropic full-matrix least-squares refinement on F^2 with 119 variables converged at R1 = 5.17 %, for the observed data and wR2 = 13.90 % for all data. The

goodness-of-fit was 1.072 . The largest peak on the final difference map was 0.403 $e^-/Å^3$ and the largest hole was -0.249 $e^-/Å^3$. Based on the final model, the calculated density of the crystal is 1.283 g/cm^3 and F(000) amounts to 360 electrons.





A yellow needle shaped crystal of **12g** (C18 H17 N) (two molecules in the asymmetric unit) with approximate dimensions 0.04 x 0.08 x 0.35 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured at 298(2) K, on a Bruker SMART APEX CCD area detector system equipped with a graphite monochromator and a MoKα fine-focus sealed tube (λ = 0.71073Å) operated at 1600 watts power (50 kV, 32 mA). The detector was placed at a distance of 5.8 cm from the crystal.

A total of 1850 frames were collected with a scan width of 0.3º in ω and an exposure time of 10 seconds/frame. The total data collection time was about 8 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame integration algorithm. The integration of the data using a Triclinic unit cell yielded a total of 8236 reflections to a maximum θ angle of 28.27 ° (0.90 Å resolution), of which 6090 were independent, completeness = 89.6 %, R_{int} = 0.0758, R_{sig} = 0.2293 and 1717 were greater than 2σ(I). The final cell constants: a = 5.538(3)Å, b = 9.573(5)Å, c = 25.815(14)Å, α = 90°, β = 87.270(12)°, γ = 90°, volume = 1367.1(13)$Å^3$, are based upon the refinement of the XYZ-centroids of 715 reflections above 20σ(I) with 2.269° <θ <27.720° . Analysis of the data showed negligible decay during data collection. Data were corrected for absorption effects using the multiscan technique (SADABS). The ratio of minimum to maximum apparent transmission was 0.03821.

The structure was solved and refined using the Bruker SHELXTL (Version 6.1) Software

Package, using the space group P-1, with Z = 2 for the formula unit, C36 H34 N2 . The final anisotropic full-matrix least-squares refinement on F^2 with 345 variables converged at R1 = 8.23 %, for the observed data and wR2 = 21.67 % for all data. The goodness-of-fit was 0.829 . The largest peak on the final difference map was 0.233 $e^-/Å^3$ and the largest hole was -0.247 $e^-/Å^3$. Based on the final model, the calculated density of the crystal is 1.202 g/cm^3 and F(000) amounts to 528 electrons.