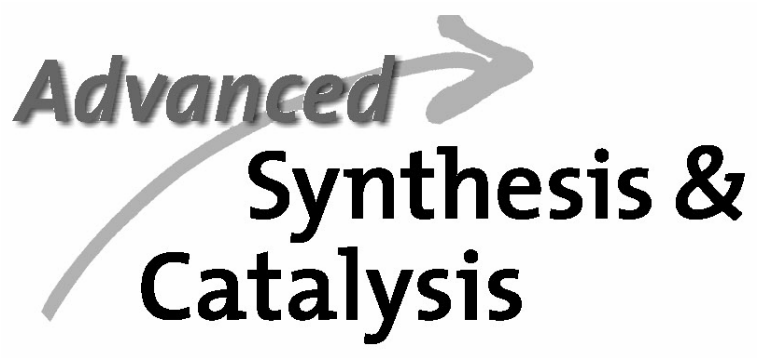

Supporting Information

Supporting information

for

Substituted Benzocarbocycles by Palladium-Catalyzed Cascade Reactions Featuring a C(sp^3)–H Activation Step

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Table of Contents

I. Synthesis of the Dihalophenylacetonitrile Substrates
Characterization data for compounds **1b-d**.

II. C(sp^3)–H Activation / Heck Cyclization / Heck Arylation Sequence
Characterization data for compounds **5a-b**, **5d-h**.

III. C(sp^3)–H Activation / Heck Reactions / Hydrogenation Sequences
Characterization data for compounds **6b-d**.
Transmission electronic microscopy (TEM).

IV. Inhibition of Microtubule Assembly

V. Copies of NMR Spectra for target compounds **3a**, **4a**, **5a-h**, **6a-d** including NOESY spectra of compounds **5b**, **5g**, **6a**, **6d**.

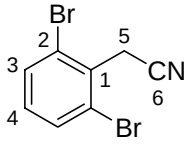
I. Preparation of the Dihalophenylacetonitrile Substrates[1] (Scheme 2, Tables 1-3)

Representatitive procedure (Preparation of compound **1b**)

N-bromosuccinimide (4.04 g, 22.7 mmol) and benzoyl peroxide (191 mg, 0.79 mmol) were added to a stirred solution of 2,6-dibromotoluene (5.15 g, 20.6 mmol) in carbon tetrachloride (50 mL). The mixture was refluxed for 2 h. After cooling to r.t. it was filtered through celite and the solvent was removed under vacuum. The resulting white powder was dissolved in acetonitrile (100 mL). Trimethylsilyl cyanide (4.1 mL, 30.9 mmol) and tetrabutylammonium fluoride (31 mL of a 1 M solution in THF, 30.9 mmol) were carefully added at r.t. and the mixture was stirred for 2 h. The solvent was then removed under reduced pressure and the residue was purified by flash chromatography (silica gel, heptanes/diethyl ether 9:1) to afford 2,6-dibromophenylacetonitrile as a white solid (4.60 g, 81 % over 2 steps).

Lithium bis(trimethylsilyl)amide (67 mL of a 1 M solution in THF, 67.0 mmol) was added dropwise to a solution of 2,6-dibromophenylacetonitrile (4.60 g, 16.7 mmol) and DMPU[2] (8.1 mL, 66.9 mmol) in THF (180 mL) under argon. After the reaction mixture had been stirred for 45 min at r.t., iodoethane (5.4 mL, 66.9 mmol) was added with a syringe and the reaction mixture was stirred for 2 h. The reaction was quenched with a saturated NH_4Cl solution (100 mL) and the aqueous layer was extracted with diethyl ether (150 mL × 3). The combined organic layers were washed with a 1 M HCl solution (150 mL) and with a saturated solution of $NaHCO_3$ (150 mL). The extracts were then dried over magnesium sulfate and evaporated under reduced pressure. The residue was purified by flash chromatography (silica gel, heptanes/ethyl acetate 95:5) to afford **1b** as a colourless oil (4.45 g, 80 %).

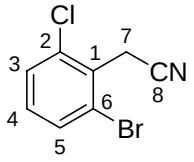
Products characterisation



2,6-dibromophenylacetonitrile

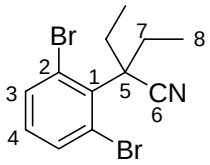
^{1}H NMR (300 MHz, *CDCl*$_3$) δ = 4.06 (s, 2H, H_5), 7.10 (t, *J* = 8.0 Hz, 1H, H_4), 7.60 (d, *J* = 8.0 Hz, 2H, H_3) ppm; ^{13}C NMR (75.5 MHz, *CDCl*$_3$) δ = 25.9 (C_5), 115.4 (C_6), 125.3 (C_2), 130.2 (C_1), 131.1 (C_4), 132.6 (C_3) ppm; GC/MS (EI, DB-5ms column, 1 min at 120 °C then 120 → 250 °C at 8 °C/min): 9.7 min (m/z: 274 [$M^{+\bullet}$]); IR (film) ν = 2257, 2974 cm^{-1}.

[1] a) Bromination: M. Tashiro, K. Nakayama, *OPPI Briefs* **1984**, *16*, 379-381; b) Cyanation: E. D. Soli, A. S. Manoso, M. C. Patterson, P. DeShong, D. A. Favor, R. Hirschmann, A. B. Smith, III, *J. Org. Chem.* **1999**, *64*, 3171-3177.
[2] DMPU: 1,3-dimethyl-3,4,5,6-tetrahydro-2(1*H*)-pyrimidinone.



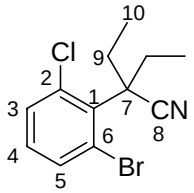
2-bromo-6-chlorophenylacetonitrile

^{1}H NMR (500 MHz, *CDCl*$_3$) δ = 4.06 (s, 2H, H$_7$), 7.18 (dd, *J* = 8.2, 8.2 Hz, H$_4$), 7.42 (dd, J = 8.1, 1.2 Hz, H$_3$), 7.55 (dd, *J* = 7.9, 1.3 Hz, H$_5$) ppm; ^{13}C NMR (75.5 MHz, *CDCl*$_3$) δ = 22.8 (C$_7$), 115.4 (C$_8$), 125.5 (C$_6$), 128.3 (C$_1$), 129.2 (C$_3$), 130.7 (C$_4$), 131.9 (C$_5$), 135.5 (C$_2$) ppm; GC/MS (EI, DB-5ms column, 1 min at 120 °C then 120 → 250 °C at 8 °C/min): 8.1 min (m/z: 229 [M$^{+\bullet}$]); IR (film) ν = 2257, 2952 cm^{-1}.



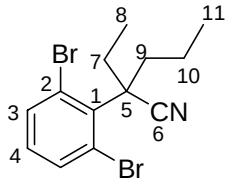
1b

^{1}H NMR (300 MHz, *CDCl*$_3$) δ = 1.08 (dd, *J* = 7.4, 7.4 Hz, 6H, H$_8$), 2.05 (dq, *J* = 14.7, 7.4, 2H, H$_{7a}$), 2.95 (dq, *J* = 14.6, 7.3 Hz, 2H, H$_{7b}$), 6.93 (t, *J* = 7.5 Hz, 1H, H$_4$), 7.68 (d, *J* = 7.5 Hz, 2H, H$_3$) ppm; ^{13}C NMR (75.5 MHz, *CDCl*$_3$) δ = 10.0 (C$_8$), 32.0 (C$_7$), 52.5 (C$_5$), 123.0 (C$_6$), 124.0 (C$_2$), 129.5 (C$_4$), 134.0 (C$_1$), 136.0 (C$_3$) ppm; GC/MS (EI, DB-5ms column, 1 min at 120 °C then 120 → 250 °C at 8 °C/min): 13.0 min (m/z: 329 [M$^{+\bullet}$]); IR (film) ν = 1458, 1576, 2225, 2970 cm^{-1}.



1c

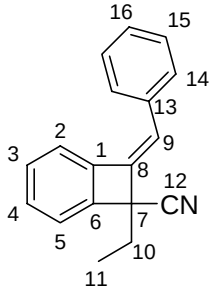
^{1}H NMR (300 MHz, *CDCl*$_3$) δ = 1.06 (dd, *J* = 7.7, 7.7 Hz, 6H, H$_{10}$), 2.09 (dq, *J* = 14.7, 7.4 Hz, 2H, H$_{9a}$), 2.88 (dq, *J* = 14.6, 7.3, 2H, H$_{9b}$), 7.03 (dd, *J* = 8.0, 8.0, 1H, H$_4$), 7.37 (dd, *J* = 8.0, 1.5 Hz, 1H, H$_3$), 7.58 (dd, *J* = 8.0, 1.5 Hz, 1H, H$_5$) ppm; ^{13}C NMR (75.5 MHz, *CDCl*$_3$) δ = 10.0 (C$_{10}$), 32.3 (C$_9$), 51.5 (C$_7$), 123.2 (C$_8$), 123.7 (C$_6$), 129.2 (C$_4$), 132.0 (C$_3$), 132.9 (C$_1$), 135.4 (C$_2$, C$_5$) ppm; GC/MS (EI, DB-5ms column, 1 min at 120 °C then 120 → 250 °C at 8 °C/min): 12.0 min (m/z: 285 [M$^{+\bullet}$]); IR (film) ν = 1458, 1573, 2225, 2970 cm^{-1}.



1d

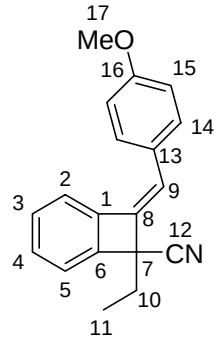
^{1}H NMR (500 MHz, *CDCl*$_3$) δ = 0.97 (dd, *J* = 7.4, 7.4 Hz, 3H, H_{11}), 1.08 (dd, *J* = 7.4, 7.4 Hz, 3H, H_8), 1.30-1.40 (m, 1H, H_{10a}), 1.57-1.68 (m, 1H, H_{10b}), 1.95-2.04 (m, 1H, H_{9a}), 2.10 (dq, *J* = 14.6, 7.5 Hz, 1H, H_{7a}), 2.85-2.98 (m, 2H, H_{7b}, H_{9b}), 6.93 (t, *J* = 7.9 Hz, 1H, H_4), 7.63 (d, *J* = 8.2 Hz, 2H, H_3) ppm; ^{13}C NMR (75.5 MHz, *CDCl*$_3$) δ = 10.0 (C_8), 14.0 (C_{11}), 18.9 (C_{10}), 32.2 (C_7), 40.7 (C_9), 51.7 (C_5), 123.2 (C_6), 123.8 (C_2), 129.4 (C_4), 134.2 (C_1), 135.9 (C_3) ppm; GC/MS (EI, DB-5ms column, 1 min at 120 °C then 120 → 250 °C at 8 °C/min): 13.8 min (m/z: 343 [$M^{+\bullet}$]); IR (film) ν = 1464, 2224, 2961 cm^{-1}.

II. C(sp^3)–H Activation / Heck Cyclization / Heck Arylation Process (Table 2)



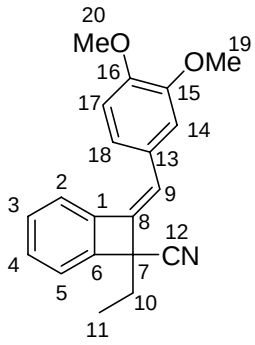
5a

^{1}H NMR (500 MHz, *CDCl*$_3$) δ = 1.23 (dd, *J* = 7.4, 7.4 Hz, 3H, H_{11}), 2.04 (dq, *J* = 14.5, 7.4, 1H, H_{10a}), 2.26 (dq, *J* = 14.5, 7.3 Hz, 1H, H_{10b}), 6.54 (s, 1H, H_9), 7.29-7.46 (m, 5H, H_3, H_4, H_{15}, H_{16} and H_2 or H_5), 7.51 (d, *J* = 7.4 Hz, 1H, H_2 or H_5), 7.56 (d, *J* = 7.9 Hz, 2H, H_{14}) ppm; ^{13}C NMR (75.5 MHz, *CDCl*$_3$) δ = 10.6 (C_{11}), 30.0 (C_{10}), 51.1 (C_7), 120.8 (C_{12}), 121.8, 121.9 (C_{16}, CH_{arom}), 122.2 (C_9), 128.0 (C_{14}), 128.1 (CH_{arom}), 128.7 (C_{15}), 130.0, 130.1 (CH_{arom}), 135.7 (C_1), 138.8 (C_8), 142.6 (C_{13}), 145.2 (C_6) ppm; GC/MS (EI, DB-5ms column, 1 min at 120 °C then 120 → 250 °C at 8 °C/min): 15.3 min (m/z: 245 [$M^{+\bullet}$]); IR (film) ν = 1459, 1599, 2229, 2968 cm^{-1}.



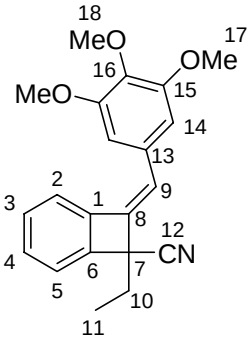
5b

^{1}H NMR (500 MHz, *CDCl*$_3$) δ = 1.23 (dd, *J* = 7.4, 7.4 Hz, 3H, H_{11}), 2.03 (dq, *J* = 14.8, 7.4 Hz, 1H, H_{10a}), 2.25 (dq, *J* = 14.7, 7.4 Hz, 1H, H_{10b}), 3.86 (s, 3H, H_{17}), 6.48 (s, 1H, H_9), 6.97 (d, *J* = 8.7 Hz, 2H, H_{15}), 7.32-7.41 (m, 3H, H_{arom}), 7.49-7.52 (m, 3H, H_{14}, H_{arom}) ppm; ^{13}C NMR (125.8 MHz, *CDCl*$_3$) δ = 10.5 (C_{11}), 30.1 (C_{10}), 51.0 (C_7), 55.4 (C_{17}), 114.2 (C_{15}), 121.0 (C_{12}), 121.6, 121.8 (CH_{arom}), 121.9 (C_9), 128.4 (C_1), 129.4 (C_{14}), 129.7, 129.9 (CH_{arom}), 136.9 (C_8), 142.8 (C_{13}), 145.1 (C_6), 159.6 (C_{16}) ppm; HRMS (ESI, MeOH + CH_2Cl_2) calcd for $C_{19}H_{17}NONa$ [$(M + Na)^+$]: 298.1208; found: 298.1194; IR (film) ν = 1247, 1509, 1605, 2228, 2967 cm^{-1}.



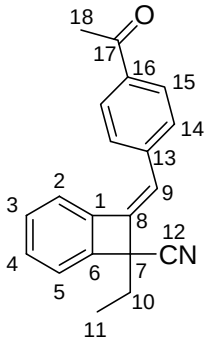
5d

^{1}H NMR (300 MHz, *CDCl*$_3$) δ = 1.24 (dd, *J* = 7.4, 7.4 Hz, 3H, H_{11}), 2.03 (dq, *J* = 14.3, 7.3 Hz, 1H, H_{10a}), 2.26 (dq, *J* = 14.3, 7.4 Hz, 1H, H_{10b}), 3.93 (s, 3H, H_{19} or H_{20}), 3.96 (s, 3H, H_{19} or H_{20}), 6.48 (s, 1H, H_9), 6.94 (d, *J* = 8.3 Hz, 1H, H_{17}), 7.10 (d, *J* = 2.0 Hz, 1H, H_{14}), 7.14 (dd, *J* = 8.3 Hz, 2.0 Hz, 1H, H_{18}), 7.32-7.42 (m, 3H, H_{arom}), 7.49-7.53 (m, 1H, H_{arom}); ^{13}C NMR (75.5 MHz, *CDCl*$_3$) δ = 10.4 (C_{11}), 30.0 (C_{10}), 51.0 (C_7), 56.0, 56.1 (C_{19}, C_{20}), 110.9 (C_{14}), 111.2 (C_{17}), 120.9 (C_{12}), 121.2 (C_{18}), 121.4, 121.9 (CH_{arom}), 122.1 (C_9), 128.6 (C_1), 129.8, 129.9 (CH_{arom}), 137.0 (C_8), 142.7 (C_{13}), 145.1 (C_6), 149.0, 149.1 (C_{15}, C_{16}) ppm; HRMS (ESI, MeOH + CH_2Cl_2) calcd for $C_{20}H_{19}NO_2Na$ [$(M + Na)^+$]: 328.1313; found: 328.1294; IR (film) ν = 1236, 1258, 1512, 1599, 2227, 2966 cm^{-1}.



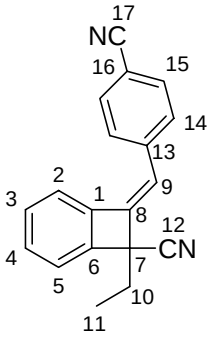
5e

^{1}H NMR (500 MHz, *CDCl*$_3$) δ = 1.24 (dd, *J* = 7.6, 7.6 Hz, 3H, H_{11}), 2.04 (dq, *J* = 14.6, 7.4 Hz, 1H, H_{10a}), 2.26 (dq, *J* = 14.3, 7.3 Hz, 1H, H_{10b}), 3.90 (s, 3H, H_{18}), 3.93 (s, 6H, H_{17}), 6.48 (s, 1H, H_9), 6.80 (s, 1H, H_{14}), 7.34-7.43 (m, 3H, $H_{3\text{-}5}$), 7.52 (d, *J* = 7.4 Hz, 1H, H_2) ppm; ^{13}C NMR (75.5 MHz, *CDCl*$_3$) δ = 10.5 (C_{11}), 30.1 (C_{10}), 51.1 (C_7), 56.3 (C_{17}), 61.0 (C_{18}), 105.4 (C_{14}), 120.7 (C_{12}), 121.5 (C_2), 122.0 (CH_{arom}), 122.2 (C_9), 130.0, 130.1 (CH_{arom}), 131.3 (C_{13}), 138.1 (C_8), 138.3 (C_{16}), 142.5 (C_1), 145.3 (C_6), 153.5 (C_{15}) ppm; HRMS (ESI, MeOH + CH_2Cl_2) calcd for $C_{21}H_{21}NO_3Na$ [$(M + Na)^+$]: 358.1419; found: 358.1382; IR (film) ν = 1520, 1604, 2228, 2967 cm^{-1}.



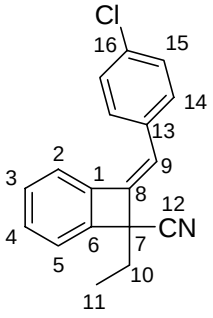
5f

^{1}H NMR (300 MHz, *CDCl*$_3$) δ = 1.24 (dd, *J* = 7.4, 7.4 Hz, 3H, H_{11}), 2.06 (dq, *J* = 14.5, 7.3 Hz, 1H, H_{10a}), 2.28 (dq, *J* = 14.5, 7.2 Hz, 1H, H_{10b}), 2.63 (s, 3H, H_{18}), 6.57 (s, 1H, H_9), 7.37-7.46 (m, 3H, H_{arom}), 7.49-7.55 (m, 1H, H_{arom}), 7.65 (d, *J* = 8.2 Hz, 2H, H_{14}), 8.03 (d, *J* = 8.4 Hz, 2H, H_{15}) ppm; ^{13}C NMR (75.5 MHz, *CDCl*$_3$) δ = 10.4 (C_{11}), 26.6 (C_{18}), 30.0 (C_{10}), 51.3 (C_7), 120.5 (C_{12}), 121.0 (C_9), 122.1 (2×CH_{arom}), 128.0 (C_{14}), 128.8 (C_{15}), 130.2, 130.9 (CH_{arom}), 136.3 (C_{16}), 140.5 (C_{13}), 141.1 (C_8), 142.0 (C_1), 145.5 (C_6), 197.4 (C_{17}) ppm; HRMS (ESI, MeOH + CH_2Cl_2) calcd for $C_{20}H_{17}NONa$ [$(M + Na)^+$]: 310.1208; found: 310.1178; IR (film) ν = 1600, 1678, 2230, 2968 cm^{-1}.



5g

^{1}H NMR (500 MHz, *CDCl*$_3$) δ = 1.24 (dd, *J* = 7.4, 7.4 Hz, 3H, H_{11}), 2.05 (dq, *J* = 14.7, 7.4 Hz, 1H, H_{10a}), 2.28 (dq, *J* = 14.7, 7.3 Hz, 1H, H_{10b}), 6.54 (s, 1H, H_9), 7.38-7.51 (m, 4H, $H_{2\text{-}5}$), 7.65 (d, *J* = 8.5 Hz, 2H, H_{14}), 7.72 (d, *J* = 8.3 Hz, 2H, H_{15}) ppm; ^{13}C NMR (75.5 MHz, *CDCl*$_3$) δ = 10.3 (C_{11}), 30.0 (C_{10}), 51.4 (C_7), 111.3 (C_{16}), 118.7 (C_{17}), 120.1 (C_9), 120.2 (C_{12}), 122.1, 122.2 (CH_{arom}), 128.4 (C_{14}), 130.3, 131.3 (CH_{arom}), 132.5 (C_{15}), 140.3 (C_{13}), 141.6 (C_1), 142.1 (C_8), 145.6 (C_6) ppm; GC/MS (CI (NH_3), DB-5ms column, 1 min at 150 °C then 150 → 280 °C at 8 °C/min): 15.6 min (m/z: 288 [$(M + NH_4)^+$]); IR (film) ν = 1603, 2226, 2969 cm^{-1}.

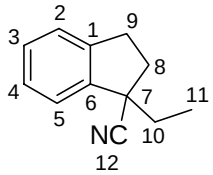


5h

^{1}H NMR (300 MHz, *CDCl*$_3$) δ = 1.22 (dd, *J* = 7.4, 7.4 Hz, 3H, H_{11}), 2.02 (dq, *J* = 15.0, 7.4 Hz, 1H, H_{10a}), 2.25 (dq, *J* = 14.8 Hz, 7.4 Hz, 1H, H_{10b}), 6.48 (s, 1H, H_9), 7.35-7.50 (m, 8H, H_{arom}) ppm; ^{13}C NMR (75.5 MHz, *CDCl*$_3$) δ = 10.4 (C_{11}), 30.0 (C_{10}), 51.2 (C_7), 120.6 (C_{12}), 120.9 (C_9), 121.8, 122.0 (CH_{arom}), 128.9 (C_{14}), 129.2 (C_{15}), 130.1, 130.4 (CH_{arom}), 133.8, 134.2 (C_1, C_{16}), 139.4 (C_8), 142.2 (C_{13}), 145.3 (C_6) ppm; GC/MS (EI, DB-5ms column, 1 min at 150 °C then 150 → 280 °C at 8 °C/min): 14.0 min (m/z: 279 [$M^{+\bullet}$]); IR (film) ν = 1092, 1592, 2230, 2968 cm^{-1}.

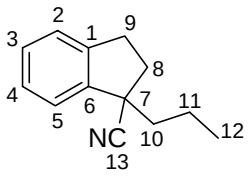
III. C(sp^3)–H Activation / Heck Reactions / Hydrogenation One-Pot Sequences (Table 3)

Products characterization



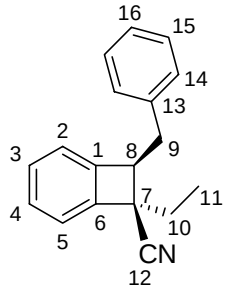
6b

^{1}H NMR (300 MHz, *CDCl*$_3$) δ = 1.15 (dd, *J* = 7.4, 7.4 Hz, 3H, H_{11}), 1.79 (dq, *J* = 14.7, 7.4 Hz, 1H, H_{10a}), 2.01 (dq, *J* = 14.9, 7.4 Hz, 1H, H_{10b}), 2.26 (ddd, *J* = 13.0, 7.6, 5.5 Hz, 1H, H_{8a}), 2.61 (ddd, *J* = 13.0, 8.6, 7.2 Hz, 1H, H_{8b}), 2.94-3.14 (m, 2H, H_9), 7.25-7.30 (m, 3H, H_{arom}), 7.36-7.42 (m, 1H, H_{arom}) ppm; ^{13}C NMR (75.5 MHz, *CDCl*$_3$) δ = 9.8 (C_{11}), 30.2 (C_9), 31.7 (C_{10}), 36.9 (C_8), 47.9 (C_7), 123.2 (C_{12}), 123.8, 125.1, 127.1, 128.6 ($C_{2\text{-}5}$), 142.3, 142.5 (C_1, C_6) ppm; GC/MS (EI, DB-5ms column, 1 min at 120 °C then 120 → 250 °C at 8 °C/min): 6.6 min (m/z: 171 [$M^{+\bullet}$]); IR (film) ν = 1458, 2232, 2969 cm^{-1}.



6c

^{1}H NMR (500 MHz, *CDCl*$_3$) δ = 0.97 (dd, *J* = 7.2, 7.2 Hz, 3H, H_{12}), 1.50-1.71 (m, 3H, H_{10a}, H_{11}), 1.91 (ddd, *J* = 18.0, 12.5, 4.3 Hz, 1H, H_{10b}), 2.24 (ddd, *J* = 13.0, 7.9, 5.4 Hz, 1H, H_{8a}), 2.59 (ddd, *J* = 12.9, 8.6, 7.2 Hz, 1H, H_{8b}), 2.95-3.09 (m, 2H, H_9), 7.23-7.30 (m, 3H, H_{arom}), 7.34-7.40 (m, 1H, H_{arom}) ppm; ^{13}C NMR (75.5 MHz, *CDCl*$_3$) δ = 14.0 (C_{12}), 18.9 (C_{11}), 30.2 (C_9), 37.5 (C_8), 40.8 (C_{10}), 47.2 (C_7), 123.4 (C_{13}), 123.8, 125.1, 127.1, 128.5 ($C_{2\text{-}5}$), 142.4, 142.5 (C_1, C_6) ppm; GC/MS (EI, DB-5ms column, 1 min at 120 °C then 120 → 250 °C at 8 °C/min): 7.7 min (m/z: 185 [$M^{+\bullet}$]); IR (film) ν = 1458, 2232, 2960 cm^{-1}.



6d

^{1}H NMR (500 MHz, *CDCl*$_3$) δ = 1.17 (dd, *J* = 7.4, 7.4 Hz, 3H, H_{11}), 2.00 (q, *J* = 7.4 Hz, 2H, H_{10}), 3.09 (dd, *J* = 13.7, 10.1 Hz, 1H, H_{9a}), 3.35 (dd, *J* = 13.8, 6.6 Hz, 1H, H_{9b}), 3.65 (dd, *J* = 10.1, 6.6 Hz, 1H, H_8), 6.79 (d, *J* = 6.3 Hz, 1H, H_2), 7.21-7.39 (m, 8H, H_{arom}) ppm; ^{13}C NMR (125.8 MHz, *CDCl*$_3$) δ = 10.7 (C_{11}), 30.8 (C_{10}), 38.1 (C_9), 50.0 (C_7), 54.7 (C_8), 120.5 (C_{12}), 121.9 (CH_{arom}), 123.5

(C_2), 126.7, 128.5 (CH_{arom}), 128.6 (C_{15}), 129.1 (C_{14}), 129.2 (CH_{arom}), 138.9 (C_{13}), 142.3 (C_6), 145.7 (C_1) ppm; GC/MS (CI (NH_3), DB-5ms column, 1 min at 120 °C then 120 → 250 °C at 8 °C/min): 13.7 min (m/z: 265 [$(M + NH_4)^+$]); IR (film) ν = 1455, 1496, 2230, 2966 cm^{-1}.

Transmission Electronic Microscopy

Electron microscopy observations were performed with a scanning transmission electron microscope STEM VG HB501. Particle size distribution was measured at 100% substrate conversion (for the reaction **1a** → **1b**, see Scheme 1) from 8 different images, each corresponding to a different sample zone, using the ImageJ 1.33 analysis program from the National Institutes of Health, USA (http://rsb.info.nih.gov/ij). The average particle diameter was 3.1 ± 0.2 nm.

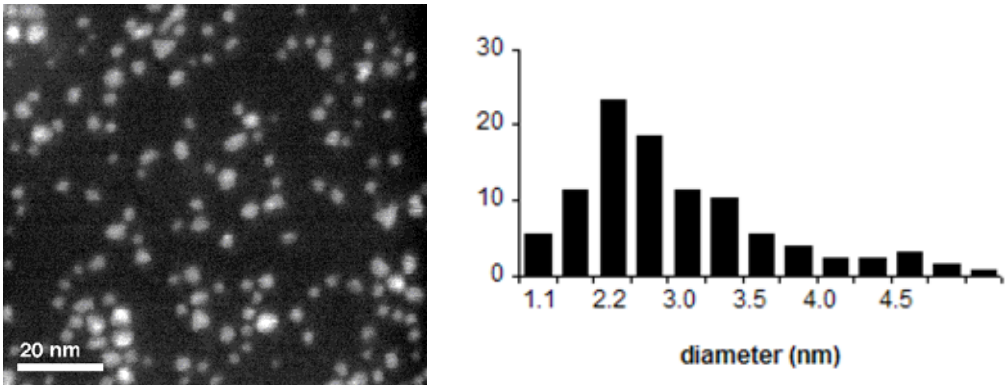
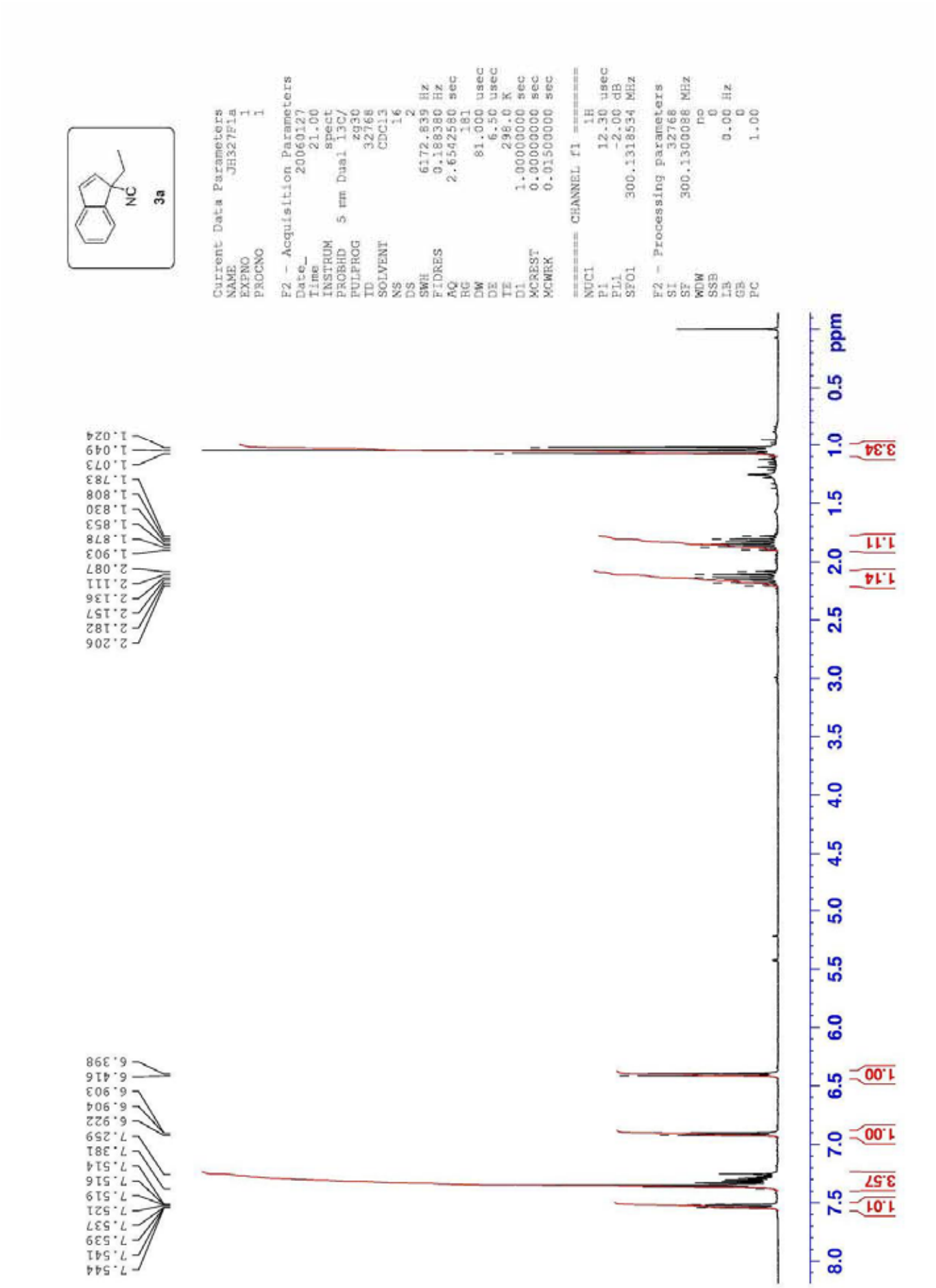


Figure S1. TEM Image and Size Distribution of Palladium Nanoparticles Formed During the Reaction **1a** → **1b** (100 % conversion)

IV. Inhibition of Microtubule Assembly (Figure 1)

The drug, dissolved in DMSO at different concentrations, was pre-incubated with a solution of tubulin at 37 °C for 10 min, then the solution was cooled to 0 °C for 5 min in order to achieve complete tubulin depolymerization. The solution was then placed in a temperature-controlled cell at 37 °C (microtubule assembly) and the increase of the optical density was monitored in a UV spectrophotometer at 350 nm for 1 min. The maximum rate of assembly was recorded and compared to a sample without drug. The IC_{50} is the concentration of compound required to inhibit 50 % of the rate of microtubule assembly. It was calculated from the effect of several concentrations and compared to the IC_{50} of colchicine obtained within the same day with the same tubulin preparation. Reported values are averages of three experiments.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PROCNO 1
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 181
DW 81.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
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P1 12.30 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
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SF 300.1300088 MHz
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SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1.024
1.049
1.073
1.783
1.808
1.830
1.853
1.878
1.903
2.087
2.111
2.136
2.157
2.182
2.206
6.398
6.416
6.903
6.904
6.922
7.259
7.381
7.514
7.516
7.519
7.521
7.537
7.539
7.541
7.544
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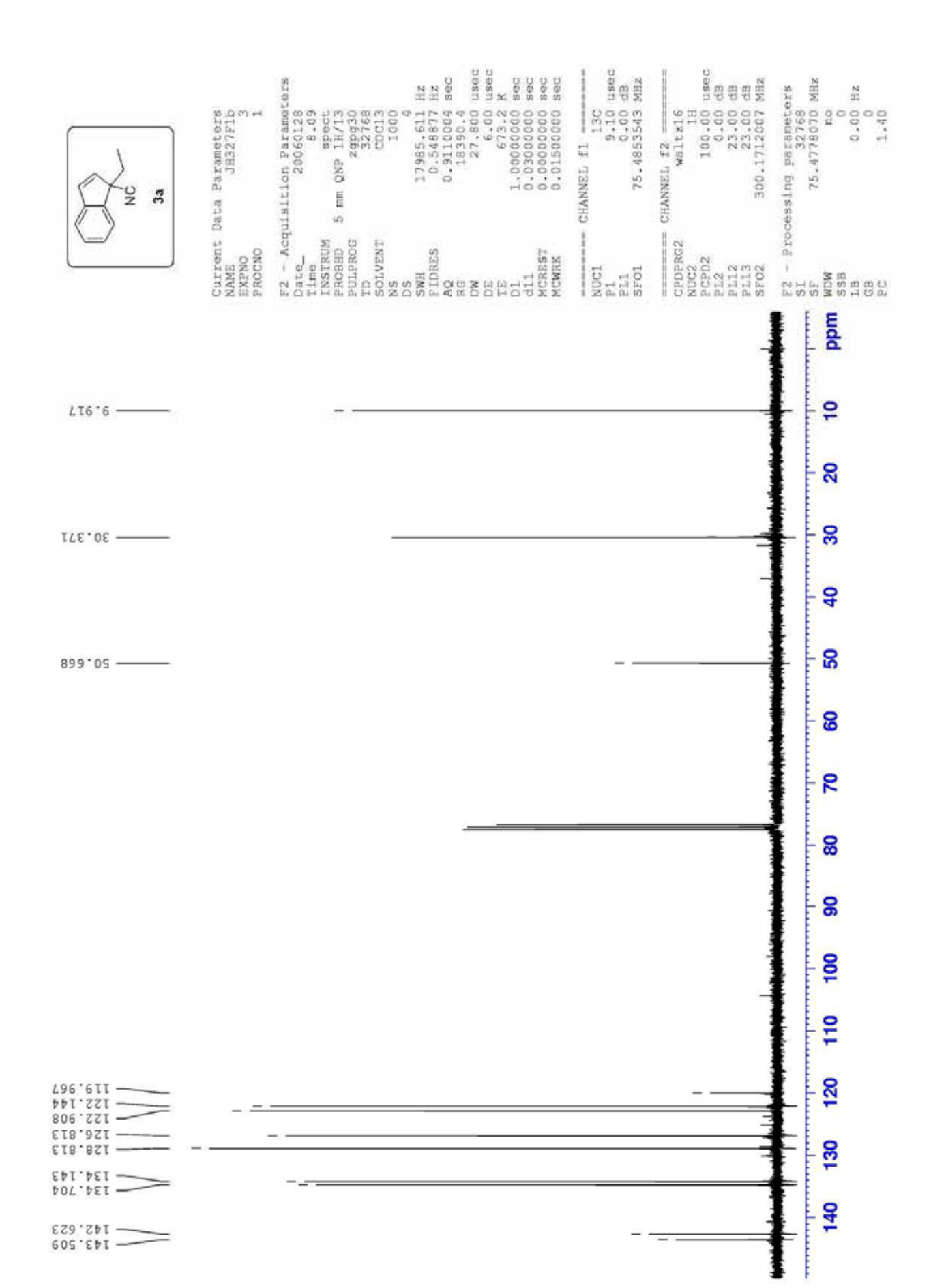
1.14
1.00
1.00
3.57
1.01
ppm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3a
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NAME JB327F1b
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
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Time 8.09
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PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1000
DS 4
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110004 sec
RG 18390.4
DW 27.800 usec
DE 6.00 usec
TE 673.2 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 9.10 usec
PL1 0.00 dB
SFO1 75.4853543 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.00 dB
PL13 23.00 dB
SFO2 300.1712007 MHz
F2 - Processing parameters
SI 32768
SF 75.4778070 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
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122.908
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128.813
134.143
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143.509
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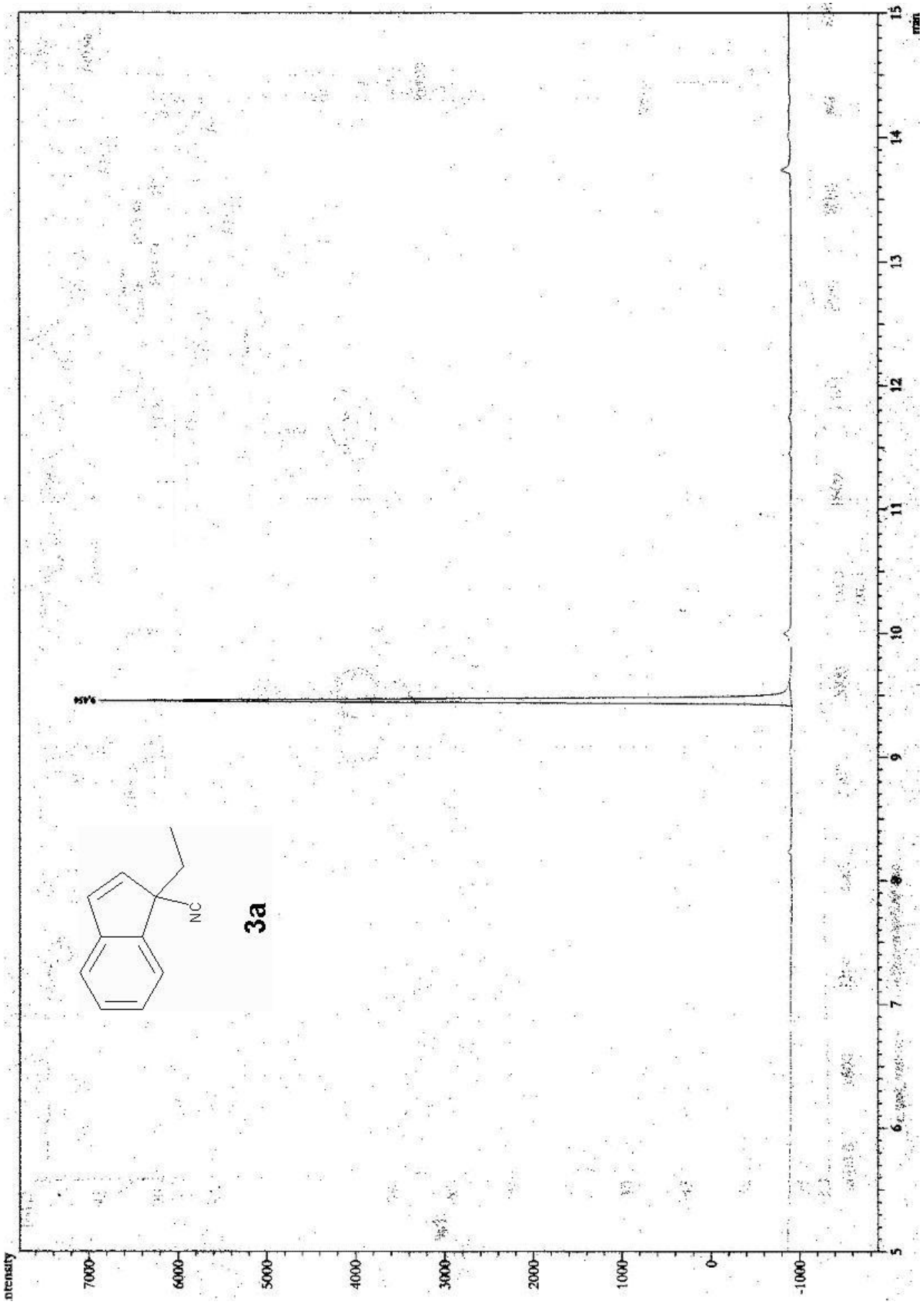
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5000
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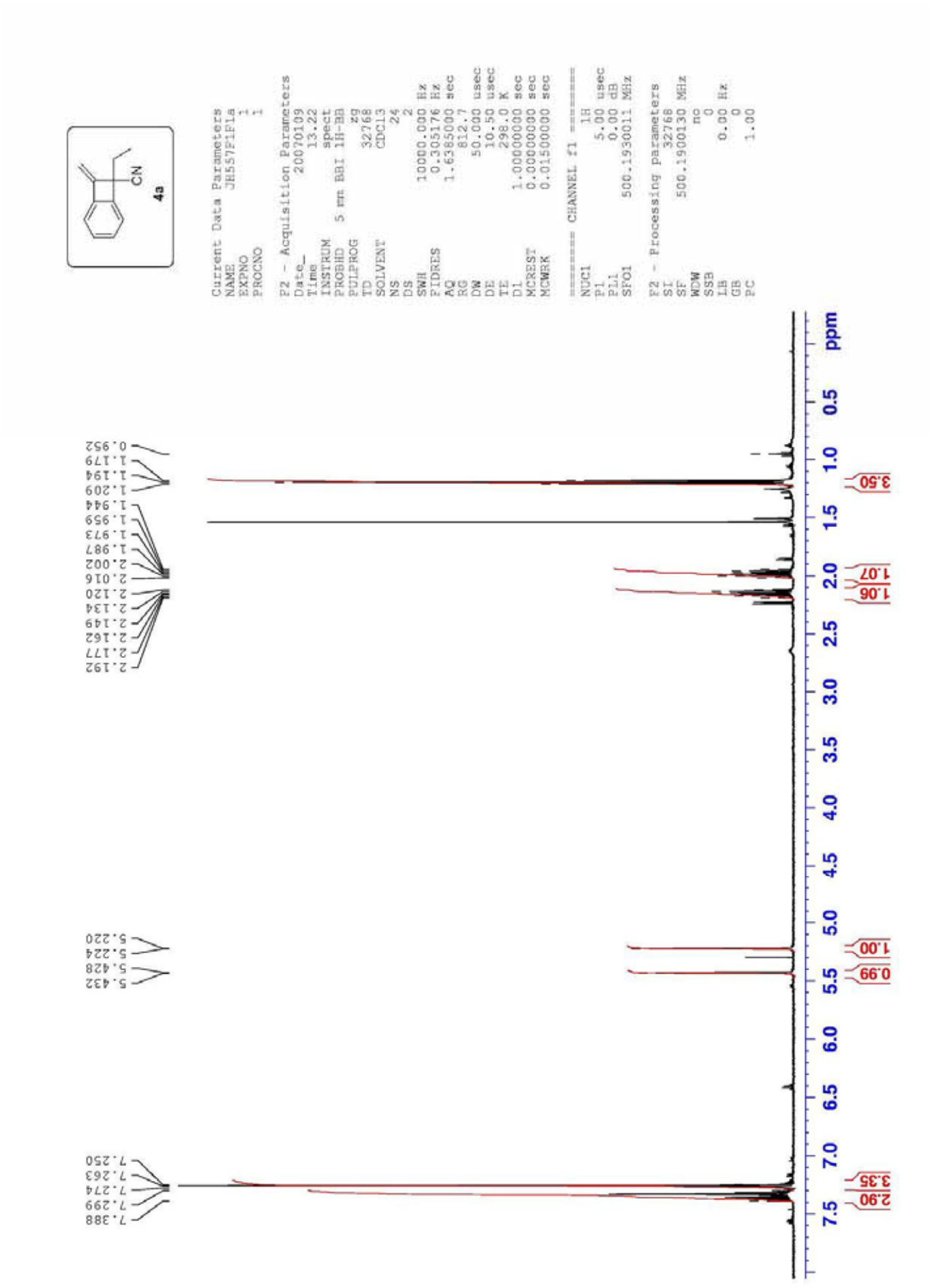
3000
2000
1000
0
-1000
5
6
7
8
9
10
11
12
13
14
15
min
NC
3a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CN
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EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
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Time 13.22
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PULPROG zg
TD 32768
SOLVENT CDCl3
NS 24
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 1.6385000 sec
RG 812.7
DW 50.000 usec
DE 10.50 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
SFO1 500.1930011 MHz
F2 - Processing parameters
SI 32768
SF 500.1900130 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
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7.299
7.274
7.263
7.250
5.432
5.428
5.224
5.220
2.192
2.177
2.162
2.149
2.134
2.120
2.016
2.002
1.987
1.973
1.959
1.944
1.209
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1.179
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3.50
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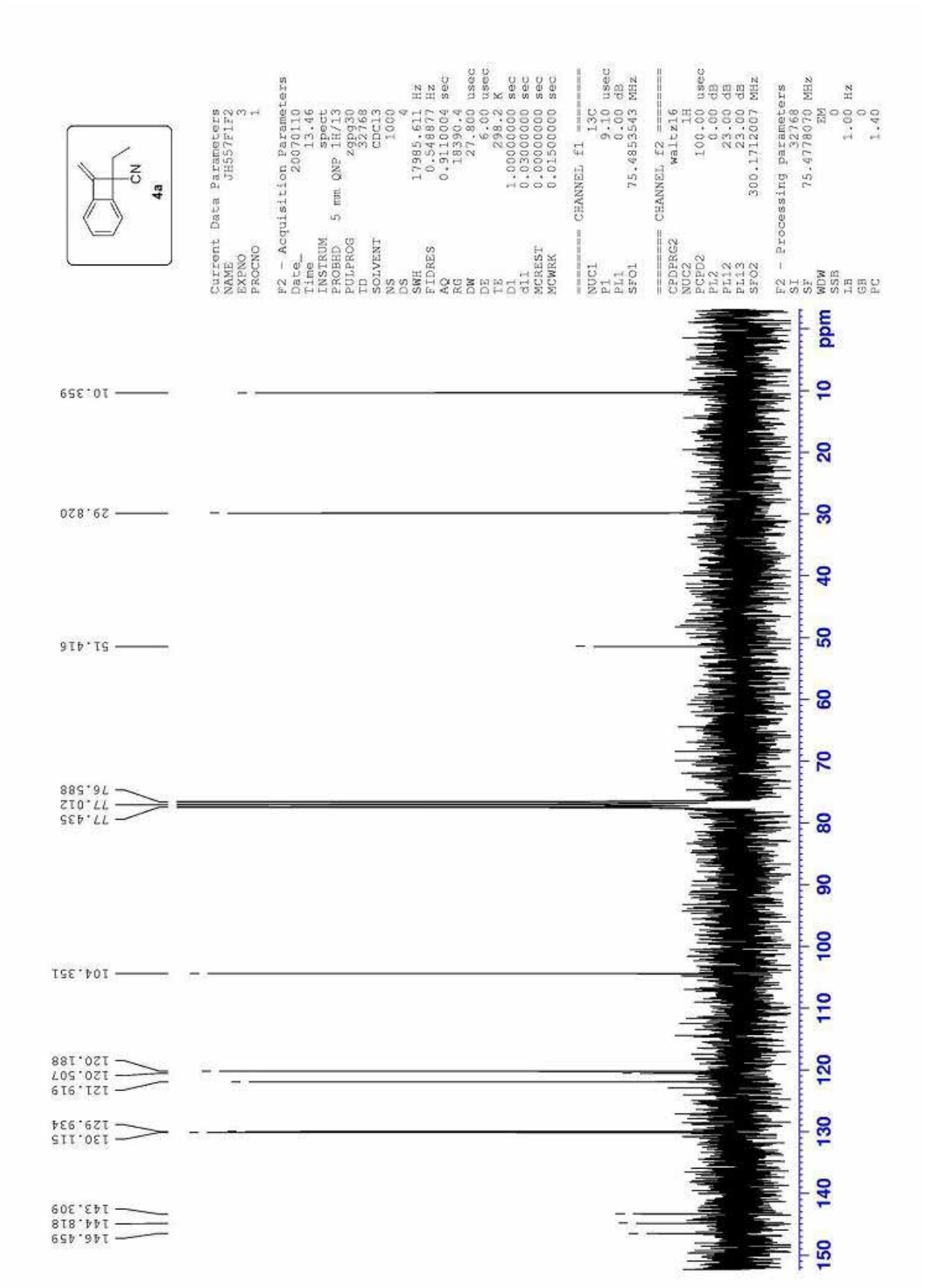
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EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
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Time 13.46
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PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1000
DS 4
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110004 sec
RG 18390.4
DW 27.800 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
NUC1 13C
P1 9.10 usec
PL1 0.00 dB
SFO1 75.4853543 MHz
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.00 dB
PL13 23.00 dB
SFO2 300.1712007 MHz
F2 - Processing parameters
SI 32768
SF 75.4778070 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
10.359
29.820
51.416
76.588
77.012
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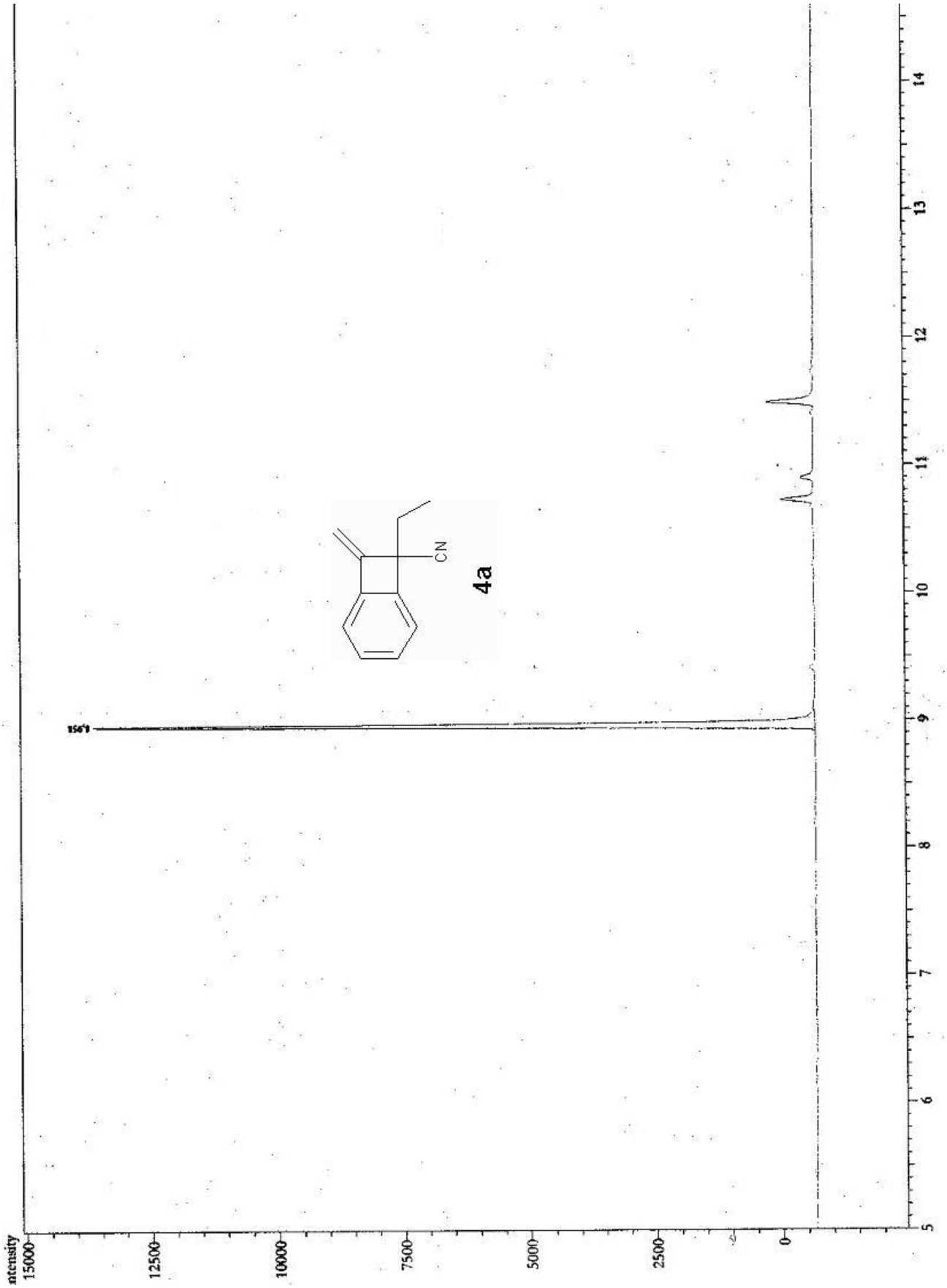
146.459
ppm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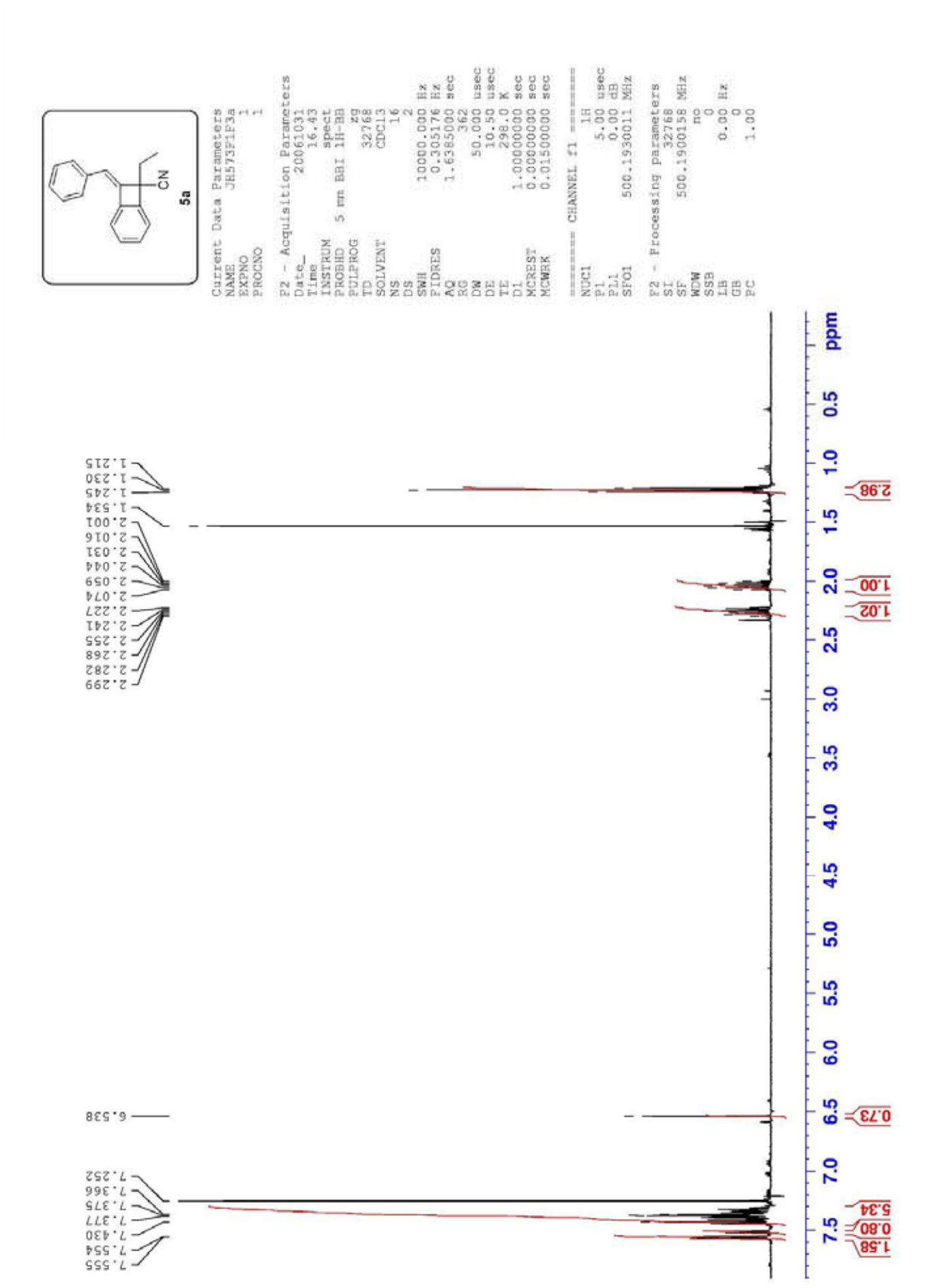
7500
5000
2500
0
5
6
7
8
9
10
11
12
13
14
CN
4a


5a
CN
Current Data Parameters
NAME JE573P1P3a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061031
Time 16.43
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 1.6385000 sec
RG 362
DW 50.000 usec
DE 10.50 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
SFO1 500.1930011 MHz
F2 - Processing parameters
SI 32768
SF 500.1900158 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.555
7.554
7.430
7.377
7.375
7.366
7.252
6.538
2.299
2.282
2.268
2.255
2.241
2.227
2.074
2.059
2.044
2.031
2.016
2.001
1.534
1.245
1.230
1.215
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
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1.02
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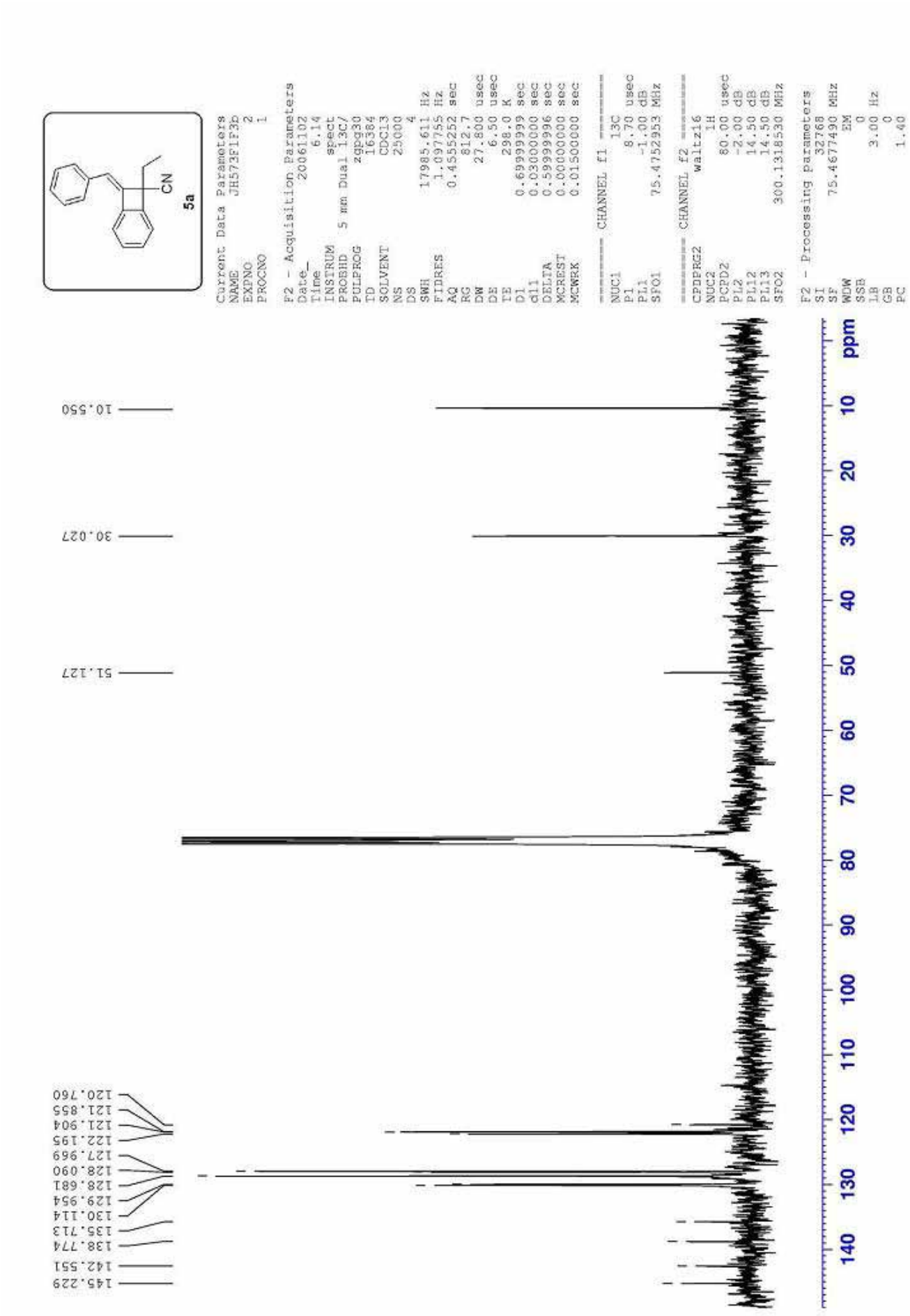
0.80
5.34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EXPNO 2
PROCNO 1
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Time 6.14
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PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 25000
DS 4
SWH 17985.611 Hz
FIDRES 1.097755 Hz
AQ 0.4555252 sec
RG 812.7
DW 27.800 usec
DE 6.50 usec
TE 298.0 K
D1 0.69999999 sec
d11 0.03000000 sec
DELTA 0.59999996 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 8.70 usec
PL1 -1.00 dB
SFO1 75.4752953 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.50 dB
PL13 14.50 dB
SFO2 300.1318530 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
145.229
142.551
138.774
135.713
130.114
129.954
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128.090
127.969
122.195
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120.760
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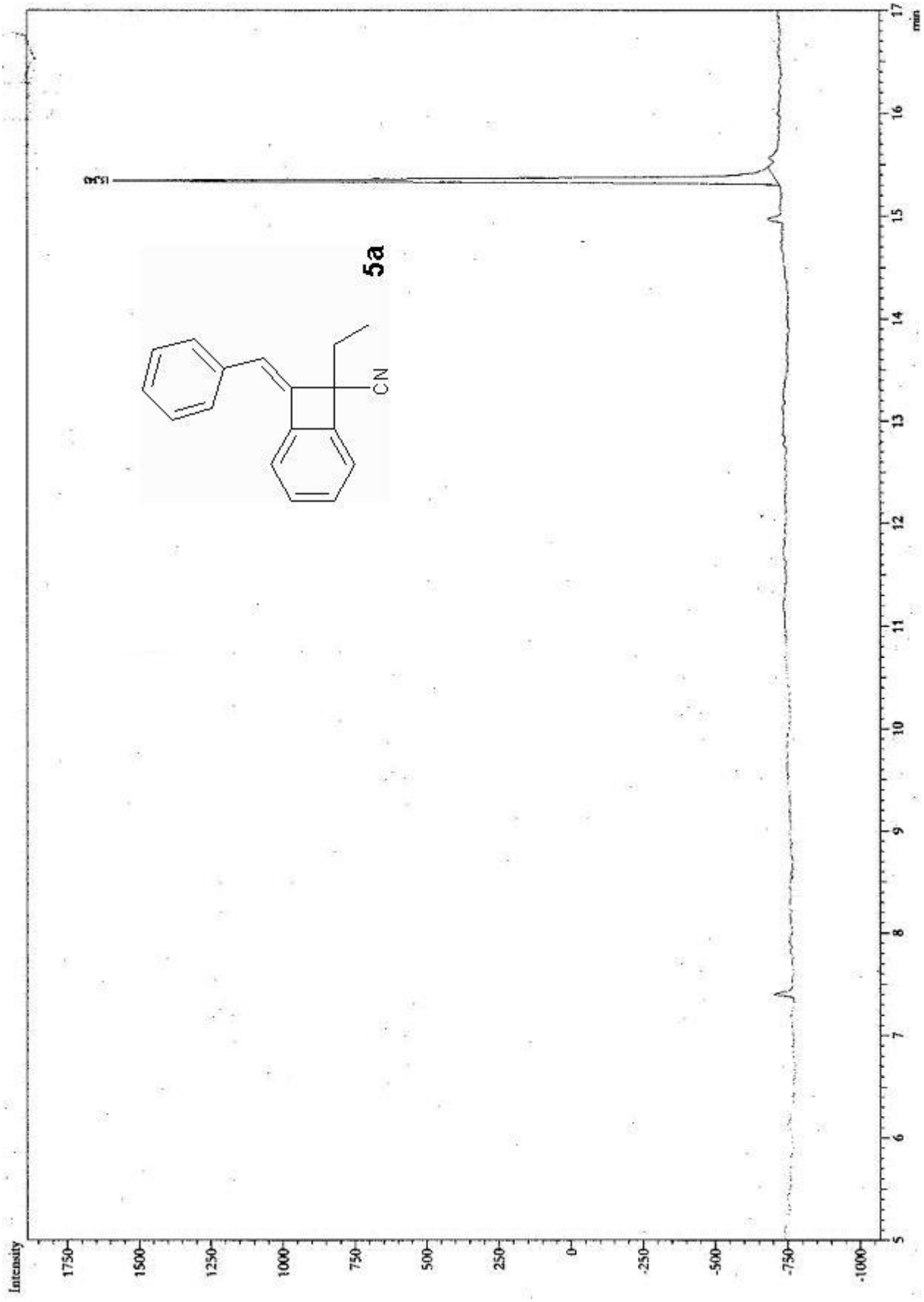
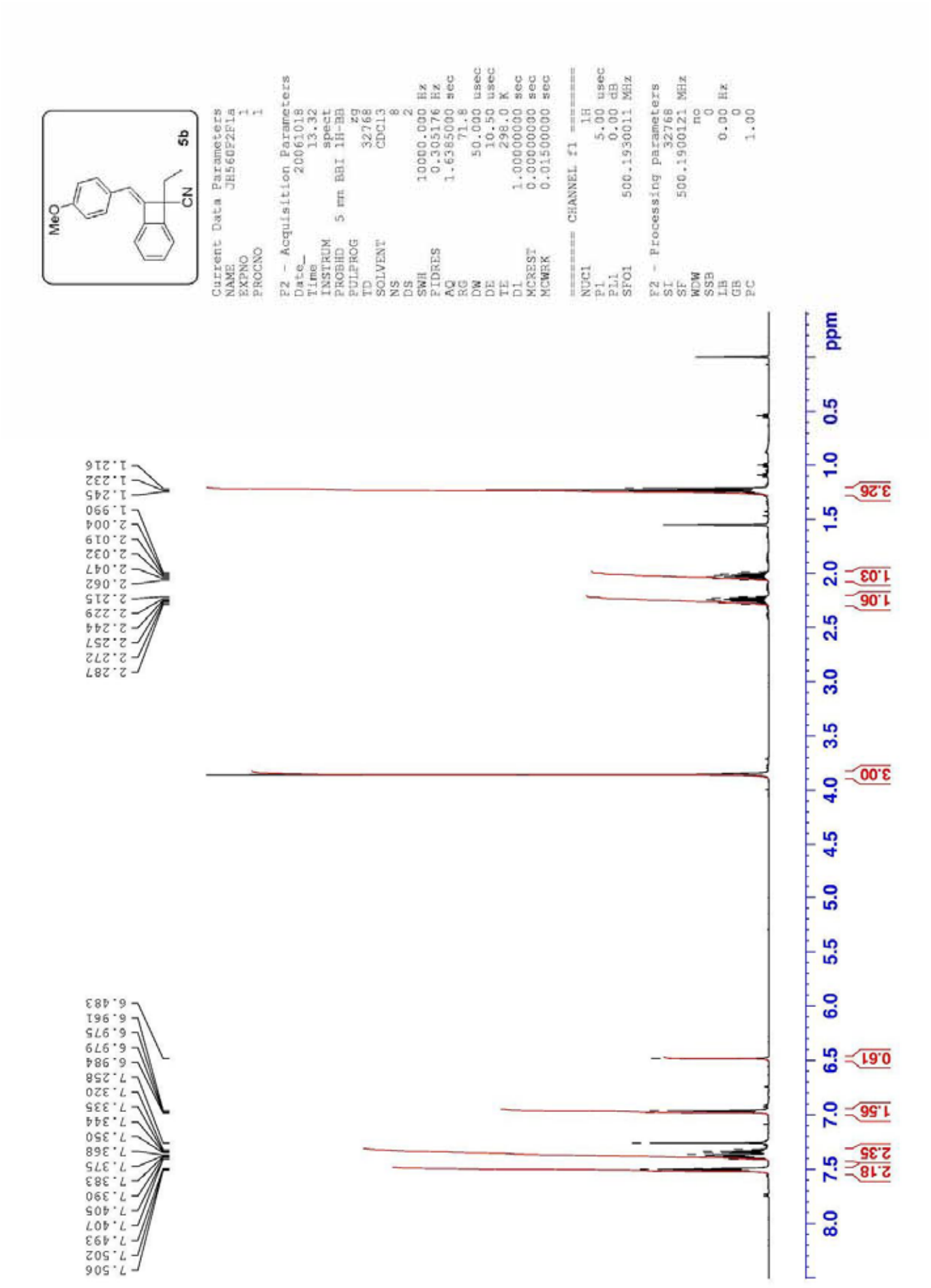
30.027
10.550
ppm


5a
CN
Intensity
min


MeO
CN
5b
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EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
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Time 13.32
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 1.6385000 sec
RG 71.8
DW 50.000 usec
DE 10.50 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
SFO1 500.1930011 MHz
F2 - Processing parameters
SI 32768
SF 500.1900121 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
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1.232
1.245
1.990
2.004
2.019
2.032
2.047
2.062
2.215
2.229
2.244
2.257
2.272
2.287
6.483
6.961
6.975
6.979
6.984
7.258
7.320
7.335
7.344
7.350
7.368
7.375
7.383
7.390
7.405
7.407
7.493
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7.506
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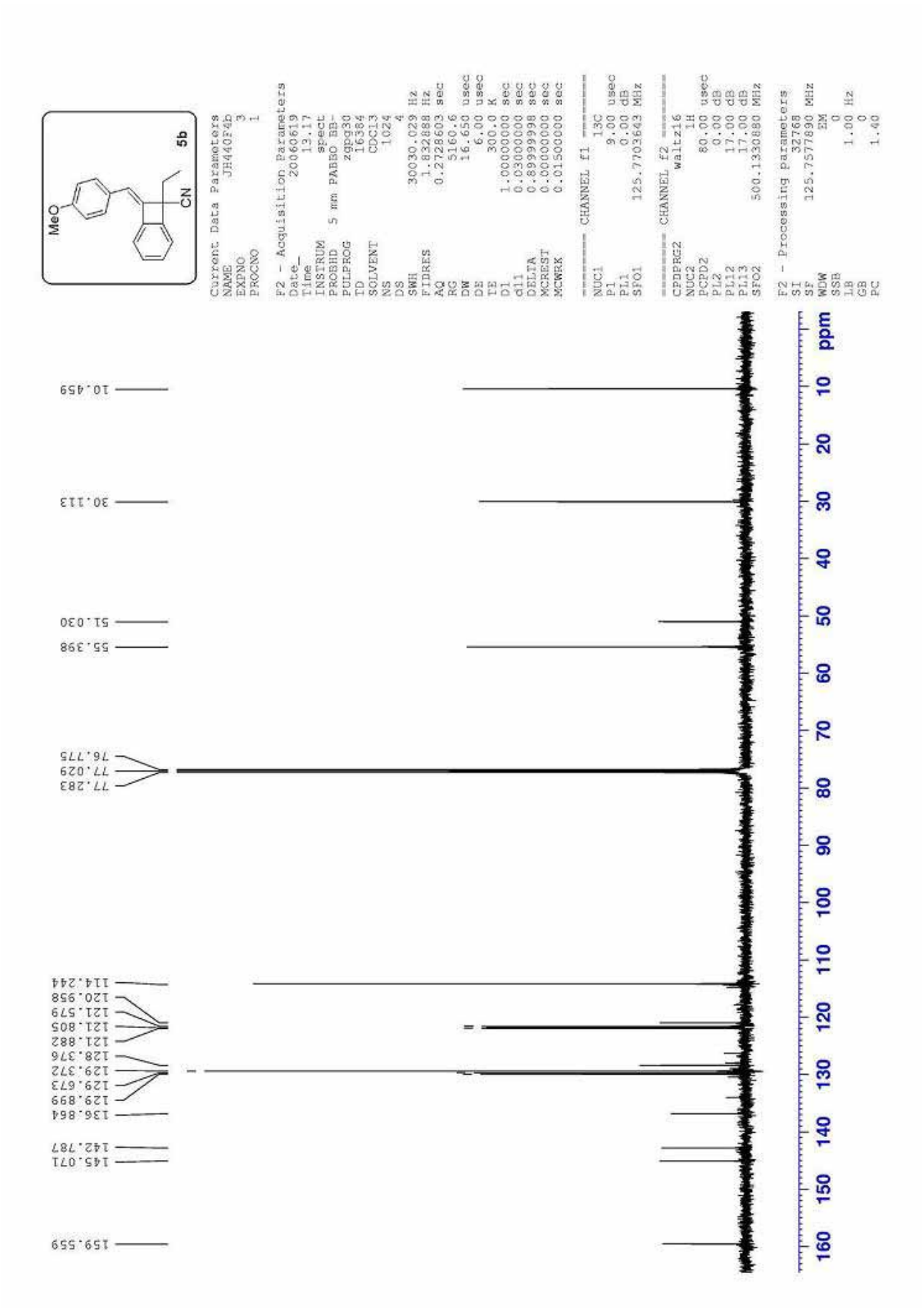
2.18
ppm


MeO
CN
5b
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EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060619
Time 13.17
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PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 1024
DS 4
SWH 30030.029 Hz
FIDRES 1.832888 Hz
AQ 0.2728603 sec
RG 5160.6
DW 16.650 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 9.00 usec
PL1 0.00 dB
SFO1 125.7703643 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 500.1330880 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
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30.113
51.030
55.398
76.775
77.029
77.283
114.244
120.958
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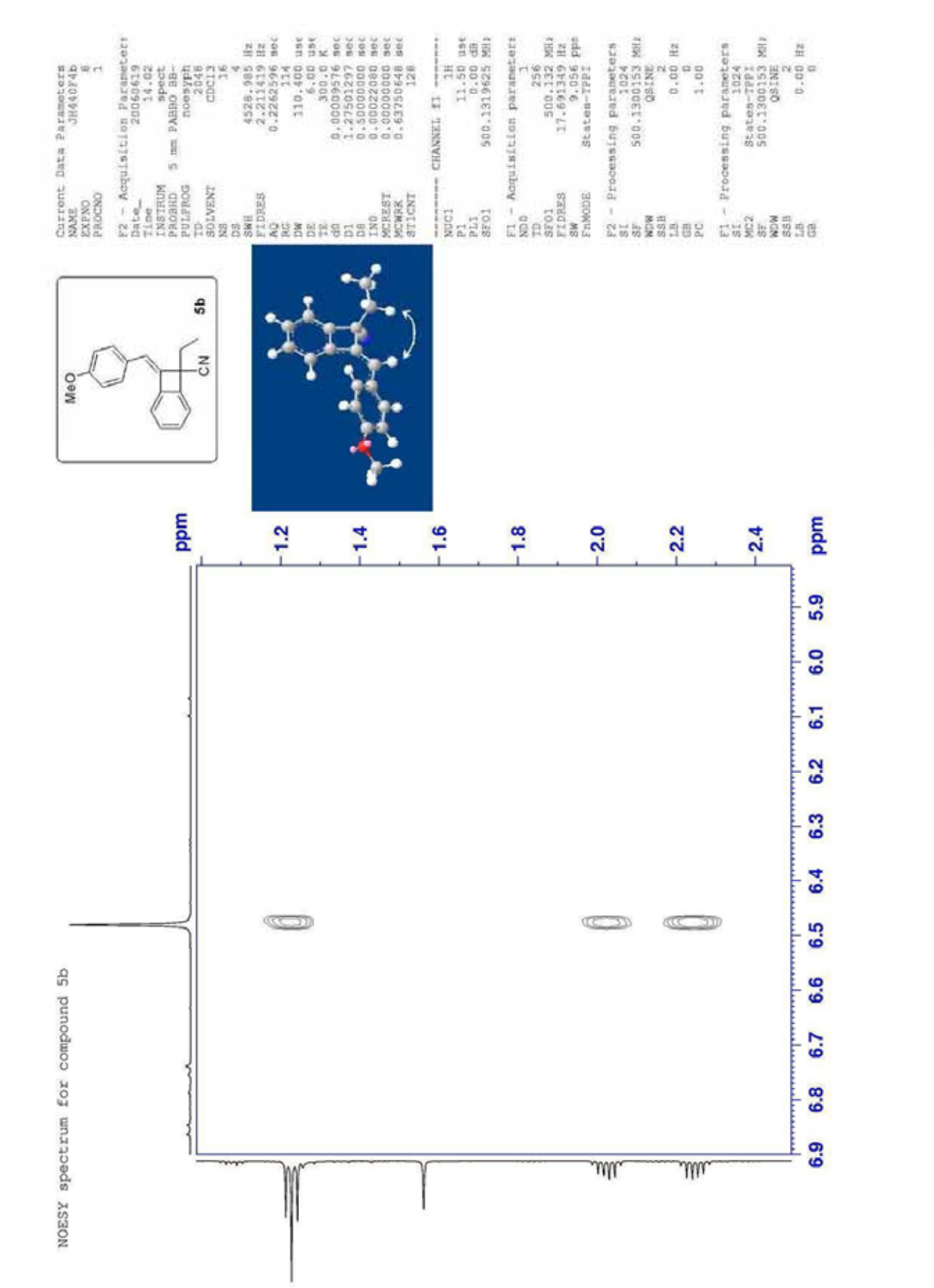
159.559
ppm


NOESY spectrum for compound 5b
MeO
CN
5b
ppm
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1.6
1.8
2.0
2.2
2.4
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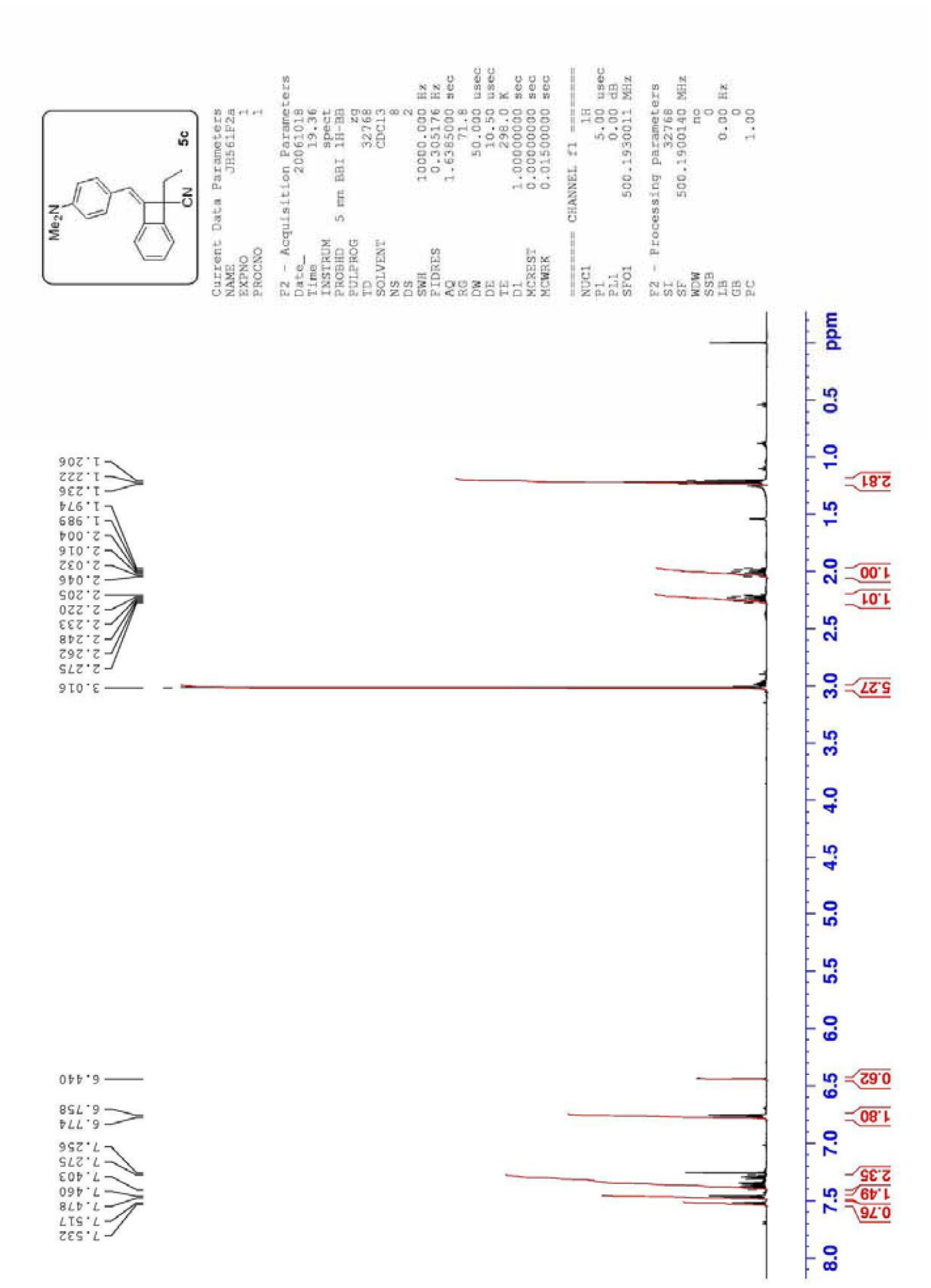
6.7
6.6
6.5
6.4
6.3
6.2
6.1
6.0
5.9
ppm


Me2N
CN
5c
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NAME JR561P2a
EXPNO 1
PROCNO 1
P2 - Acquisition Parameters
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Time 19.36
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PROBHD 5 mm BBI 1H-BB
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 1.6385000 sec
RG 71.8
DW 50.000 usec
DE 10.50 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
SFO1 500.1930011 MHz
F2 - Processing parameters
SI 32768
SF 500.1900140 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
ppm
2.81
1.00
1.01
5.27
0.62
1.80
2.35
1.49
0.76
3.016
2.275
2.262
2.248
2.233
2.220
2.205
2.046
2.032
2.016
2.004
1.989
1.974
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7.403
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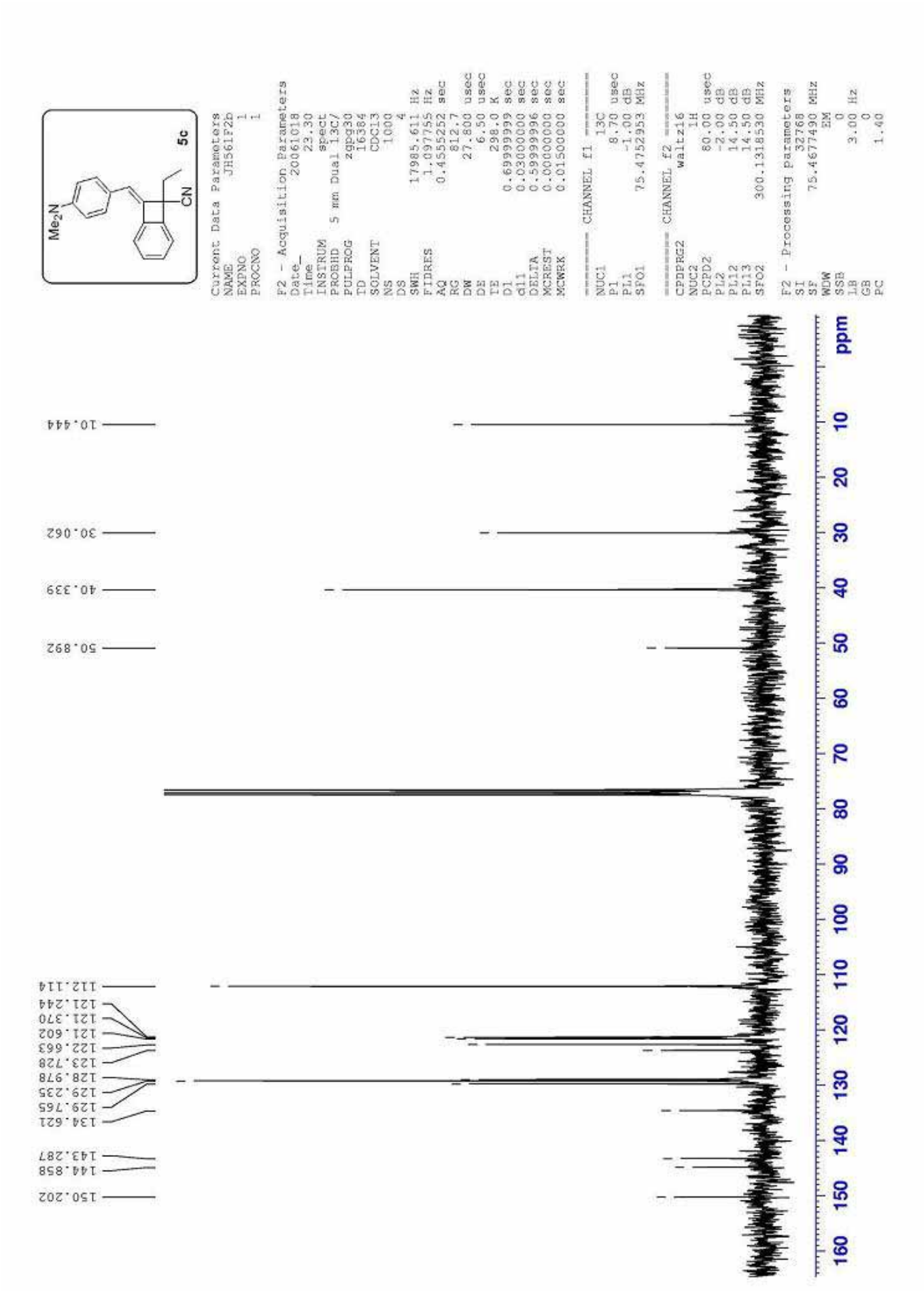
7.478
7.517
7.532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CN
5c
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PROCNO 1
F2 - Acquisition Parameters
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Time 23.30
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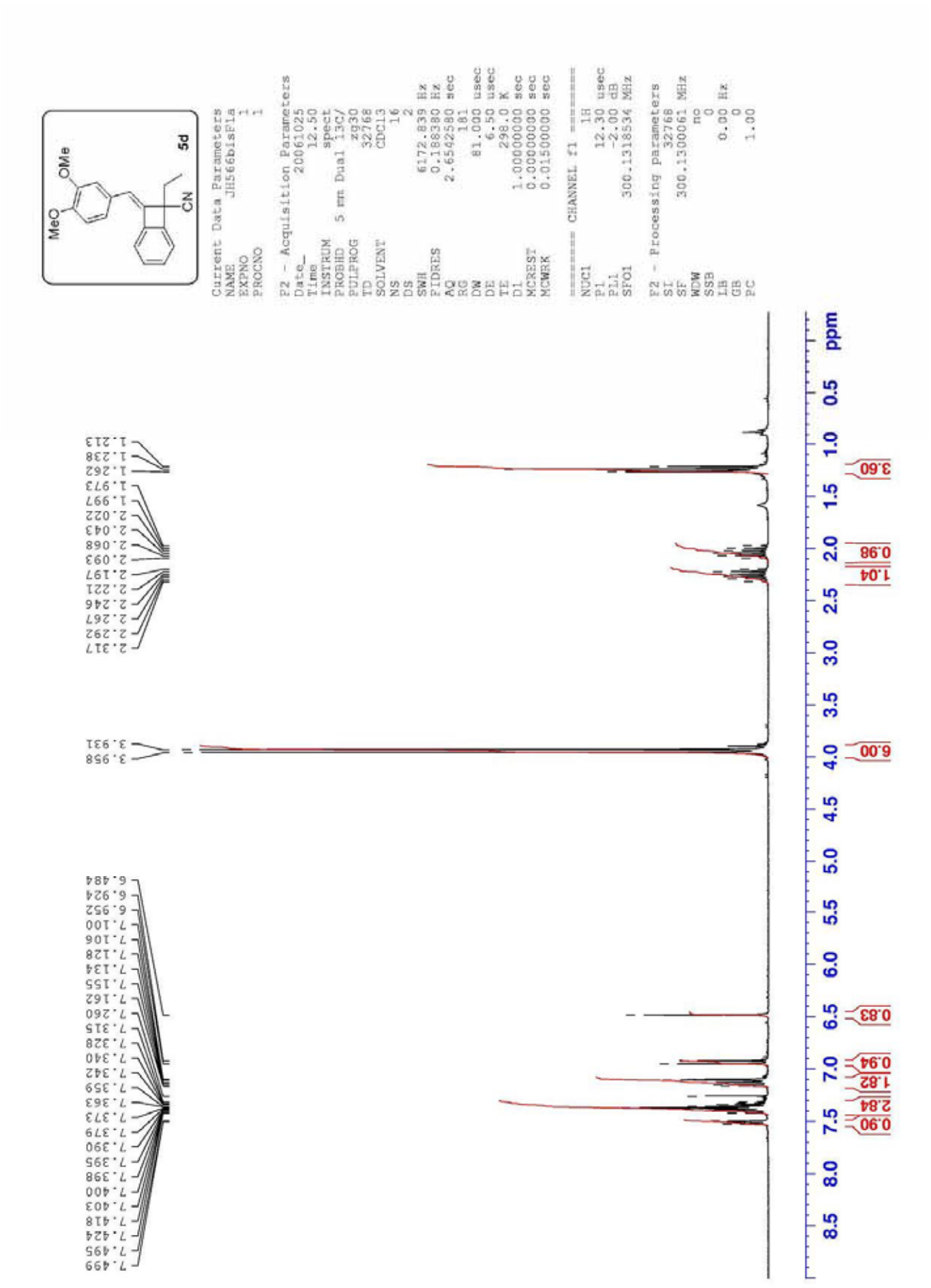
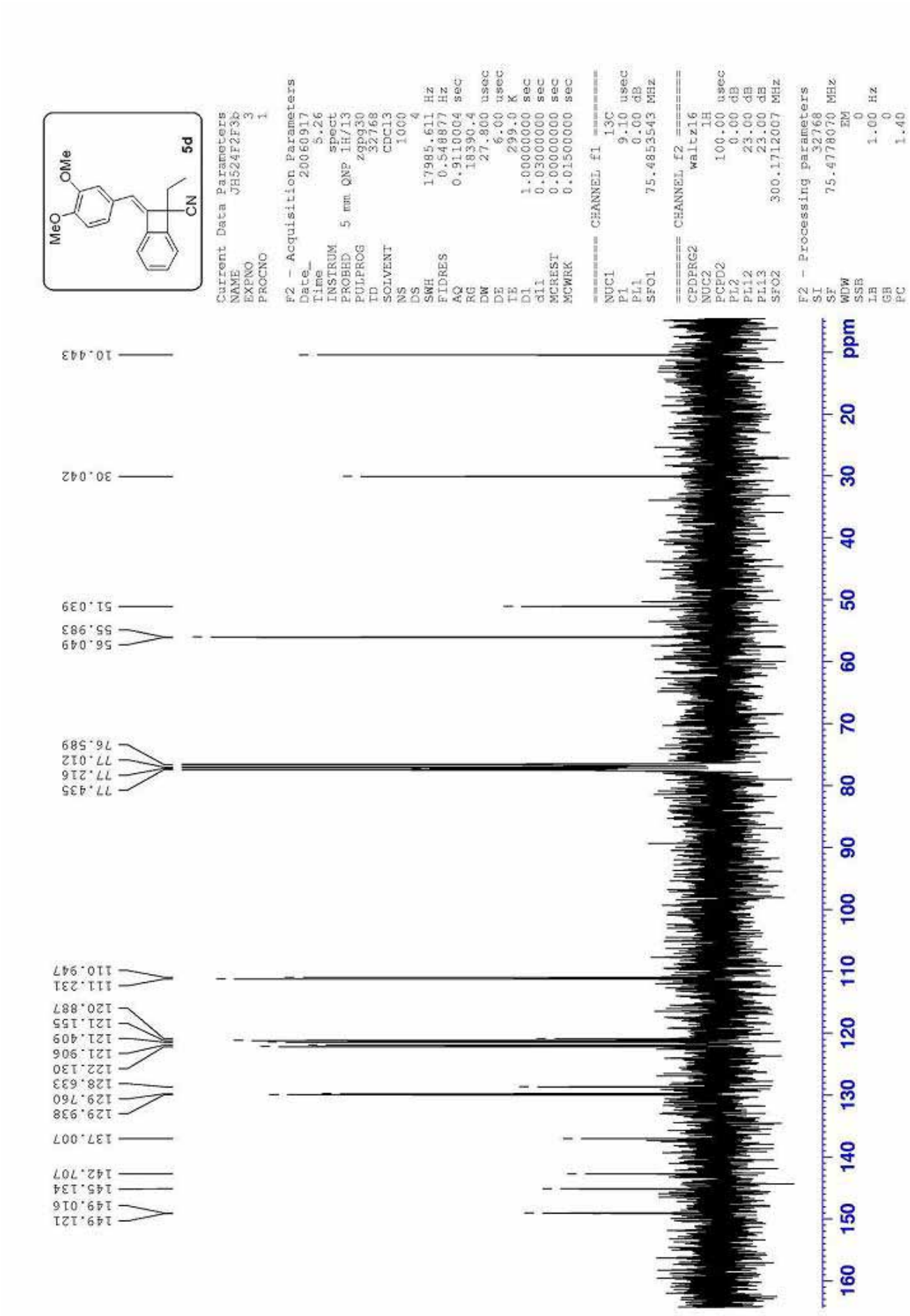
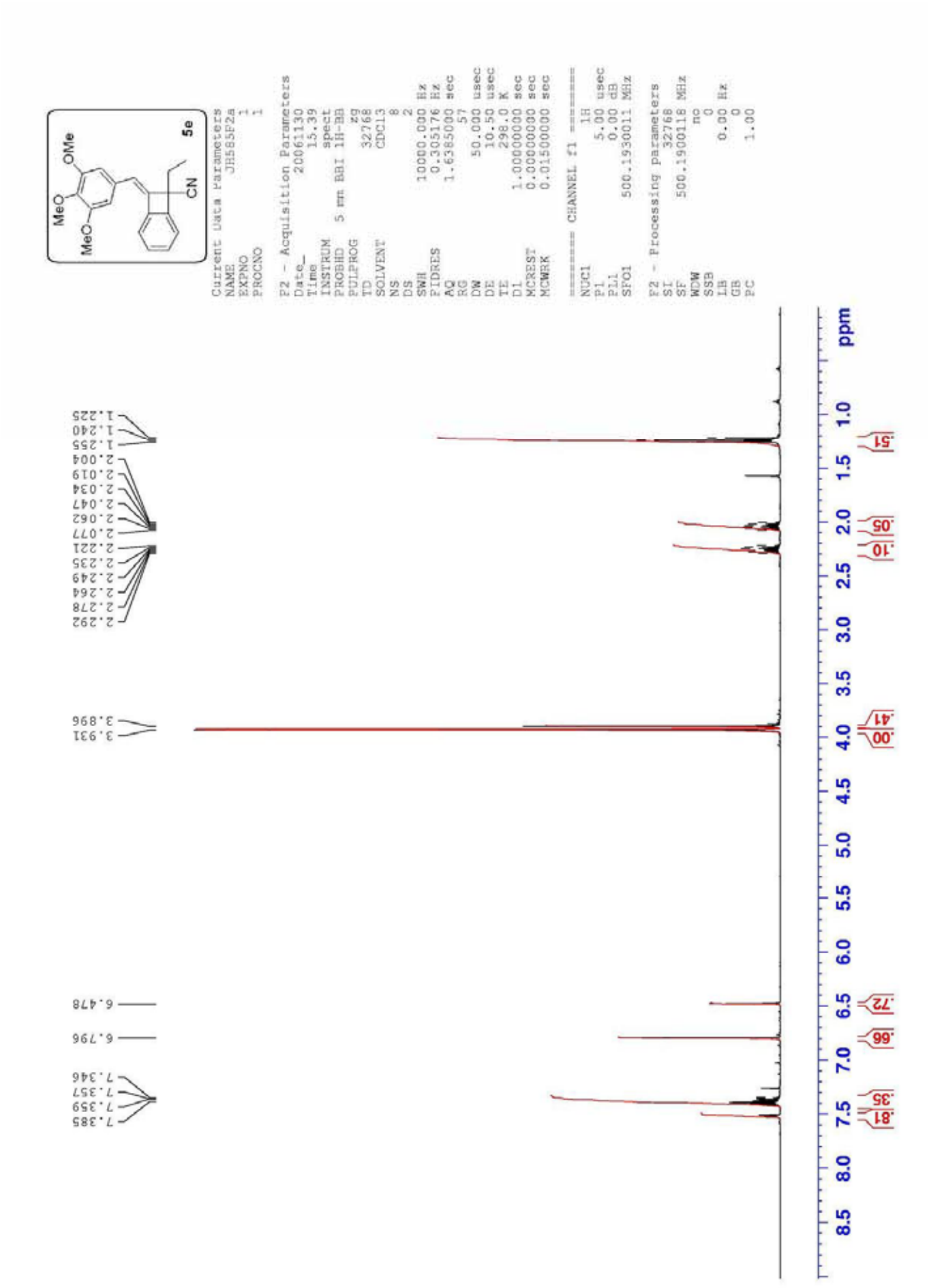
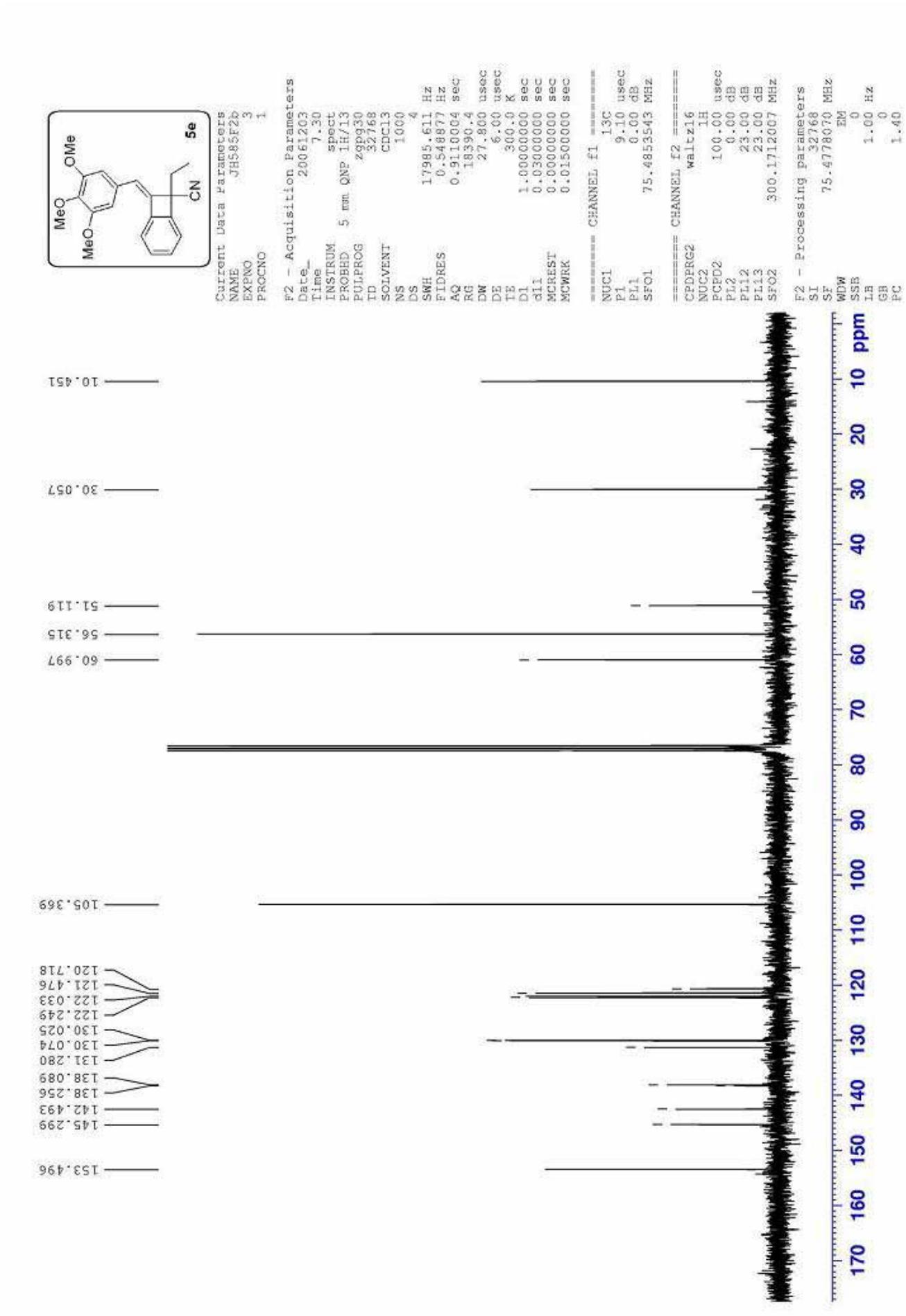
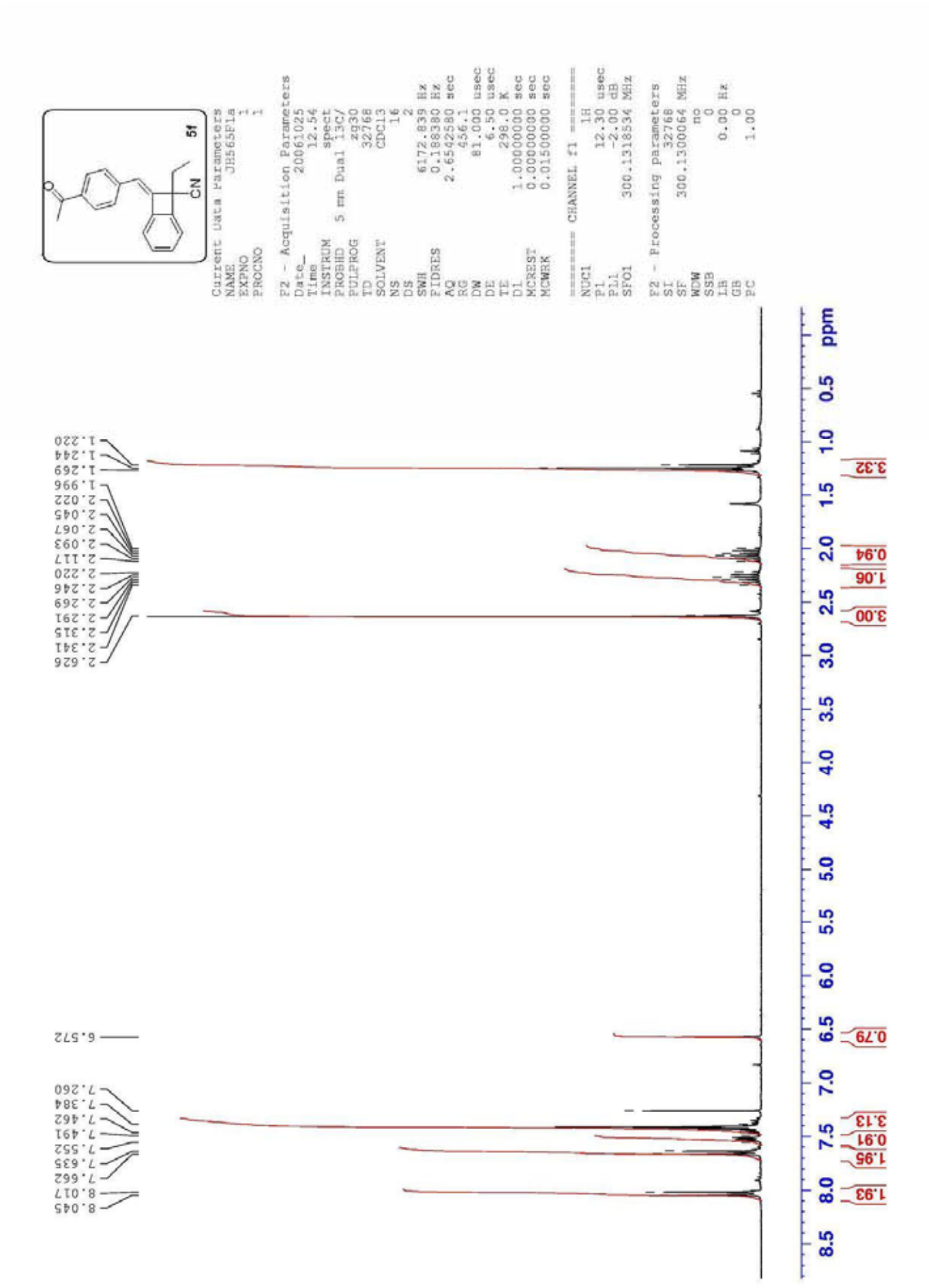
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 1000
DS 4
SWH 17985.611 Hz
FIDRES 1.097755 Hz
AQ 0.4555252 sec
RG 812.7
DW 27.800 usec
DE 6.50 usec
TE 298.0 K
D1 0.69999999 sec
d11 0.03000000 sec
DELTA 0.59999996 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 8.70 usec
PL1 -1.00 dB
SFO1 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.50 dB
PL13 14.50 dB
SFO2 300.1318530 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
10.444
30.062
40.339
50.892
112.114
121.244
121.370
121.602
122.663
123.728
128.978
129.235
129.765
134.621
143.287
144.858
150.202
ppm

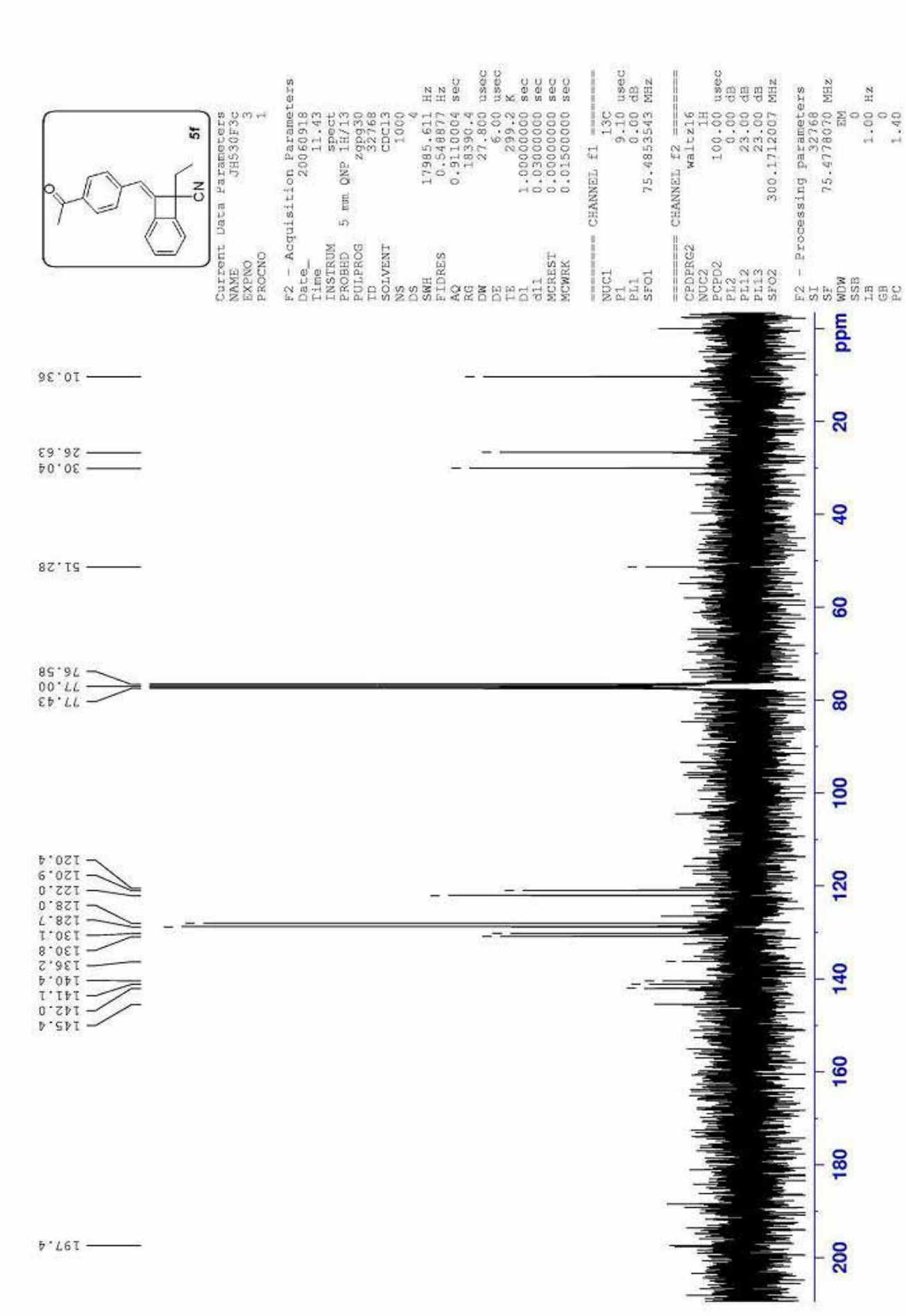

MeO
OMe
CN
5d
Current Data Parameters
NAME JH566bisF1a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061025
Time 12.50
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 181
DW 81.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.30 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300061 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.499
7.495
7.424
7.418
7.403
7.400
7.398
7.395
7.390
7.379
7.373
7.363
7.359
7.342
7.340
7.328
7.315
7.260
7.162
7.155
7.134
7.128
7.106
7.100
6.952
6.924
6.484
3.958
3.931
2.317
2.292
2.267
2.246
2.221
2.197
2.093
2.068
2.043
2.022
1.997
1.973
1.262
1.238
1.213
3.60
0.98
1.04
6.00
0.83
0.94
1.82
2.84
0.90
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

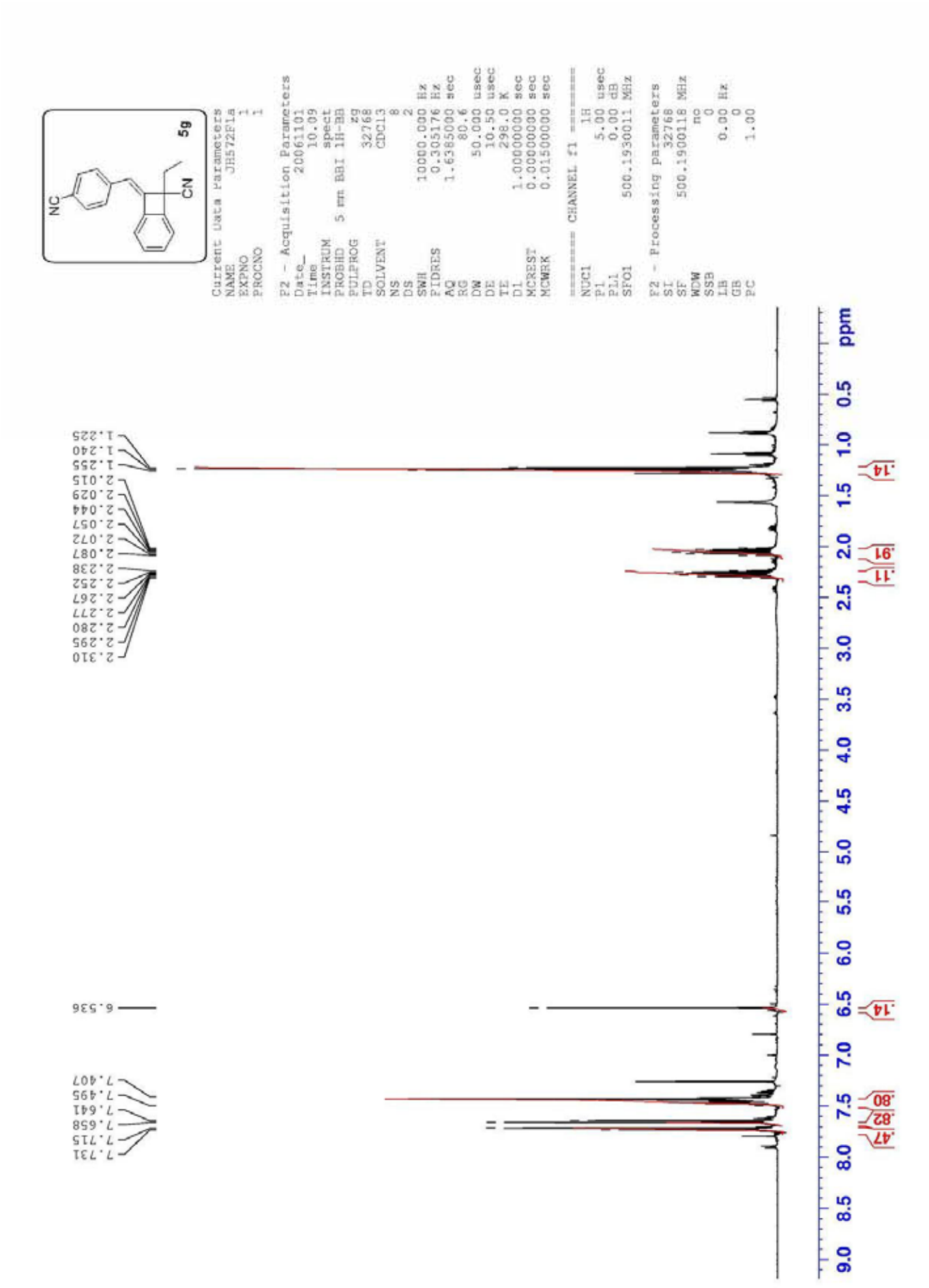

MeO
OMe
CN
5d
Current Data Parameters
NAME JH524f2f3b
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060917
Time 5.26
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1000
DS 4
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110004 sec
RG 18390.4
DW 27.800 usec
DE 6.00 usec
TE 299.0 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
NUC1 13C
P1 9.10 usec
PL1 0.00 dB
SFO1 75.4853543 MHz
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.00 dB
PL13 23.00 dB
SFO2 300.1712007 MHz
F2 - Processing parameters
SI 32768
SF 75.4778070 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
10.443
30.042
51.039
55.983
56.049
76.589
77.012
77.216
77.435
110.947
111.231
120.887
121.155
121.409
121.906
122.130
128.633
129.760
129.938
137.007
142.707
145.134
149.016
149.121
ppm

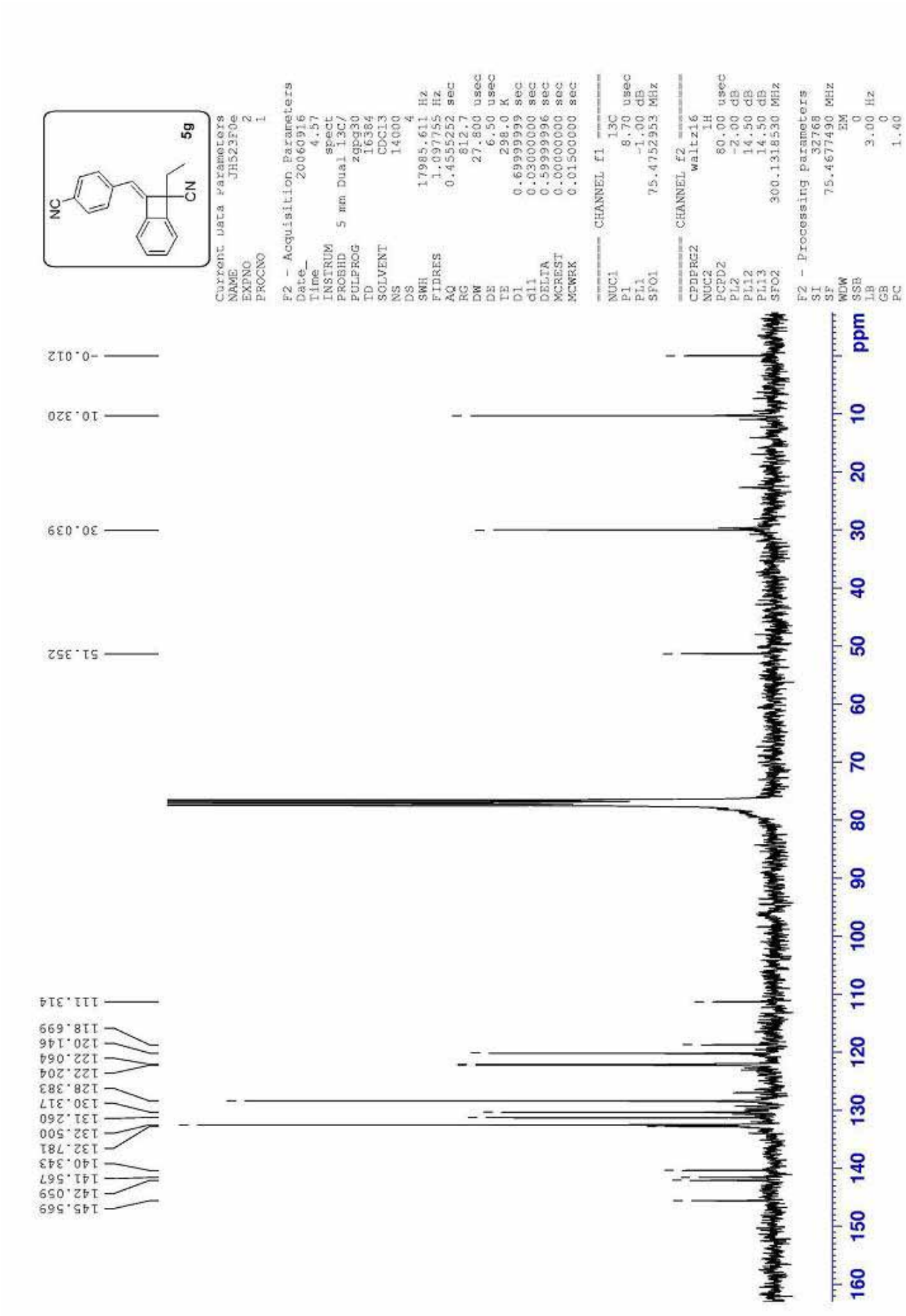

5e
OMe
MeO
MeO
CN
Current Data Parameters
NAME JR585P2a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061130
Time 15.39
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 1.6385000 sec
RG 57
DW 50.000 usec
DE 10.50 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
SFO1 500.1930011 MHz
F2 - Processing parameters
SI 32768
SF 500.1900118 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.385
7.359
7.357
7.346
6.796
6.478
3.931
3.896
2.292
2.278
2.264
2.249
2.235
2.221
2.077
2.062
2.047
2.034
2.019
2.004
1.255
1.240
1.225
ppm
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
.81
.35
.66
.72
.41
.00
.10
.05
.51

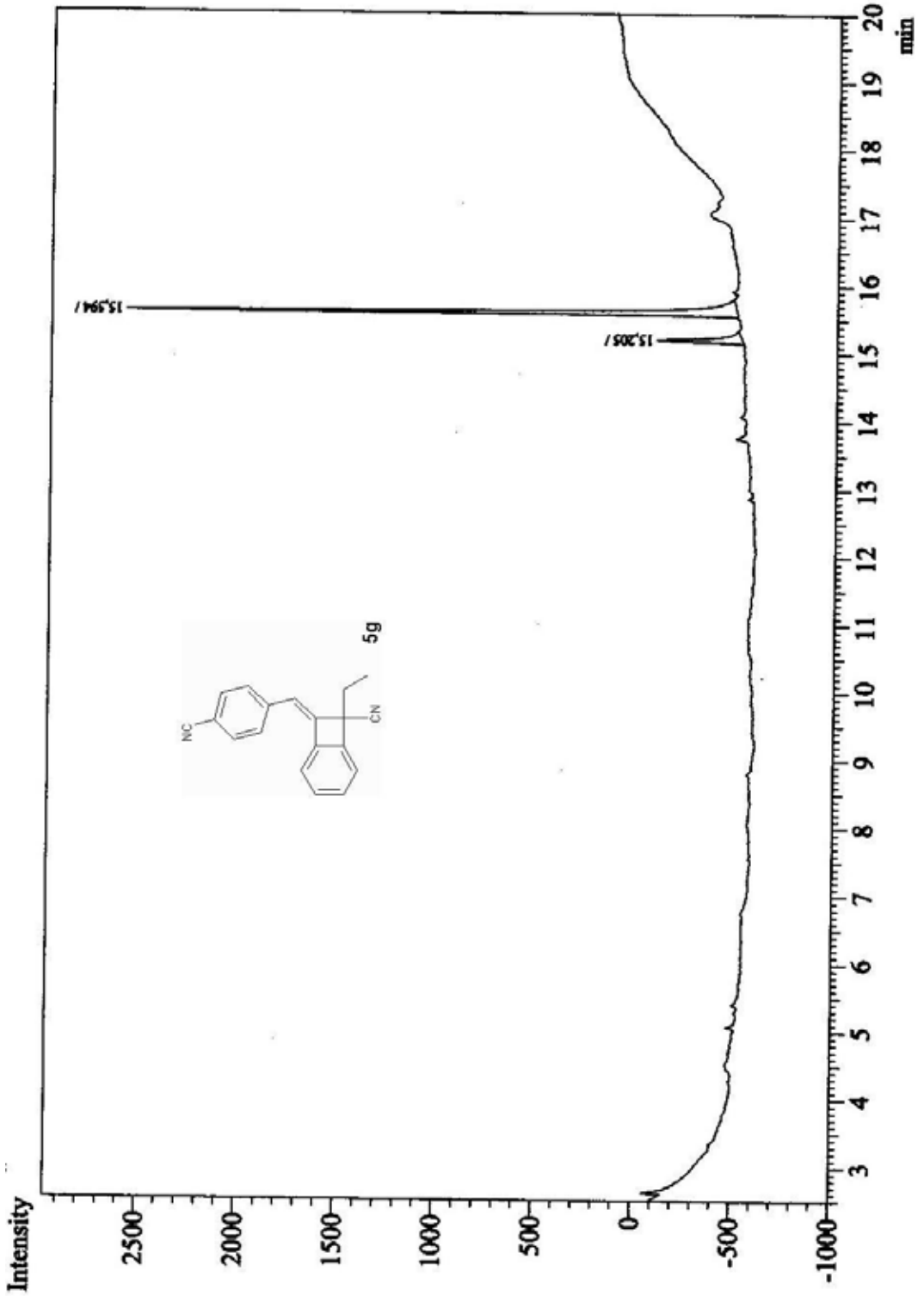

5e
OMe
MeO
MeO
CN
Current Data Parameters
NAME JH585F2b
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061203
Time 7.30
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1000
DS 4
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110004 sec
RG 18390.4
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
NUC1 13C
P1 9.10 usec
PL1 0.00 dB
SFO1 75.4853543 MHz
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.00 dB
PL13 23.00 dB
SFO2 300.1712007 MHz
F2 - Processing parameters
SI 32768
SF 75.4778070 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
10.451
30.057
51.119
56.315
60.997
105.369
120.718
121.476
122.033
122.249
130.025
130.074
131.280
138.089
138.256
142.493
145.299
153.496
ppm

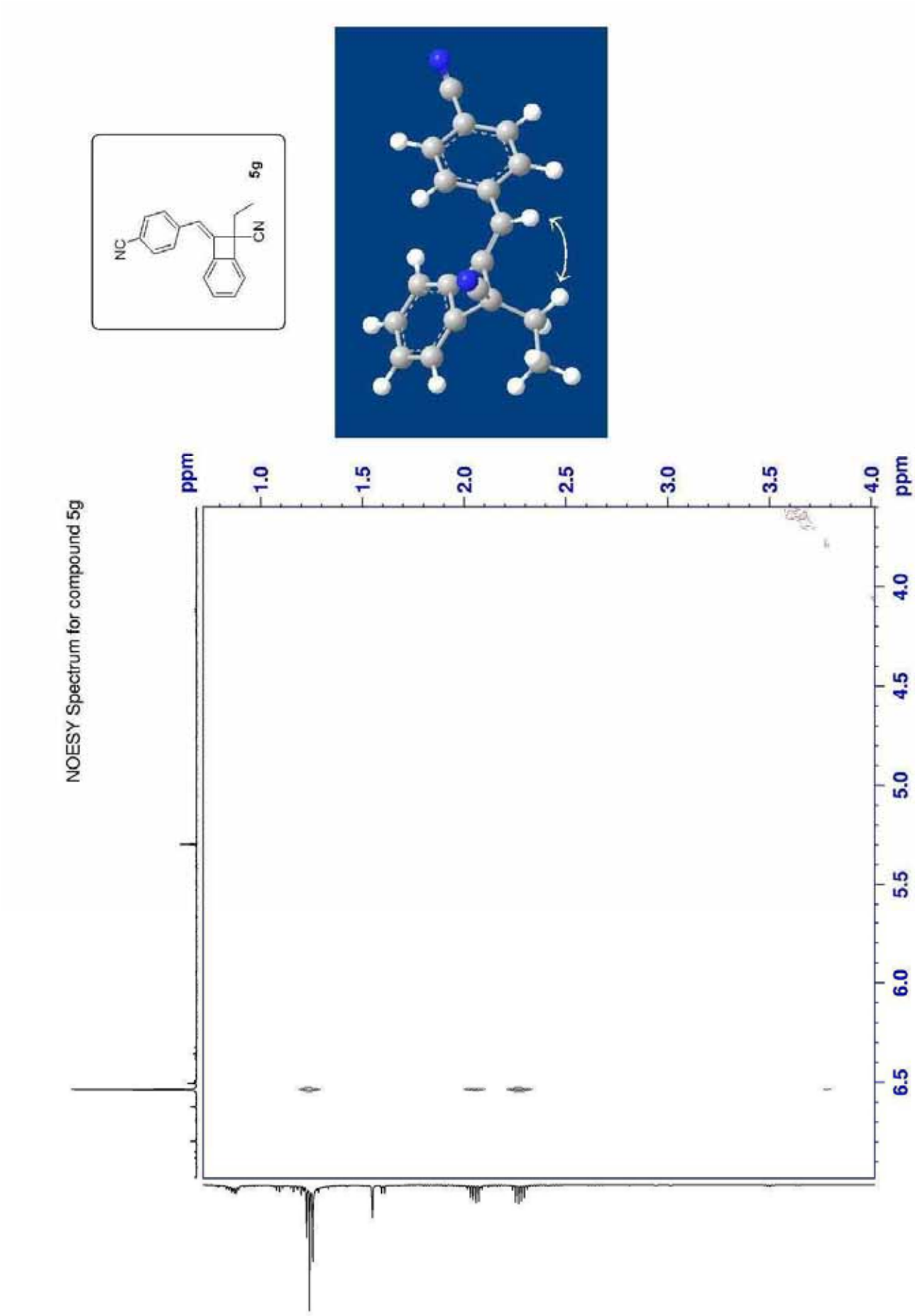

5f
CN
Current Data Parameters
NAME JB565F1a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061025
Time 12.54
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 456.1
DW 81.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.30 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.045
8.017
7.662
7.635
7.552
7.491
7.462
7.384
7.260
6.572
2.626
2.341
2.315
2.291
2.269
2.246
2.220
2.117
2.093
2.067
2.045
2.022
1.996
1.269
1.244
1.220
1.93
1.95
0.91
3.13
0.79
3.00
1.06
0.94
3.32
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

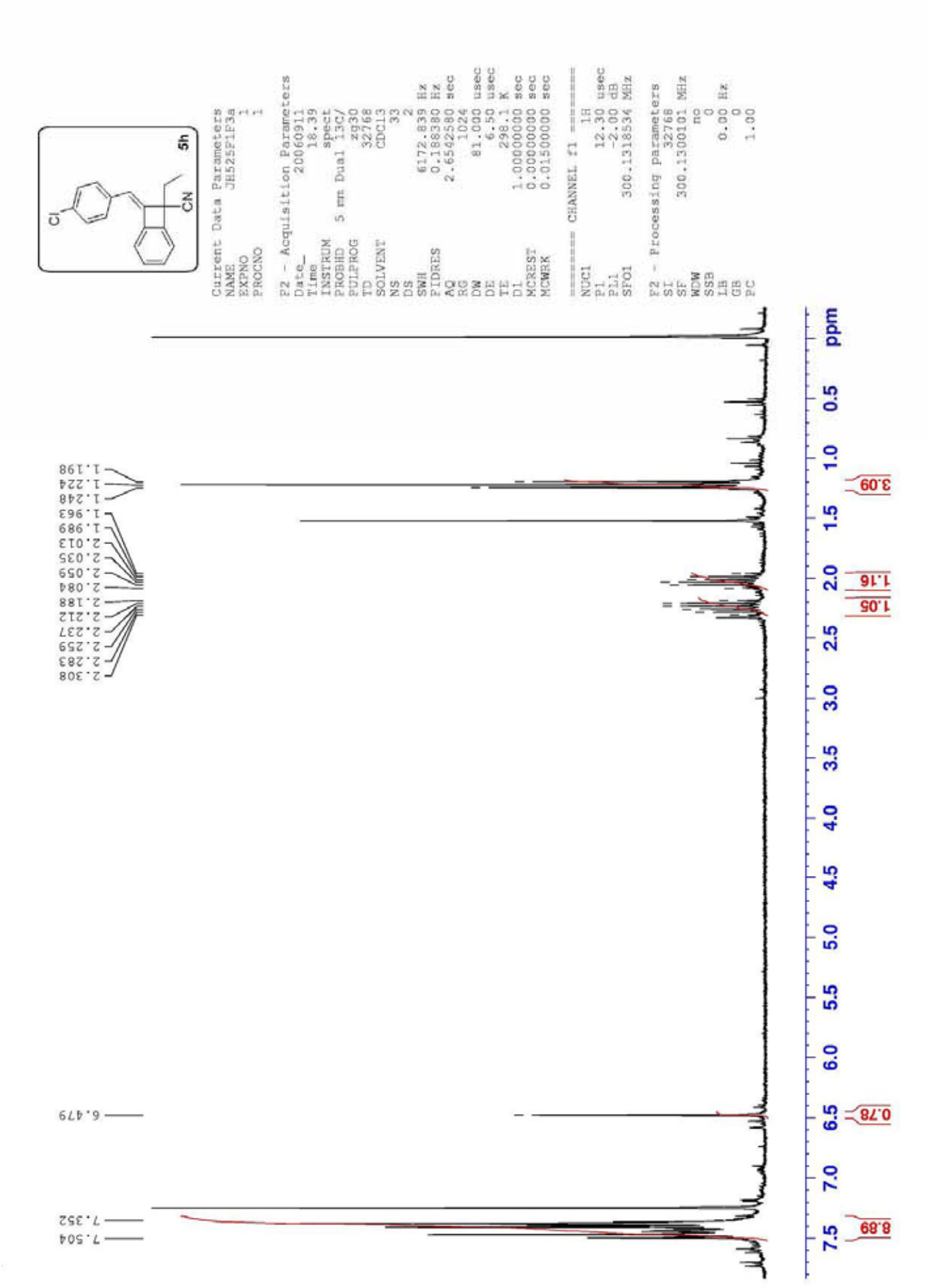

5f
CN
Current Data Parameters
NAME JH530F3c
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060918
Time 11.43
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1000
DS 4
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110004 sec
RG 18390.4
DW 27.800 usec
DE 6.00 usec
TE 299.2 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1 13C
NUC1 13C
P1 9.10 usec
PL1 0.00 dB
SFO1 75.4853543 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.00 dB
PL13 23.00 dB
SFO2 300.1712007 MHz
F2 - Processing parameters
SI 32768
SF 75.4778070 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
197.4
145.4
142.0
141.1
140.4
136.2
130.8
130.1
128.7
128.0
122.0
120.9
120.4
77.43
77.00
76.58
51.28
30.04
26.63
10.36
ppm

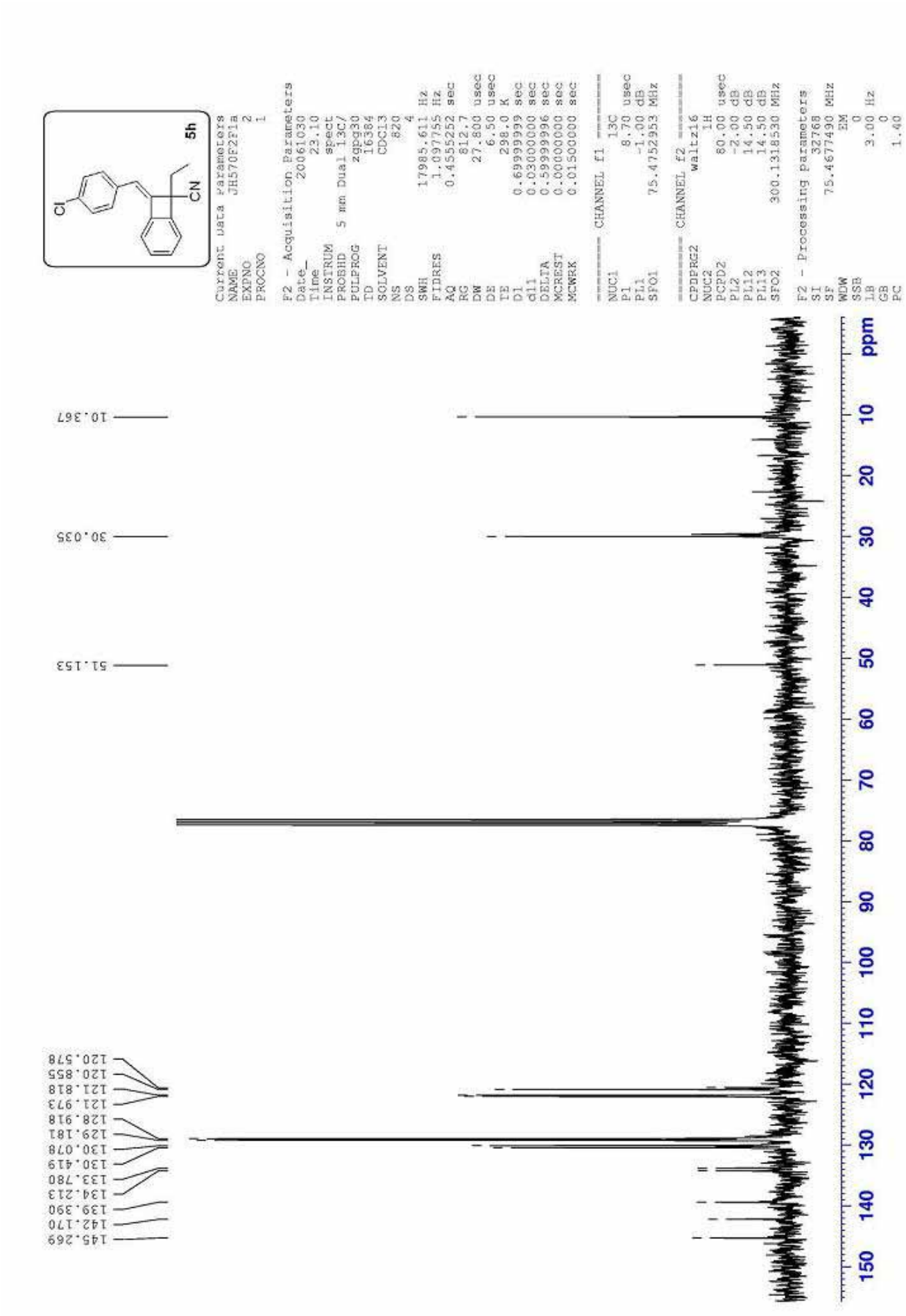

5g
NC
CN
Current Data Parameters
NAME JR572F1a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061101
Time 10.09
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 1.6385000 sec
RG 80.6
DW 50.000 usec
DE 10.50 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
SFO1 500.1930011 MHz
F2 - Processing parameters
SI 32768
SF 500.1900118 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1.225
1.240
1.255
2.015
2.029
2.044
2.057
2.072
2.087
2.238
2.252
2.267
2.277
2.280
2.295
2.310
6.536
7.407
7.495
7.641
7.658
7.715
7.731
14.
9.1
11.
14.
80.
82.
47.
ppm

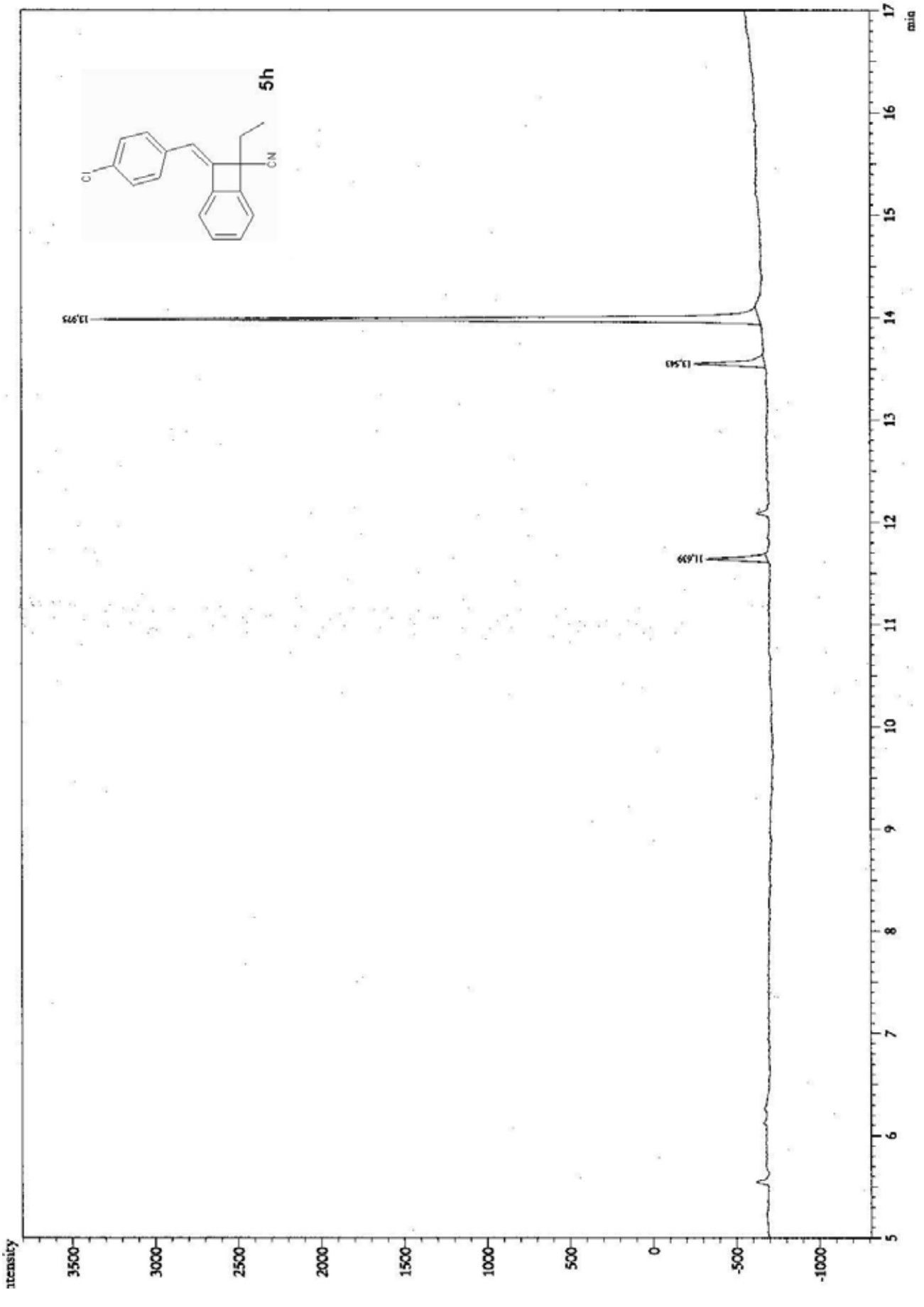

5g
NC
CN
Current Data Parameters
NAME JH523F0e
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060916
Time 4.57
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 14000
DS 4
SWH 17985.611 Hz
FIDRES 1.097755 Hz
AQ 0.4555252 sec
RG 812.7
DW 27.800 usec
DE 6.50 usec
TE 298.0 K
D1 0.69999999 sec
d11 0.03000000 sec
DELTA 0.59999996 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 8.70 usec
PL1 -1.00 dB
SFO1 75.4752953 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.50 dB
PL13 14.50 dB
SFO2 300.1318530 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
-0.012
10.320
30.039
51.352
111.314
118.699
120.146
122.064
122.204
128.383
130.317
131.260
132.500
132.781
140.343
141.567
142.059
145.569
ppm

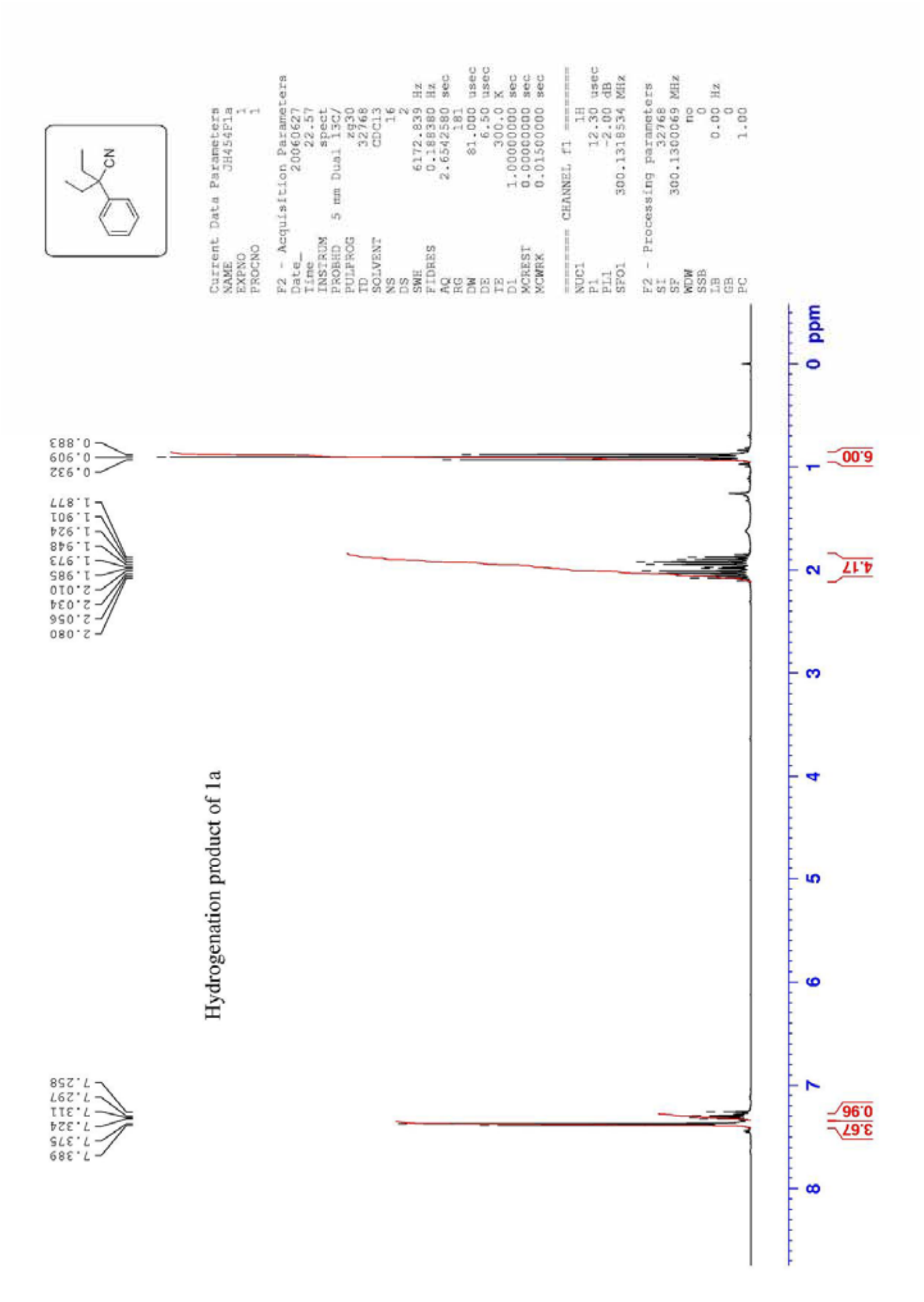

Intensity
2500
2000
1500
1000
500
0
-500
-1000
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
min
15.394
15.205
5g
NC
CN


5g
NC
CN
NOESY Spectrum for compound 5g
ppm
1.0
1.5
2.0
2.5
3.0
3.5
4.0
ppm
4.0
4.5
5.0
5.5
6.0
6.5


5h
Cl
CN
Current Data Parameters
NAME JH525P1P3a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060911
Time 18.39
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 33
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 1024
DW 81.000 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 1H
P1 12.30 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300101 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1.198
1.224
1.248
1.963
1.989
2.013
2.035
2.059
2.084
2.188
2.212
2.237
2.259
2.283
2.308
6.479
7.352
7.504
ppm
3.09
1.16
1.05
0.78
8.89


5h
Cl
CN
Current data parameters
NAME JH570F2Fla
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061030
Time 23.10
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 820
DS 4
SWH 17985.611 Hz
FIDRES 1.097755 Hz
AQ 0.4555252 sec
RG 812.7
DW 27.800 usec
DE 6.50 usec
TE 298.0 K
D1 0.69999999 sec
d11 0.03000000 sec
DELTA 0.59999996 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 8.70 usec
PL1 -1.00 dB
SFO1 75.4752953 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.50 dB
PL13 14.50 dB
SFO2 300.1318530 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
10.367
30.035
51.153
120.578
120.855
121.818
121.973
128.918
129.181
130.078
130.419
133.780
134.213
139.390
142.170
145.269
ppm


5h
Cl
CN
13,975
13,563
11,639
Intensity
3500
3000
2500
2000
1500
1000
500
0
-500
-1000
5
6
7
8
9
10
11
12
13
14
15
16
17
min


Hydrogenation product of 1a
CN
Current Data Parameters
NAME JH454P1a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060627
Time 22.57
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 181
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 1H
P1 12.30 usec
PL1 -2.00 dB
SFO1 300.1318534 MHz
F2 - Processing parameters
SI 32768
SF 300.1300069 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
0.883
0.909
0.932
1.877
1.901
1.924
1.948
1.973
1.985
2.010
2.034
2.056
2.080
7.258
7.297
7.311
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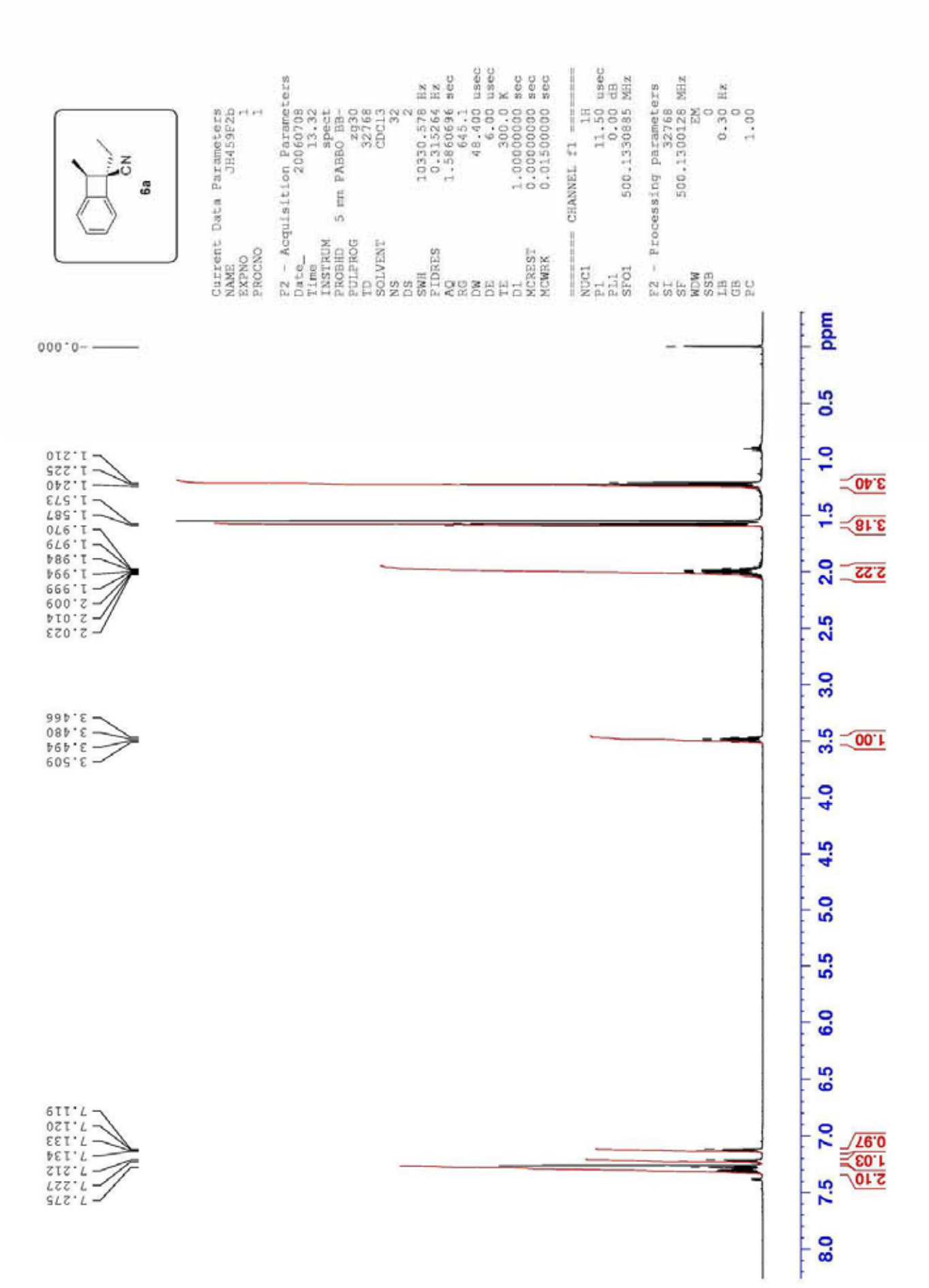
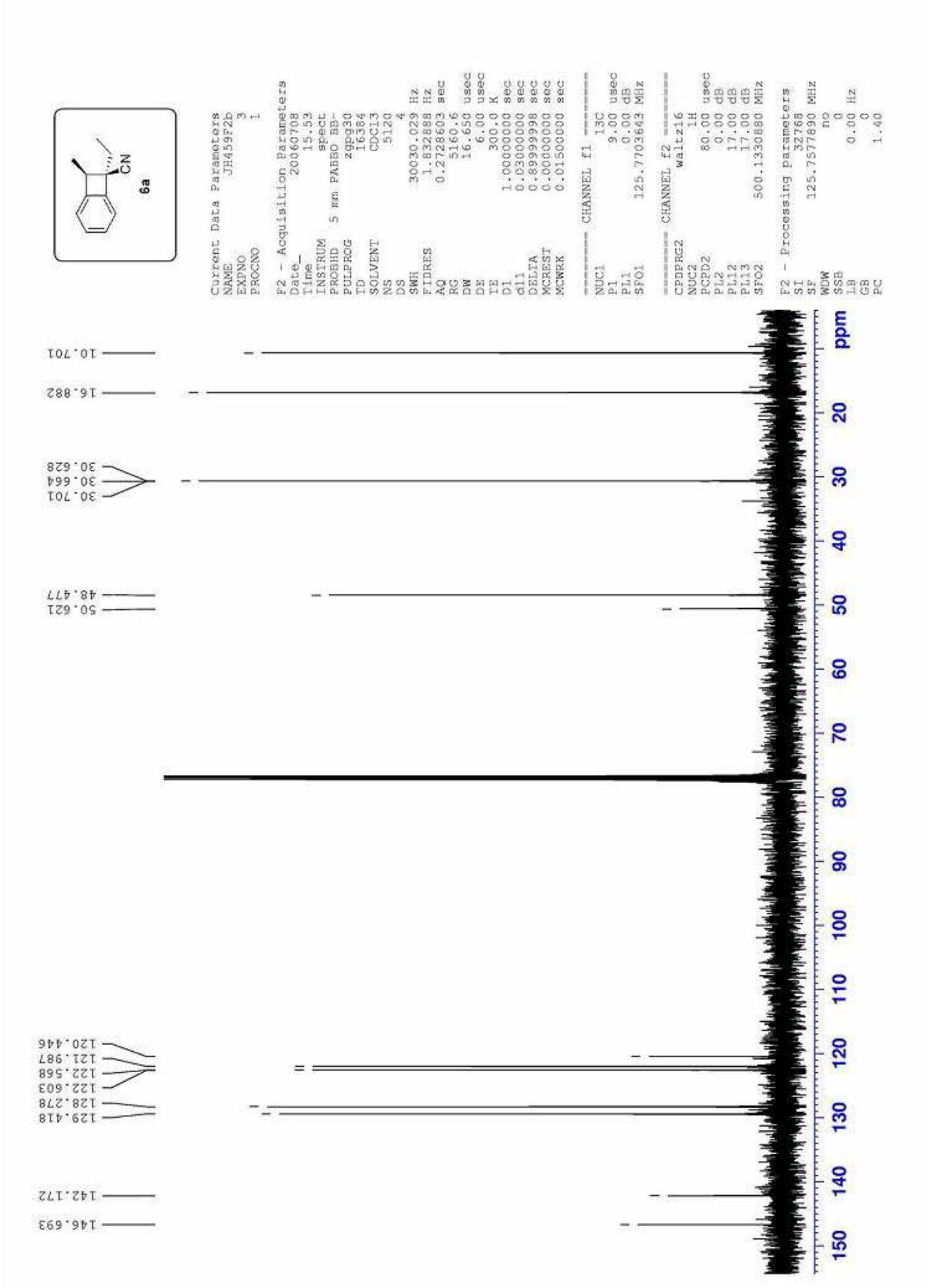
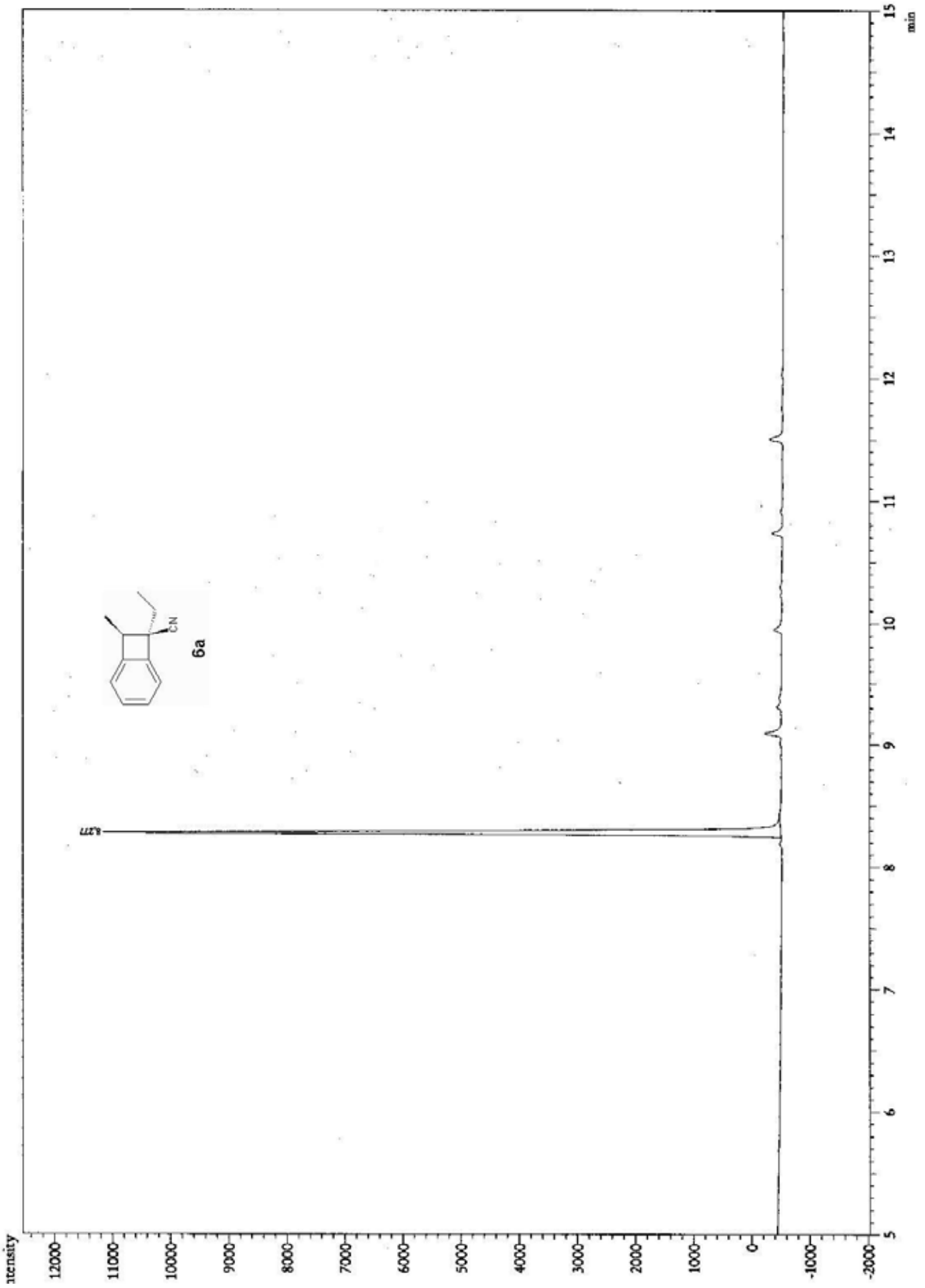
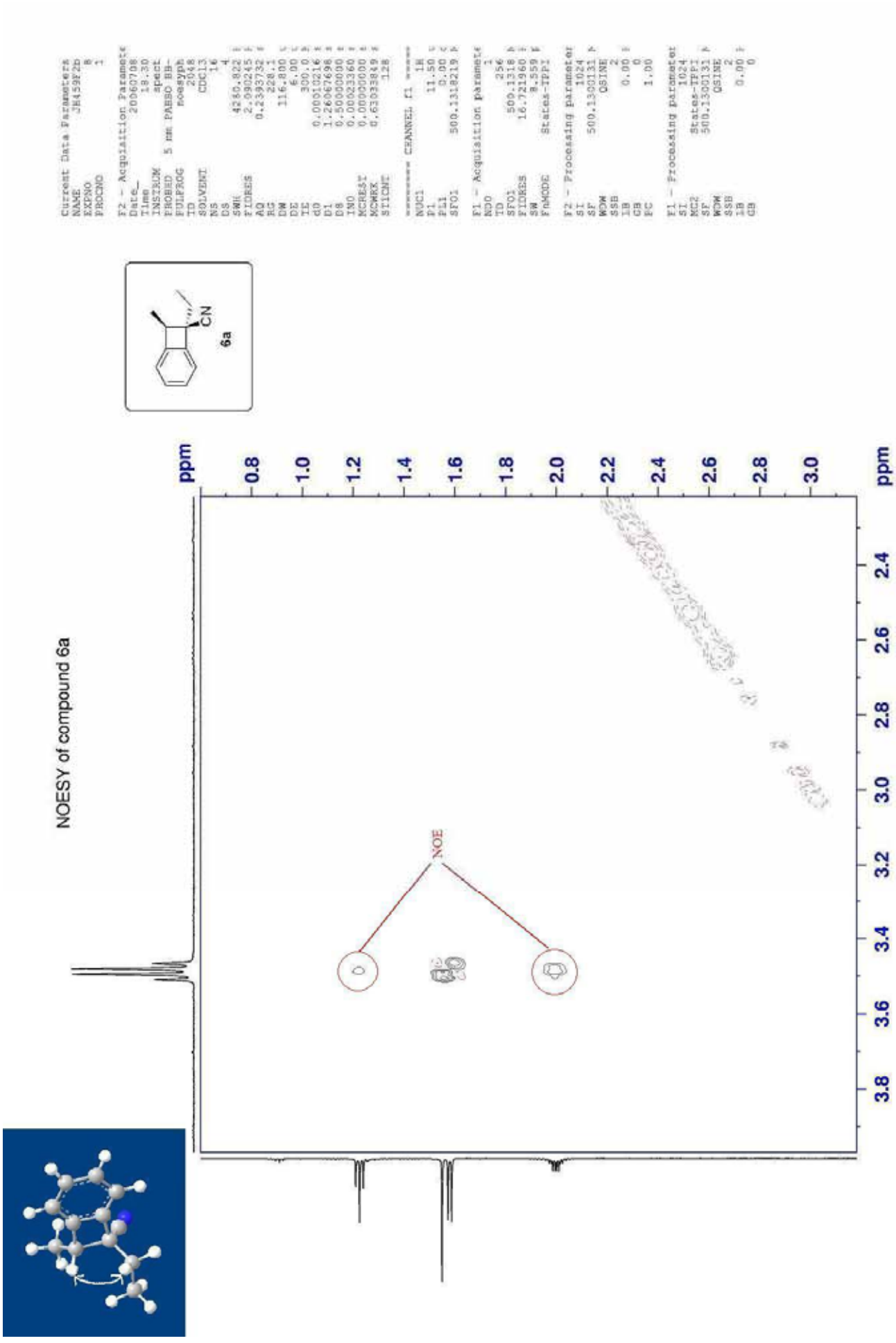
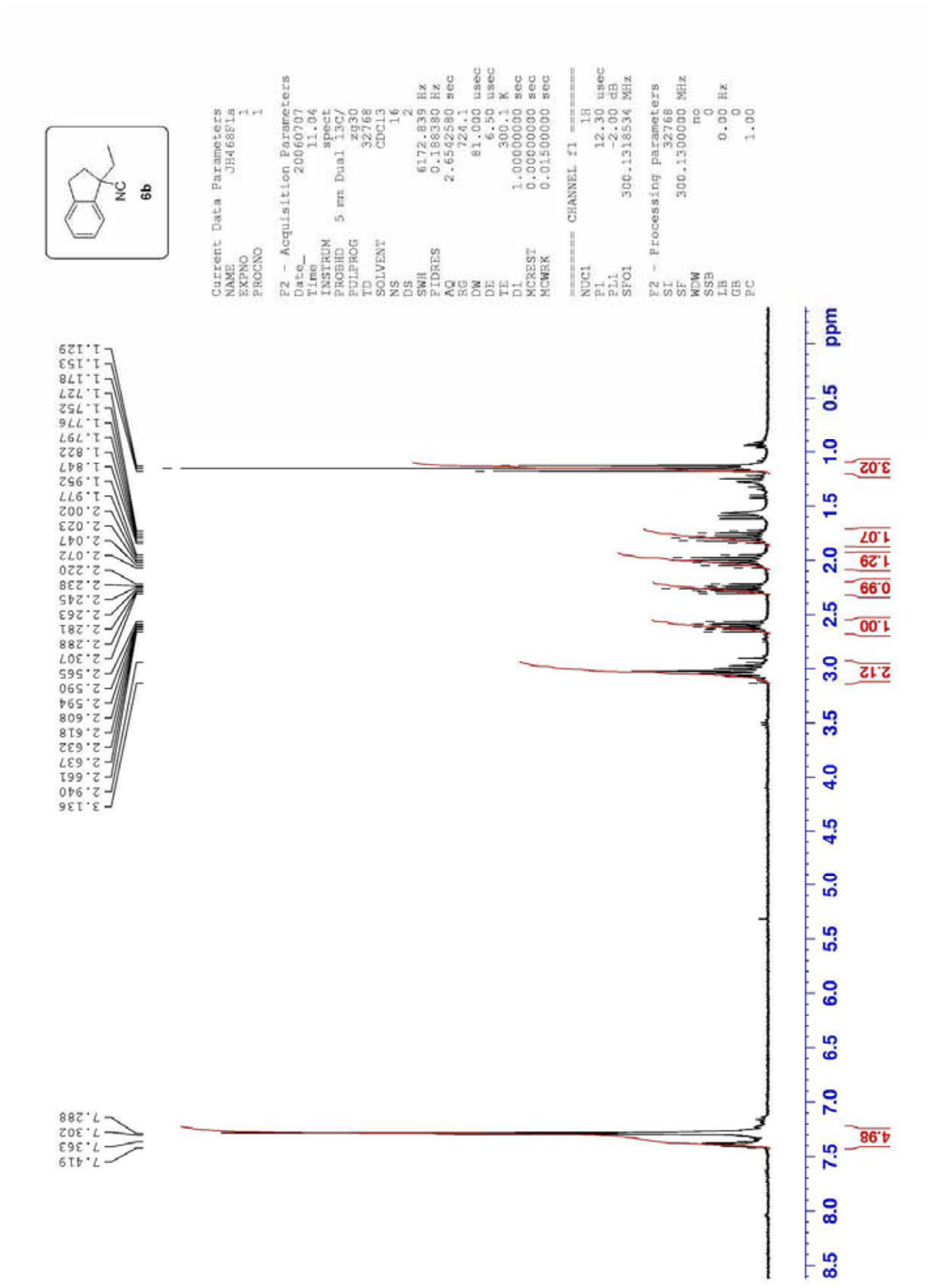
7.389
6.00
4.17
3.67
0.96
ppm

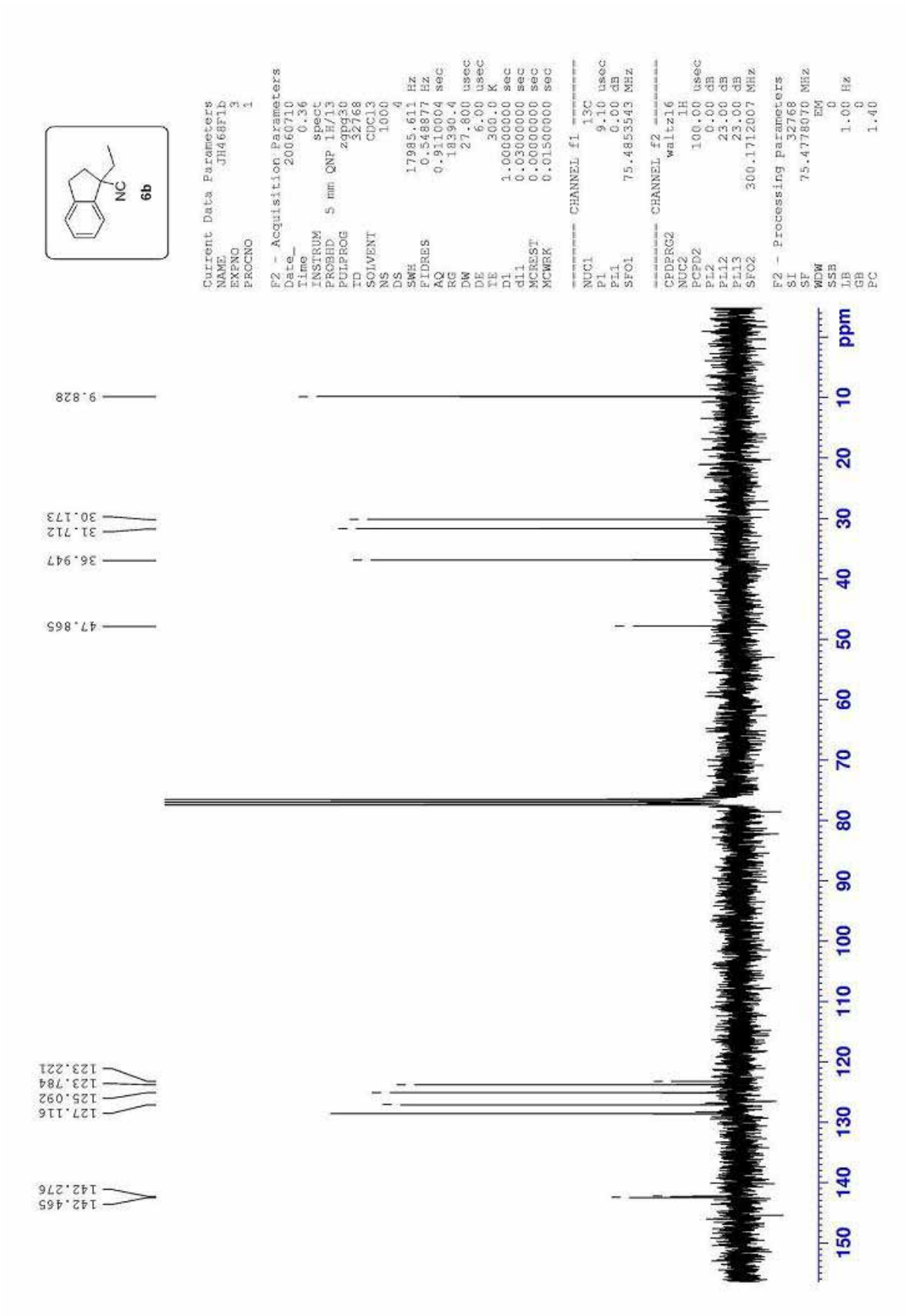

6a
CN
Current Data Parameters
NAME JH459P2b
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060708
Time 13.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.5860696 sec
RG 645.1
DW 48.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 0.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300128 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
1.210
1.225
1.240
1.573
1.587
1.970
1.979
1.984
1.994
1.999
2.009
2.014
2.023
3.466
3.480
3.494
3.509
7.119
7.120
7.133
7.134
7.212
7.227
7.275
3.40
3.18
2.22
1.00
0.97
1.03
2.10
ppm

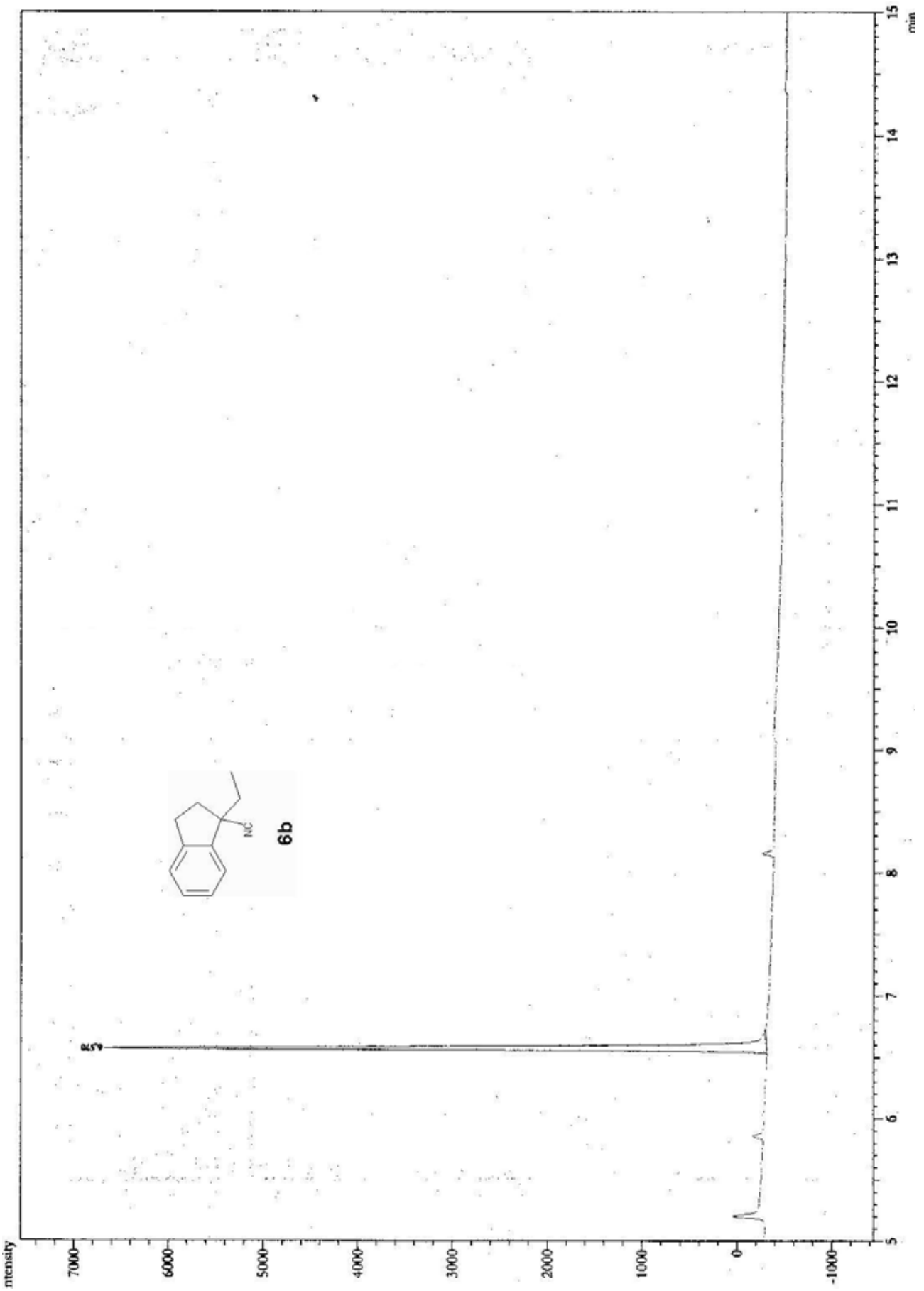

CN
6a
Current Data Parameters
NAME JH459F2b
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060708
Time 15.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 5120
DS 4
SWH 30030.029 Hz
FIDRES 1.832888 Hz
AQ 0.2728603 sec
RG 5160.6
DW 16.650 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 9.00 usec
PL1 0.00 dB
SFO1 125.7703643 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 500.1330880 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
10.701
16.882
30.628
30.664
30.701
48.477
50.621
120.446
121.987
122.568
122.603
128.278
129.418
142.172
146.693
ppm

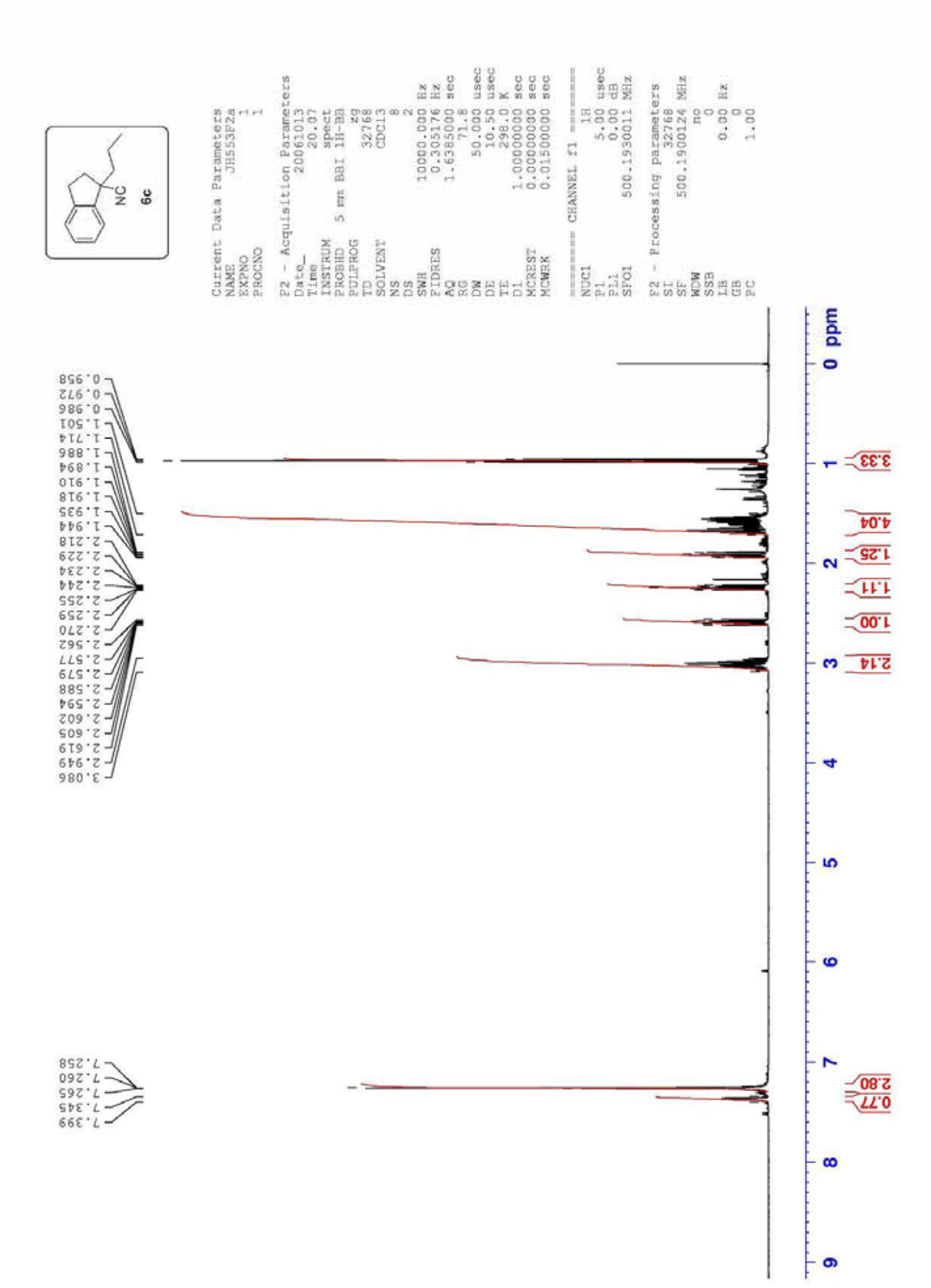

CN
6a
Intensity
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
5
6
7
8
9
10
11
12
13
14
15
min

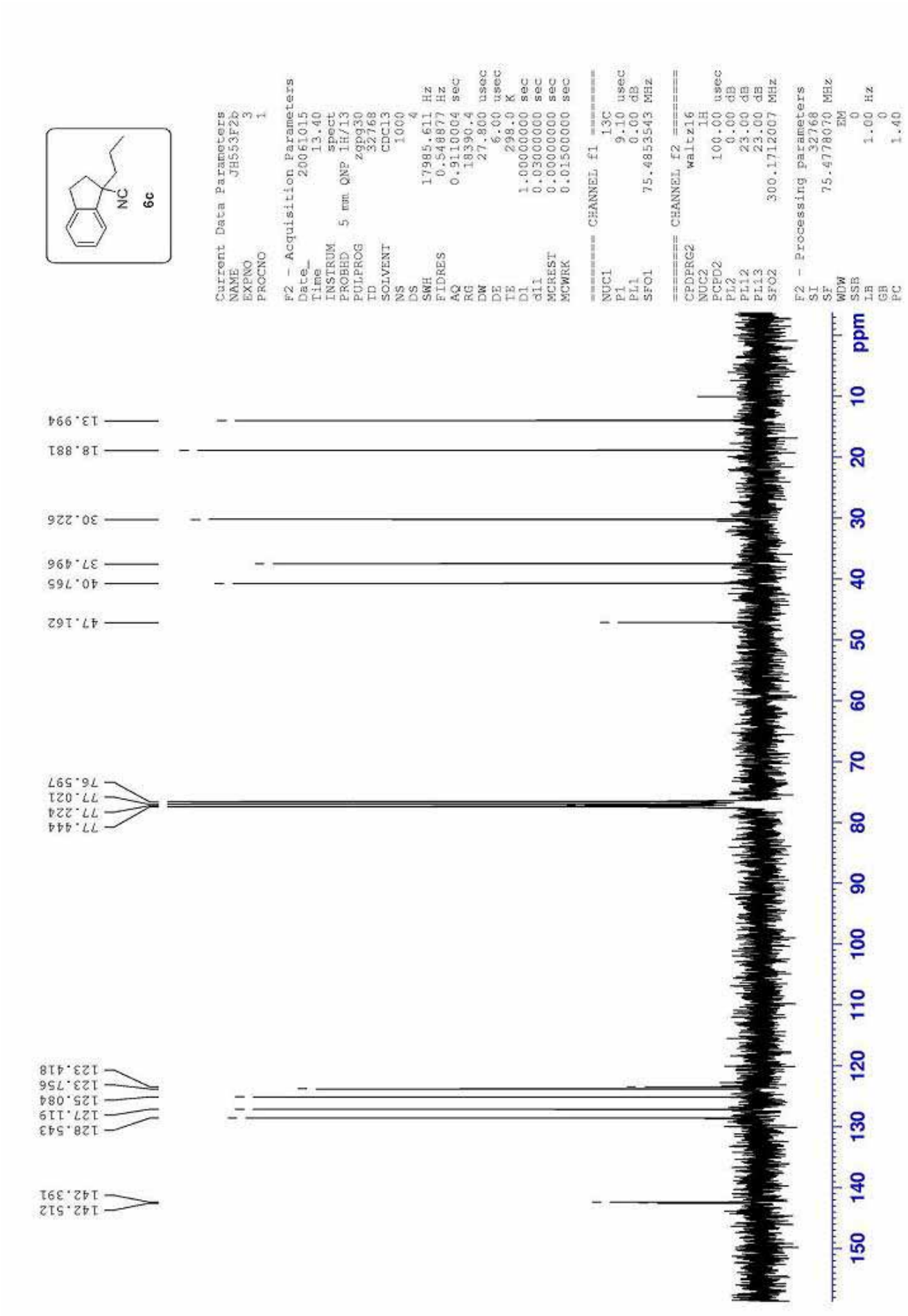

NOESY of compound 6a
ppm
0.8
1.0
1.2
1.4
1.6
1.8
2.0
2.2
2.4
2.6
2.8
3.0
3.2
3.4
3.6
3.8
NOE
CN
6a

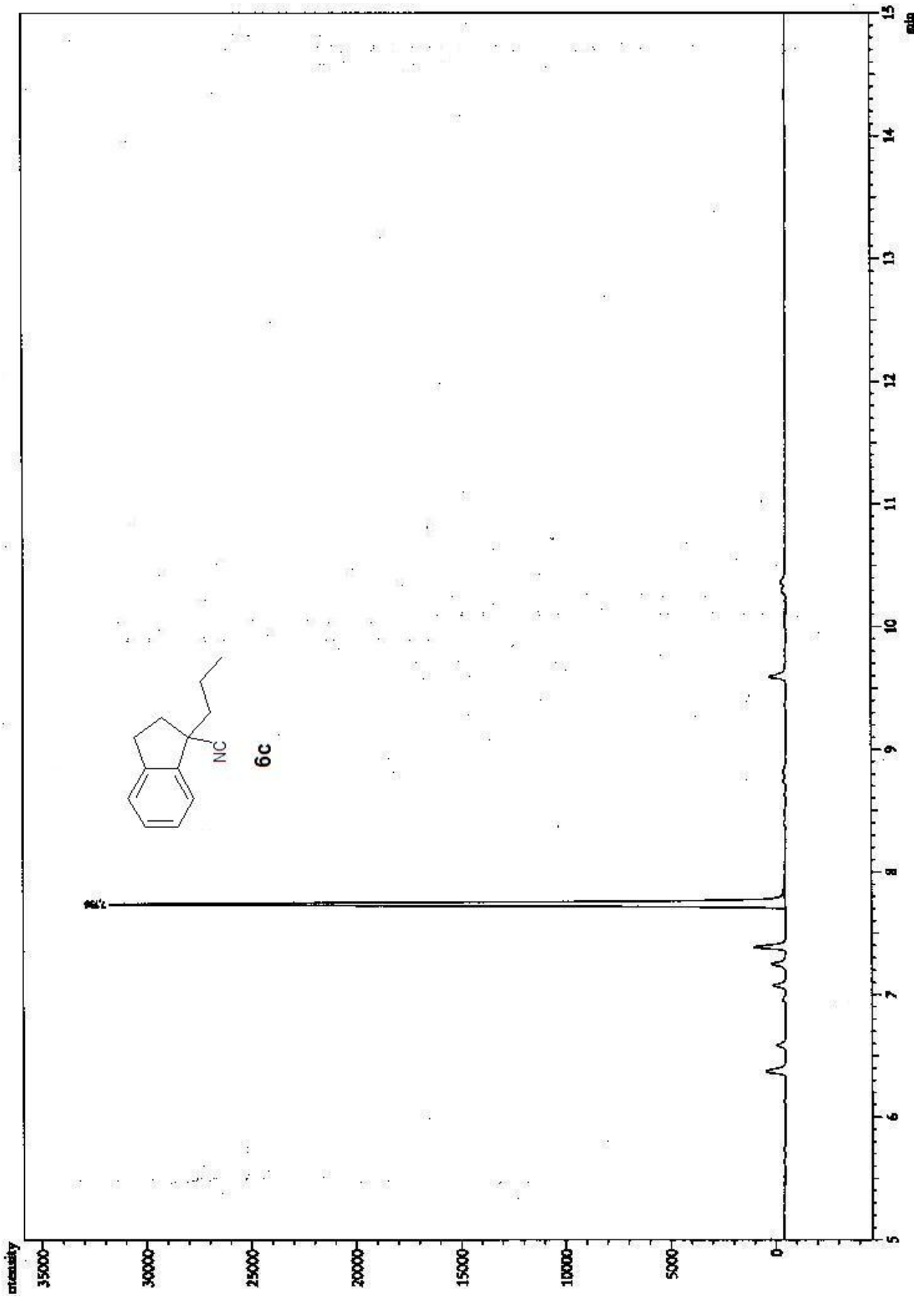

6b
Current Data Parameters
NAME JH468F1a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060707
Time 11.04
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 724.1
DW 81.000 usec
DE 6.50 usec
TE 300.1 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 1H
P1 12.30 usec
PL1 -2.00 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
ppm
3.02
1.07
1.29
0.99
1.00
2.12
4.98

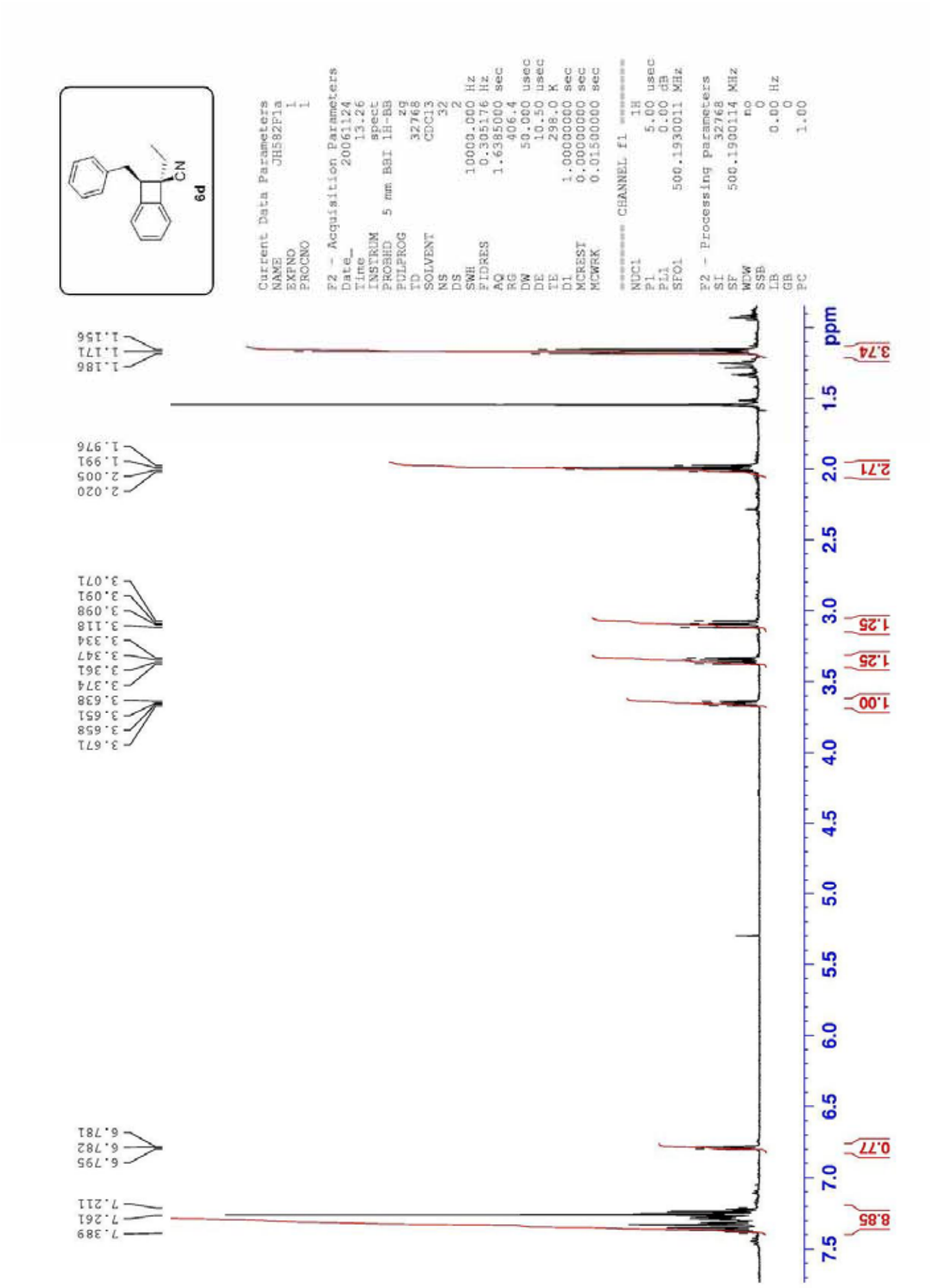

NC
6b
Current Data Parameters
NAME JH468f1b
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060710
Time 0.36
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1000
DS 4
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110004 sec
RG 18390.4
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 9.10 usec
PL1 0.00 dB
SFO1 75.4853543 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.00 dB
PL13 23.00 dB
SFO2 300.1712007 MHz
F2 - Processing parameters
SI 32768
SF 75.4778070 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
9.828
30.173
31.712
36.947
47.865
123.221
123.784
125.092
127.116
142.465
142.276
ppm

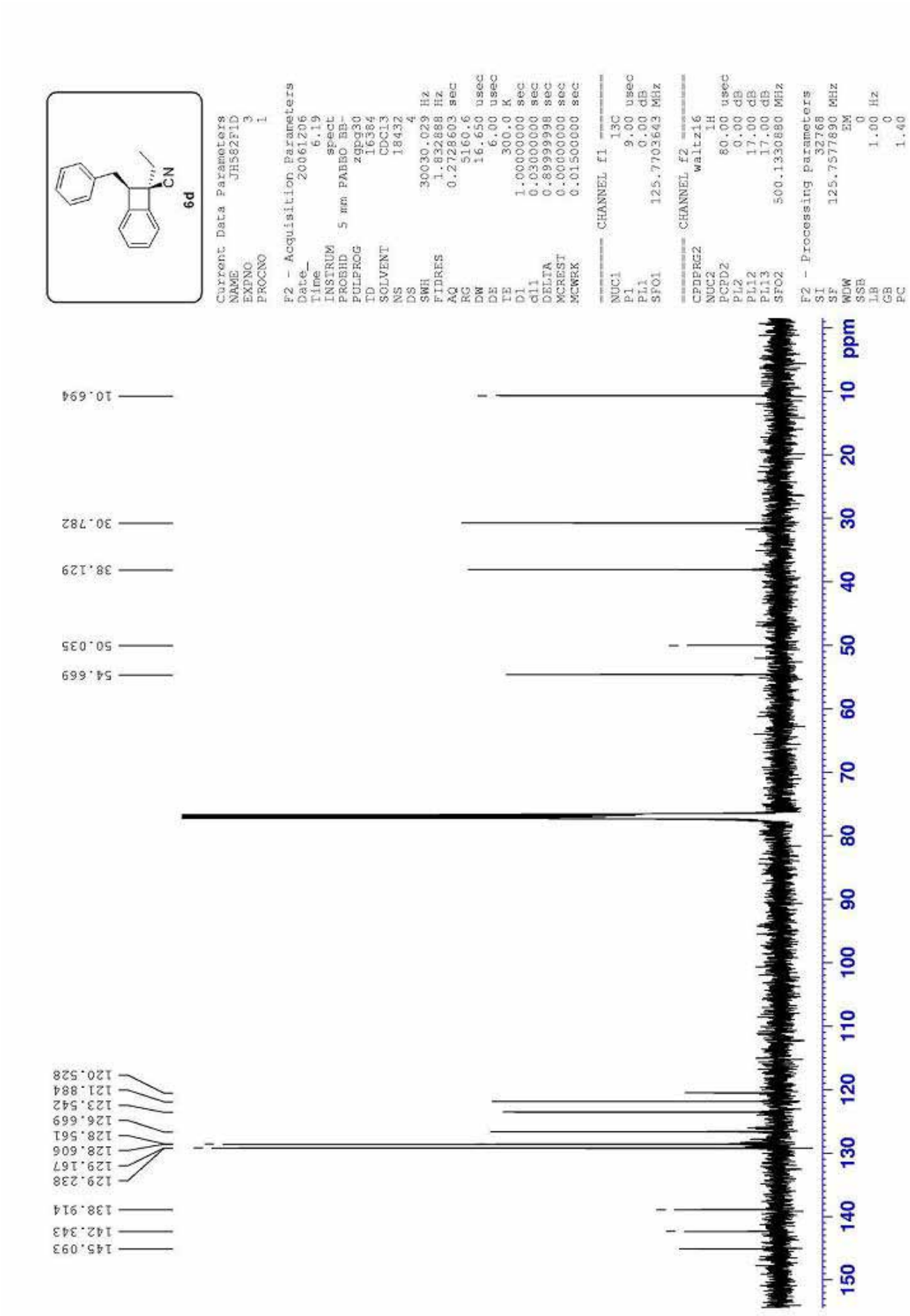

6.578
NC
6b
Intensity
7000
6000
5000
4000
3000
2000
1000
0
-1000
5
6
7
8
9
10
11
12
13
14
15
min

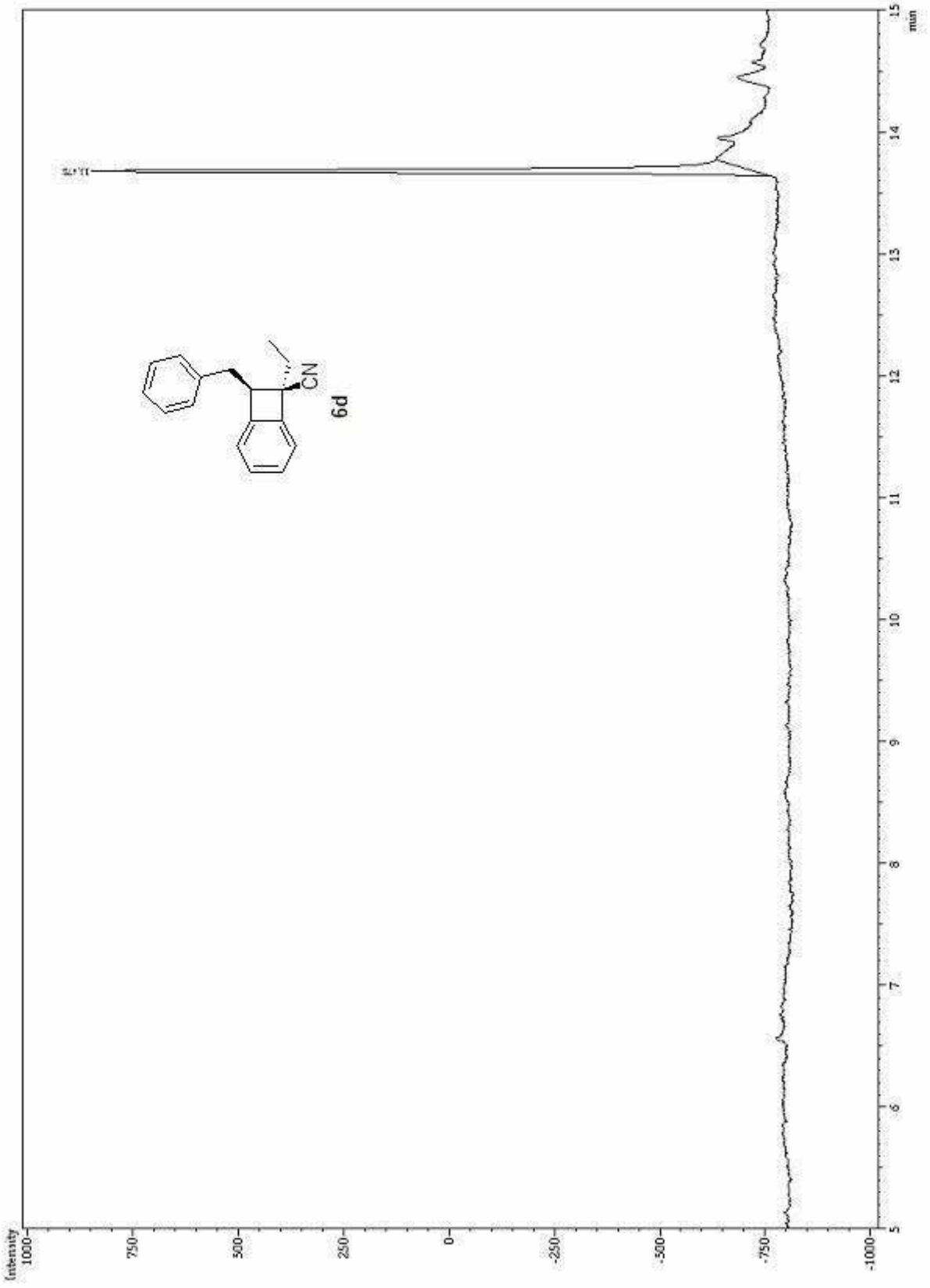

6c
Current Data Parameters
NAME JR553P2a
EXPNO 1
PROCNO 1
P2 - Acquisition Parameters
Date_ 20061013
Time 20.07
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 1.6385000 sec
RG 71.8
DW 50.000 usec
DE 10.50 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
SFO1 500.1930011 MHz
F2 - Processing parameters
SI 32768
SF 500.1900124 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
0.958
0.972
0.986
1.501
1.714
1.886
1.894
1.910
1.918
1.935
1.944
2.218
2.229
2.234
2.244
2.255
2.259
2.270
2.562
2.577
2.579
2.588
2.594
2.602
2.605
2.619
2.949
3.086
7.258
7.260
7.265
7.345
7.399
3.33
4.04
1.25
1.11
1.00
2.14
2.80
0.77
0 ppm

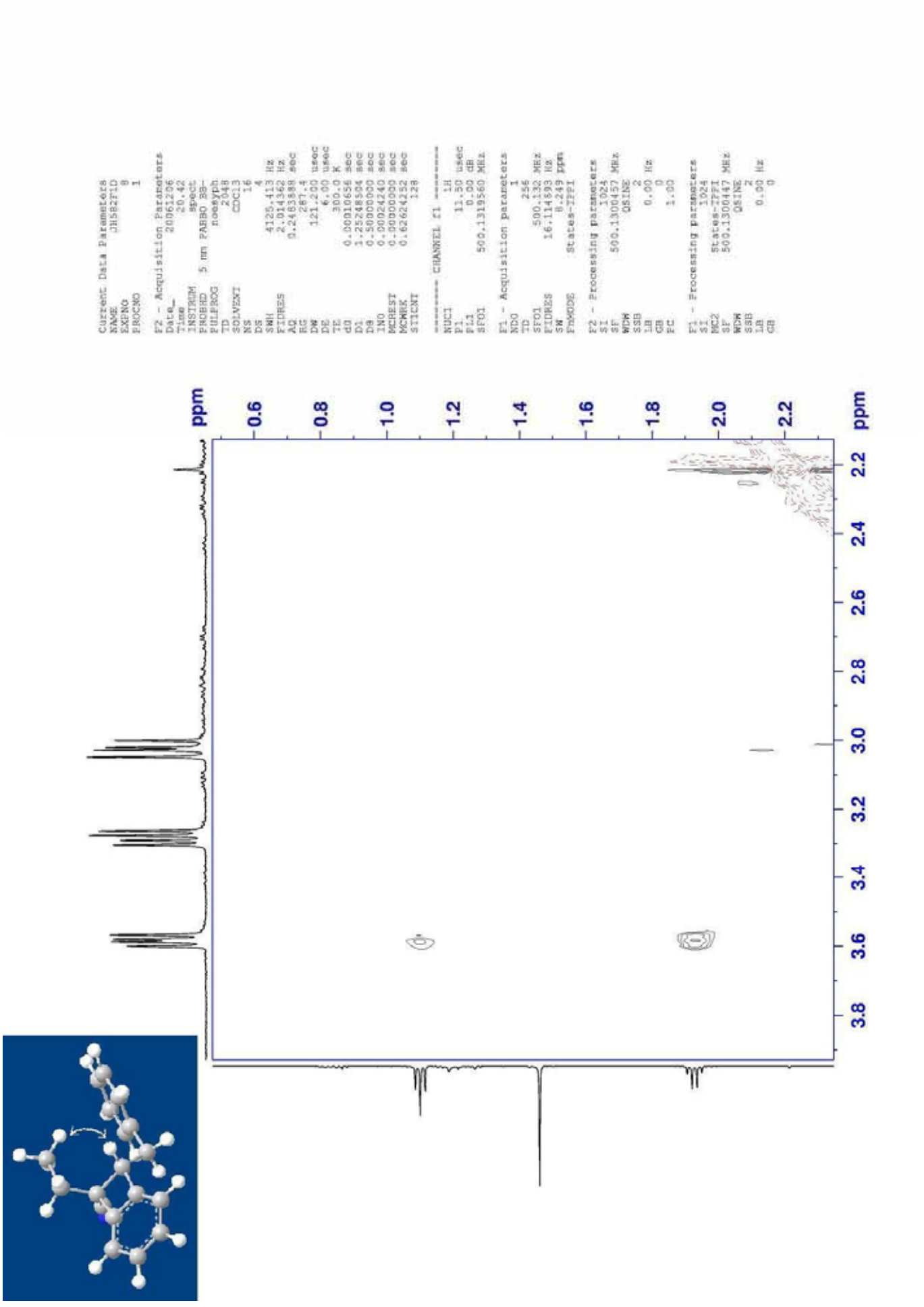

NC
6c
Current Data Parameters
NAME JH553F2b
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061015
Time 13.40
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 1000
DS 4
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110004 sec
RG 18390.4
DW 27.800 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 9.10 usec
PL1 0.00 dB
SFO1 75.4853543 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.00 dB
PL13 23.00 dB
SFO2 300.1712007 MHz
F2 - Processing parameters
SI 32768
SF 75.4778070 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
13.994
18.881
30.226
37.496
40.765
47.162
76.597
77.021
77.224
77.444
123.418
123.756
125.084
127.119
128.543
142.391
142.512
ppm


intensity
35000
30000
25000
20000
15000
10000
5000
0
5
6
7
8
9
10
11
12
13
14
15
min
NC
6c


6d
CN
Current Data Parameters
NAME JH582Pla
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061124
Time 13.26
INSTRUM spect
PROBHD 5 mm BBI 1H-BB
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 1.6385000 sec
RG 406.4
DW 50.000 usec
DE 10.50 usec
TE 298.0 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
SFO1 500.1930011 MHz
F2 - Processing parameters
SI 32768
SF 500.1900114 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1.186
1.171
1.156
1.976
1.991
2.005
2.020
3.071
3.091
3.098
3.118
3.334
3.347
3.361
3.374
3.638
3.651
3.658
3.671
6.781
6.782
6.795
7.211
7.261
7.389
ppm
3.74
2.71
1.25
1.25
1.00
0.77
8.85


6d
CN
Current Data Parameters
NAME JH582F1D
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061206
Time 6.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 18432
DS 4
SWH 30030.029 Hz
FIDRES 1.832888 Hz
AQ 0.2728603 sec
RG 5160.6
DW 16.650 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 9.00 usec
PL1 0.00 dB
SFO1 125.7703643 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 500.1330880 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
10.694
30.782
38.129
50.035
54.669
120.528
121.884
123.542
126.669
128.561
128.606
129.167
129.238
138.914
142.343
145.093
ppm


CN
6d
Intensity
1000
750
500
250
0
-250
-500
-750
-1000
5
6
7
8
9
10
11
12
13
14
15
min


Current Data Parameters
NAME JH582FID
EXPNO 8
PROCNO 1
F2 - Acquisition Parameters
Date_ 20061206
Time 20.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG noesyph
TD 2048
SOLVENT CDCl3
NS 16
DS 4
SWH 4126.413 Hz
FIDRES 2.014362 Hz
AQ 0.2483388 sec
RG 287.4
DW 121.200 usec
DE 6.00 usec
TE 300.0 K
d0 0.00010656 sec
D1 1.25248504 sec
D8 0.50000000 sec
IN0 0.00024240 sec
MCREST 0.00000000 sec
MCWRK 0.62624252 sec
STSCNT 128
======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 0.00 dB
SFO1 500.1319560 MHz
F1 - Acquisition parameters
NDO 1
TD 256
SFO1 500.132 MHz
FIDRES 16.114893 Hz
SW 8.249 ppm
FnMODE States-TPPI
F2 - Processing parameters
SI 1024
SF 500.1300457 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.00
F1 - Processing parameters
SI 1024
MC2 States-TPPI
SF 500.1300447 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
ppm
0.6
0.8
1.0
1.2
1.4
1.6
1.8
2.0
2.2
ppm
2.2
2.4
2.6
2.8
3.0
3.2
3.4
3.6
3.8