Supporting Information

Selective *N*-alkylation of indoles with primary alcohols by Pt/HBEA catalyst

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Experimental Section

General: Commercially available organic and inorganic compounds (from Tokyo Chemical Industry, Wako Pure Chemical Industries, Kishida Chemical, or Mitsuwa Chemicals) were used without further purifications. Benzyl-α,α-d_2 alcohol ($BzCD_2OH$) with 99.5 atom % D was purchased from Aldrich. The GC (Shimadzu GC-14B) and GCMS (Shimadzu GCMS-QP2010) analyses were carried out with Ultra ALLOY capillary column UA+-5 (Frontier Laboratories Ltd.) using nitrogen or He as the carrier gas. ^1H and ^{13}C NMR spectra were recorded using at ambient temperature on JEOL-ECX 600 operating at 600.17 and 150.92 MHz, respectively with tetramethylsilane as an internal standard.

Catalyst Preparation: H^+-type BEA zeolite (HBEA, SiO_2/Al_2O_3 = 25±5, JRC-Z-HB25), CeO_2 (JRC-CEO-1), MgO (JRC-MGO-3), TiO_2 (JRC-TIO-4), HY (JRC-Z-HY5.5) and SiO_2-Al_2O_3 (JRC-SAL-2) were supplied by Catalysis Society of Japan. HMFI (SiO_2/Al_2O_3 = 22.3) was kindly supplied by Tosoh Co. γ-Al_2O_3 was prepared by calcination of γ-AlOOH (Catapal B Alumina purchased from Sasol) at 900 °C for 3 h. Hydroxide of Zr was prepared by hydrolysis of zirconium oxynitrate 2-hydrate in distilled water by gradually adding an aqueous NH_4OH solution (1.0 mol dm^{-3}), followed by filtration of precipitate, washing with distilled water three times, drying at 100 °C for 12 h. ZrO_2 was prepared by calcination of this hydroxide at 500 °C for 3 h. Active carbon (296 m^2 g^{-1}) was purchased from Kishida Chemical. SiO_2 (Q-10, 300 m^2 g^{-1}) was kindly supplied by Fuji Silysia Chemical Ltd.

According to our recent report[23] HBEA-supported Pt nanocluster (5 wt%) was prepared by impregnation method; a mixture of HBEA powder and aqueous HNO_3 solution of $Pt(NH_3)_2(NO_3)_2$ was evaporated, followed by drying at 90 °C. The precursor in a pyrex tube was reduced under a flow of H_2 (20 cm^3 min^{-1}) at 300 °C (0.5 h), and the pre-reduced catalyst (Pt/HBEA) was obtained. Platinum oxides-loaded HBEA (PtOx/HBEA), as a comparative catalyst, was prepared by calcination of the precursor in air at 300 °C for 3 h. By using various supports, several pre-reduced Pt catalysts were prepared by the same method as Pt/HBEA. FAU–supported metal catalysts, M/FAU (M = Ir, Re, Pd, Rh, Ru, Ag, Co, Ni, Cu) with metal loading of 5 wt% were prepared by impregnation method in the similar manner as Pt/HBEA using aqueous solution of metal nitrates (for Ag, Ni, Cu), $RuCl_3$, $IrCl_3$•nH_2O, NH_4ReO_4 or aqueous HNO_3 solution of $Rh(NO_3)_3$ or $Pd(NO_3)_2$.

Characterization: The number of surface metal atoms in Pt/HBEA, in situ reduced under H_2 at 300 °C, was estimated by the CO pulse-adsorption experiment at room temperature in a flow of He using BELCAT (BELL Japan Inc.). The average Pt particle size was calculated from the CO uptake assuming that CO was adsorbed on the surface of spherical Pt particles at CO/(surface Pt atom) = 1/1 stoichiometry. Transmission electron microscopy (TEM)

measurements were carried out by using a JEOL JEM-2100F TEM operated at 200 kV. X-ray diffraction (XRD) patterns of the powdered catalysts were recorded with a Rigaku MiniFlex II/AP diffractometer with Cu Kα radiation.

X-ray absorption near-edge structures (XANES) and extended X-ray absorption fine structure (EXAFS) at Pt L_3-edge were measured at the BL14B2 in the SPring-8 (Proposal No. 2012A1734) in a transmittance mode. The storage ring was operated at 8 GeV. A Si(111) single crystal was used to obtain a monochromatic X-ray beam. The Pt/HBEA catalyst pre-reduced in a flow of 100% H_2 (20 cm^3 min^{-1}) for 0.5 h at 300 °C was cooled to room temperature in the flow of H_2 and was sealed in cells made of polyethylene under N_2, and then the EXAFS spectrum was taken at room temperature. The spectrum of Pt foil was recorded without the pre-reduction treatment. The EXAFS analysis was performed using the REX version 2.5 program (RIGAKU). The parameters for Pt–Pt shells were provided by the FEFF6.

In situ IR (infrared) spectra were recorded by connecting a conventional flow reaction system with a JASCO FT/IR-4200 equipped with a quartz IR cell at 40 °C. Self-supporting wafer (φ = 2 cm) was prepared by pressing 40 mg of the sample and mounted in to the quartz IR cell with CaF_2 windows. Spectra were measured accumulating 30 scans at a resolution of 4 cm^{-1}. A reference spectrum of the catalyst wafer was subtracted from each spectrum, which was measured at same temperature in He. Prior to the experiment the disk of Pt/HBEA was heated in H_2 flow (20 cm^3 min^{-1}) at 300 °C for 0.5 h, followed by cooling to 40 °C and purging with He. Then, the catalyst was exposed to a flow of CO(5%)/He (20 cm^3 min^{-1}) for 180 s, followed by purging with He (40 cm^3 min^{-1}) for 600 s.

Typical Procedure of Catalytic Reactions: Typically, the mixture indole (1.0 mmol) and 1-octanol (1.0 mmol) in *o*-xylene (1 g) was injected to the pre-reduced catalyst inside the reactor (cylindrical glass tube) through a septum inlet, followed by heating at 130 °C under 1 atm N_2. After cooling the mixture, followed by removal of the catalyst by filtration, evacuation, and by purification by column chromatography with silica gel 60 (spherical) 63-210 μm (Kanto Chemical Co. Ltd.) using eluting solvent of hexane/ethyl acetate (50/1) to isolate *N*-adducts in Table 2. For the standard reaction of indole and 1-octanol for catalyst screening (Table 1) and kinetic studies, conversion and yields of products were determined by GC using *n*-dodecane as an internal standard adopting the GC sensitivity estimated using the isolated product.

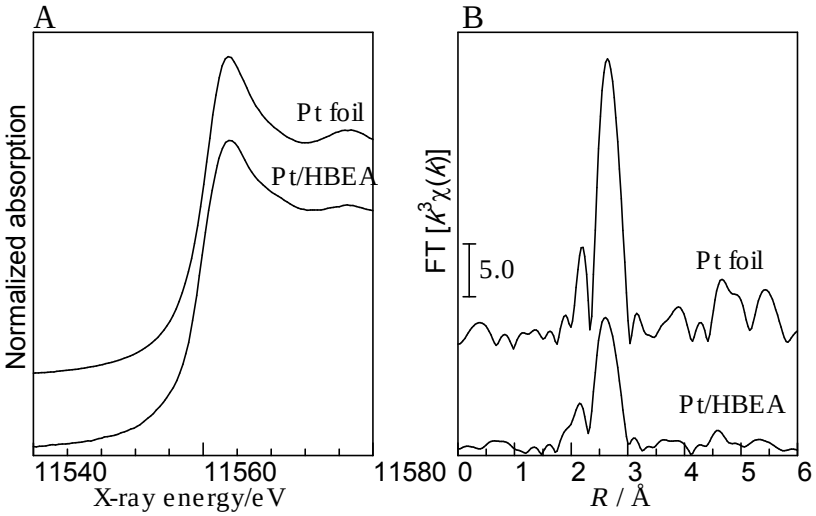


Figure S1 Pt L_3-edge XANES spectra (A) and EXAFS Fourier transforms (B).

Table S1. Curve-fitting analysis of Pt L_3-edge EXAFS.

Sample	Shell	N [a]	*R* /Å [b]	σ /Å [c]	R_f /% [d]
Pt/HBEA	Pt	10.2	2.74	0.080	1.8
Pt metal[e]	Pt	12	2.76	-	-

[a] Coordination numbers. [b] Bond distance. [c] Debye-Waller factor. [d] Residual factor.

[e] Crystallographic data of Pt metal.

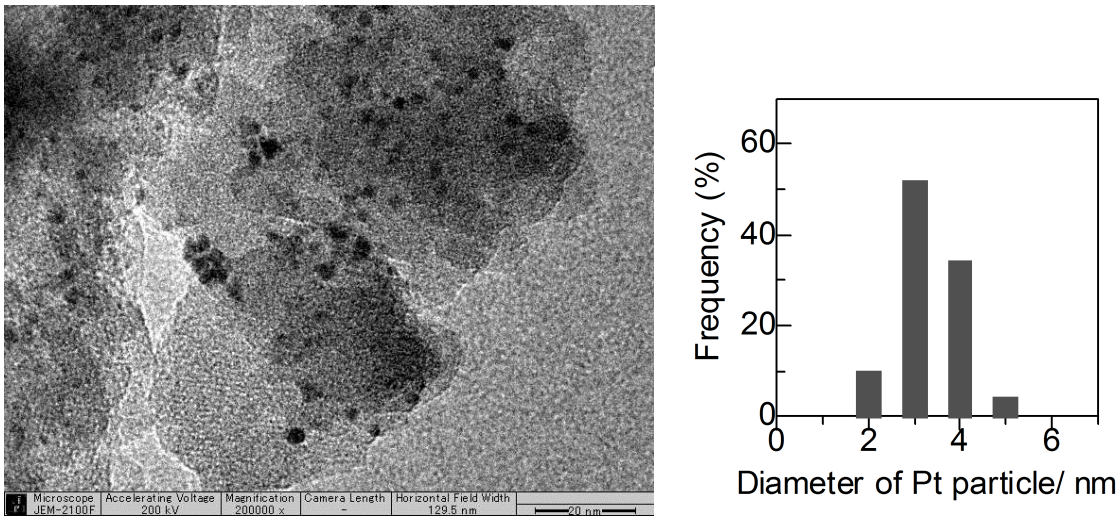


Figure S2. Typical TEM image and Pt particle size distribution of Pt/HBEA (TEM analysis). Average size is 3.3 ± 0.7 nm.

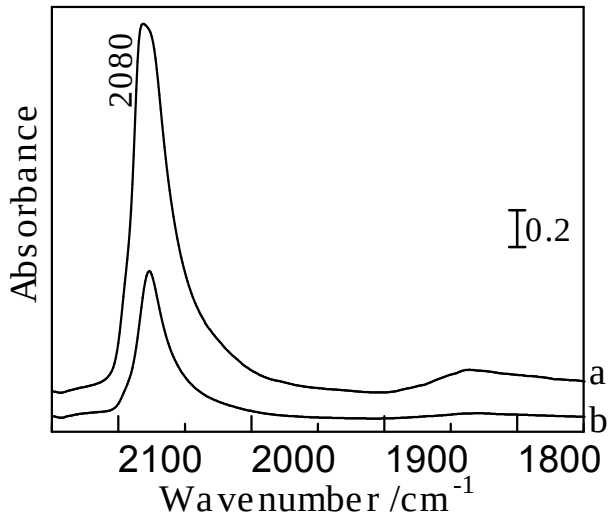


Figure S3. IR spectra of CO adsorbed at 40 °C on (a) as-reduced Pt/HBEA and (b) Pt/HBEA after re-oxidation in air at room temperature.

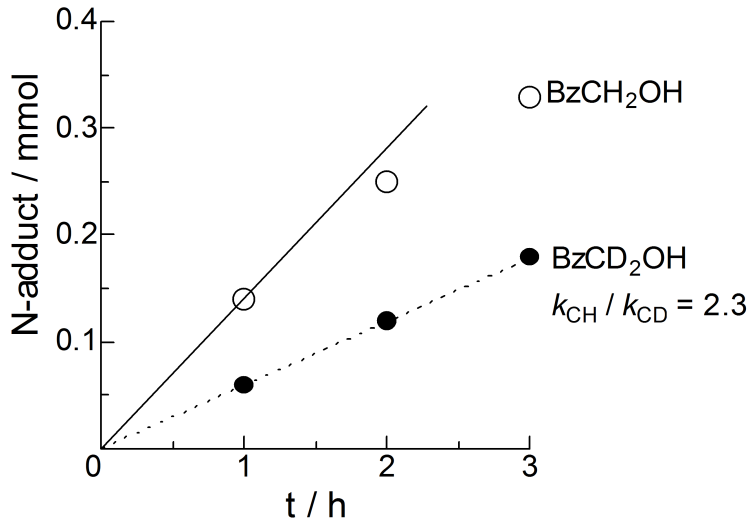


Figure S4. Kinetic isotopic effect (KIE) in the reaction of benzylalcohol (1 mmol) with indole (1 mmol) by Pt/HBEA (1 mol%) at 130 °C. The KIE estimated from the zero order rate constants (the slope of the lines) is 2.3.

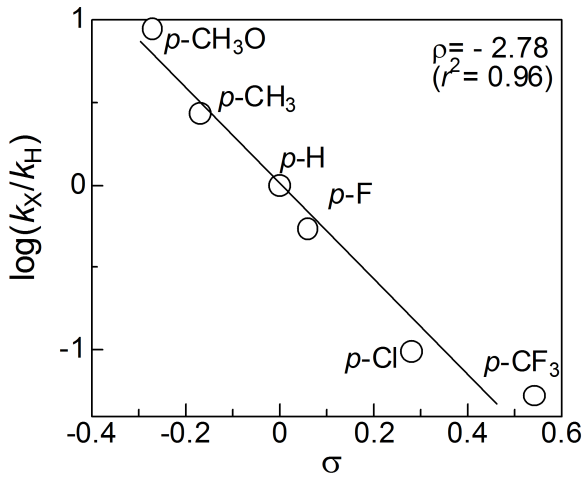
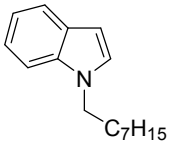


Figure S5. Hammett plot for N-alkylation of indole (1 mmol) with *p*-substituted benzylalcohols (1 mmol) by Pt/HBEA (1 mol%) at 130 °C.

NMR and GC/MS analysis

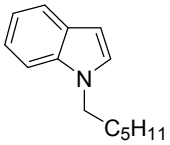
^{1}H and ^{13}C NMR spectra for alkylated/arylated indole of Table-3 and Table-4 were assigned and reproduced to the corresponding literature. ^{1}H and ^{13}C NMR spectra were recorded using at ambient temperature on JEOL-ECX 600 operating at 600.17 and 150.92 MHz, respectively with tetramethylsilane as an internal standard. All chemical shifts (δ) are reported in ppm and coupling constants (*J*) in Hz. All chemical shifts are reported relative to tetramethylsilane and *d*-solvent peaks (77.00 ppm chloroform, 40.45 ppm dimethylsulfoxide), respectively. Abbreviations used in the NMR experiments: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. GC-MS spectra was taken by SHIMADZU QP2010.

1-Octyl-1*H*-indole:[1]



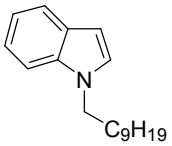
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.67 (d, *J* = 8.94 Hz, 1H), 7.38 (d, *J* = 8.22 Hz, 1H), 7.24 (m, 1H), 7.14-7.12 (m, 2H), 6.52 (d, *J* = 2.76 Hz, 1H), 4.13 (t, *J* = 6.61 Hz, 2H), 1.87-1.85 (t, *J* = 6.54 Hz, 2H), 1.41-1.22 (m, 10H), 0.92 (t, *J* = 6.61 Hz, 3H); ^{13}C NMR (150.92 MHz, $CDCl_3$): δ 135.88, 128.51, 127.74, 121.21, 120.87, 119.07, 109.34, 100.73, 46.36, 31.75, 30.12, 29.19, 29.14, 26.98, 22.59, 14.05; GC-MS m/e 229.185.

1-Hexyl-1*H*-indole:[2]



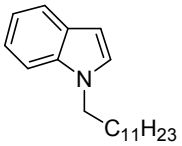
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.71 (d, *J* = 7.56 Hz, 1H), 7.41 (d, *J* = 6.85 Hz, 1H), 7.29-7.27 (m, 1H), 7.18-7.15 (m, 2H), 6.56 (d like s, 1H), 4.16 (t, *J* = 6.60 Hz, 2H), 1.90-1.88 (m, 2H), 1.37-1.34 (m, 6H), 0.95 (t, *J* = 6.60 Hz, 3H); ^{13}C NMR (150.92 MHz, $CDCl_3$): δ 135.87, 128.50, 127.73, 121.20, 120.85, 119.06, 109.32, 100.73, 46.33, 31.39, 30.16, 26.62, 22.50, 13.97; GC-MS m/e 201.155.

1-Decyl-1*H*-indole:[1]



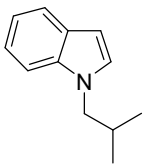
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.68 (d, *J* = 8.22 Hz, 1H), 7.38 (d, *J* = 8.28 Hz, 1H), 7.26-7.23 (m, 1H), 7.15-7.12 (m, 2H), 6.53 (d, *J* = 2.76 Hz, 1H), 4.13 (t, *J* = 6.60 Hz, 2H), 1.87-1.85 (m, 2H), 1.34-1.29 (m, 14H), 0.93 (t, *J* = 6.60 Hz, 3H); ^{13}C NMR (150.92 MHz, $CDCl_3$): δ 135.88, 128.51, 127.73, 121.21, 120.87, 119.07, 109.33, 100.73, 46.35, 31.84, 30.21, 29.50, 29.48, 29.25, 29.23, 26.97, 21.52, 14.09; GC-MS m/e 257.215.

1-Dodecyl-1*H*-indole:[1]



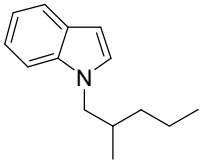
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.78 (d, *J* = 7.56 Hz, 1H), 7.49 (d, *J* = 8.22 Hz, 1H), 7.37-7.34 (m, 1H), 7.24-7.23 (m, 2H), 6.63 (d, *J* = 3.42 Hz, 1H), 4.25 (t, *J* = 6.87 Hz, 2H), 1.98-1.96 (m, 2H), 1.45-1.39 (m, 18H), 1.03 (t, *J* = 6.87 Hz, 3H); ^{13}C NMR (150.92 MHz, $CDCl_3$): δ 135.89, 128.51, 127.75, 121.22, 120.87, 119.08, 109.34, 100.74, 46.38, 31.89, 30.23, 29.58 (C×2), 29.55, 29.49, 29.32, 29.23, 26.99, 22.67, 14.11; GC-MS m/e 285.245.

1-Isobutyl-1*H*-indole:[2]



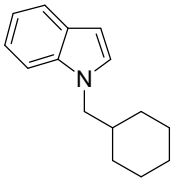
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.67 (d, *J* = 7.56 Hz, 1H), 7.37 (d, *J* = 8.28 Hz, 1H), 7.25-7.22 (m, 1H), 7.15-7.10 (m, 2H), 6.53 (d, *J* = 3.42 Hz, 1H), 3.94 (d, *J* = 7.56 Hz, 2H), 2.25-2.23 (m, 1H), 0.97 (d, *J* = 6.90 Hz, 6H); ^{13}C NMR (150.92 MHz, $CDCl_3$): δ 136.21, 128.50, 128.37, 121.22, 120.85, 119.08, 109.56, 100.69, 54.10, 29.49, 20.29 (C×2); GC-MS m/e 173.125.

1-(2-Methyl-pentyl)-1*H*-indole:[5]



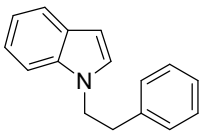
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.67 (d, J = 7.56 Hz, 1H), 7.36 (d, J = 8.22 Hz, 1H), 7.25-7.23 (m, 1H), 7.14-7.09 (m, 2H), 6.51 (d, J = 3.48 Hz, 1H), 4.08-4.05 (m, 1H), 3.88-3.85 (m, 1H), 2.10-2.07 (m, 1H), 1.51-1.45 (m, 1H), 1.39-1.30 (m, 2H), 1.21-1.19 (m, 1H), 0.93-0.89 (m, 6H); ^{13}C NMR (150.92 MHz, $CDCl_3$): δ 136.25, 128.50, 128.45, 121.22, 120.85, 119.07, 109.54, 100.67, 52.95, 36.73, 34.01, 19.99, 17.71, 14.22; GC-MS m/e 201.155.

1-Cyclohexylmethyl-1*H*-indole:[3]



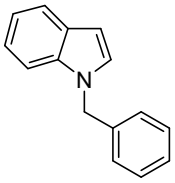
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.63-7.61 (m, 1H), 7.33-7.32 (m, 1H), 7.21-7.17 (m, 1H), 7.08-7.06 (m, 1H), 7.05-7.04 (m, 1H), 6.46 (d, J = 3.42 Hz, 1H), 3.92-3.91 (m, 2H) 1.85-1.82 (m, 1H), 1.72-1.68 (m, 2H), 1.64-1.58 (m, 3H), 1.22-1.14 (m, 3H), 0.99-0.95 (m, 2H); ^{13}C NMR (150.92 MHz, $CDCl_3$): δ 136.22, 128.54, 128.42, 121.17, 120.83, 119.04, 109.60, 100.54, 52.95, 38.78, 31.06(C×2), 26.29, 25.70 (C×2); GC-MS m/e 213.155.

1-Phenethyl-1*H*-indole:[2]



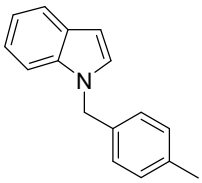
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.63-7.61 (m, 1H), 7.33-7.31 (m, 1H), 7.25-7.19 (m, 4H), 7.10-7.05 (m, 3H), 6.89-6.88 (m, 1H), 6.43-6.42 (m, 1H), 4.30 (t, J = 7.56 Hz, 2H), 3.06 (t, J = 7.56 Hz, 2H); ^{13}C NMR (150.92 MHz, $CDCl_3$): δ 138.47, 135.63, 128.70 (C×2), 128.60 (C×2), 128.55, 127.83, 126.58, 121.36, 120.95, 119.26, 109.21, 100.91, 48.04, 36.67; GC-MS m/e 221.125.

1-Benzyl-1*H*-indole:[4]



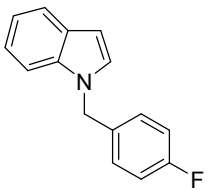
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.73 (d, J = 7.56 Hz, 1H), 7.35-7.30 (m, 4H), 7.27-7.23 (m, 1H), 7.21-7.15 (m, 4H), 6.63 (d, J = 3.41 Hz, 1H), 5.35 (s, 2H); ^{13}C NMR (150.92 MHz, $CDCl_3$): δ 137.47, 136.21, 128.67 (C×2), 128.63, 128.20, 127.50, 126.68 (C×2), 121.61, 120.90, 119.46, 109.63, 101.60, 49.95; GC-MS m/e 207.105.

1-(4-Methyl-benzyl)-1*H*-indole:[5]



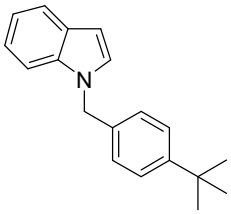
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.63 (d, J = 7.56 Hz, 1H), 7.27 (d, J = 8.22 Hz, 1H), 7.15 (t, J = 6.90 Hz, 1H), 7.10-7.07 (m, 4H), 6.99 (d, J = 8.28 Hz, 2H), 6.52 (d, J = 2.70 Hz, 1H), 5.24 (s, 2H), 2.29 (s, 3H); ^{13}C NMR (150.92 MHz, $CDCl_3$): δ 137.24, 136.23, 134.43, 129.36 (C×2), 128.66, 128.15, 126.76 (C×2), 121.57, 120.90, 119.41, 109.66, 101.50, 49.80, 21.04; GC-MS m/e 221.125.

1-(4-Fluoro-benzyl)-1*H*-indole:[6]



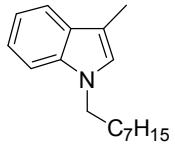
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.64 (d, J = 7.56 Hz, 1H), 7.24 (d, J = 8.28 Hz, 1H), 7.16 (t, J = 7.56 Hz, 1H), 7.12-7.09 (m, 2H), 7.06-7.04 (m, 2H), 6.95 (t, J = 8.94 Hz, 2H), 6.54 (d, J = 3.42 Hz, 1H), 5.26 (s, 2H); ^{13}C NMR (150.92 MHz, $CDCl_3$): δ 162.16 (d, J = 245.29 Hz, 4-F-C), 136.11, 133.23, 128.72, 128.37 (d, J = 8.06 Hz, *meta to* 4-F, C×2), 128.03, 121.74, 121.02, 119.60, 115.61 (d, J = 21.88 Hz, *ortho* to 4-F, C×2), 109.55, 101.83, 49.36; GC-MS m/e 225.105.

1-(4-*tert*-Butyl-benzyl)-1*H*-indole:[7]



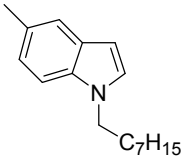
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): 7.65 (d, *J* = 7.56 Hz, 1H), 7.30-7.28 (m, 3H), 7.18-7.155 (m, 1H), 7.11-7.08 (m, 2H), 7.05-7.03 (m, 2H), 6.54-6.53 (m, 1H), 5.27 (s, 2H), 1.27 (s, 9H); ^{13}C NMR (150.92 MHz, $CDCl_3$): δ 150.50, 136.27, 134.50, 128.64, 128.20, 126.51 (C×2), 125.61 (C×2), 121.56, 120.90, 119.41, 109.67, 101.52, 49.66, 34.46, 31.40 (C×3); GC-MS m/e 263.165.

3-Methyl-1-octyl-1*H*-indole:



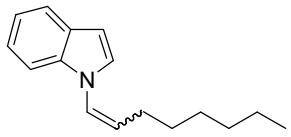
^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.55 (d, *J* = 8.28 Hz, 1H), 7.28 (d, *J* = 8.28 Hz, 1H), 7.19-7.17 (m, 1H), 7.09-7.07 (m, 1H), 6.84 (d, *J* = 3.42 Hz, 1H), 4.01 (t, *J* = 7.20 Hz, 2H), 2.32 (s, 3H), 1.79-1.76 (m, 2H), 1.29-1.23 (m, 10H), 0.86 (t, *J* = 7.20 Hz, 3H); ^{13}C NMR (150.92 MHz, $CDCl_3$): δ 136.21, 128.62, 125.41, 121.14, 118.92, 118.31, 109.88, 109.12, 46.05, 31.77, 30.36, 29.24, 29.15, 27.02, 22.60, 14.06, 9.57; GC-MS m/e 243.200.

5-Methyl-1-octyl-1*H*-indole:



^{1}H NMR (600.17 MHz, $CDCl_3$, TMS): δ 7.43-7.40 (m, 1H), 7.23-7.20 (m, 1H), 7.03-7.02 (m, 2H), 6.39 (d, *J* = 3.42 Hz, 1H), 4.05 (t, *J* = 7.56 Hz, 2H), 2.46-2.43 (m, 3H), 1.81-1.79 (m, 2H), 1.31-1.25 (m, 10H), 0.87 (t, *J* = 7.56 Hz, 3H); ^{13}C NMR (150.92 MHz, $CDCl_3$) δ 134.29, 128.79, 128.23, 127.82, 122.84, 120.51, 109.03, 100.13, 46.40, 31.77, 30.22, 29.19 (C×2), 26.99, 22.62, 21.36, 14.06; GC-MS m/e 243.200.

1-Oct-1-enyl-1*H*-indole: GC-MS m/e 227.165.



References:

1 E. G. Pérez, B. K. Cassels, C. Eibl and D. Gündisch, *Bioorg. Med. Chem.,* 2012, **20**, 3719.

2 S. Bahn, S. Imm, K. Mevius, L. Neubert, A. Tillack, J. M. J. Williams and M.Beller, *Chem. Eur. J.,* 2010, **16**, 3590.

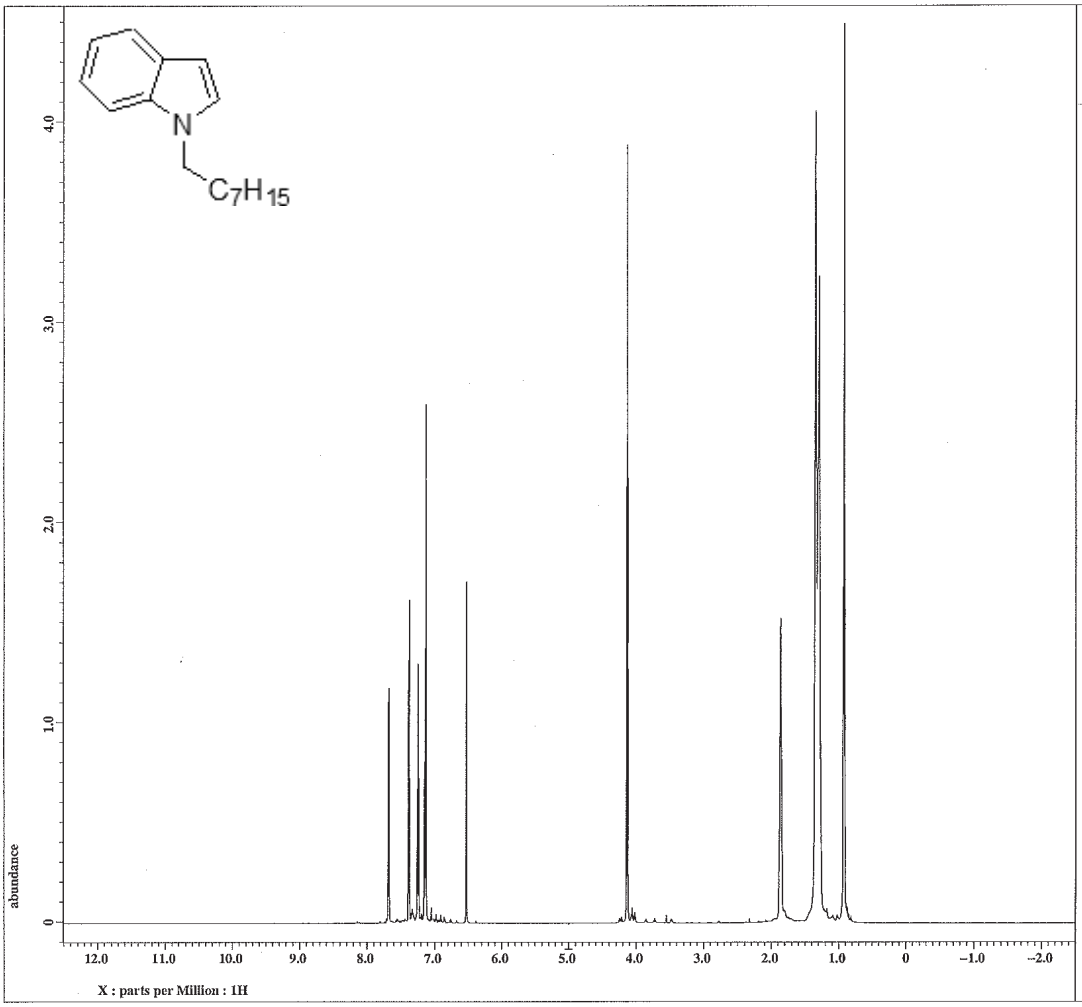
3 I. Deb, D. J. Coiro and D. Seidel, *Chem. Commun.,* 2011, **47**, 6473.

4 I. Deb, D. Das and D.Seidel, *Org. Lett.,* 2011, **13**, 812.

5 G. A. Molander, D. W. Ryu, M. H. Sarvari, R. Devulapally and D. G. Seapy, *J. Org. Chem.,* 2013, **78**, 6448.

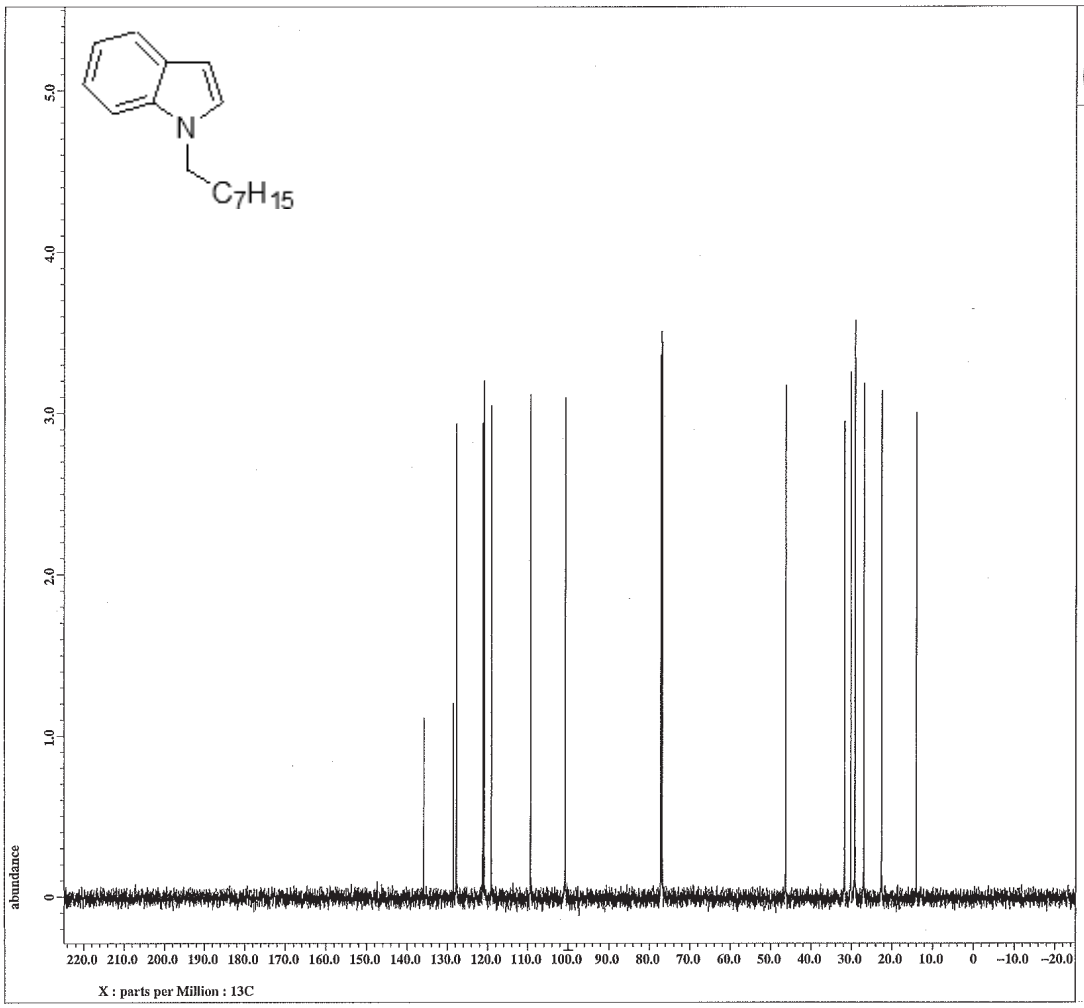
6 P. S. Fier and J. F. Hartwig, *J. Am. Chem. Soc.*, 2012, **134**, 10795.

7 Y. H. Lim, Q. Ong, H. A. Duong, T. M. Nguyen and C. W. Johannes, *Org. Lett.,* 2012, **14**, 5676.



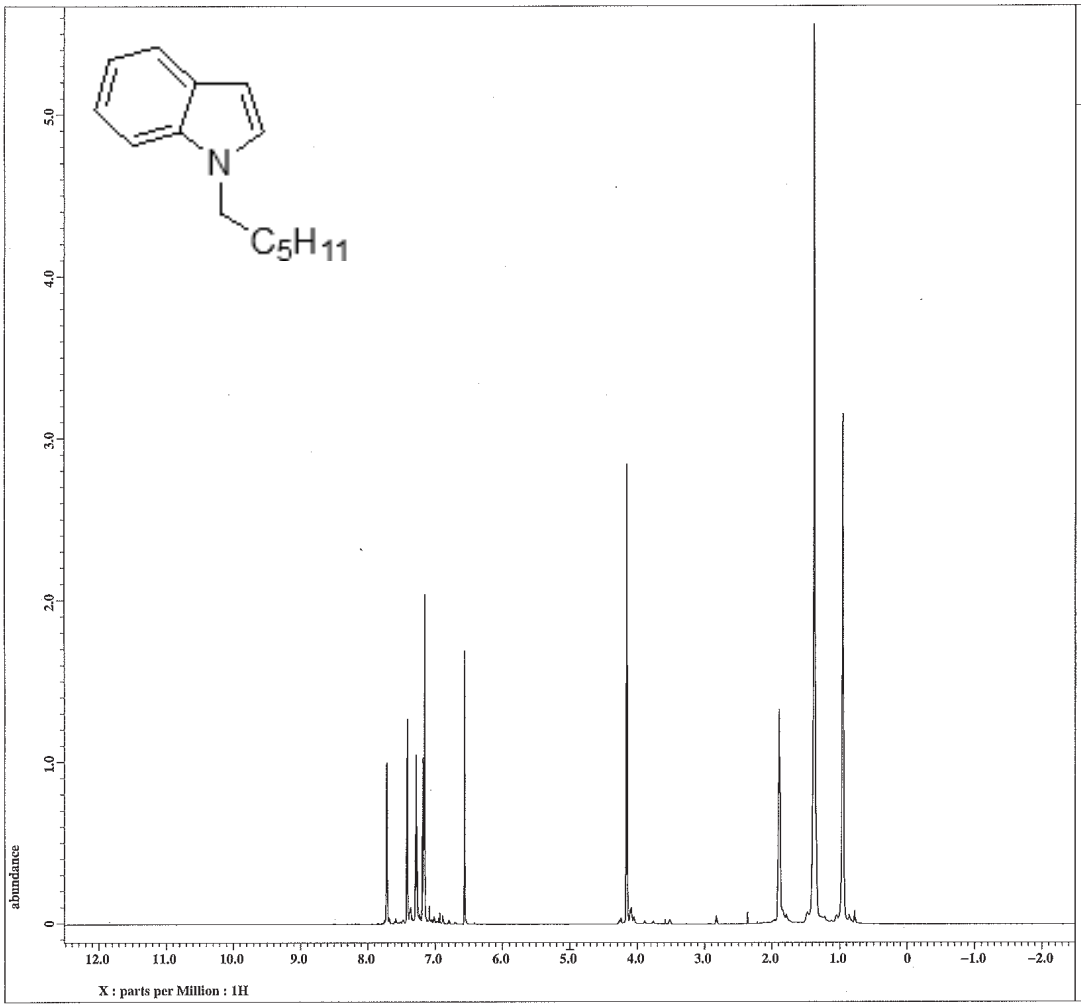
JEOL

```
Filename          = Exp-hakim-C-N-oct-pro
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-C-N-oct-pro
Solvent           = CHLOROFORM-D
Creation_time     =  6-NOV-2013 14:04:40
Revision_time     =  6-NOV-2013 14:16:16
Current_time      =  6-NOV-2013 14:16:29

Content           = Exp-hakim-C-N-oct-pro
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 1.4548992[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.68733284[Hz]
X_sweep           = 11.26126126[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 600.1723046[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 13[us]
X_acq_time        = 1.4548992[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 34
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 20.2[dC]
```



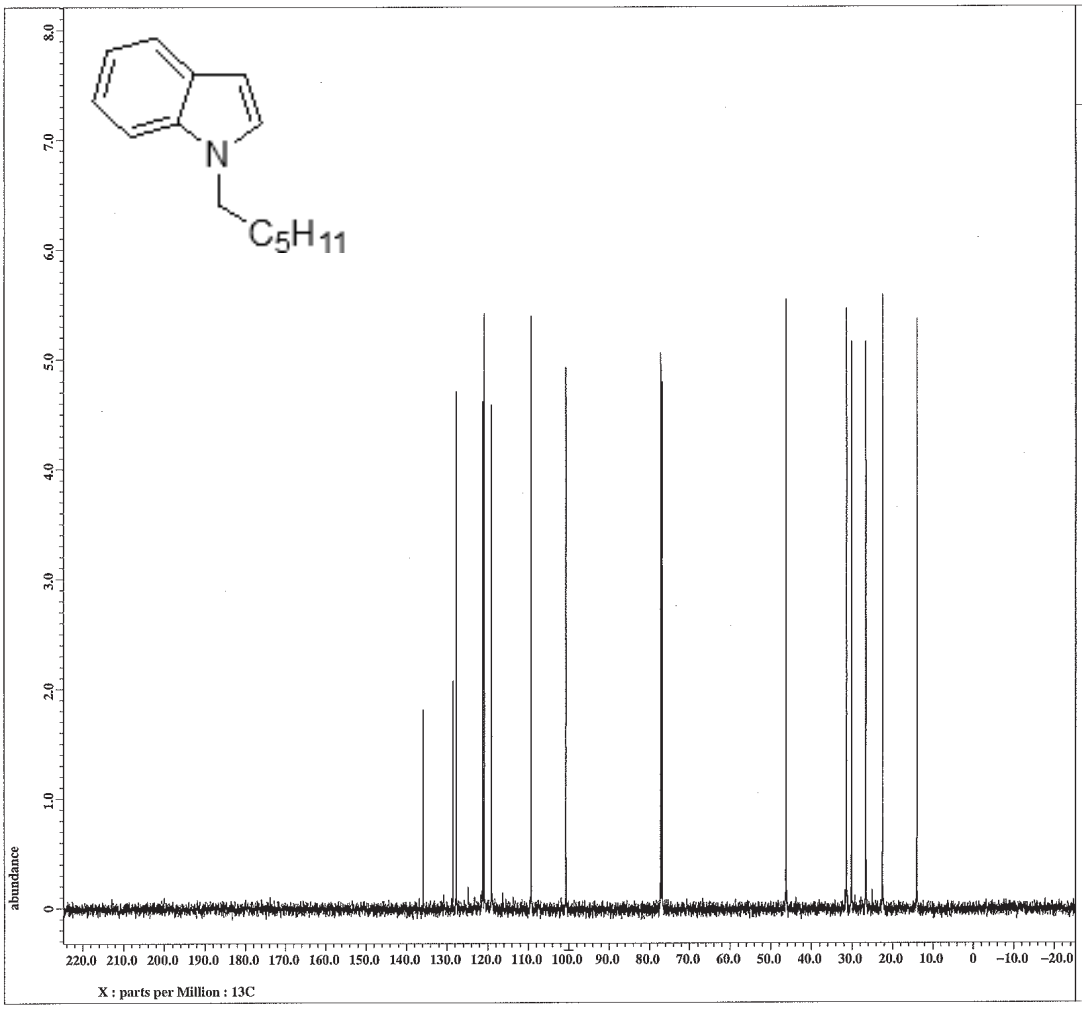
JEOL

```
Filename          = Exp-hakim-C-N-oct-car
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-C-N-oct-car
Solvent           = CHLOROFORM-D
Creation_time     =  6-NOV-2013 14:09:50
Revision_time     =  6-NOV-2013 14:23:03
Current_time      =  6-NOV-2013 14:23:15

Content           = Exp-hakim-C-N-oct-car
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.868352[s]
X_domain          = 13C
X_freq            = 150.91343039[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.15160672[Hz]
X_sweep           = 37.73584906[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 75
Total_scans       = 75

X_90_width        = 11.4[us]
X_acq_time        = 0.868352[s]
X_angle           = 30[deg]
X_atn             = 7.5[dB]
X_pulse           = 3.8[us]
Irr_atn_dec       = 19.391[dB]
Irr_atn_noe       = 19.391[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2.5[s]
Recvr_gain        = 76
Relaxation_delay  = 2.5[s]
Repetition_time   = 3.368352[s]
Temp_get          = 21.1[dC]
```



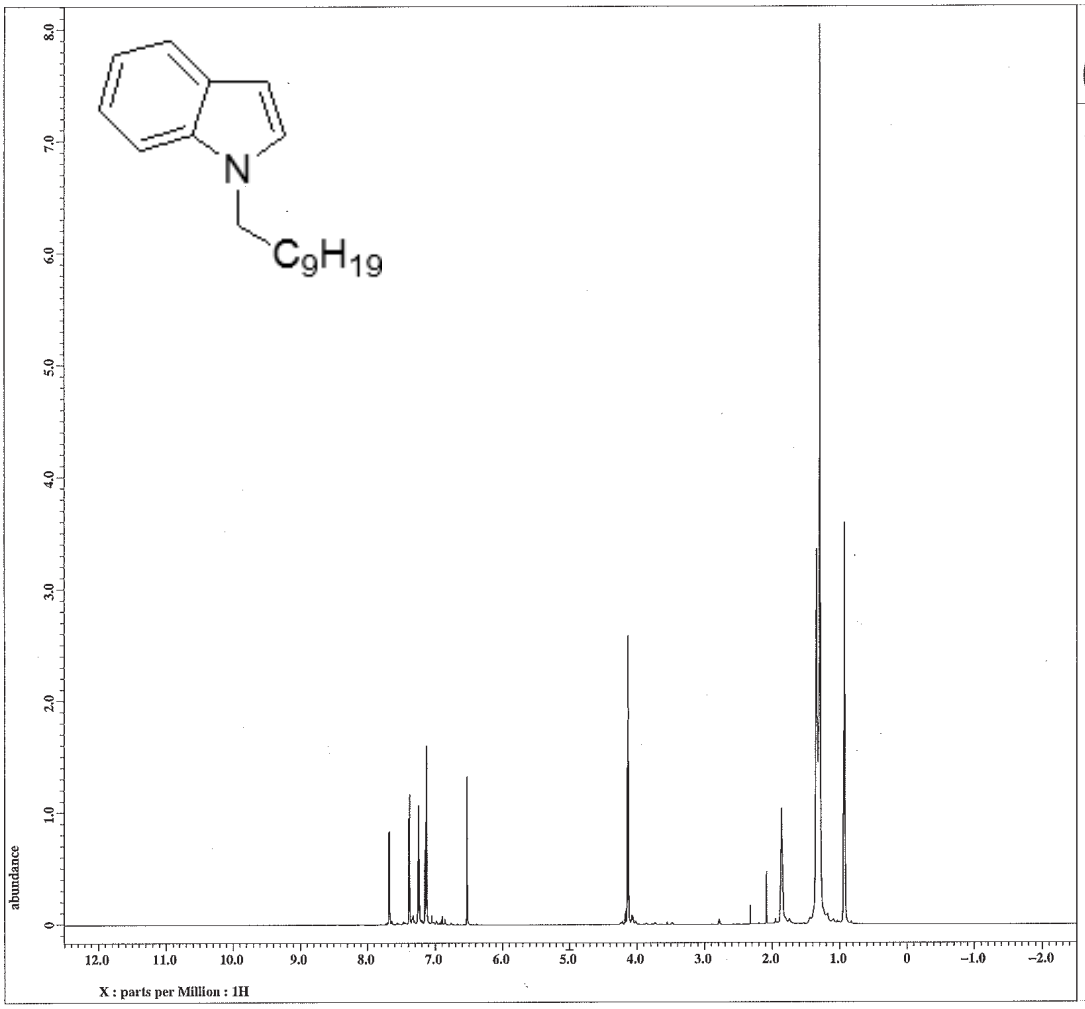
JEOL

```
Filename          = Exp-hakim-C-N-hexa-pr
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-C-N-hexa-pr
Solvent           = CHLOROFORM-D
Creation_time     =  6-NOV-2013 14:16:33
Revision_time     =  6-NOV-2013 14:27:49
Current_time      =  6-NOV-2013 14:27:55

Content           = Exp-hakim-C-N-hexa-pr
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 1.4548992[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.68733284[Hz]
X_sweep           = 11.26126126[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 600.1723046[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 13[us]
X_acq_time        = 1.4548992[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 30
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 19.9[dC]
```

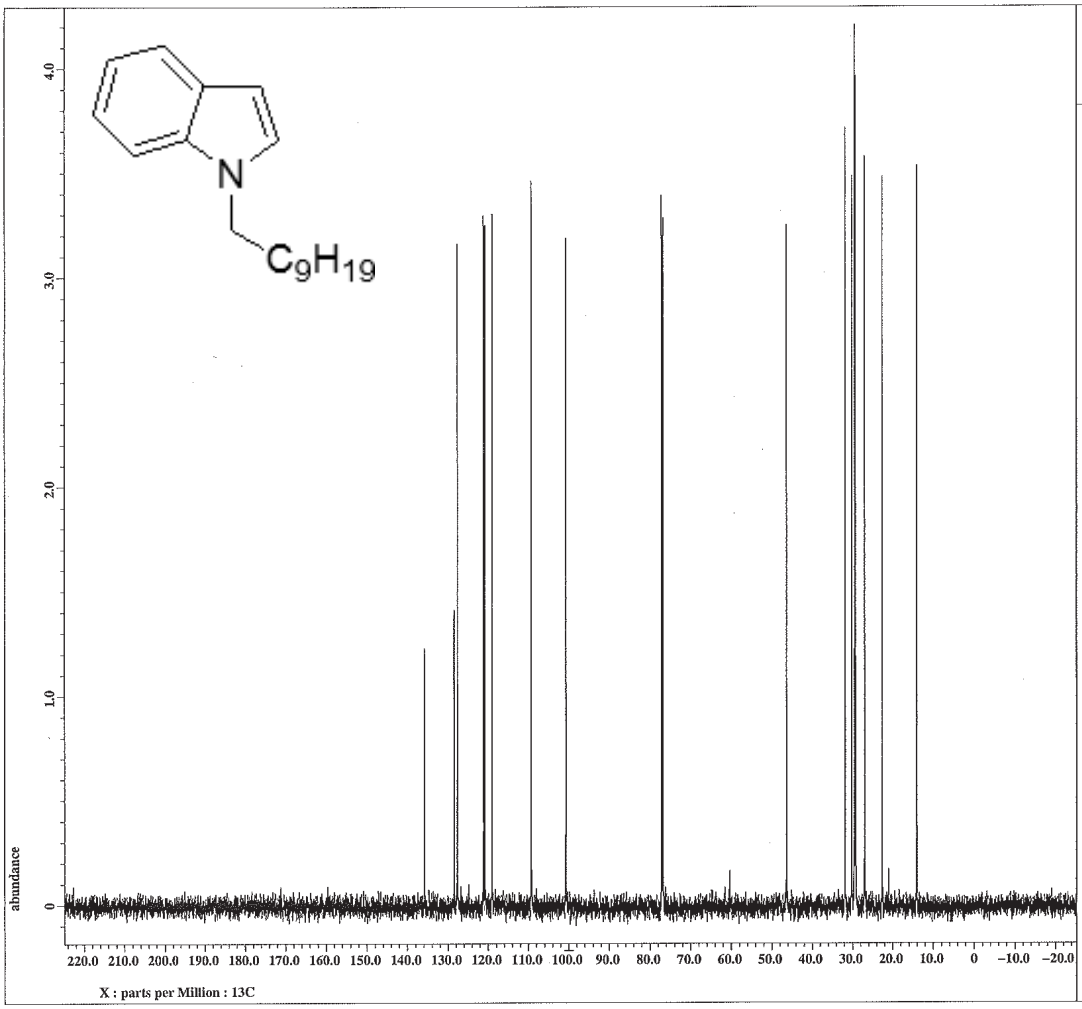


JEOL

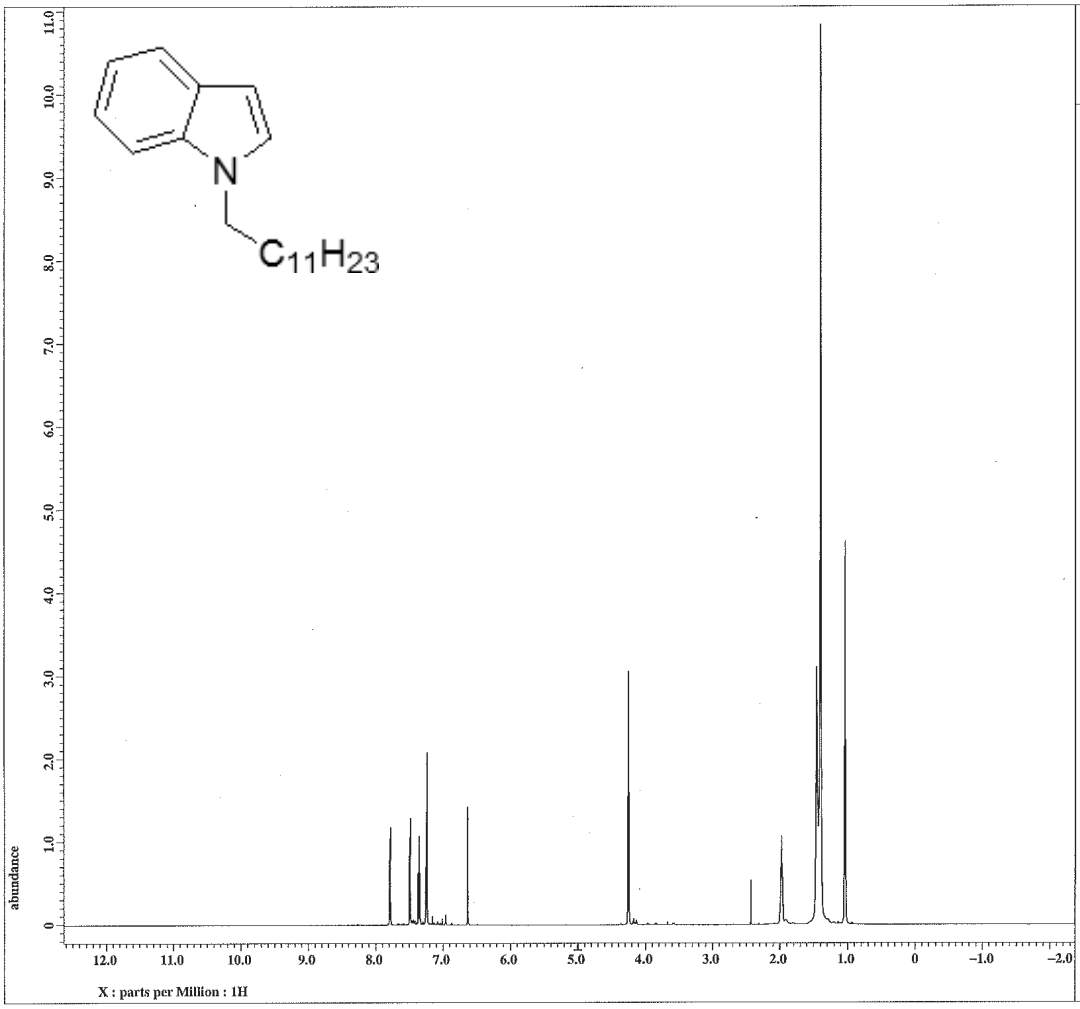
```
Filename          = Exp-hakim-C-N-hexa-ca
Author            = delta
Experiment        = single_pulse_dec '
Sample_id         = Exp-hakim-C-N-hexa-ca
Solvent           = CHLOROFORM-D
Creation_time     =  6-NOV-2013 14:23:18
Revision_time     =  6-NOV-2013 14:36:16
Current_time      =  6-NOV-2013 14:36:28

Content           = Exp-hakim-C-N-hexa-ca
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.868352[s]
X_domain          = 13C
X_freq            = 150.91343039[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.15160672[Hz]
X_sweep           = 37.73584906[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 101
Total_scans       = 101

X_90_width        = 11.4[us]
X_acq_time        = 0.868352[s]
X_angle           = 30[deg]
X_atn             = 7.5[dB]
X_pulse           = 3.8[us]
Irr_atn_dec       = 19.391[dB]
Irr_atn_noe       = 19.391[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2.5[s]
Recvr_gain        = 78
Relaxation_delay  = 2.5[s]
Repetition_time   = 3.368352[s]
Temp_get          = 20.8[dC]
```



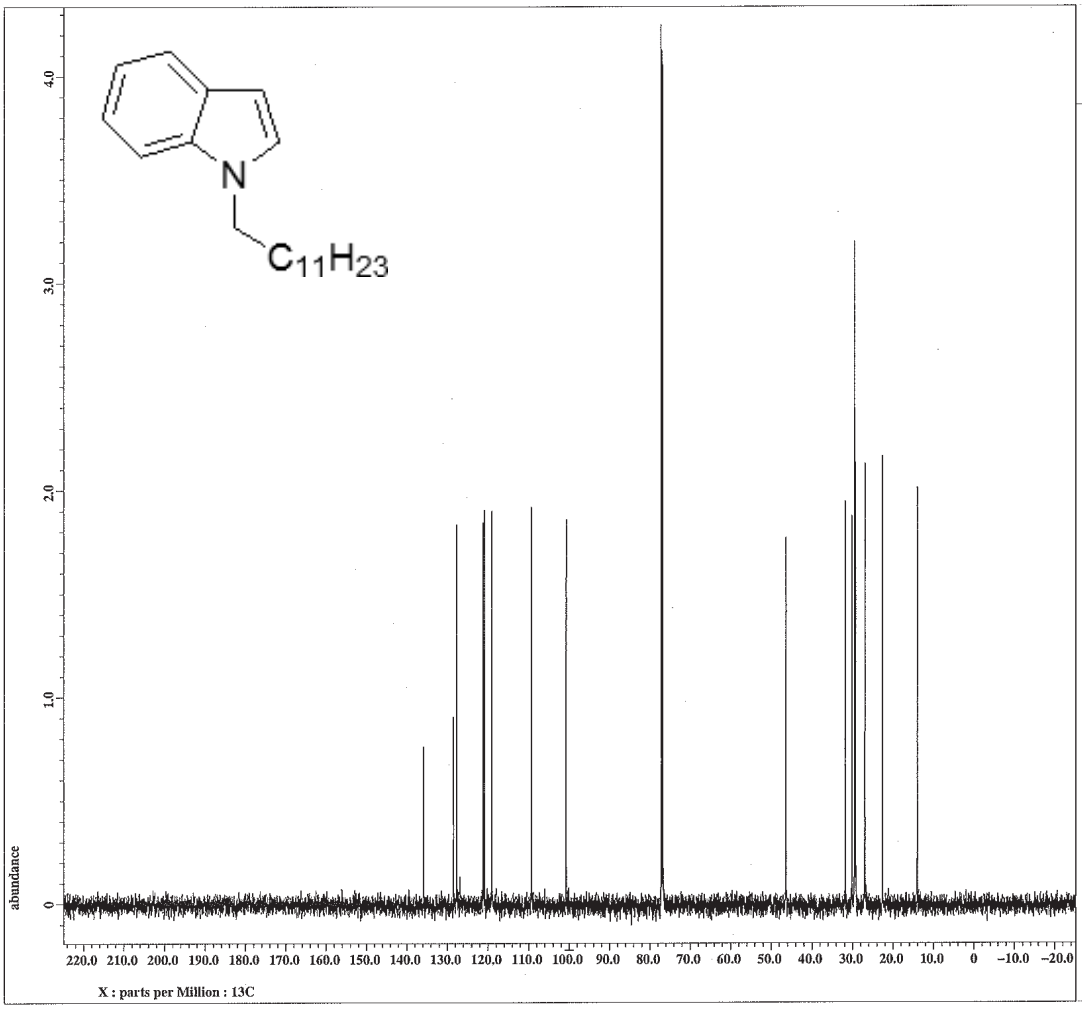

```
Filename          = Exp-hakim-C-N-deca-pr
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-C-N-deca-pr
Solvent           = CHLOROFORM-D
Creation_time     =  6-NOV-2013 14:42:33
Revision_time     =  6-NOV-2013 14:53:46
Current_time      =  6-NOV-2013 14:53:50

Content           = Exp-hakim-C-N-deca-pr
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 1.4548992[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.68733284[Hz]
X_sweep           = 11.26126126[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 600.1723046[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 13[us]
X_acq_time        = 1.4548992[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 30
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 20.1[dC]
```



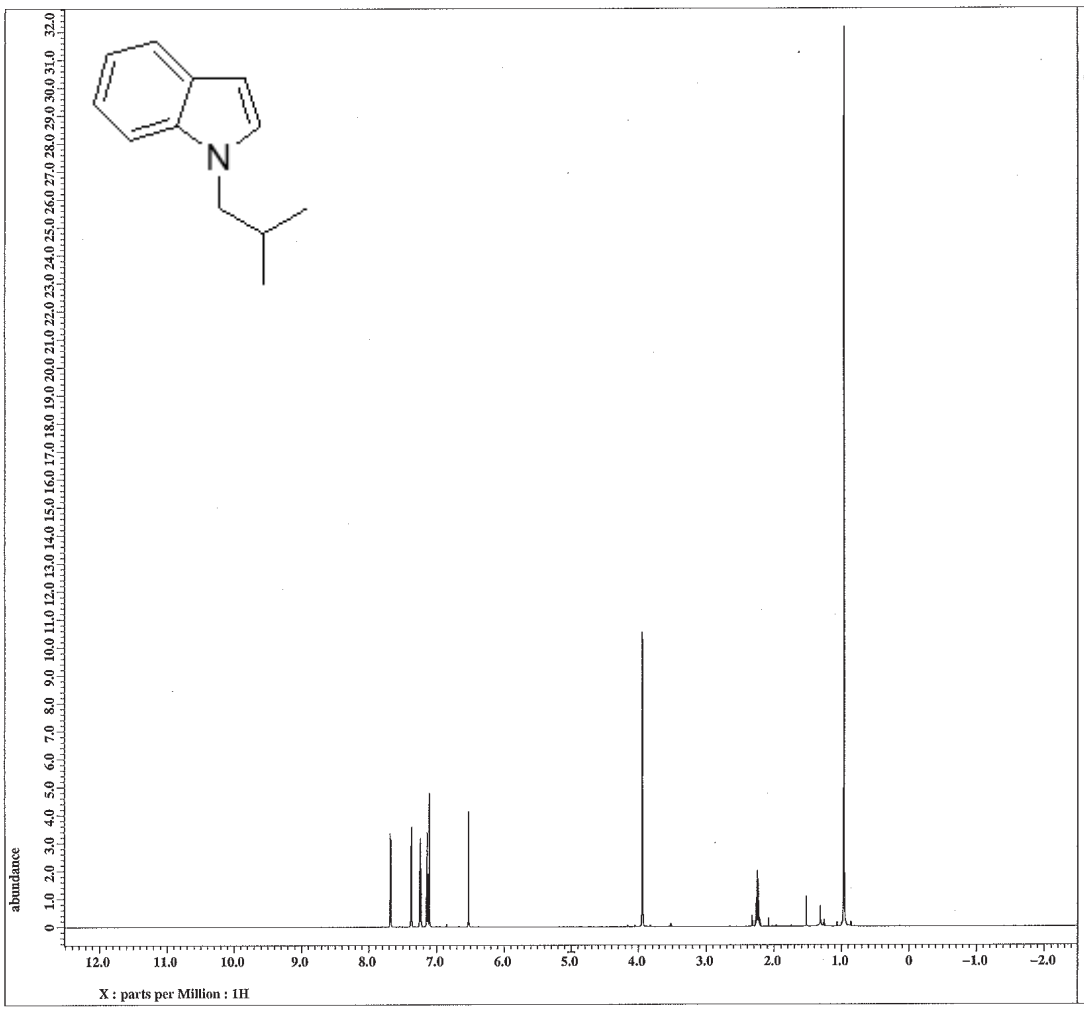

```
Filename          = Exp-hakim-C-N-deca-ca
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-C-N-deca-ca
Solvent           = CHLOROFORM-D
Creation_time     =  6-NOV-2013 14:48:00
Revision_time     =  6-NOV-2013 15:00:48
Current_time      =  6-NOV-2013 15:00:52

Content           = Exp-hakim-C-N-deca-ca
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.868352[s]
X_domain          = 13C
X_freq            = 150.91343039[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.15160672[Hz]
X_sweep           = 37.73584906[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 80
Total_scans       = 80

X_90_width        = 11.4[us]
X_acq_time        = 0.868352[s]
X_angle           = 30[deg]
X_atn             = 7.5[dB]
X_pulse           = 3.8[us]
Irr_atn_dec       = 19.391[dB]
Irr_atn_noe       = 19.391[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2.5[s]
Recvr_gain        = 76
Relaxation_delay  = 2.5[s]
Repetition_time   = 3.368352[s]
Temp_get          = 20.5[dC]
```




```
Filename          = Exp-hakim-C-N-dodeca-
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-C-N-dodeca-
Solvent           = CHLOROFORM-D
Creation_time     =  6-NOV-2013 14:29:47
Revision_time     =  6-NOV-2013 14:41:03
Current_time      =  6-NOV-2013 14:41:07

Content           = Exp-hakim-C-N-dodeca-
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 1.4548992[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.68733284[Hz]
X_sweep           = 11.26126126[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 600.1723046[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 13[us]
X_acq_time        = 1.4548992[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 34
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 20[dC]
```

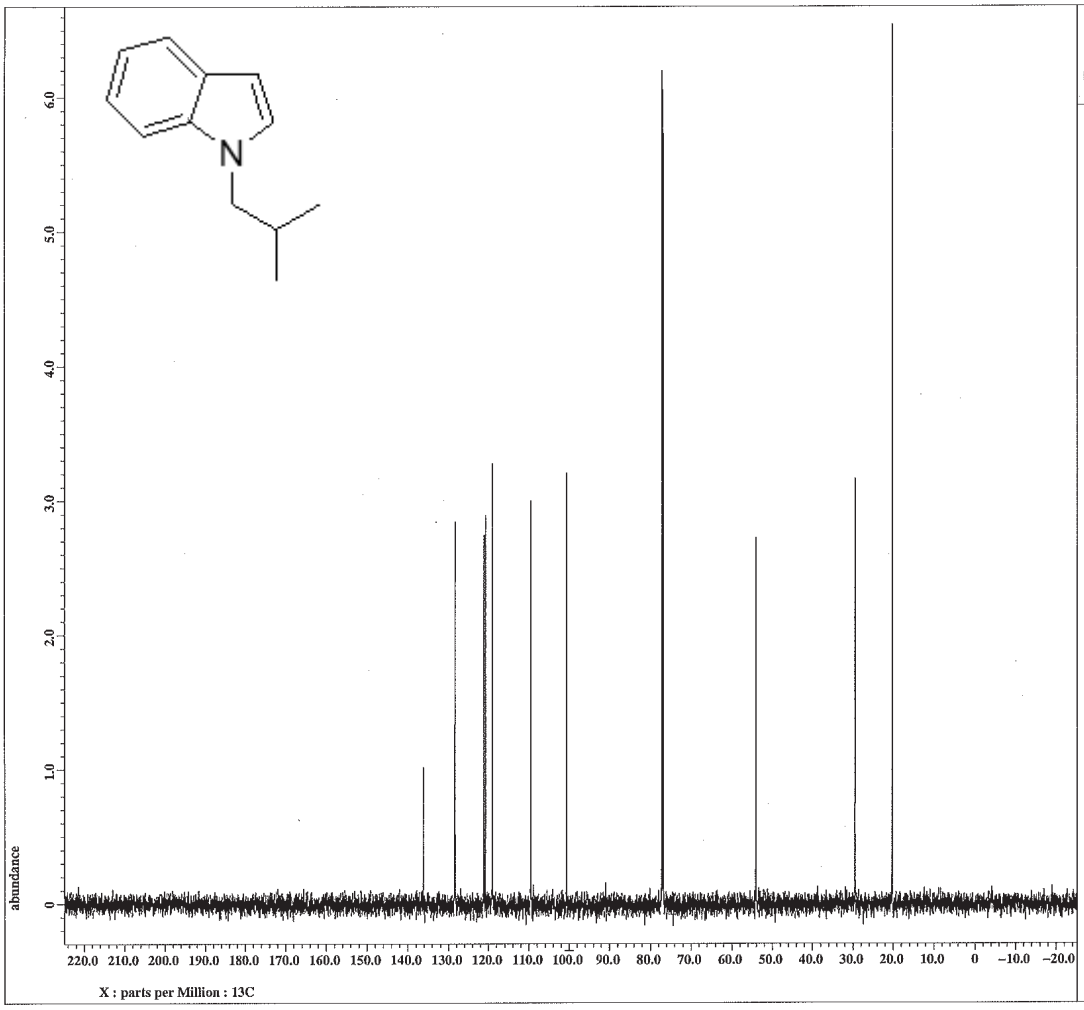



```
Filename          = Exp-hakim-C-N-dodeca-
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-C-N-dodeca-
Solvent           = CHLOROFORM-D
Creation_time     =  6-NOV-2013 14:36:18
Revision_time     =  6-NOV-2013 14:48:48
Current_time      =  6-NOV-2013 14:48:52

Content           = Exp-hakim-C-N-dodeca-
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.868352[s]
X_domain          = 13C
X_freq            = 150.91343039[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.15160672[Hz]
X_sweep           = 37.73584906[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 99
Total_scans       = 99

X_90_width        = 11.4[us]
X_acq_time        = 0.868352[s]
X_angle           = 30[deg]
X_atn             = 7.5[dB]
X_pulse           = 3.8[us]
Irr_atn_dec       = 19.391[dB]
Irr_atn_noe       = 19.391[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2.5[s]
Recvr_gain        = 76
Relaxation_delay  = 2.5[s]
Repetition_time   = 3.368352[s]
Temp_get          = 20.8[dC]
```



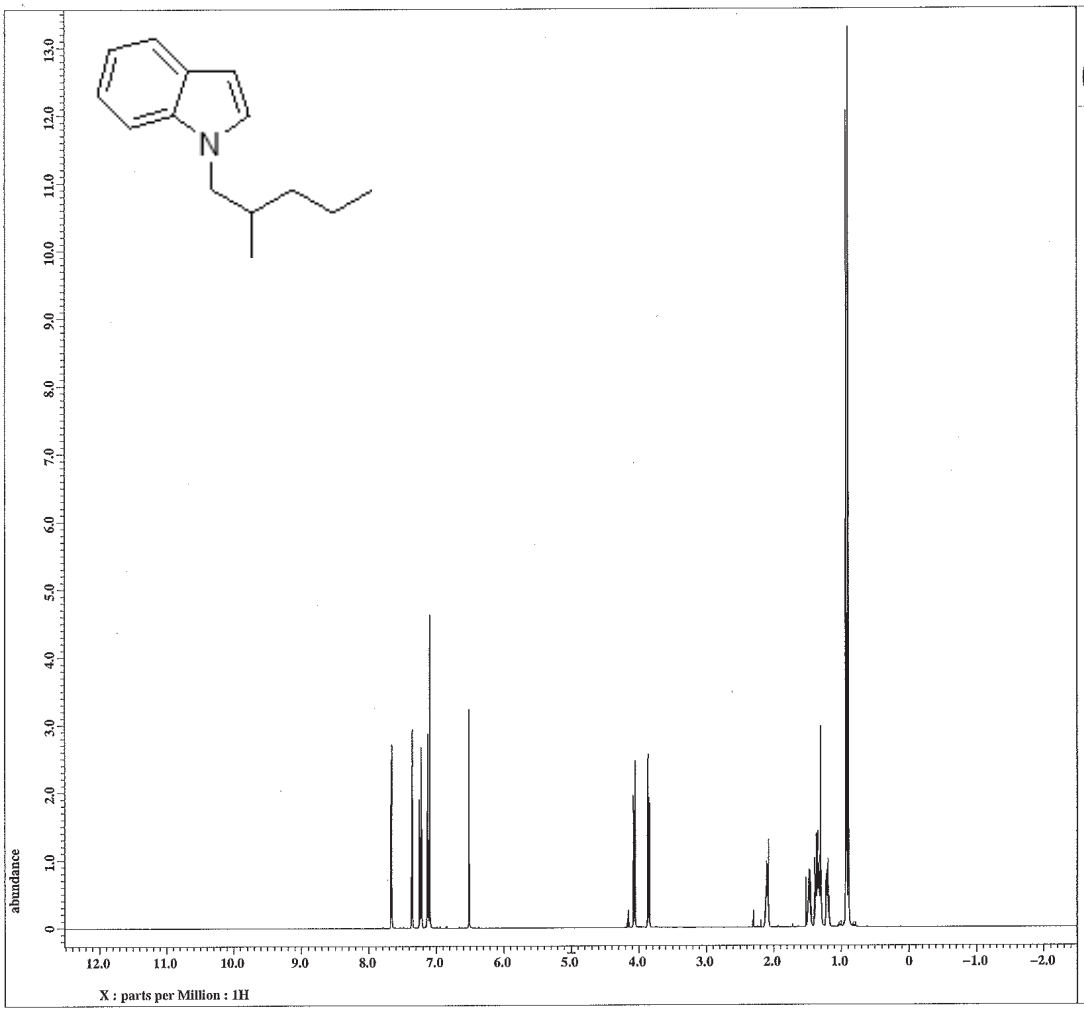
JEOL

Filename	= Exp-hakim-236-1a-prot
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= Exp-hakim-236-1a-prot
Solvent	= CHLOROFORM-D
Creation_time	= 10-DEC-2013 21:50:29
Revision_time	= 10-DEC-2013 22:04:20
Current_time	= 10-DEC-2013 22:04:25
Content	= Exp-hakim-236-1a-prot
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 600
Spectrometer	= DELTA2_NMR
Field_strength	= 14.09636928[T] (600[M
X_acq_duration	= 1.4548992[s]
X_domain	= 1H
X_freq	= 600.1723046[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.68733284[Hz]
X_sweep	= 11.26126126[kHz]
Irr_domain	= 1H
Irr_freq	= 600.1723046[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 600.1723046[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 13[us]
X_acq_time	= 1.4548992[s]
X_angle	= 45[deg]
X_atn	= 3.6[dB]
X_pulse	= 6.5[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 40
Relaxation_delay	= 5[s]
Repetition_time	= 6.4548992[s]
Temp_get	= 23.8[dC]

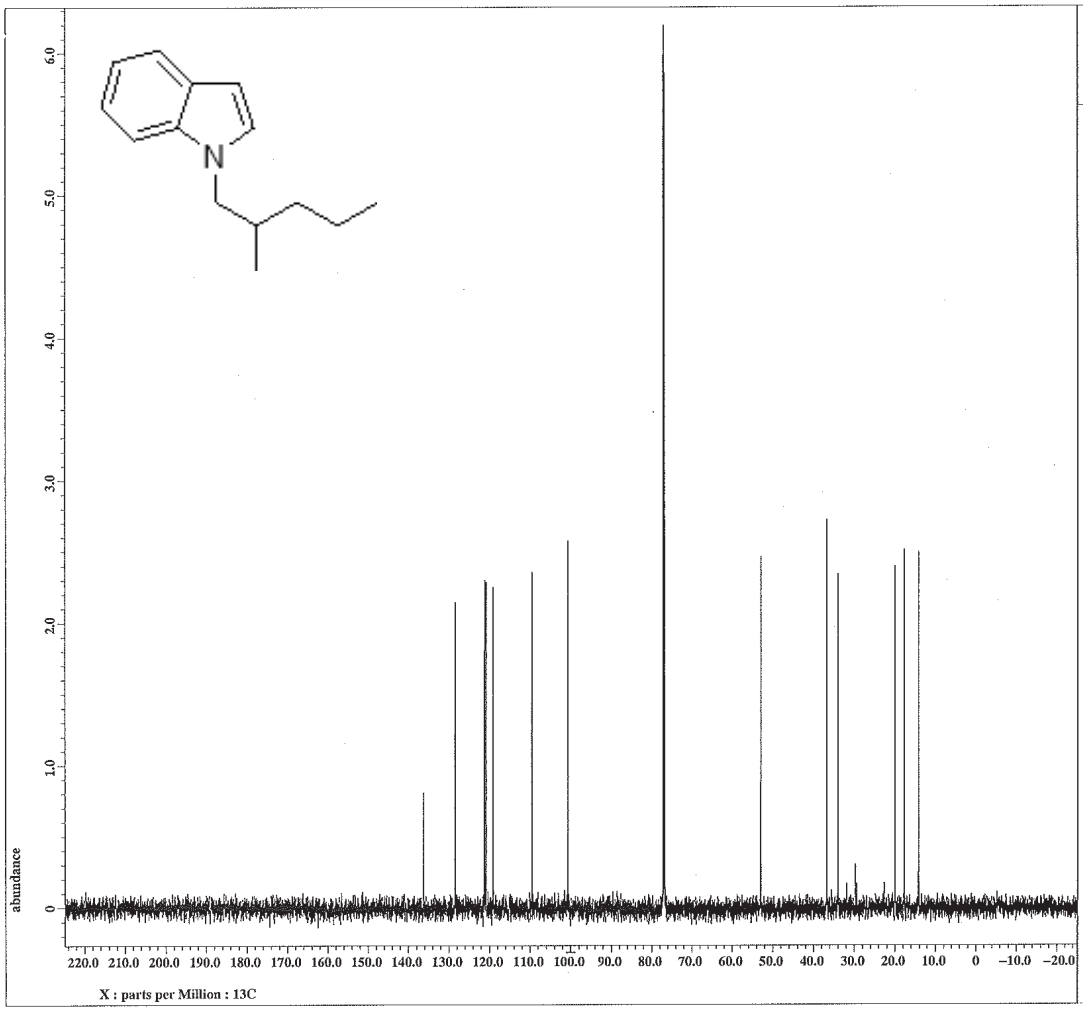


JEOL

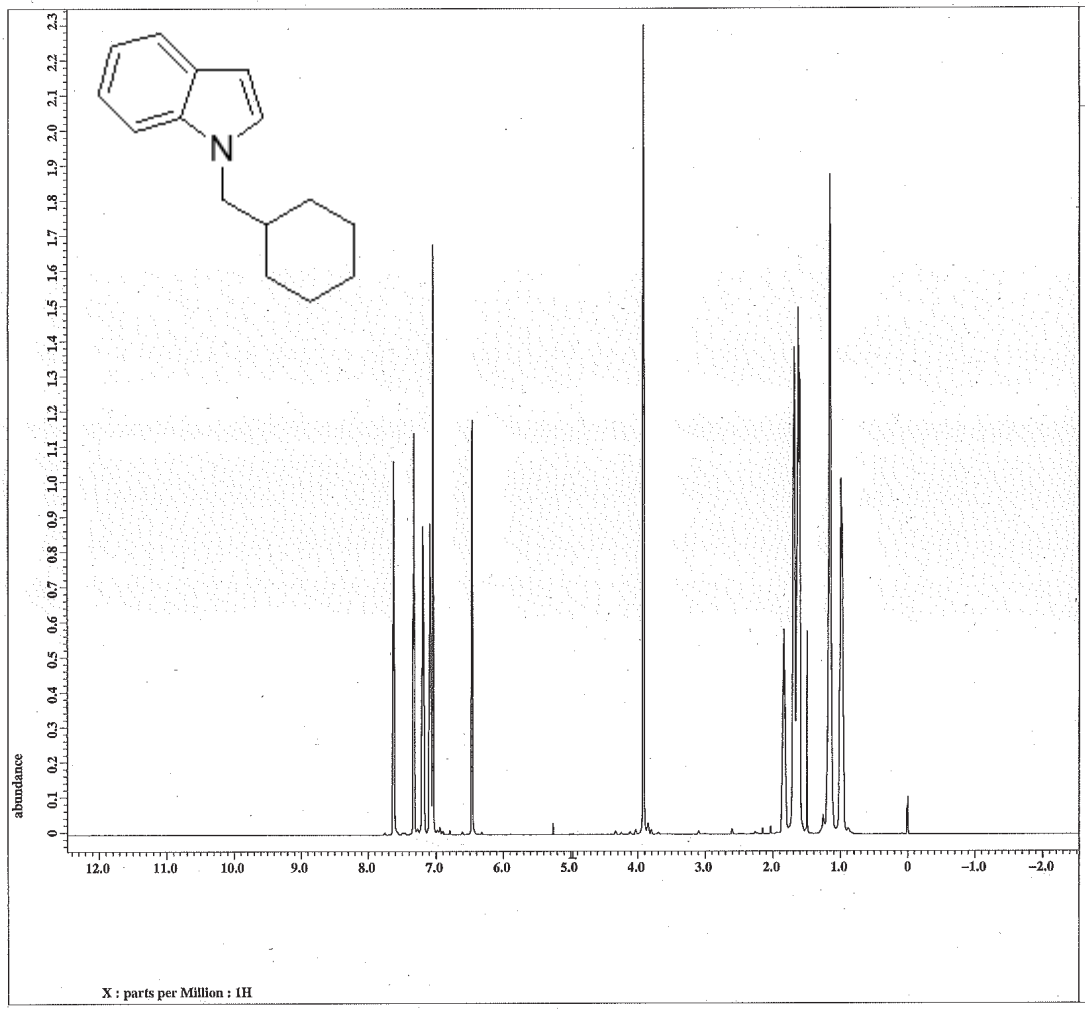
Filename	= Exp-hakim-236-1a-carb
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= Exp-hakim-236-1a-carb
Solvent	= CHLOROFORM-D
Creation_time	= 10-DEC-2013 21:54:49
Revision_time	= 10-DEC-2013 22:09:44
Current_time	= 10-DEC-2013 22:09:50
Content	= Exp-hakim-236-1a-carb
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 600
Spectrometer	= DELTA2_NMR
Field_strength	= 14.09636928[T] (600[M
X_acq_duration	= 0.868352[s]
X_domain	= 13C
X_freq	= 150.91343039[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 1.15160672[Hz]
X_sweep	= 37.73584906[kHz]
Irr_domain	= 1H
Irr_freq	= 600.1723046[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 58
Total_scans	= 58
X_90_width	= 11.4[us]
X_acq_time	= 0.868352[s]
X_angle	= 30[deg]
X_atn	= 7.5[dB]
X_pulse	= 3.8[us]
Irr_atn_dec	= 19.391[dB]
Irr_atn_noe	= 19.391[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2.5[s]
Recvr_gain	= 78
Relaxation_delay	= 2.5[s]
Repetition_time	= 3.368352[s]
Temp_get	= 24.4[dC]




```
Filename          = Exp-hakim-236-2a-prot
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-236-2a-prot
Solvent           = CHLOROFORM-D
Creation_time     = 10-DEC-2013 22:01:01
Revision_time     = 10-DEC-2013 22:14:38
Current_time      = 10-DEC-2013 22:14:42

Content           = Exp-hakim-236-2a-prot
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 1.4548992[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.68733284[Hz]
X_sweep           = 11.26126126[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 600.1723046[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 13[us]
X_acq_time        = 1.4548992[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 23.9[dC]
```

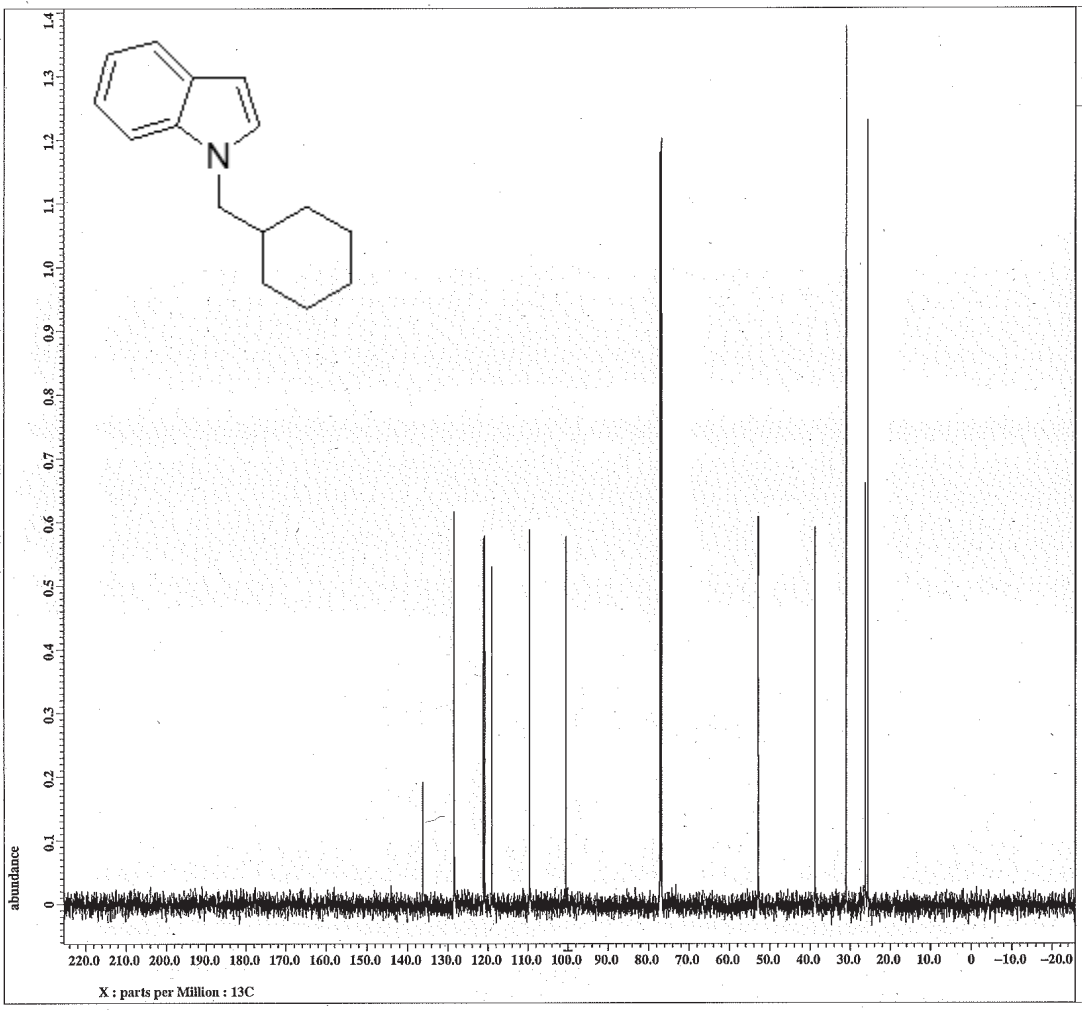



```
Filename          = Exp-hakim-236-2a-carb
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-236-2a-carb
Solvent           = CHLOROFORM-D
Creation_time     = 10-DEC-2013 22:05:51
Revision_time     = 16-DEC-2013 17:48:45
Current_time      = 16-DEC-2013 17:48:49

Content           = Exp-hakim-236-2a-carb
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.868352[s]
X_domain          = 13C
X_freq            = 150.91343039[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.15160672[Hz]
X_sweep           = 37.73584906[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 67
Total_scans       = 67

X_90_width        = 11.4[us]
X_acq_time        = 0.868352[s]
X_angle           = 30[deg]
X_atn             = 7.5[dB]
X_pulse           = 3.8[us]
Irr_atn_dec       = 19.391[dB]
Irr_atn_noe       = 19.391[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2.5[s]
Recvr_gain        = 78
Relaxation_delay  = 2.5[s]
Repetition_time   = 3.368352[s]
Temp_get          = 24.5[dC]
```



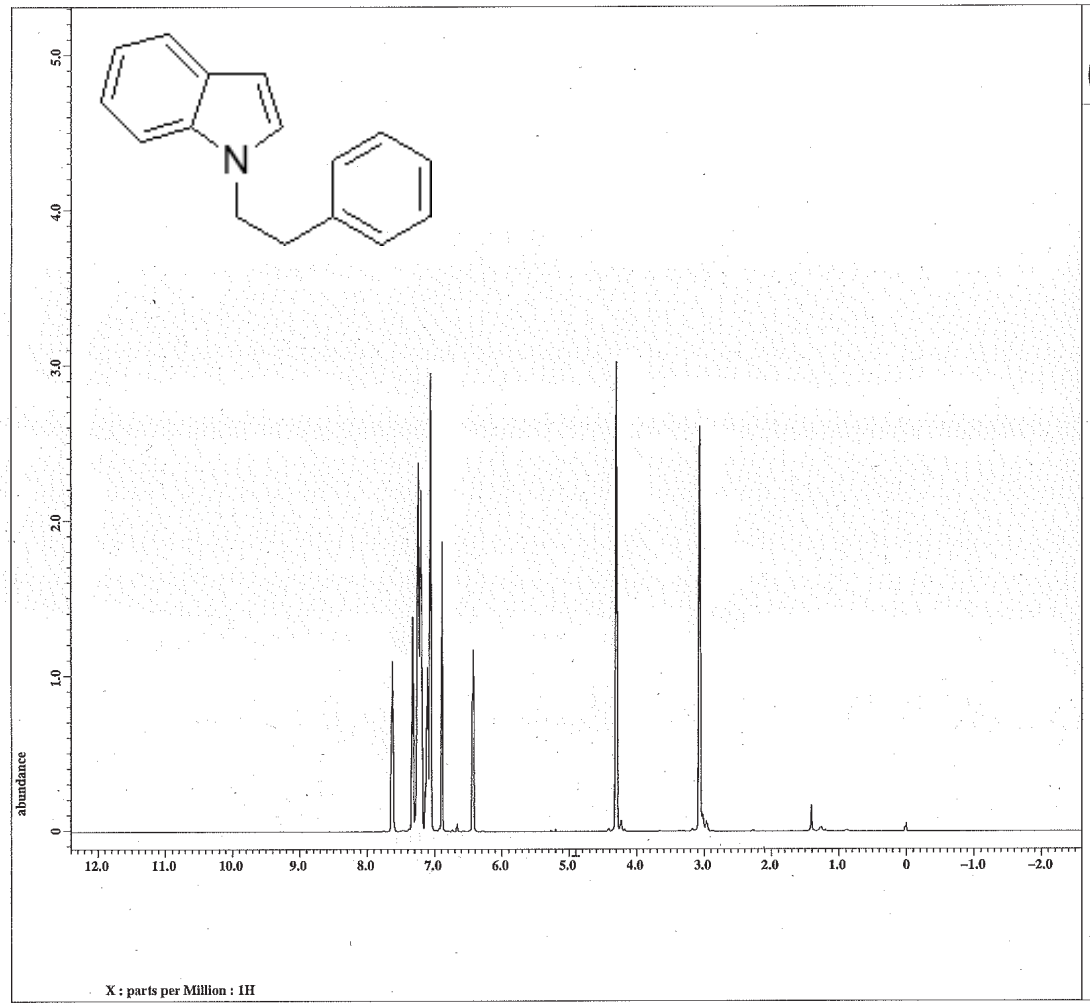
JEOL

Filename	= Exp-7-C-N-proton-5.jd
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= Exp-7-C-N-proton
Solvent	= CHLOROFORM-D
Creation_time	= 22-MAY-2013 09:57:08
Revision_time	= 22-MAY-2013 10:07:07
Current_time	= 22-MAY-2013 10:07:11
Content	= Exp-7-C-N-proton
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 600
Spectrometer	= DELTA2_NMR
Field_strength	= 14.09636928[T] (600[M
X_acq_duration	= 1.4548992[s]
X_domain	= 1H
X_freq	= 600.1723046[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.68733284[Hz]
X_sweep	= 11.26126126[kHz]
Irr_domain	= 1H
Irr_freq	= 600.1723046[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 600.1723046[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 12.4[us]
X_acq_time	= 1.4548992[s]
X_angle	= 45[deg]
X_atn	= 3.6[dB]
X_pulse	= 6.2[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 38
Relaxation_delay	= 5[s]
Repetition_time	= 6.4548992[s]
Temp_get	= 20[dC]



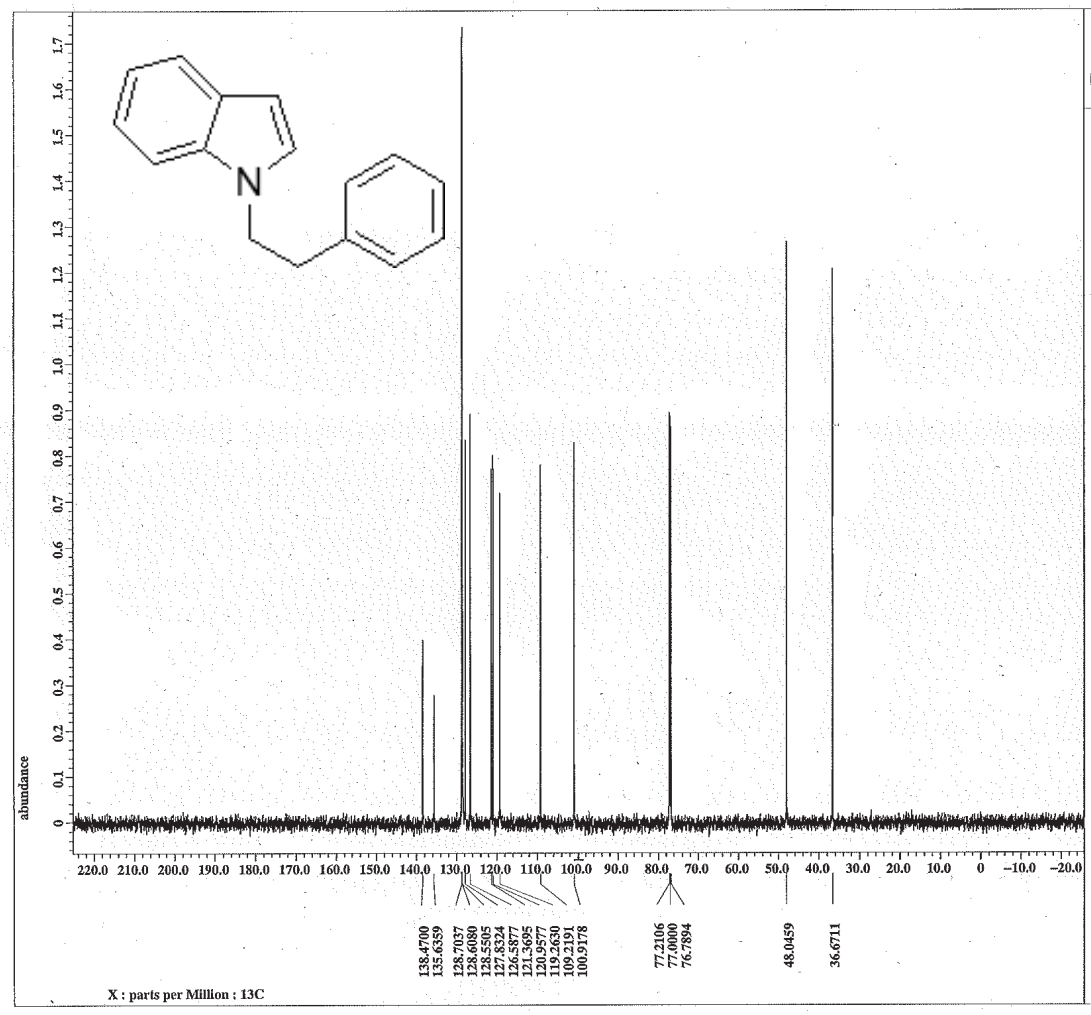
JEOL

Filename	= Exp-7-C-N-carbon-3.jd
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= Exp-7-C-N-carbon
Solvent	= CHLOROFORM-D
Creation_time	= 22-MAY-2013 10:02:11
Revision_time	= 22-MAY-2013 10:10:35
Current_time	= 22-MAY-2013 10:10:44
Content	= Exp-7-C-N-carbon
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 600
Spectrometer	= DELTA2_NMR
Field_strength	= 14.09636928[T] (600[M
X_acq_duration	= 0.69206016[s]
X_domain	= 13C
X_freq	= 150.91343039[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 1.44496109[Hz]
X_sweep	= 47.34848485[kHz]
Irr_domain	= 1H
Irr_freq	= 600.1723046[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 97
Total_scans	= 97
X_90_width	= 11.3[us]
X_acq_time	= 0.69206016[s]
X_angle	= 30[deg]
X_atn	= 8[dB]
X_pulse	= 3.76666667[us]
Irr_atn_dec	= 19.34784[dB]
Irr_atn_noe	= 19.34784[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 2.69206016[s]
Temp_get	= 20.5[dC]



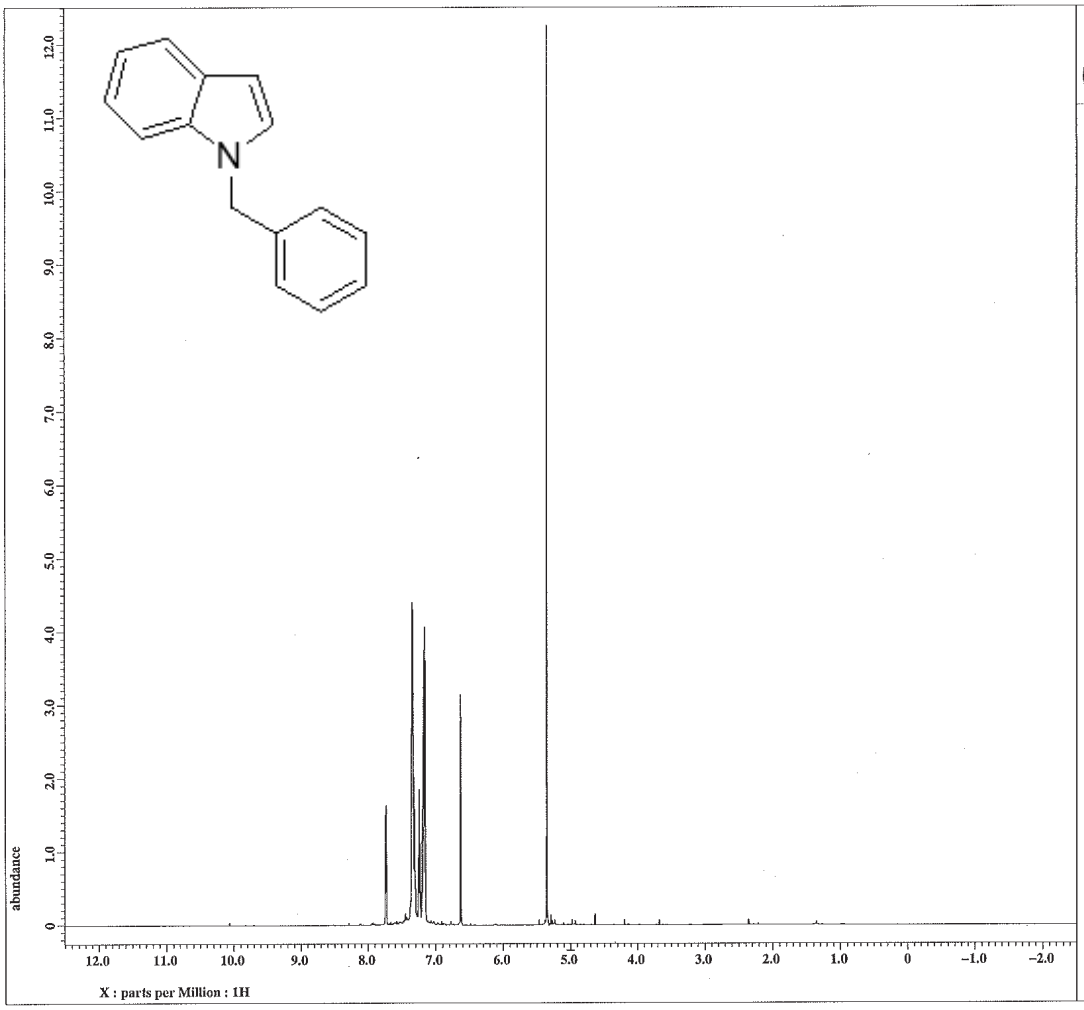
JEOL

Filename	= Exp-6-C-N-proton-5.jd
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= Exp-6-C-N-proton
Solvent	= CHLOROFORM-D
Creation_time	= 22-MAY-2013 09:43:27
Revision_time	= 22-MAY-2013 09:55:15
Current_time	= 22-MAY-2013 09:55:28
Content	= Exp-6-C-N-proton
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 600
Spectrometer	= DELTA2_NMR
Field_strength	= 14.09636928[T] (600[M
X_acq_duration	= 1.4548992[s]
X_domain	= 1H
X_freq	= 600.1723046[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.68733284[Hz]
X_sweep	= 11.26126126[kHz]
Irr_domain	= 1H
Irr_freq	= 600.1723046[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 600.1723046[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 12.4[us]
X_acq_time	= 1.4548992[s]
X_angle	= 45[deg]
X_atn	= 3.6[dB]
X_pulse	= 6.2[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 36
Relaxation_delay	= 5[s]
Repetition_time	= 6.4548992[s]
Temp_get	= 19.9[dC]



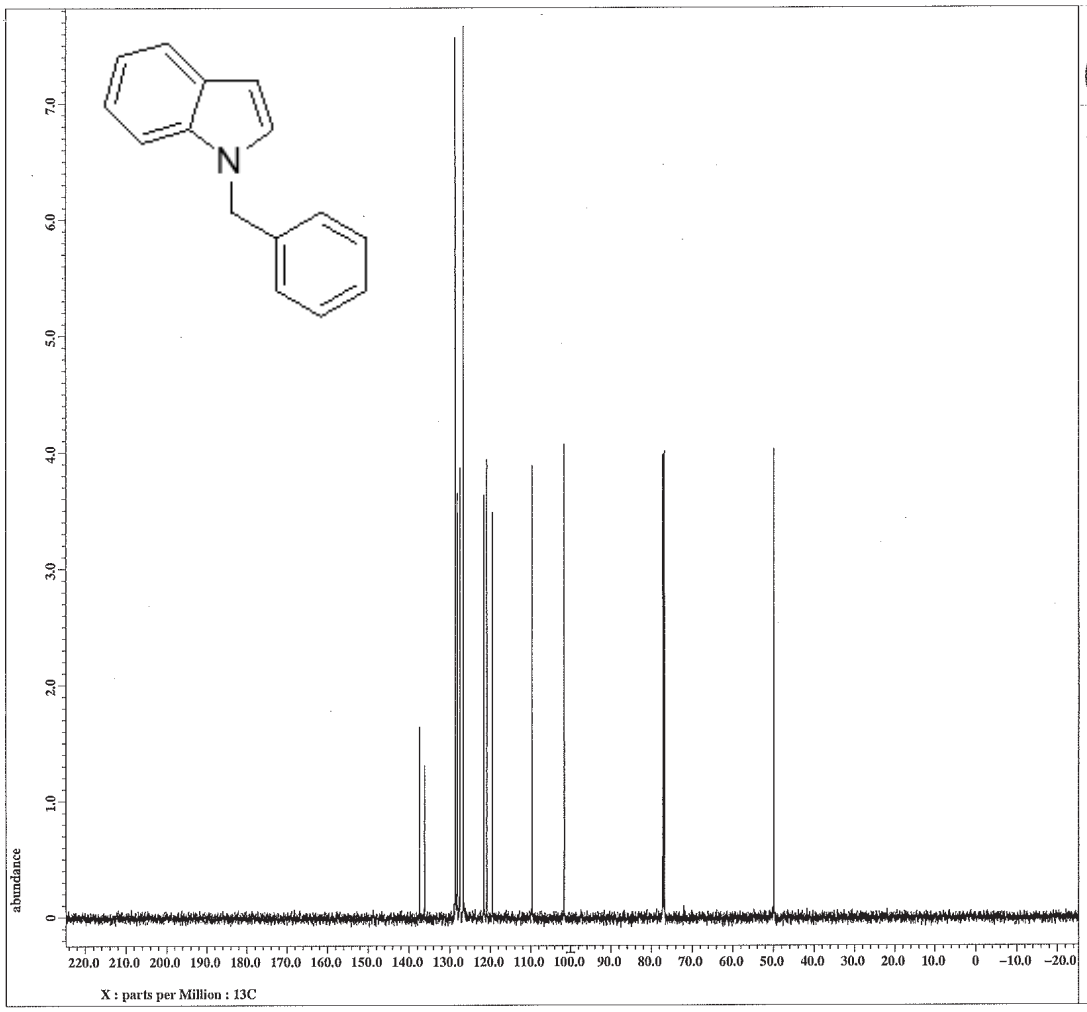
JEOL

Filename	= Exp-6-C-N-carbon-3.jd
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= Exp-6-C-N-carbon
Solvent	= CHLOROFORM-D
Creation_time	= 22-MAY-2013 09:49:49
Revision_time	= 22-MAY-2013 10:00:10
Current_time	= 22-MAY-2013 10:00:19
Content	= Exp-6-C-N-carbon
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 600
Spectrometer	= DELTA2_NMR
Field_strength	= 14.09636928[T] (600[M
X_acq_duration	= 0.69206016[s]
X_domain	= 13C
X_freq	= 150.91343039[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 1.44496109[Hz]
X_sweep	= 47.34848485[kHz]
Irr_domain	= 1H
Irr_freq	= 600.1723046[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 126
Total_scans	= 126
X_90_width	= 11.3[us]
X_acq_time	= 0.69206016[s]
X_angle	= 30[deg]
X_atn	= 8[dB]
X_pulse	= 3.76666667[us]
Irr_atn_dec	= 19.34784[dB]
Irr_atn_noe	= 19.34784[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 2.69206016[s]
Temp_get	= 21[dC]



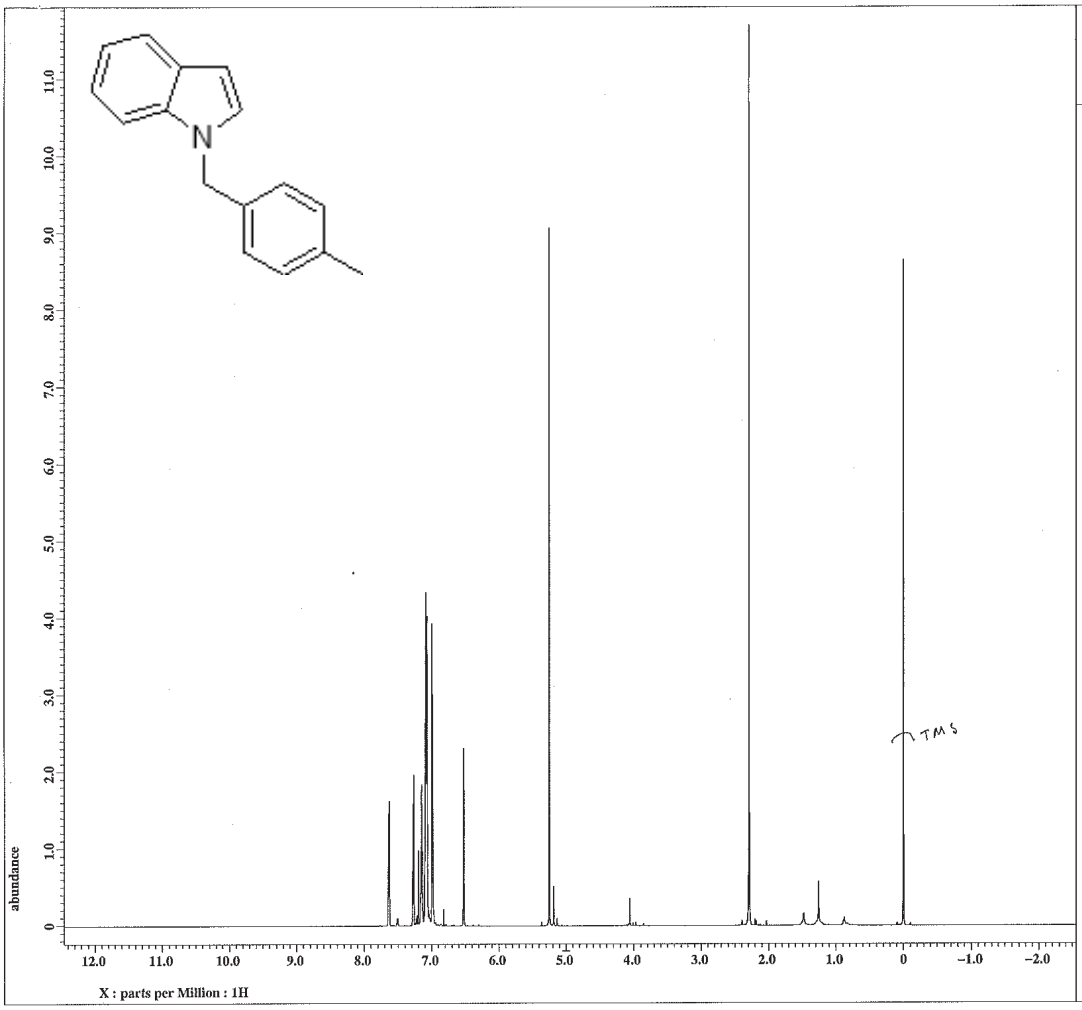
JEOL

Filename	= Exp-hakim-C-N-benz-pr
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= Exp-hakim-C-N-benz-pr
Solvent	= CHLOROFORM-D
Creation_time	= 6-NOV-2013 14:54:02
Revision_time	= 6-NOV-2013 15:05:21
Current_time	= 6-NOV-2013 15:05:25
Content	= Exp-hakim-C-N-benz-pr
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 600
Spectrometer	= DELTA2_NMR
Field_strength	= 14.09636928[T] (600[M
X_acq_duration	= 1.4548992[s]
X_domain	= 1H
X_freq	= 600.1723046[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.68733284[Hz]
X_sweep	= 11.26126126[kHz]
Irr_domain	= 1H
Irr_freq	= 600.1723046[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 600.1723046[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 13[us]
X_acq_time	= 1.4548992[s]
X_angle	= 45[deg]
X_atn	= 3.6[dB]
X_pulse	= 6.5[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 36
Relaxation_delay	= 5[s]
Repetition_time	= 6.4548992[s]
Temp_get	= 19.8[dC]

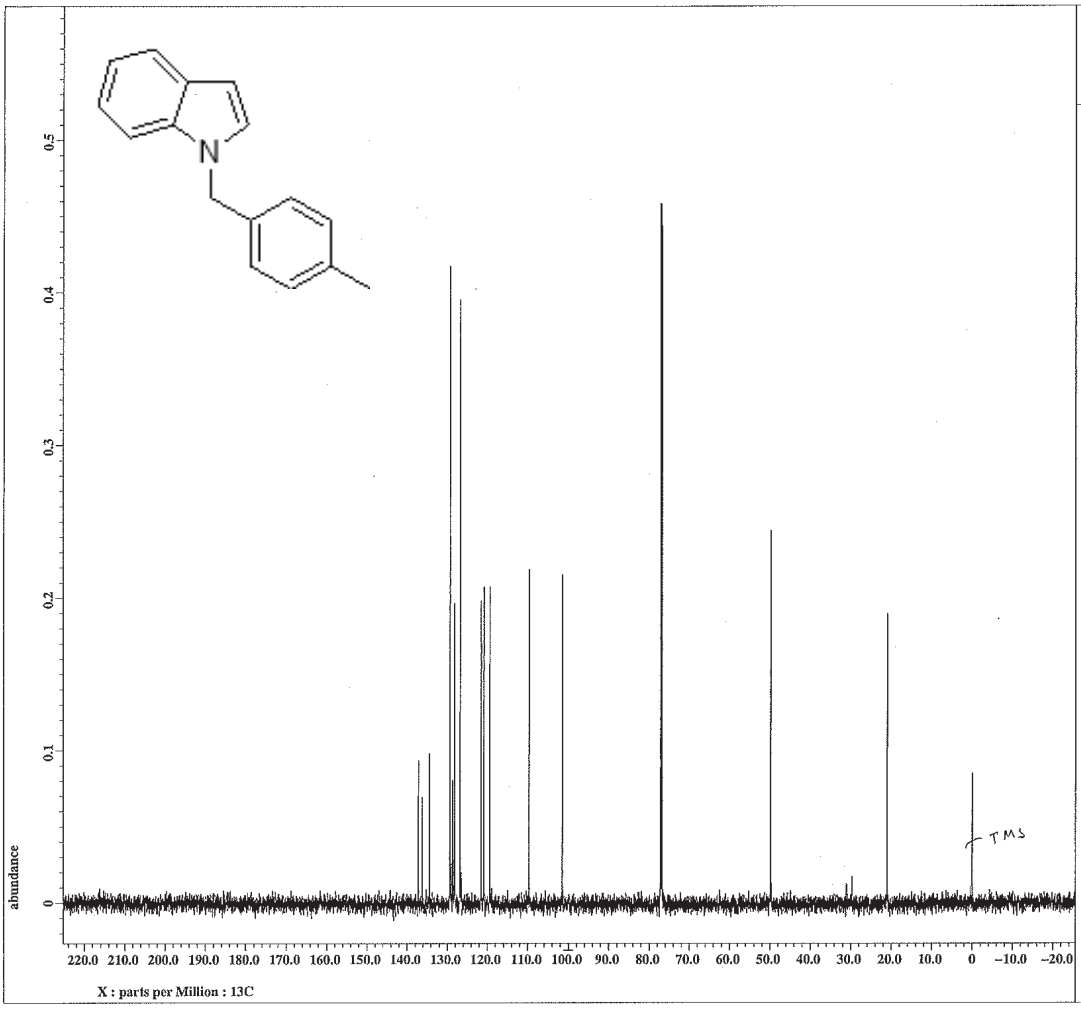


JEOL

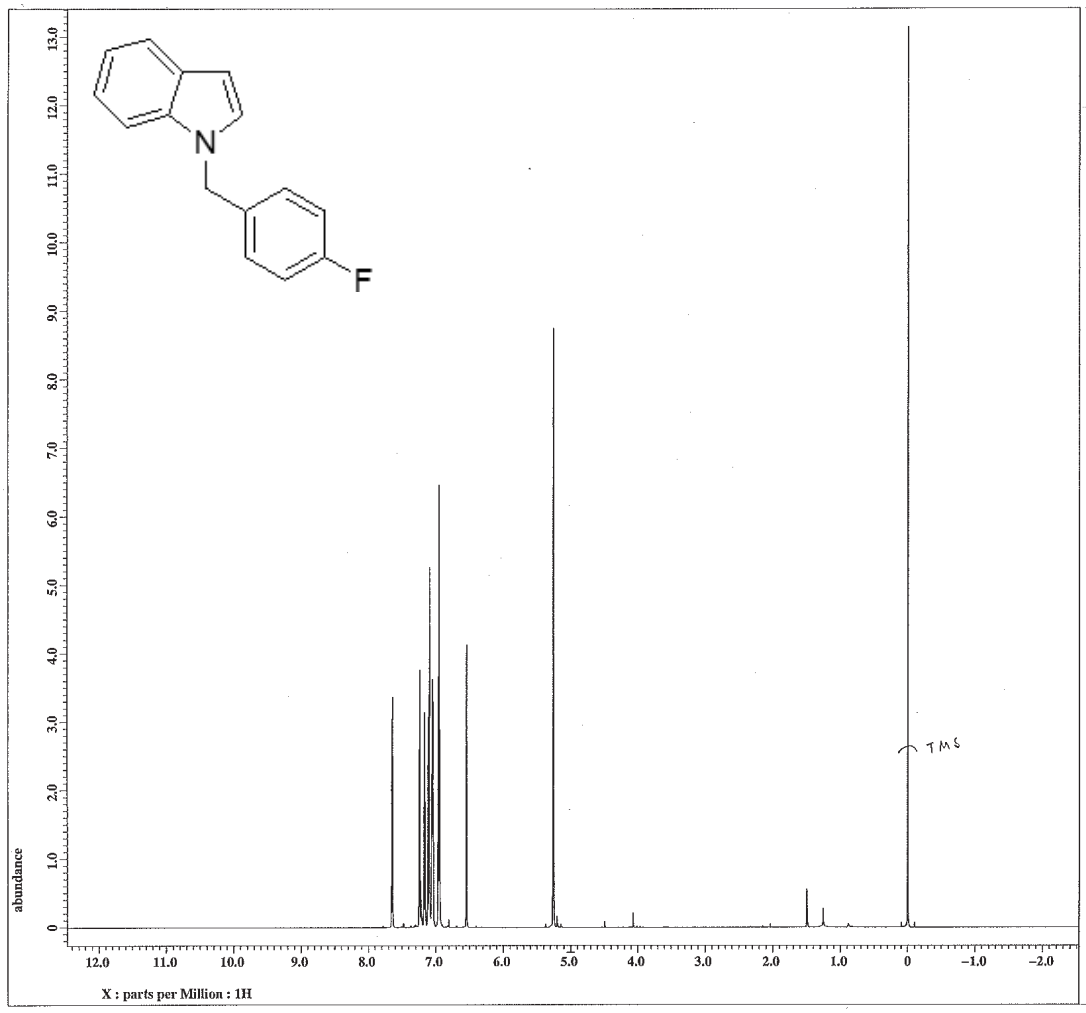
Filename	= Exp-hakim-C-N-benz-ca
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= Exp-hakim-C-N-benz-ca
Solvent	= CHLOROFORM-D
Creation_time	= 6-NOV-2013 15:00:19
Revision_time	= 16-DEC-2013 17:47:56
Current_time	= 16-DEC-2013 17:48:00
Content	= Exp-hakim-C-N-benz-ca
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 600
Spectrometer	= DELTA2_NMR
Field_strength	= 14.09636928[T] (600[M
X_acq_duration	= 0.868352[s]
X_domain	= 13C
X_freq	= 150.91343039[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 1.15160672[Hz]
X_sweep	= 37.73584906[kHz]
Irr_domain	= 1H
Irr_freq	= 600.1723046[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 95
Total_scans	= 95
X_90_width	= 11.4[us]
X_acq_time	= 0.868352[s]
X_angle	= 30[deg]
X_atn	= 7.5[dB]
X_pulse	= 3.8[us]
Irr_atn_dec	= 19.391[dB]
Irr_atn_noe	= 19.391[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2.5[s]
Recvr_gain	= 76
Relaxation_delay	= 2.5[s]
Repetition_time	= 3.368352[s]
Temp_get	= 20.4[dC]



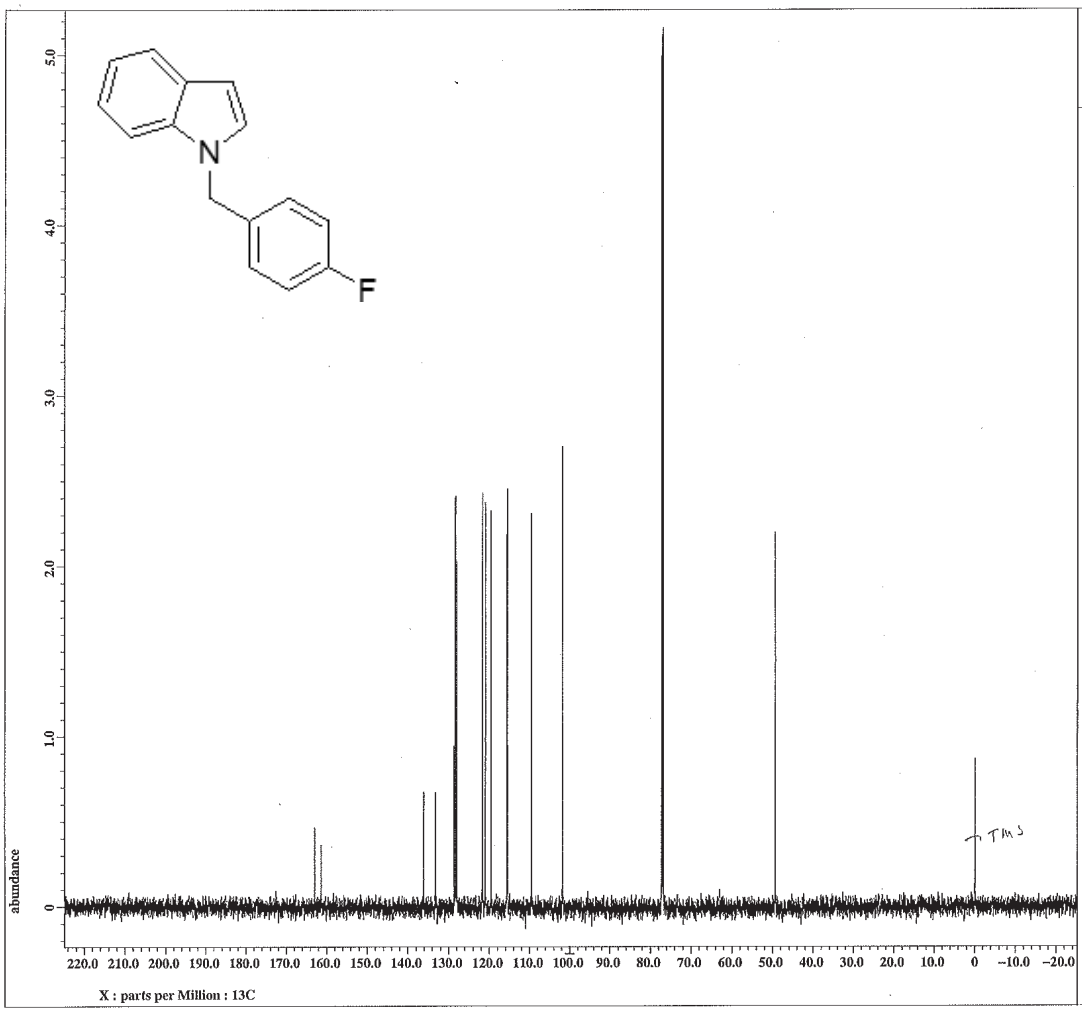

```
Filename            = Exp-hakim-307-1b-prot
Author              = delta
Experiment          = single_pulse.ex2
Sample_id           = Exp-hakim-307-1b-prot
Solvent             = CHLOROFORM-D
Creation_time       = 14-MAY-2014 13:22:34
Revision_time       = 14-MAY-2014 13:42:27
Current_time        = 14-MAY-2014 13:42:31

Content             = Exp-hakim-307-1b-prot
Data_format         = 1D COMPLEX
Dim_size            = 13107
Dim_title           = 1H
Dim_units           = [ppm]
Dimensions          = X
Site                = ECA 600
Spectrometer        = DELTA2_NMR

Field_strength      = 14.09636928[T] (600[M
X_acq_duration      = 1.4548992[s]
X_domain            = 1H
X_freq              = 600.1723046[MHz]
X_offset            = 5[ppm]
X_points            = 16384
X_prescans          = 1
X_resolution        = 0.68733284[Hz]
X_sweep             = 11.26126126[kHz]
Irr_domain          = 1H
Irr_freq            = 600.1723046[MHz]
Irr_offset          = 5[ppm]
Tri_domain          = 1H
Tri_freq            = 600.1723046[MHz]
Tri_offset          = 5[ppm]
Clipped             = FALSE
Mod_return          = 1
Scans               = 8
Total_scans         = 8

X_90_width          = 13[us]
X_acq_time          = 1.4548992[s]
X_angle             = 45[deg]
X_atn               = 3.6[dB]
X_pulse             = 6.5[us]
Irr_mode            = Off
Tri_mode            = Off
Dante_presat        = FALSE
Initial_wait        = 1[s]
Recvr_gain          = 40
Relaxation_delay    = 5[s]
Repetition_time     = 6.4548992[s]
Temp_get            = 20.8[dC]
```



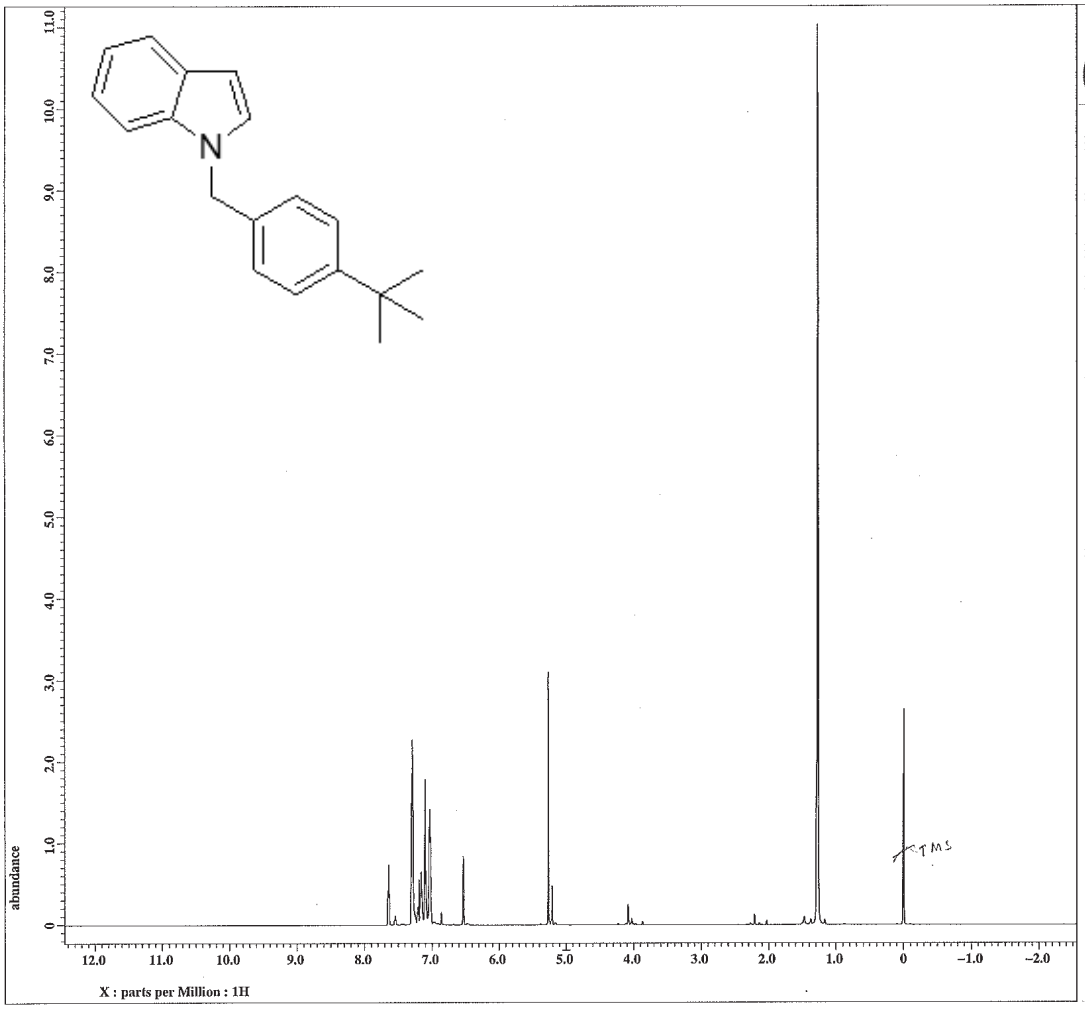

```
Filename            = Exp-hakim-307-1a-carb
Author              = delta
Experiment          = single_pulse_dec
Sample_id           = Exp-hakim-307-1a-carb
Solvent             = CHLOROFORM-D
Creation_time       = 13-MAY-2014 18:04:34
Revision_time       = 13-MAY-2014 18:24:48
Current_time        = 13-MAY-2014 18:25:09

Content             = Exp-hakim-307-1a-carb
Data_format         = 1D COMPLEX
Dim_size            = 26214
Dim_title           = 13C
Dim_units           = [ppm]
Dimensions          = X
Site                = ECA 600
Spectrometer        = DELTA2_NMR

Field_strength      = 14.09636928[T] (600[M
X_acq_duration      = 0.69206016[s]
X_domain            = 13C
X_freq              = 150.91343039[MHz]
X_offset            = 100[ppm]
X_points            = 32768
X_prescans          = 4
X_resolution        = 1.44496109[Hz]
X_sweep             = 47.34848485[kHz]
Irr_domain          = 1H
Irr_freq            = 600.1723046[MHz]
Irr_offset          = 5[ppm]
Clipped             = FALSE
Mod_return          = 1
Scans               = 90
Total_scans         = 90

X_90_width          = 11.4[us]
X_acq_time          = 0.69206016[s]
X_angle             = 30[deg]
X_atn               = 7.5[dB]
X_pulse             = 3.8[us]
Irr_atn_dec         = 19.391[dB]
Irr_atn_noe         = 19.391[dB]
Irr_noise           = WALTZ
Decoupling          = TRUE
Initial_wait        = 1[s]
Noe                 = TRUE
Noe_time            = 2[s]
Recvr_gain          = 50
Relaxation_delay    = 2[s]
Repetition_time     = 2.69206016[s]
Temp_get            = 21.4[dC]
```



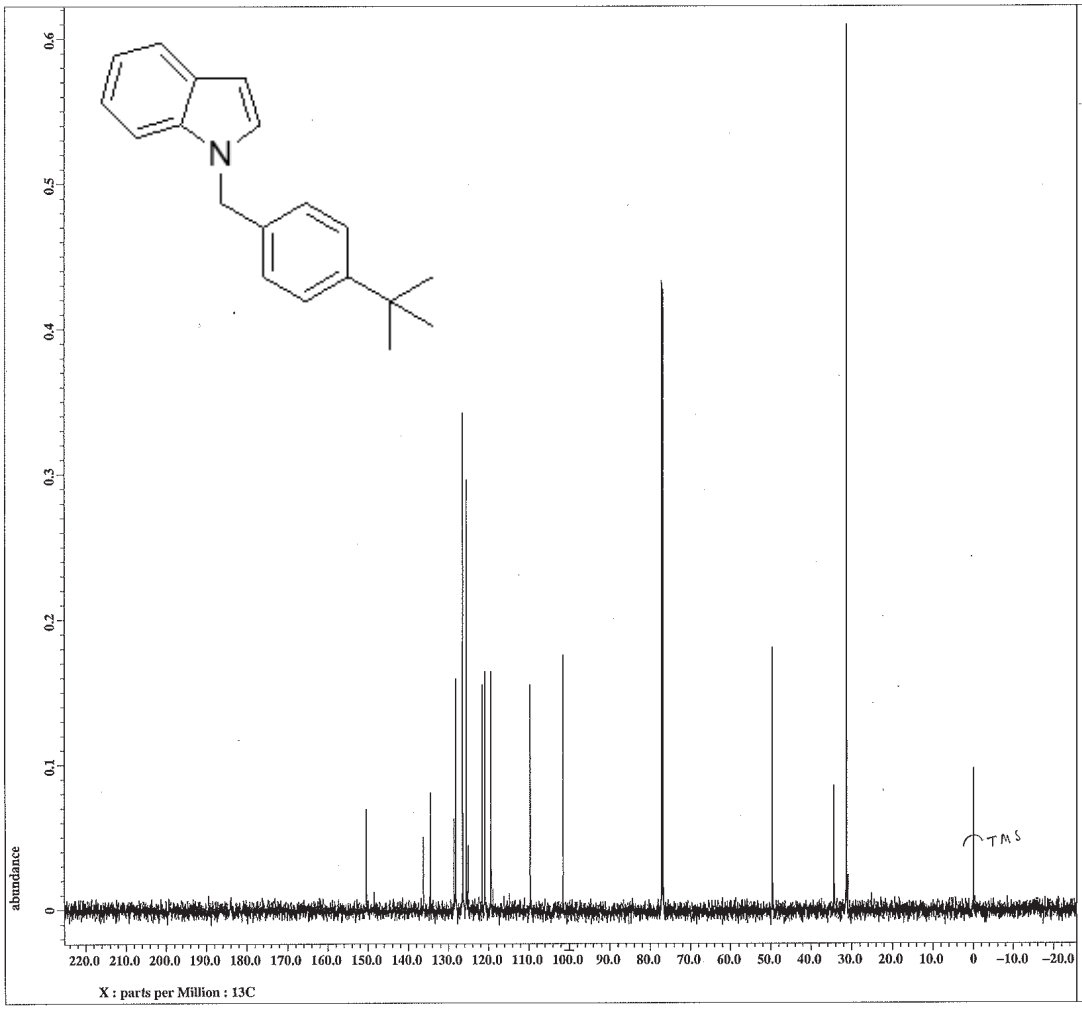

```
Filename          = Exp-hakim-307-3-proto
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-307-3-proto
Solvent           = CHLOROFORM-D
Creation_time     = 13-MAY-2014 14:07:09
Revision_time     = 13-MAY-2014 14:26:20
Current_time      = 13-MAY-2014 14:26:22

Content           = Exp-hakim-307-3-proto
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 1.4548992[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.68733284[Hz]
X_sweep           = 11.26126126[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 600.1723046[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 13[us]
X_acq_time        = 1.4548992[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 42
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 20.7[dC]
```



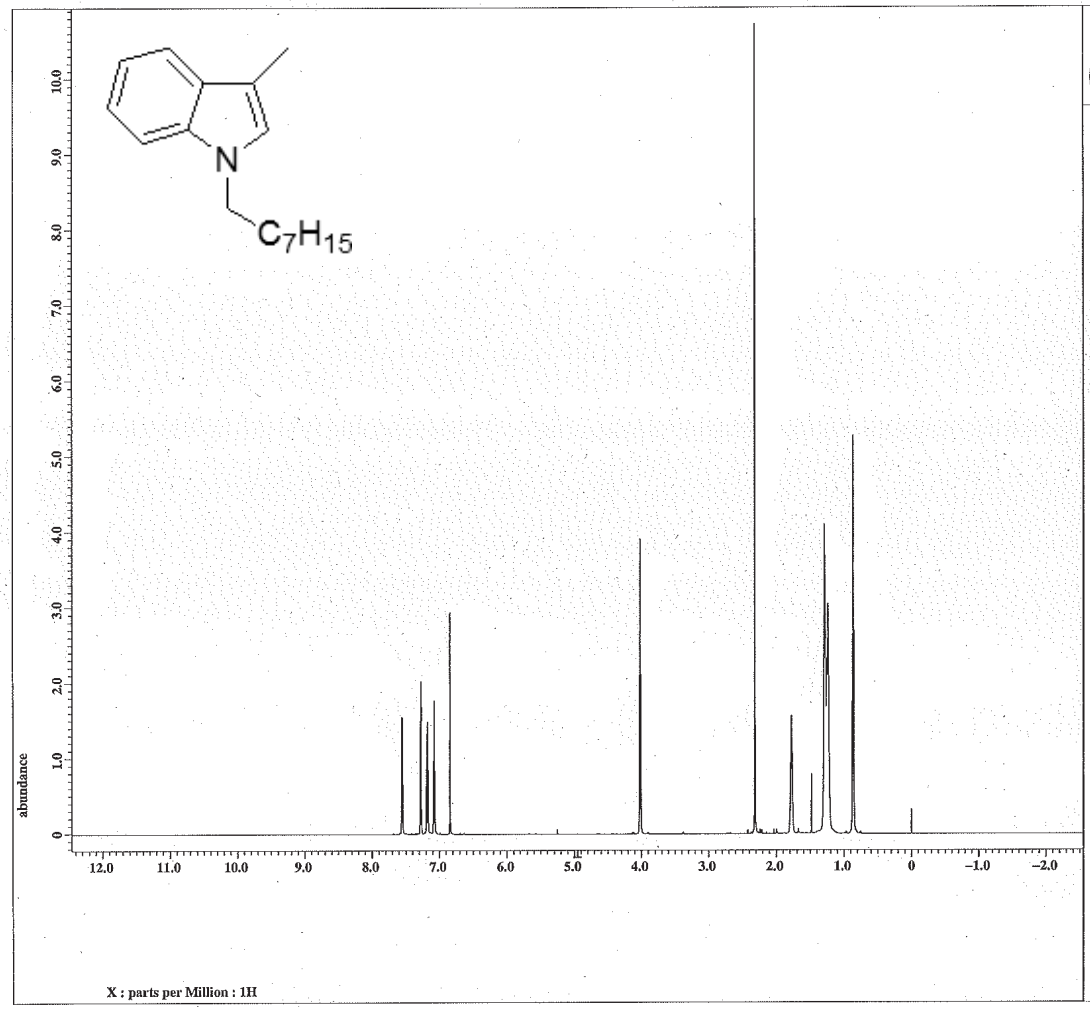

```
Filename          = Exp-hakim-307-3-carbo
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-307-3-carbo
Solvent           = CHLOROFORM-D
Creation_time     = 13-MAY-2014 14:13:26
Revision_time     = 13-MAY-2014 14:31:57
Current_time      = 13-MAY-2014 14:32:01

Content           = Exp-hakim-307-3-carbo
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.868352[s]
X_domain          = 13C
X_freq            = 150.91343039[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.15160672[Hz]
X_sweep           = 37.73584906[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 93
Total_scans       = 93

X_90_width        = 11.4[us]
X_acq_time        = 0.868352[s]
X_angle           = 30[deg]
X_atn             = 7.5[dB]
X_pulse           = 3.8[us]
Irr_atn_dec       = 19.391[dB]
Irr_atn_noe       = 19.391[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2.5[s]
Recvr_gain        = 78
Relaxation_delay  = 2.5[s]
Repetition_time   = 3.368352[s]
Temp_get          = 21.5[dC]
```



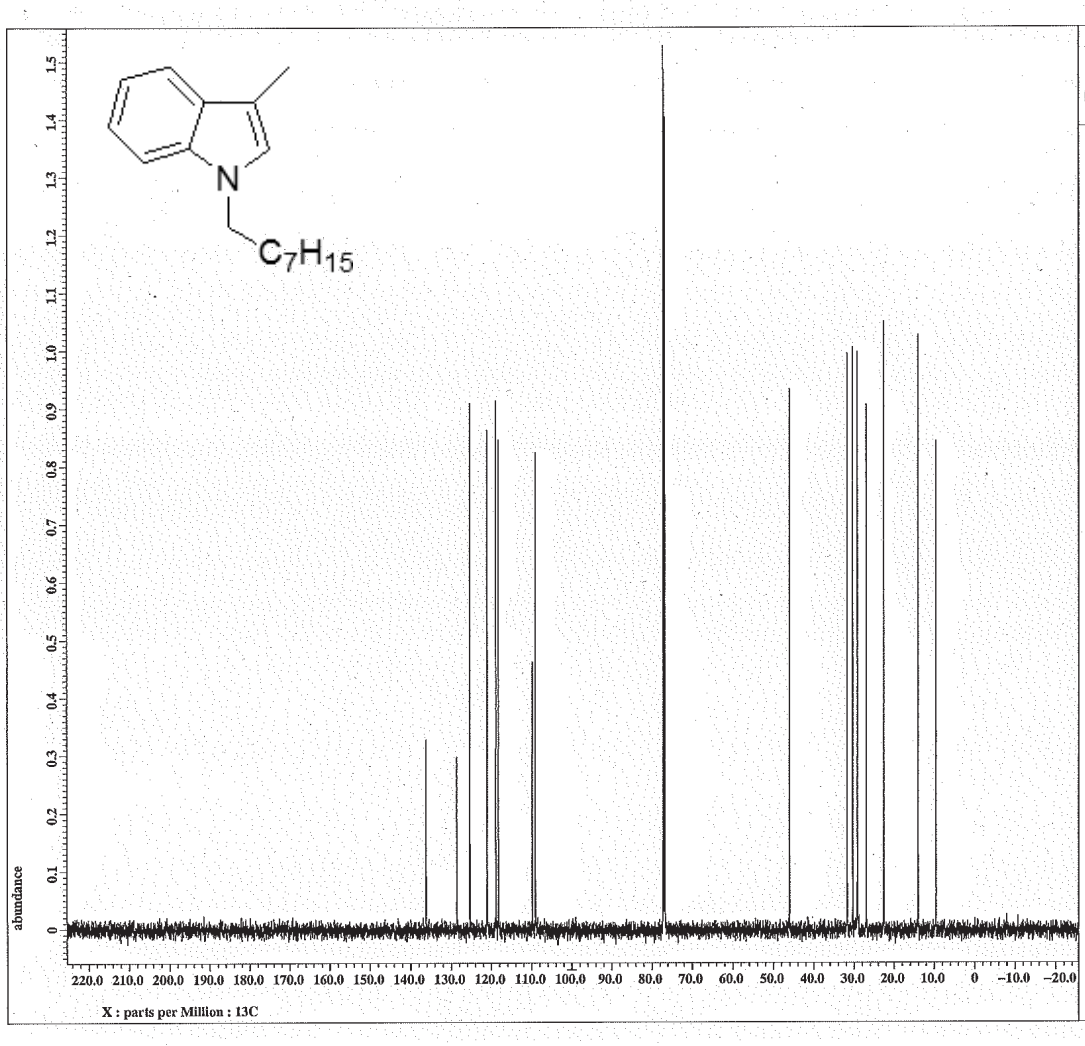

```
Filename          = Exp-hakim-307-3b-prot
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-hakim-307-3b-prot
Solvent           = CHLOROFORM-D
Creation_time     = 14-MAY-2014 13:29:08
Revision_time     = 14-MAY-2014 14:40:35
Current_time      = 14-MAY-2014 14:40:39

Content           = Exp-hakim-307-3b-prot
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 1.4548992[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.68733284[Hz]
X_sweep           = 11.26126126[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 600.1723046[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 13[us]
X_acq_time        = 1.4548992[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.5[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 20.8[dC]
```



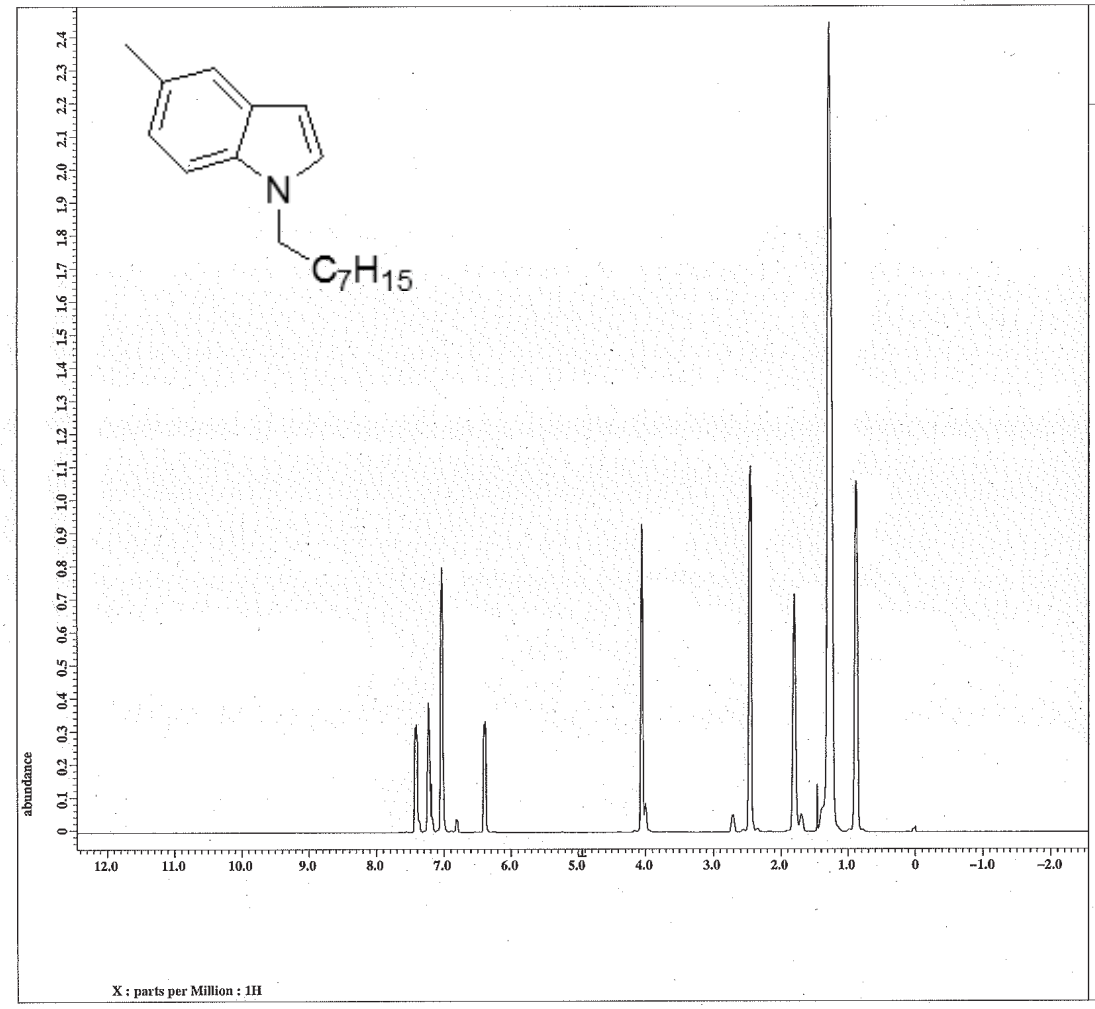

```
Filename          = Exp-hakim-307-3a-carb
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-hakim-307-3a-carb
Solvent           = CHLOROFORM-D
Creation_time     = 13-MAY-2014 18:38:36
Revision_time     = 13-MAY-2014 18:56:48
Current_time      = 13-MAY-2014 18:56:52

Content           = Exp-hakim-307-3a-carb
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.69206016[s]
X_domain          = 13C
X_freq            = 150.91343039[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.44496109[Hz]
X_sweep           = 47.34848485[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 76
Total_scans       = 76

X_90_width        = 11.4[us]
X_acq_time        = 0.69206016[s]
X_angle           = 30[deg]
X_atn             = 7.5[dB]
X_pulse           = 3.8[us]
Irr_atn_dec       = 19.391[dB]
Irr_atn_noe       = 19.391[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 50
Relaxation_delay  = 2[s]
Repetition_time   = 2.69206016[s]
Temp_get          = 21.2[dC]
```




```
Filename          = Exp-8-C-N-proton-5.jd
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = Exp-8-C-N-proton
Solvent           = CHLOROFORM-D
Creation_time     = 22-MAY-2013 10:07:54
Revision_time     = 22-MAY-2013 10:21:57
Current_time      = 22-MAY-2013 10:21:59

Content           = Exp-8-C-N-proton
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 1.4548992[s]
X_domain          = 1H
X_freq            = 600.1723046[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.68733284[Hz]
X_sweep           = 11.26126126[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 600.1723046[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 12.4[us]
X_acq_time        = 1.4548992[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.2[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 5[s]
Repetition_time   = 6.4548992[s]
Temp_get          = 20.4[dC]
```

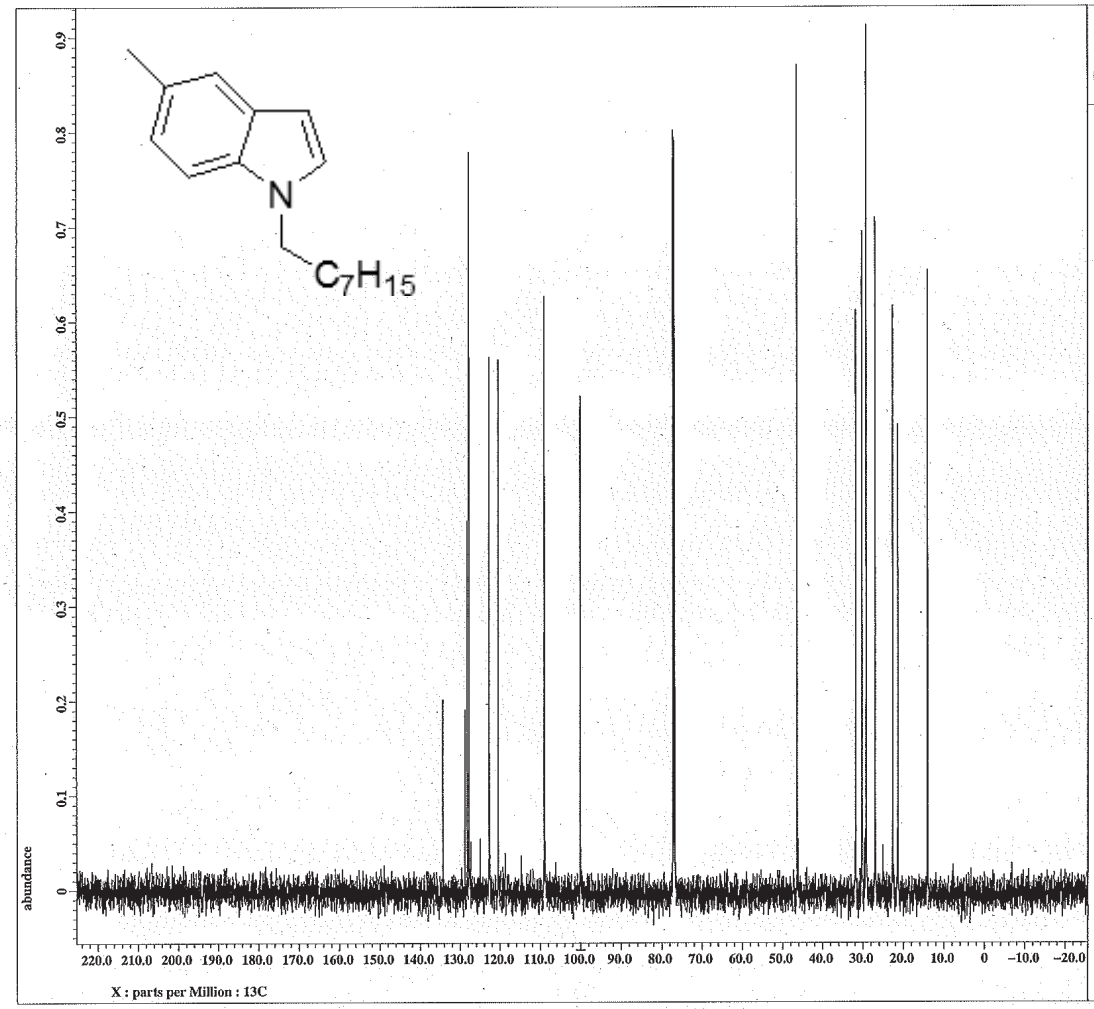



```
Filename          = Exp-8-C-N-carbon-3.jd
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Exp-8-C-N-carbon
Solvent           = CHLOROFORM-D
Creation_time     = 22-MAY-2013 10:15:29
Revision_time     = 22-MAY-2013 10:23:57
Current_time      = 22-MAY-2013 10:24:03

Content           = Exp-8-C-N-carbon
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 600
Spectrometer      = DELTA2_NMR

Field_strength    = 14.09636928[T] (600[M
X_acq_duration    = 0.69206016[s]
X_domain          = 13C
X_freq            = 150.91343039[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.44496109[Hz]
X_sweep           = 47.34848485[kHz]
Irr_domain        = 1H
Irr_freq          = 600.1723046[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 153
Total_scans       = 153

X_90_width        = 11.3[us]
X_acq_time        = 0.69206016[s]
X_angle           = 30[deg]
X_atn             = 8[dB]
X_pulse           = 3.76666667[us]
Irr_atn_dec       = 19.34784[dB]
Irr_atn_noe       = 19.34784[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 2.69206016[s]
Temp_get          = 20.8[dC]
```



JEOL

Filename	= Exp-9-C-N-proton-5.jd
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= Exp-9-C-N-proton
Solvent	= CHLOROFORM-D
Creation_time	= 22-MAY-2013 10:21:08
Revision_time	= 22-MAY-2013 10:30:32
Current_time	= 22-MAY-2013 10:30:39
Content	= Exp-9-C-N-proton
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 600
Spectrometer	= DELTA2_NMR
Field_strength	= 14.09636928[T] (600[M
X_acq_duration	= 1.4548992[s]
X_domain	= 1H
X_freq	= 600.1723046[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.68733284[Hz]
X_sweep	= 11.26126126[kHz]
Irr_domain	= 1H
Irr_freq	= 600.1723046[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 600.1723046[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 12.4[us]
X_acq_time	= 1.4548992[s]
X_angle	= 45[deg]
X_atn	= 3.6[dB]
X_pulse	= 6.2[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 30
Relaxation_delay	= 5[s]
Repetition_time	= 6.4548992[s]
Temp_get	= 20.2[dC]



JEOL

Filename	= Exp-9-C-N-carbon-3.jd
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= Exp-9-C-N-carbon
Solvent	= CHLOROFORM-D
Creation_time	= 22-MAY-2013 10:25:50
Revision_time	= 22-MAY-2013 10:34:41
Current_time	= 22-MAY-2013 10:34:47
Content	= Exp-9-C-N-carbon
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 600
Spectrometer	= DELTA2_NMR
Field_strength	= 14.09636928[T] (600[M
X_acq_duration	= 0.69206016[s]
X_domain	= 13C
X_freq	= 150.91343039[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 1.44496109[Hz]
X_sweep	= 47.34848485[kHz]
Irr_domain	= 1H
Irr_freq	= 600.1723046[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 89
Total_scans	= 89
X_90_width	= 11.3[us]
X_acq_time	= 0.69206016[s]
X_angle	= 30[deg]
X_atn	= 8[dB]
X_pulse	= 3.76666667[us]
Irr_atn_dec	= 19.34784[dB]
Irr_atn_noe	= 19.34784[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 2.69206016[s]
Temp_get	= 20.7[dC]