

Highly Selective Cascade Couplings for the Syntheses of Functionalized Piperidinones and Bispidines

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Supporting Information

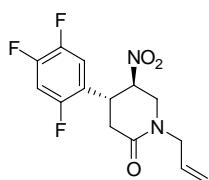
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General. Unless otherwise noted, all reactions were conducted under N₂ atmosphere using standard air-free manipulation techniques. Solvents were purchased from Fisher Scientific Company or Aldrich and used without further purification. Commercial reagents were purchased either from Aldrich and used without further purification. Chiral substrates **4** were prepared according to Deng's or Evan's procedures¹.

High performance liquid chromatography (HPLC) analysis was performed using Agilent Technology 1100 series instrument with YMC Pack Pro C18 (240 x 4.6 mm I.D., 5 μm particle size) column. Proton nuclear magnetic resonance (¹H NMR) spectra were measured on Bruker Avance-400 or 500 instrument (400 or 500 MHz). Carbon nuclear magnetic resonance (¹³C NMR) spectra were measured on Bruker Avance-400 or 500 instrument (100 or 125 MHz) with complete proton decoupling. Chemical shifts are reported in ppm downfield from tetramethylsilane (TMS).

1. Procedure for the one-pot preparation of *trans* pyrrolidinones 1

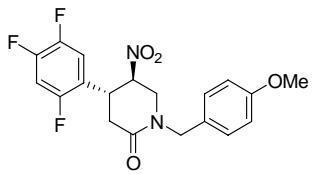


(4R,5R)-1-allyl-5-nitro-4-(2,4,5-trifluorophenyl)piperidin-2-one (1a):

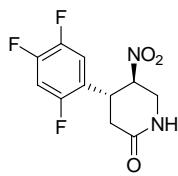
To a solution of nitro malonate **4a** (100 g, 0.2983 mol) and allyl amine (22.14 g, 0.3878 mol) in iPrOH (500 mL) and water (100 mL) at 50 °C was added 37 wt% HCHO (25.42 g, 0.3132 mol) dropwise over 1-2 h and the reaction solution was stirred for additional 2-3 h at 50 °C. 5N NaOH (104.4 mL, 0.522 mol) was added in one portion at 50 °C. The reaction solution was stirred additional 1-2 h at 50 °C. Conc. HCl (54.2 mL, 0.6563 mol) was added dropwise over 30 min. between 50 – 60 °C. The reaction solution was stirred at 55- 60 °C for 2-3 h and then cooled to 45 °C and 5% NaHCO₃ (ca. 100 mL) was added dropwise to pH = 7-8. Then, water (100 mL) was added over 30 min. The resulting slurry was stirred at 45 °C for additional 1-2 h and cooled to ambient temperature slowly. After aging overnight, the slurry was filtered. The wet cake was displacement washed with 50% aq. iPrOH (150ml x 2), and suction dried at ambient temperature to give 72 g of off-white to yellowish solid. 76 % yield. The isolated crystalline *trans* pyrrolidinone **1a** is stable at ambient temperature and no epimerization to its corresponding *cis* isomer was observed within a year.

¹H-NMR (500 MHz, CDCl₃): δ 7.04 (m, 1 H), 7.01 (m, 1 H), 5.76 (m, 1 H), 5.26 (m, 2 H), 5.07 (m, 1 H), 4.13 (dd, *J* = 6.2, 14.9 Hz, 1 H), 4.02 (m, 2 H), 3.91 (dd, *J* = 6.9, 13.2 Hz, 1 H), 3.72 (dd, *J* = 5.1, 13.2 Hz, 1 H), 2.84 (dd, *J* = 6.2, 17.5 Hz, 1 H), 2.72 (dd, *J* = 9.8, 17.5 Hz, 1 H). ¹³C-NMR (125 MHz, CDCl₃): δ 166.7, 156.0 (ddd, *J* = 2.5, 10.0, 245.0 Hz), 150.1 (td, *J* = 13.6, 251.3 Hz), 147.3 (ddd, *J* = 3.8, 12.5, 246.3 Hz), 131.7, 121.6 (td, *J* = 5.0, 15.0 Hz), 119.6, 117.1 (ddd, *J* = 1.3, 6.3, 20.0 Hz), 106.9 (dd, *J* = 21.3, 27.5 Hz), 83.1 (d, *J* = 1.3 Hz), 49.4, 47.7, 36.7, 35.0. Anal calc'd for C₁₄H₁₃F₃N₂O₃: C, 53.51; H, 4.17; N, 8.91; Found: C, 53.59; H, 4.12; N, 8.90.

¹ (a) Evans, D. A.; Seidel, D. *J. Am. Chem. Soc.* **2005**, 127, 9958. (b) Li, H.; Wang, Y.; Tang, L.; Deng, L. *J. Am. Chem. Soc.* **2004**, 127, 9906.

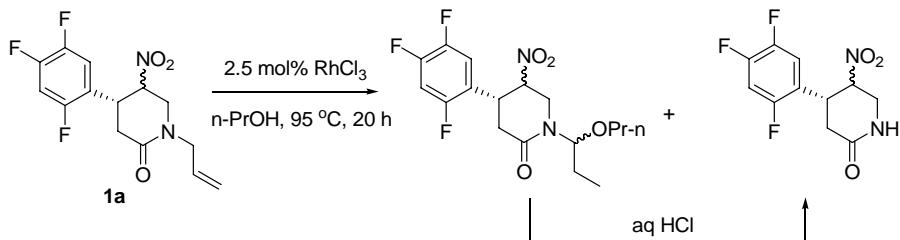


(4R,5R)-1-(4-methoxybenzyl)-5-nitro-4-(2,4,5-trifluorophenyl)piperidin-2-one: The title compound was prepared under similar conditions described above. $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ 7.19 (m, 2 H), 6.98 (m, 1 H), 6.86 (m, 2 H), 4.98 (ddd, $J = 5.2, 6.9, 8.3$ Hz, 1 H), 4.76 (d, $J = 14.4$ Hz, 1 H), 4.37 (d, $J = 14.4$ Hz, 1 H), 3.98 (m, 1 H), 3.80 (dd, $J = 6.5, 13.1$ Hz, 1 H), 3.78 (s, 3 H), 3.58 (dd, $J = 5.1, 13.1$ Hz, 1 H), 2.85 (dd, $J = 6.2, 17.6$ Hz, 1 H), 2.72 (dd, $J = 9.4, 17.6$ Hz, 1 H). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3): δ 166.8, 159.6, 155.84 (ddd, $J = 3.2, 9.6, 245.9$ Hz), 150.0 (ddd, $J = 13.6, 14.4, 253.1$ Hz), 147.2 (ddd, $J = 4.0, 12.8, 247.5$ Hz), 129.9, 127.7, 121.5 (td, $J = 4.8, 16.0$ Hz), 117.0 (dd, $J = 5.6, 20.0$ Hz), 114.4, 106.7 (dd, $J = 20.8, 28.0$ Hz) 82.8, 55.4, 49.7, 47.4, 37.3, 34.9. HRMS calc'd for $\text{C}_{19}\text{H}_{17}\text{F}_3\text{N}_2\text{O}_3$ [$\text{M}+\text{H}]^+$ 395.1213, found 395.1221.



(4R,5R)-5-nitro-4-(2,4,5-trifluorophenyl)piperidin-2-one: The title compound and its cis isomer were prepared by deprotection of **1a** in the presence of catalytic amount of RhCl_3 in $n\text{-PrOH}$ at 95 °C followed by aqueous HCl treatment as shown in Scheme S1. The crystalline trans product could be prepared under similar isomerization conditions described above. $^1\text{H-NMR}$ (400 MHz, $d_4\text{-MeOH}$): δ 7.38 (m, 1 H), 7.20 (m, 1 H), 5.31 (m, 1 H), 4.11 (m, 1 H), 4.02 (m, 2 H), 3.86 (dd, $J = 7.7, 12.9$ Hz, 1 H), 2.70 (dd, $J = 7.2, 17.7$ Hz, 1 H), 2.64 (dd, $J = 9.6, 17.7$ Hz, 1 H). $^{13}\text{C-NMR}$ (100 MHz, $d_4\text{-MeOH}$): δ 172.3, 157.5 (ddd, $J = 2.4, 9.6, 244.9$ Hz), 151.1 (ddd, $J = 12.8, 14.5, 250.6$ Hz), 148.5 (ddd, $J = 3.2, 12.0, 244.9$ Hz), 124.2 (td, $J = 4.8, 16.1$ Hz), 118.2 (dd, $J = 5.6, 21.7$ Hz), 107.4 (dd, $J = 20.9, 28.9$ Hz), 84.2, 44.5, 37.8, 36.0. HRMS calc'd for $\text{C}_{11}\text{H}_8\text{F}_3\text{N}_2\text{O}_3$ [$\text{M}+\text{H}]^+$ 275.0638, found 275.0641.

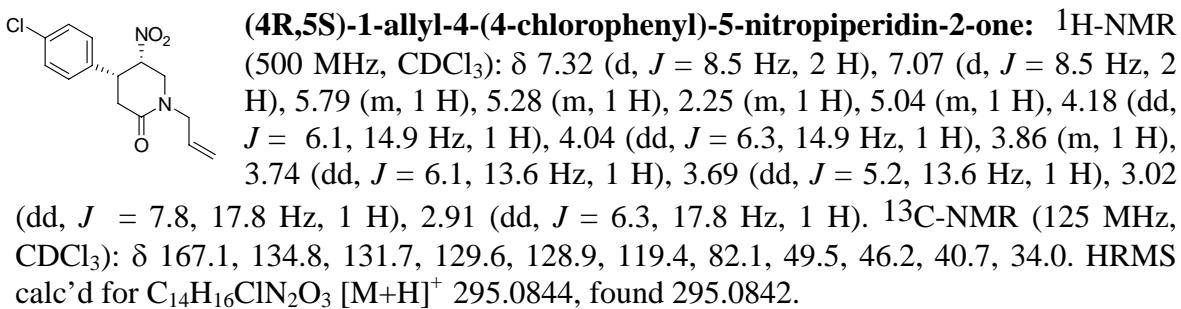
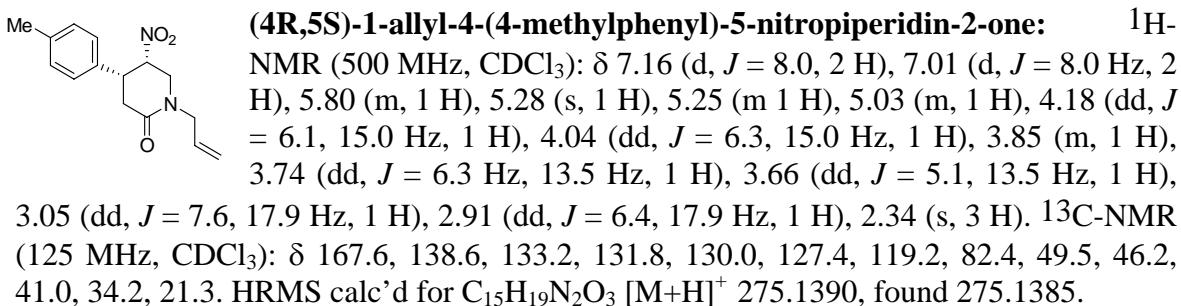
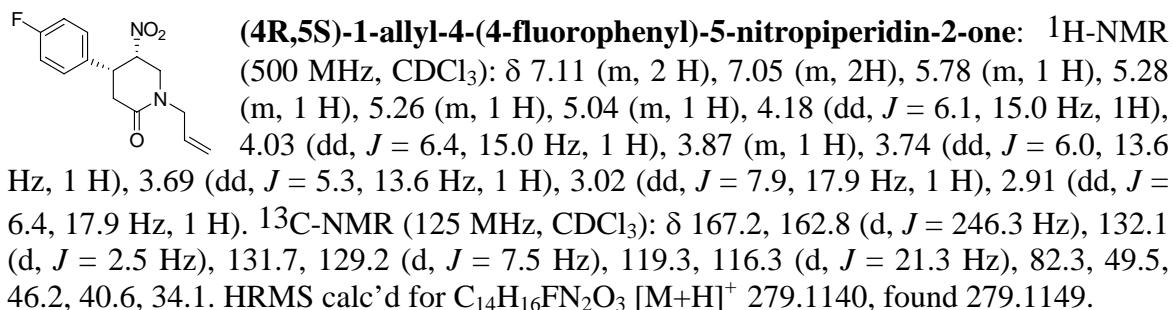
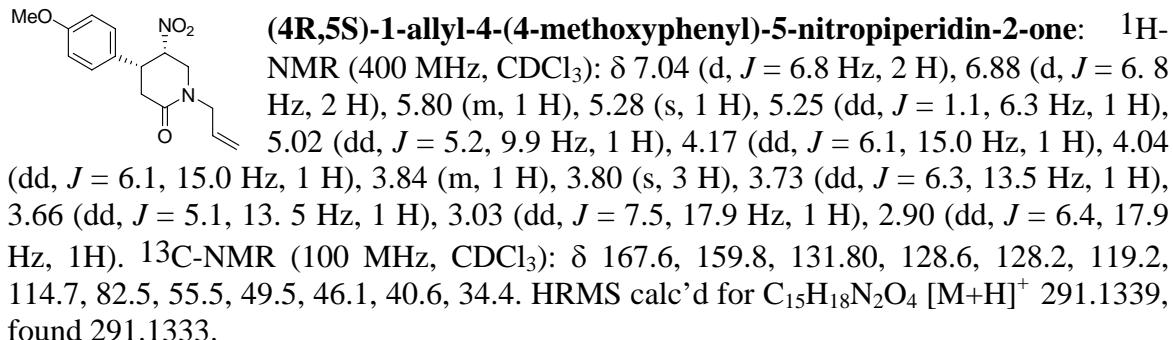
Scheme S1

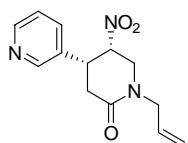


2. General procedure for the one-pot preparation of *cis* pyrrolidinones 1

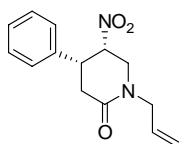
To a solution of nitro malonate **4** (3.0 mmol) and allyl amine (223 mg, 3.9 mmol) in *i*- PrOH (5 mL) and water (1 mL) at 50–55 °C was added a solution of 37 wt% HCHO (256 mg, 3.15 mmol) in water (1 mL) dropwise over 1.5 h and the reaction solution was stirred for additional 3 h at 55 °C. 5N NaOH (0.9 mL, 4.5 mmol) was added in one portion at 55 °C. The reaction solution was stirred additional 2 h at 55 °C. Conc. HCl (1.25 mL, 15 mmol) was added dropwise between 50 – 60 °C. The reaction solution was stirred at 55–

60 °C for 3-5 h and then cooled to ambient temperature. EtOAc (80 mL) was added and the organic phase was washed with water (10 mL) followed by brine (10 mL). Upon concentration, the residue was purified on silica gel column eluting with EtOAc/hexane to afford the desired *cis* pyrrolidinone **1**.





(4R,5S)-1-allyl-5-nitro-4-pyridin-3-ylpiperidin-2-one: $^1\text{H-NMR}$ (500 MHz, CDCl_3): δ 8.52 (m, 2 H), 7.55 (m, 1 H), 7.28 (m, 1 H), 5.70 (m, 1 H), 5.21 (m, 2 H), 5.01 (m, 1 H), 4.07 (m, 1 H), 3.99 (dd, $J = 6.3, 14.9$, Hz, 1 H), 3.86 (m, 2 H), 3.67 (dd, $J = 5.2, 13.1$ Hz, 1 H), 2.82 (dd, $J = 6.1, 17.7$ Hz, 1 H), 2.68 (dd, $J = 9.6, 17.7$ Hz, 1 H). $^{13}\text{C-NMR}$ (125 MHz, CDCl_3): δ 166.6, 149.7, 148.9, 134.6, 133.8, 131.5, 124.1, 119.3, 84.4, 49.2, 47.5, 40.3, 35.8. HRMS calc'd for $\text{C}_{13}\text{H}_{16}\text{N}_3\text{O}_3$ $[\text{M}+\text{H}]^+$ 262.1186, found 262.1190.



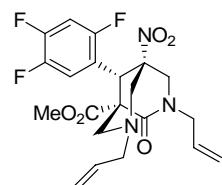
(4R,5S)-1-allyl-5-nitro-4-phenylpiperidin-2-one: $^1\text{H-NMR}$ (500 MHz, CDCl_3): δ 7.35 (m, 3 H), 7.13 (m, 2 H), 5.79 (m, 1 H), 5.28 (s, 1 H), 5.26 (m, 1 H), 5.07 (m, 1 H), 4.19 (dd, $J = 6.1, 15.0$ Hz, 1 H), 4.05 (dd, $J = 6.3, 15.0$ Hz, 1 H), 3.88 (m, 1 H), 3.75 (dd, $J = 6.1, 13.5$ Hz, 1 H), 3.69 (dd, $J = 5.1, 13.5$ Hz, 1 H), 3.08 (dd, $J = 7.8, 17.9$ Hz, 1 H), 2.93 (dd, $J = 6.4, 17.9$ Hz, 1 H). $^{13}\text{C-NMR}$ (125 MHz, CDCl_3): δ 167.3, 136.1, 131.6, 129.1, 128.5, 127.3, 119.0, 82.2, 49.3, 46.1, 41.0, 33.8. HRMS calc'd for $\text{C}_{14}\text{H}_{17}\text{N}_2\text{O}_3$ $[\text{M}+\text{H}]^+$ 261.1234, found 262.1235.

3. General procedure for the one-step preparation of bispidines 2

To a solution of nitro malonate **4** (1.5 mmol) in i-PrOH (7 mL) and water (1.5 mL) at ambient temperature was added t-BuCO₂H (77 mg, 0.75 mmol) followed by allyl amine (214 mg, 3.75 mmol). Then, the reaction solution was heated to 55 °C and a solution of 37 wt% HCHO (268 mg, 3.3 mmol) in water (1 mL) was added dropwise over 2.5 h. After addition, the reaction solution was agitated at 55 °C for 5 – 10 h. EtOAc (80 mL) was added and the organic phase was washed with water (10 mL) followed by brine (10 mL). Upon concentration, the residue was purified on silica gel column eluting with EtOAc/hexane to afford the desired **2**.

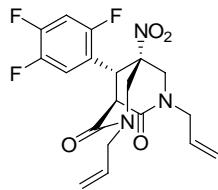
4. General procedure for the one-step preparation of bispidines 3

To a solution of nitro malonate **4** (1.5 mmol) in i-PrOH (7 mL) and water (1.5 mL) at ambient temperature was added MeSO₃H (72 mg, 0.75 mmol) followed by allyl amine (214 mg, 3.75 mmol). Then, the reaction solution was heated to 55 °C and a solution of 37 wt% HCHO (487 mg, 6.0 mmol) in water (1 mL) was added dropwise over 2.5 h. After addition, the reaction solution was agitated at 55 °C for 5 – 10 h. EtOAc (80 mL) was added and the organic phase was washed with water (10 mL) followed by brine (10 mL). Upon concentration, the residue was purified on silica gel column eluting with EtOAc/hexane to afford the desired **3**.

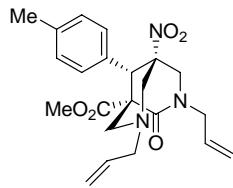


Methyl (1S,5S,9S)-3,7-diallyl-5-nitro-2-oxo-9-(2,4,5-trifluorophenyl)-3,7-diazabicyclo[3.3.1]nonane-1-carboxylate (3a): $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ 7.15 (m, 1 H), 6.92 (m, 1 H), 5.88 (m, 1

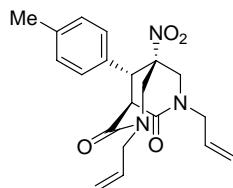
H), 5.72 (m, 1 H), 5.45 (dd, $J = 0.7$, 17.2 Hz, 1 H), 5.39 (d, $J = 10.1$ Hz, 1 H), 5.23 (m, 2 H), 4.40 (dd, $J = 5.9$, 14.7 Hz, 1 H), 4.29 (s, 1 H), 4.17 (dd, $J = 1.6$, 13.3 Hz, 1 H), 3.99 (dd, $J = 6.8$, 14.7 Hz, 1 H), 3.77 (d, $J = 13.3$ Hz, 1 H), 3.53 (dd, $J = 1.3$, 11.6 Hz, 1 H), 3.46 (s, 3 H), 3.37 (dd, $J = 1.0$, 10.4 Hz, 1 H), 3.24 (dd, $J = 6.0$, 13.6 Hz, 1 H), 3.10 (dd, $J = 6.9$, 13.6 Hz, 1 H), 2.94 (dd, $J = 1.2$, 10.3 Hz, 1 H), 2.86 (d, $J = 11.5$ Hz, 1 H). ^{13}C -NMR (100 MHz, CDCl_3): δ 168.1, 166.3, 156.8 (ddd, $J = 3.2$, 8.8, 248.2 Hz), 150.0 (td, $J = 13.7$, 253.8 Hz), 146.9 (ddd, $J = 3.2$, 12.0, 245.7 Hz), 133.0, 130.8, 120.5, 120.1, 118.0 (td, $J = 5.3$, 13.7 Hz), 117.4 (m), 106.0 (dd, $J = 20.0$, 30.0 Hz), 84.2, 65.7, 60.4, 60.0, 56.5, 52.7, 50.1, 49.9, 43.6. HRMS calc'd for $\text{C}_{21}\text{H}_{23}\text{F}_3\text{N}_3\text{O}_5$ $[\text{M}+\text{H}]^+$ 454.1584, found 454.1594.



(1R,5R,9R)-3,7-diallyl-5-nitro-9-(2,4,5-trifluorophenyl)-3,7-diaza-bicyclo[3.3.1]nonane-2,8-dione (2a): ^1H -NMR (500 MHz, CDCl_3): δ 6.99 (m, 1 H), 6.85 (m, 1 H), 5.80 (m, 2 H), 5.39 (d, $J = 7.9$ Hz, 1 H), 5.36 (dd, $J = 1.1$, 8.0 Hz, 1 H), 5.33 (s, 1 H), 5.28 (dd, $J = 1.2$, 17.1 Hz, 1 H), 4.45 (s, 1 H), 4.22 (dd, $J = 1.1$, 12.8 Hz, 1 H), 4.15 (m, 4 H), 4.05 (dd, $J = 6.4$, 15.0 Hz, 1 H), 3.71 (d, $J = 2.3$ Hz, 1 H), 3.70 (s, 1 H), 3.68 (s, 1 H). ^{13}C -NMR (125 MHz, CDCl_3): δ 163.7, 162.3, 156.4 (ddd, $J = 2.8$, 9.2, 248.6 Hz), 150.5 (td, $J = 14.2$, 255.3 Hz), 147.4 (ddd, $J = 3.7$, 12.6, 247.3 Hz), 131.2, 130.6, 121.5, 120.2, 117.5 (td, $J = 4.9$, 15.1 Hz), 116.4 (ddd, $J = 1.5$, 4.3, 20.3 Hz), 107.0 (dd, $J = 17.0$, 24.0 Hz), 84.2, 56.9, 52.8, 50.6, 50.1, 49.1, 39.3. HRMS calc'd for $\text{C}_{19}\text{H}_{19}\text{F}_3\text{N}_3\text{O}_4$ $[\text{M}+\text{H}]^+$ 410.1322, found 410.1311. Anal calc'd for $\text{C}_{19}\text{H}_{18}\text{F}_3\text{N}_3\text{O}_4$: C, 55.75; H, 4.43; N, 10.26; Found: C, 55.67; H, 4.41; N, 10.22.

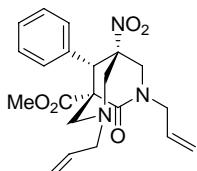


Methyl (1S,5S,9S)-3,7-diallyl-9-(4-methylphenyl)-5-nitro-2-oxo-3,7-diazabicyclo[3.3.1]nonane-1-carboxylate: ^1H -NMR (500 MHz, CDCl_3): δ 7.08 (d, $J = 8.4$ Hz, 2 H), 7.06 (d, $J = 8.4$ Hz, 2 H), 5.89 (m, 1 H), 5.73 (m, 1 H), 5.43 (m, 1 H), 5.33 (m, 1 H), 5.23 (m, 1 H), 5.20 (m, 1 H), 4.56 (m, 1 H), 4.13 (dd, $J = 2.1$, 12.9 Hz, 1 H), 3.84 (dd, $J = 7.0$, 14.8 Hz, 1 H), 3.78 (s, 1 H), 3.64 (dd, $J = 0.9$, 12.9 Hz, 1 H), 3.47 (dd, $J = 1.6$, 11.2 Hz, 1 H), 3.33 (s, 3 H), 3.32 (dd, $J = 1.6$, 11.2 Hz, 1 H), 3.21 (dd, $J = 1.3$, 6.0, 13.5 Hz, 1 H), 3.06 (dd, $J = 6.9$, 13.5 Hz, 1 H), 2.99 (dd, $J = 2.0$, 10.3 Hz, 1 H), 2.86 (d, $J = 11.2$ Hz, 1 H), 2.29 (s, 3 H). ^{13}C -NMR (125 MHz, CDCl_3): δ 168.9, 166.6, 138.5, 133.3, 131.4, 129.7, 129.6, 129.3, 119.39, 119.37, 85.1, 65.1, 60.9, 59.9, 56.5, 54.1, 52.1, 49.6, 49.4, 21.0. HRMS calc'd for $\text{C}_{22}\text{H}_{28}\text{N}_3\text{O}_5$ $[\text{M}+\text{H}]^+$ 414.2024, found 414.2012.

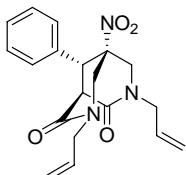


(1R,5R,9R)-3,7-diallyl-9-(4-methylphenyl)-5-nitro-3,7-diaza-bicyclo[3.3.1]nonane-2,8-dione: ^1H -NMR (500 MHz, CDCl_3): δ 7.14 (d, $J = 8.1$ Hz, 2 H), 6.93 (d, $J = 8.2$ Hz, 2 H), 5.76 (m, 1 H), 5.29 (m, 2 H), 4.27 (dd, $J = 6.2$, 14.6 Hz, 1 H), 4.15 (m, 3 H), 4.06 (dd, $J = 6.2$, 15.1 Hz, 1 H), 3.96 (dd, $J = 7.0$, 14.6 Hz, 1 H), 3.82 (d, $J = 13.1$ Hz, 1 H), 3.81 (d, $J = 2.0$ Hz, 1 H), 3.68 (d, $J = 13.1$ Hz, 1 H),

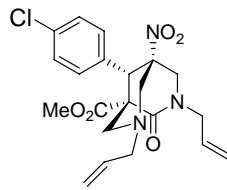
3.52 (dd, $J = 1.7, 12.5$ Hz, 1 H), 2.33 (s, 3 H). ^{13}C -NMR (125 MHz, CDCl_3): δ 164.7, 163.3, 139.2, 131.2, 131.0, 130.13, 130.09, 127.3, 120.5, 119.5, 85.4, 55.9, 52.7, 49.88, 49.87, 48.6, 45.4, 21.0. HRMS calc'd for $\text{C}_{20}\text{H}_{24}\text{N}_3\text{O}_4$ [$\text{M}+\text{H}]^+$ 370.1761, found 370.1759. Anal calc'd for $\text{C}_{20}\text{H}_{23}\text{N}_3\text{O}_4$: C, 65.03; H, 6.28; N, 11.37; Found: C, 64.97; H, 6.32; N, 11.39.



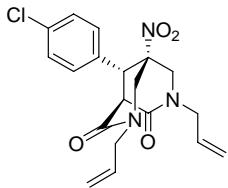
Methyl (1S,5S,9S)-3,7-diallyl-9-phenyl-5-nitro-2-oxo-3,7-diazabicyclo[3.3.1]nonane-1-carboxylate: ^1H -NMR (500 MHz, CDCl_3): δ 7.27 (m, 3 H), 7.21 (m, 2 H), 5.89 (m, 1 H), 5.74 (m, 1 H), 5.43 (dd, $J = 1.3, 17.2$ Hz, 1 H), 5.34 (dd, $J = 0.9, 10.3$ Hz, 1 H), 5.24 (m, 1 H), 5.21 (s, 1 H), 4.58 (m, 1 H), 4.15 (dd, $J = 2.1, 13.0$ Hz, 1 H), 3.85 (dd, $J = 6.9, 14.9$ Hz, 1 H), 3.81 (s, 1 H), 3.67 (d, $J = 13.0$ Hz, 1 H), 3.48 (dd, $J = 1.5, 11.2$ Hz, 1 H), 3.34 (dd, $J = 1.4, 10.3$ Hz, 1 H), 3.30 (s, 1 H), 3.22 (dd, $J = 6.0, 13.5$ Hz, 1 H), 3.07 (dd, $J = 6.9, 13.5$ Hz, 1 H), 2.99 (dd, $J = 2.0, 10.3$ Hz, 1 H), 2.88 (d, $J = 11.2$ Hz, 1 H). ^{13}C -NMR (125 MHz, CDCl_3): δ 169.1, 166.7, 133.4, 133.1, 131.5, 130.0, 128.9, 128.8, 119.7, 85.2, 65.4, 61.1, 60.1, 56.8, 54.7, 52.3, 49.8, 49.6. HRMS calc'd for $\text{C}_{21}\text{H}_{26}\text{N}_3\text{O}_5$ [$\text{M}+\text{H}]^+$ 400.1867, found 400.1865. Anal calc'd for $\text{C}_{21}\text{H}_{25}\text{N}_3\text{O}_5$: C, 63.14; H, 6.31; N, 10.52; Found: C, 62.79; H, 6.30; N, 10.38.



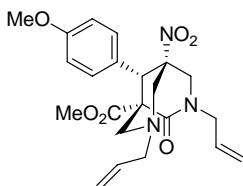
(1R,5R,9R)-3,7-diallyl-5-nitro-9-phenyl-3,7-diazabicyclo[3.3.1]nonane-2,8-dione: ^1H -NMR (500 MHz, CDCl_3): δ 7.35 (m, 3 H), 7.06 (m, 2 H), 5.79 (m, 2 H), 5.30 (m, 4 H), 4.30 (m, 1 H), 4.18 (m, 2 H), 4.14 (dd, $J = 6.1, 15.1$ Hz, 1 H), 4.07 (dd, $J = 6.1, 15.1$ Hz, 1 H), 3.95 (dd, $J = 7.0, 14.6$ Hz, 1 H), 3.84 (d, $J = 2.0$ Hz, 1 H), 3.82 (d, $J = 13.1$ Hz, 1 H), 3.69 (d, $J = 13.1$ Hz, 1 H), 3.54 (dd, $J = 1.8, 12.6$ Hz, 1 H). ^{13}C -NMR (125 MHz, CDCl_3): δ 164.8, 163.4, 133.4, 131.4, 131.1, 129.7, 129.5, 127.8, 120.8, 119.8, 85.5, 56.2, 52.8, 50.09, 50.07, 48.8, 45.9. HRMS calc'd for $\text{C}_{19}\text{H}_{22}\text{ClN}_3\text{O}_4$ [$\text{M}+\text{H}]^+$ 356.1605, found 356.1596.



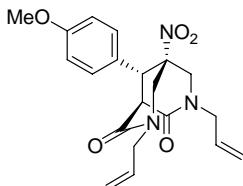
Methyl (1S,5S,9S)-3,7-diallyl-9-(4-chlorophenyl)-5-nitro-2-oxo-3,7-diazabicyclo[3.3.1]nonane-1-carboxylate: ^1H -NMR (500 MHz, CDCl_3): δ 7.25 (m, 2 H), 7.16 (m, 2 H), 5.88 (m, 1 H), 5.73 (m, 1 H), 5.43 (m, 1 H), 5.34 (dd, $J = 1.1, 10.2$ Hz, 1 H), 5.21 (m, 2 H), 4.53 (m, 1 H), 4.07 (dd, $J = 2.1, 13.1$ Hz, 1 H), 3.85 (6.9, 14.8 Hz, 1 H), 3.80 (s, 1 H), 3.67 (d, $J = 13.1$ Hz, 1 H), 3.49 (dd, $J = 1.5, 11.3$ Hz, 1 H), 3.37 (s, 3 H), 3.34 (dd, $J = 1.5$ Hz, 10.3 Hz, 1 H), 3.21 (m, 1 H), 3.07 (dd, $J = 7.0, 13.5$ Hz, 1 H), 2.97 (dd, $J = 2.0, 10.3$ Hz, 1 H), 2.85 (d, $J = 11.3$ Hz, 1 H). ^{13}C -NMR (125 MHz, CDCl_3): δ 168.8, 166.5, 135.0, 133.3, 131.2, 131.4, 131.3, 129.0, 119.9, 119.8, 85.1, 65.3, 61.0, 60.1, 56.6, 53.9, 52.5, 49.9, 49.5. HRMS calc'd for $\text{C}_{21}\text{H}_{25}\text{ClN}_3\text{O}_5$ [$\text{M}+\text{H}]^+$ 434.1477, found 434.1477.



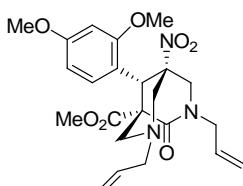
(1R,5R,9R)-3,7-diallyl-9-(4-chlorophenyl)-5-nitro-3,7-diaza-bicyclo[3.3.1]nonane-2,8-dione: $^1\text{H-NMR}$ (500 MHz, CDCl_3): δ 7.33 (m, 2 H), 7.00 (m, 2 H), 5.77 (m, 2 H), 5.30 (m, 4 H), 4.27 (m, 1 H), 4.15 (m, 2 H), 4.13 (m, 1 H), 4.06 (m, 1 H), 3.97 (dd, $J = 7.0, 14.5$ Hz, 1 H), 3.81 (d, $J = 2.0$ Hz, 1 H), 3.79 (d, $J = 12.8$ Hz, 1 H), 3.69 (d, $J = 13.1$ Hz, 1 H), 3.56 (dd, $J = 1.8, 12.8$ Hz, 1 H). $^{13}\text{C-NMR}$ (125 MHz, CDCl_3): δ 164.5, 163.1, 135.7, 131.9, 131.3, 130.9, 129.9, 129.2, 121.0, 119.9, 85.2, 56.1, 52.7, 50.1, 50.0, 48.8, 45.3. HRMS calc'd for $\text{C}_{19}\text{H}_{21}\text{ClN}_3\text{O}_4$ $[\text{M}+\text{H}]^+$ 390.1215, found 390.1213.



Methyl (1S,5S,9S)-3,7-diallyl-9-(4-methoxyphenyl)-5-nitro-2-oxo-3,7-diazabicyclo[3.3.1]nonane-1-carboxylate: $^1\text{H-NMR}$ (500 MHz, CDCl_3): δ 7.12 (d, $J = 8.9$ Hz, 2 H), 6.78 (d, $J = 8.9$ Hz, 2 H), 5.88 (m, 1 H), 5.73 (m, 1 H), 5.43 (m, 1 H), 5.33 (m, 1 H), 5.23 (m, 1 H), 5.20 (s, 1 H), 4.57 (m, 1 H), 4.11 (dd, $J = 1.9, 13.0$ Hz, 1 H), 3.83 (dd, $J = 7.0, 14.8$ Hz, 1 H), 3.77 (s, 4 H), 3.65 (d, $J = 13.0$ Hz, 1 H), 3.47 (dd, $J = 1.3, 11.2$ Hz, 1 H), 3.34 (s, 3 H), 3.32 (d, $J = 10.3$ Hz, 1 H), 3.21 (ddd, $J = 1.2, 6.0, 13.5$ Hz, 1 H), 3.06 (dd, $J = 0.9, 7.0, 13.5$ Hz, 1 H), 2.98 (dd, $J = 1.5, 10.3$ Hz, 1 H), 2.86 (d, $J = 11.2$ Hz, 1 H). $^{13}\text{C-NMR}$ (125 MHz, CDCl_3): δ 169.1, 166.8, 159.9, 133.5, 131.6, 131.2, 124.8, 119.6, 114.2, 85.3, 65.3, 61.1, 60.1, 56.8, 55.4, 54.0, 52.4, 49.8, 49.5. Anal calc'd for $\text{C}_{22}\text{H}_{27}\text{N}_3\text{O}_6$: C, 61.53; H, 6.34; N, 9.78; Found: C, 61.36; H, 6.32; N, 9.77.

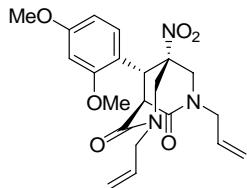


(1R,5R,9R)-3,7-diallyl-5-nitro-9-(4-methoxyphenyl)-3,7-diaza-bicyclo[3.3.1]nonane-2,8-dione: $^1\text{H-NMR}$ (500 MHz, CDCl_3): δ 6.97 (d, $J = 8.8$ Hz, 2 H), 6.85 (d, $J = 8.8$ Hz, 2 H), 5.78 (m, 2 H), 5.31 (m, 3 H), 5.26 (m, 1 H), 4.29 (dd, $J = 6.2, 14.6$ Hz, 1 H), 4.14 (m, 3 H), 4.05 (dd, $J = 6.2, 15.1$ Hz, 1 H), 3.95 (dd, $J = 7.0, 14.6$ Hz, 1 H), 3.81 (m, 2 H), 3.80 (s, 3 H), 3.68 (d, $J = 13.1$ Hz, 1 H), 3.52 (dd, $J = 1.7, 12.5$ Hz, 1 H). $^{13}\text{C-NMR}$ (125 MHz, CDCl_3): δ 164.9, 163.5, 160.3, 131.4, 131.2, 129.0, 125.1, 120.7, 119.7, 115.0, 85.7, 56.0, 55.0, 53.0, 50.09, 50.05, 48.8, 45.3. HRMS calc'd for $\text{C}_{20}\text{H}_{24}\text{N}_3\text{O}_5$ $[\text{M}+\text{H}]^+$ 386.1711, found 386.1714. Anal calc'd for $\text{C}_{20}\text{H}_{23}\text{N}_3\text{O}_5$: C, 62.33; H, 6.01; N, 10.90; Found: C, 62.26; H, 6.58; N, 10.98.

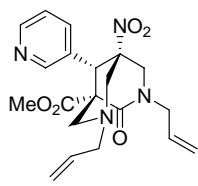


Methyl (1S,5S,9S)-3,7-diallyl-9-(2,4-dimethoxyphenyl)-5-nitro-2-oxo-3,7-diazabicyclo[3.3.1]nonane-1-carboxylate: $^1\text{H-NMR}$ (500 MHz, CDCl_3): δ 7.13 (d, $J = 9.0$ Hz, 1 H), 6.41 (m, 2 H), 5.87 (m, 1 H), 5.76 (m, 1 H), 5.42 (dd, $J = 1.3, 17.1$ Hz, 1 H), 5.33 (d, $J = 17.1$ Hz, 1 H), 5.23 (dd, $J = 1.1, 9.1$ Hz, 1 H), 5.21 (s, 1 H), 4.56 (s, 1 H), 4.48 (dd, $J = 4.9, 14.7$ Hz, 1 H), 4.32 (d, $J = 13.0$ Hz, 1 H), 3.80 (dd, $J = 5.8, 14.0$ Hz, 1 H), 3.77 (s, 3 H), 3.76 (s, 3 H), 3.66 (d, $J = 13.0$ Hz, 1 H), 3.45 (dd, $J = 1.2, 11.3$ Hz, 1 H), 3.35 (s, 3 H), 3.30 (dd, $J = 0.9, 10.2$ Hz, 1 H), 3.23 (dd, $J = 6.0, 13.5$ Hz, 1 H), 3.09 (dd, $J = 6.9, 13.5$ Hz, 1 H), 3.03 (dd, $J = 1.3, 10.2$ Hz, 1 H), 2.92 (d, $J =$

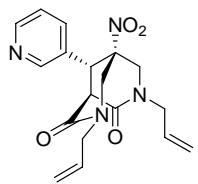
11.3 Hz, 1 H). ^{13}C -NMR (125 MHz, CDCl_3): δ 169.1, 167.2, 160.7, 159.4, 133.47, 131.6, 129.1, 119.7, 119.6, 114.8, 104.8, 98.9, 84.8, 65.7, 60.9, 60.3, 56.7, 56.2, 55.4, 52.2, 50.5, 49.8, 43.8. HRMS calc'd for $\text{C}_{23}\text{H}_{30}\text{N}_3\text{O}_7$ [$\text{M}+\text{H}]^+$ 460.2078, found 460.2068.



(1R,5R,9R)-3,7-diallyl-5-nitro-9-(2,4-dimethoxyphenyl)-3,7-diazabicyclo[3.3.1]nonane-2,8-dione: ^1H -NMR (500 MHz, CDCl_3): δ 6.82 (d, $J = 9.2$ Hz, 1 H), 6.42 (m, 2 H), 5.78 (m, 2 H), 5.29 (m, 4 H), 4.04 (s, 1 H), 4.37 (d, $J = 13.0$ Hz, 1 H), 4.25 (dd, $J = 6.3, 14.6$ Hz, 1 H), 4.16 (m, 1 H), 4.14 (d, $J = 12.1$ Hz, 1 H), 4.07 (d, $J = 6.3, 15.0$ Hz, 1 H), 3.92 (dd, $J = 7.0, 14.5$ Hz, 1 H), 3.80 (s, 3 H), 3.72 (d, $J = 2.0$ Hz, 1 H), 3.71 (s, 3 H), 3.60 (d, $J = 12.9$ Hz, 1 H), 3.45 (dd, $J = 1.5, 12.7$ Hz, 1 H). ^{13}C -NMR (125 MHz, CDCl_3): δ 165.4, 163.8, 161.4, 159.1, 131.7, 131.5, 128.7, 120.6, 119.6, 114.3, 105.1, 99.1, 85.5, 56.2, 55.6, 55.4, 53.3, 50.9, 49.8, 49.0, 40.9. HRMS calc'd for $\text{C}_{21}\text{H}_{26}\text{N}_3\text{O}_6$ [$\text{M}+\text{H}]^+$ 416.1816, found 416.1831.



Methyl (1S,5S,9S)-3,7-diallyl-5-nitro-2-oxo-9-pyridin-3-yl-3,7-diazabicyclo[3.3.1]nonane-1-carboxylate: ^1H -NMR (500 MHz, CDCl_3): δ 8.54 (d, $J = 4.3$ Hz, 1 H), 8.47 (d, $J = 1.9$ Hz, 1 H), 7.58 (d, $J = 8.0$ Hz, 1 H), 7.23 (dd, $J = 4.8, 8.0$ Hz, 1 H), 5.91 (m, 1 H), 5.73 (m, 1 H), 5.44 (m, 1 H), 5.36 (m, 1 H), 5.24 (m, 2 H), 4.55 (dd, $J = 5.5, 14.8$ Hz, 1 H), 4.08 (dd, $J = 1.7, 13.2$ Hz, 1 H), 3.85 (dd, $J = 6.9, 14.8$ Hz, 1 H), 3.82 (s, 1 H), 3.74 (d, $J = 13.2$ Hz, 1 H), 3.53 (d, $J = 11.3$ Hz, 1 H), 3.37 (d, $J = 10.4$ Hz, 1 H), 3.34 (s, 3 H), 3.23 (dd, $J = 6.0, 13.5$ Hz, 1 H), 3.09 (dd, $J = 6.9, 13.5$ Hz, 1 H), 2.97 (dd, $J = 1.8, 10.4$ Hz, 1 H), 2.87 (d, $J = 11.4$ Hz, 1 H). ^{13}C -NMR (125 MHz, CDCl_3): δ 168.6, 166.4, 151.3, 150.2, 137.2, 133.2, 131.2, 129.3, 123.5, 120.0, 119.9, 84.9, 65.3, 60.8, 60.1, 56.6, 52.5, 52.1, 49.9, 49.5. HRMS calc'd for $\text{C}_{20}\text{H}_{25}\text{N}_4\text{O}_5$ [$\text{M}+\text{H}]^+$ 401.1820, found 401.1815.

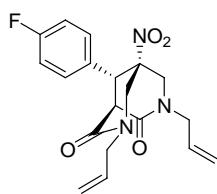


(1R,5R,9R)-3,7-diallyl-5-nitro-9-pyridin-3-yl-3,7-diazabicyclo[3.3.1]-nonane-2,8-dione: ^1H -NMR (500 MHz, CDCl_3): δ 8.62 (dd, $J = 1.5, 4.8$ Hz, 1 H), 8.38 (d, $J = 2.4$ Hz, 1 H), 7.40 (m, 1 H), 7.31 (dd, $J = 4.8, 7.9$ Hz, 1 H), 5.77 (m, 2 H), 5.32 (m, 4 H), 4.29 (m, 1 H), 4.2 (s, 1 H), 4.16 (dd, $J = 1.4, 13.1$ Hz, 1 H), 4.08 (m, 2 H), 3.97 (dd, $J = 7.0, 14.6$ Hz, 1 H), 3.85 (dd, $J = 1.0, 12.9$ Hz, 1 H), 3.81 (d, $J = 2.1$ Hz, 1 H), 3.73 (d, $J = 13.1$ Hz, 1 H), 3.65 (dd, $J = 1.7, 13.1$ Hz, 1 H). ^{13}C -NMR (125 MHz, CDCl_3): δ 164.2, 162.7, 150.9, 149.4, 135.4, 131.2, 130.8, 129.5, 124.3, 121.1, 120.0, 84.9, 56.3, 52.5, 50.1, 50.0, 48.9, 43.9. HRMS calc'd for $\text{C}_{18}\text{H}_{21}\text{N}_4\text{O}_4$ [$\text{M}+\text{H}]^+$ 357.1557, found 357.1551.



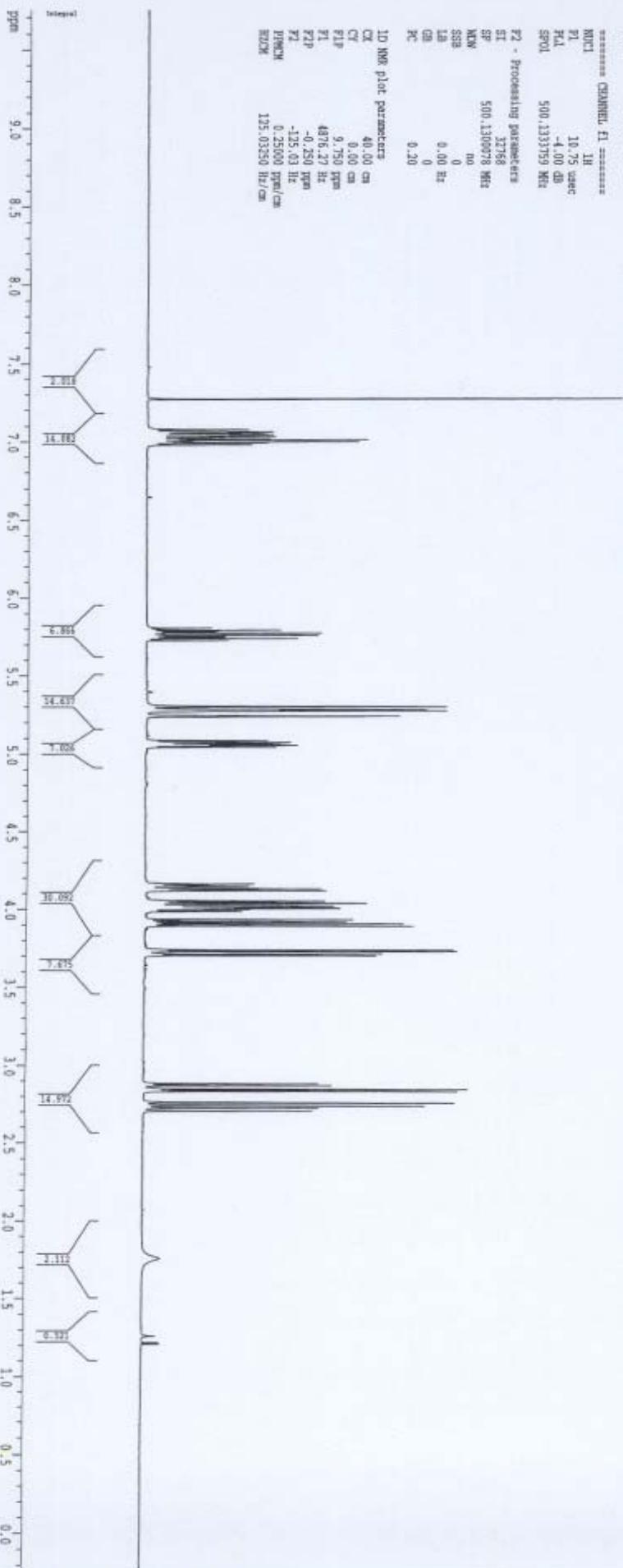
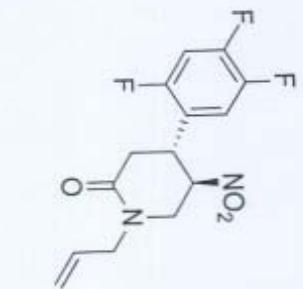
Methyl (1S,5S,9S)-3,7-diallyl-9-(4-fluorophenyl)-5-nitro-2-oxo-3,7-diazabicyclo[3.3.1]nonane-1-carboxylate: ^1H -NMR (500 MHz, CDCl_3): δ 7.20 (dd, $J = 8.4, 11.9$ Hz, 2 H), 6.96 (t, $J = 8.5$ Hz, 2 H),

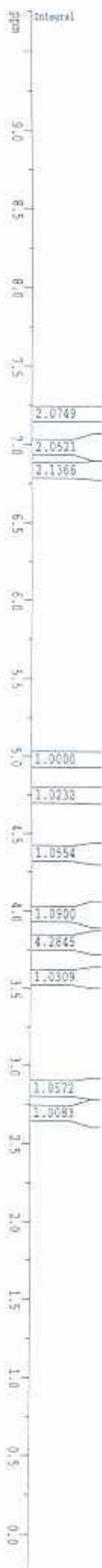
5.88 (m, 1 H), 5.73 (m 1 H), 5.43 (dd, $J = 1.3, 17.3$ Hz, 1 H), 5.34 (dd, $J = 0.9, 10.2$ Hz, 1 H), 5.23 (m, 1 H), 5.21 (s, 1 H), 4.54 (dd, $J = 5.5, 14.8$ Hz, 1 H), 4.09 (dd, $J = 2.0, 13.1$ Hz, 1 H), 3.85 (dd, $J = 7.0, 14.8$ Hz, 1 H), 3.81 (s, 1 H), 3.67 (d, $J = 13.1$ Hz, 1 H), 3.48 (dd, $J = 1.3, 11.3$ Hz, 1 H), 3.34 (s, 3 H), 3.33 (dd, $J = 1.1, 10.2$ Hz, 1 H), 3.21 (dd, $J = 6.1, 13.5$ Hz, 1 H), 3.08 (dd, $J = 7.0, 13.5$ Hz, 1 H), 2.98 (dd, $J = 1.8, 10.4$ Hz, 1 H), 2.86 (d, $J = 11.3$ Hz, 1 H). ^{13}C -NMR (125 MHz, CDCl_3): δ 168.9, 166.5, 162.9 (d, $J = 247.3$ Hz), 133.2, 131.8 (d, $J = 7.4$ Hz), 131.3, 128.8 (d, $J = 3.4$ Hz), 119.8, 115.8 (d, $J = 21.3$ Hz), 85.1, 65.2, 60.9, 60.1, 56.7, 53.8, 52.4, 49.8, 49.4. HRMS calc'd for $\text{C}_{21}\text{H}_{25}\text{FN}_3\text{O}_5$ $[\text{M}+\text{H}]^+$ 418.1773, found 418.1780.



(1R,5R,9R)-3,7-diallyl-9-(4-fluorophenyl)-5-nitro-3,7-diazabicyclo[3.3.1]nonane-2,8-dione: ^1H -NMR (500 MHz, CDCl_3): δ 7.05 (d, $J = 6.6$ Hz, 4 H), 5.78 (m, 2 H), 5.31 (m, 4 H), 4.28 (m, 1 H), 4.16 (m, 2 H), 4.12 (m, 1 H), 4.07 (m, 1 H), 3.96 (dd, $J = 7.0, 14.5$ Hz, 1 H), 3.82 (d, $J = 2.0$ Hz, 1 H), 3.80 (m, 1 H), 3.69 (d, $J = 13.1$ Hz, 1 H), 3.56 (dd, $J = 1.7, 12.7$ Hz, 1 H). ^{13}C -NMR (125 MHz, CDCl_3): δ 164.5, 163.2 (d, $J = 249.9$ Hz), 163.1, 131.3, 130.9, 129.6 (d, $J = 8.3$ Hz), 129.2 (d, $J = 3.3$ Hz), 120.9, 119.8, 116.7 (d, $J = 21.7$ Hz), 85.4, 56.1, 52.8, 50.1, 49.9, 48.8, 45.2. HRMS calc'd for $\text{C}_{19}\text{H}_{21}\text{FN}_3\text{O}_4$ $[\text{M}+\text{H}]^+$ 374.1511, found 374.1509.

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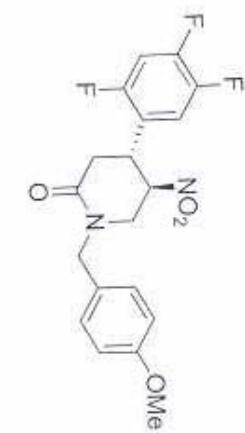


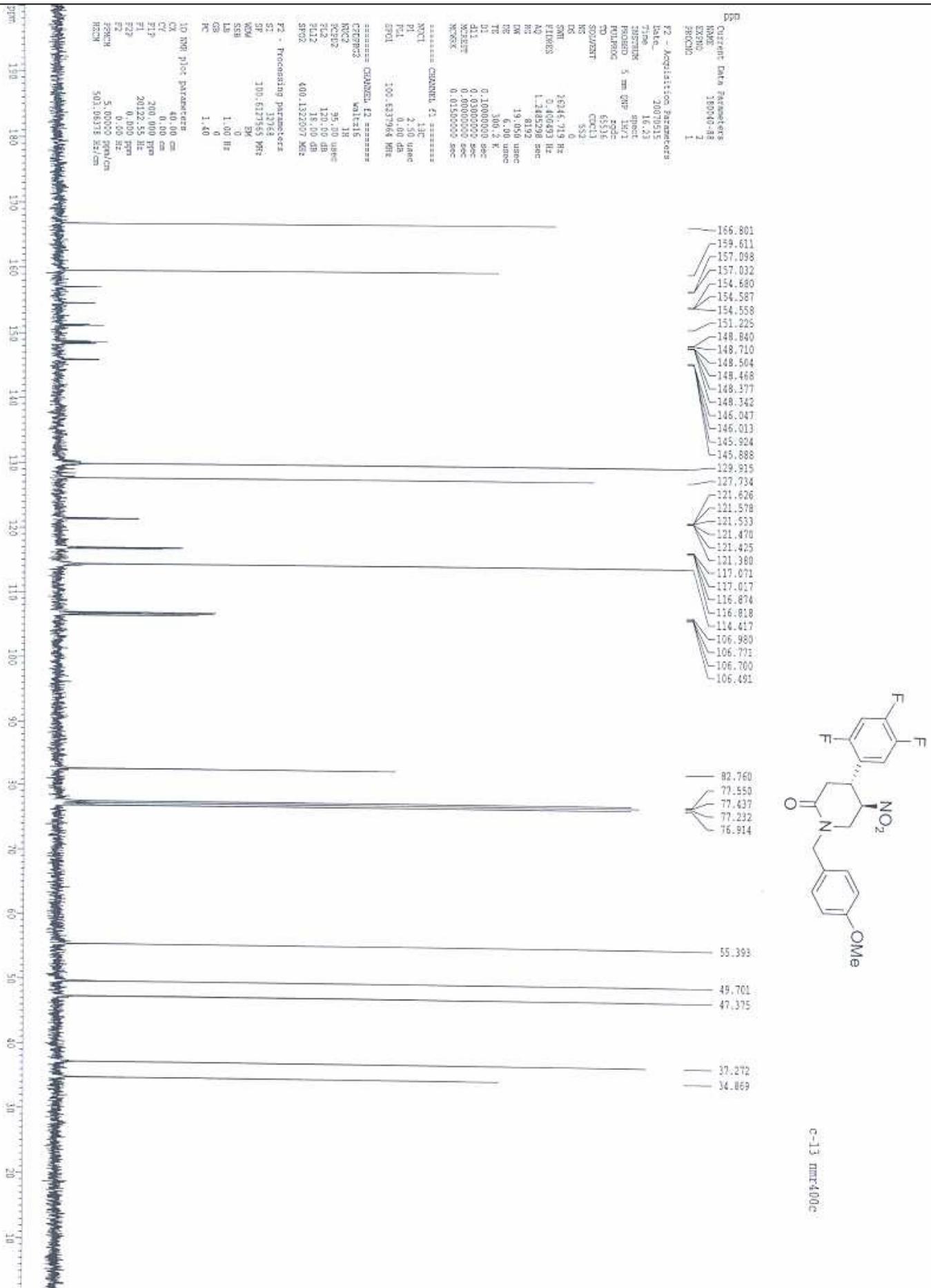
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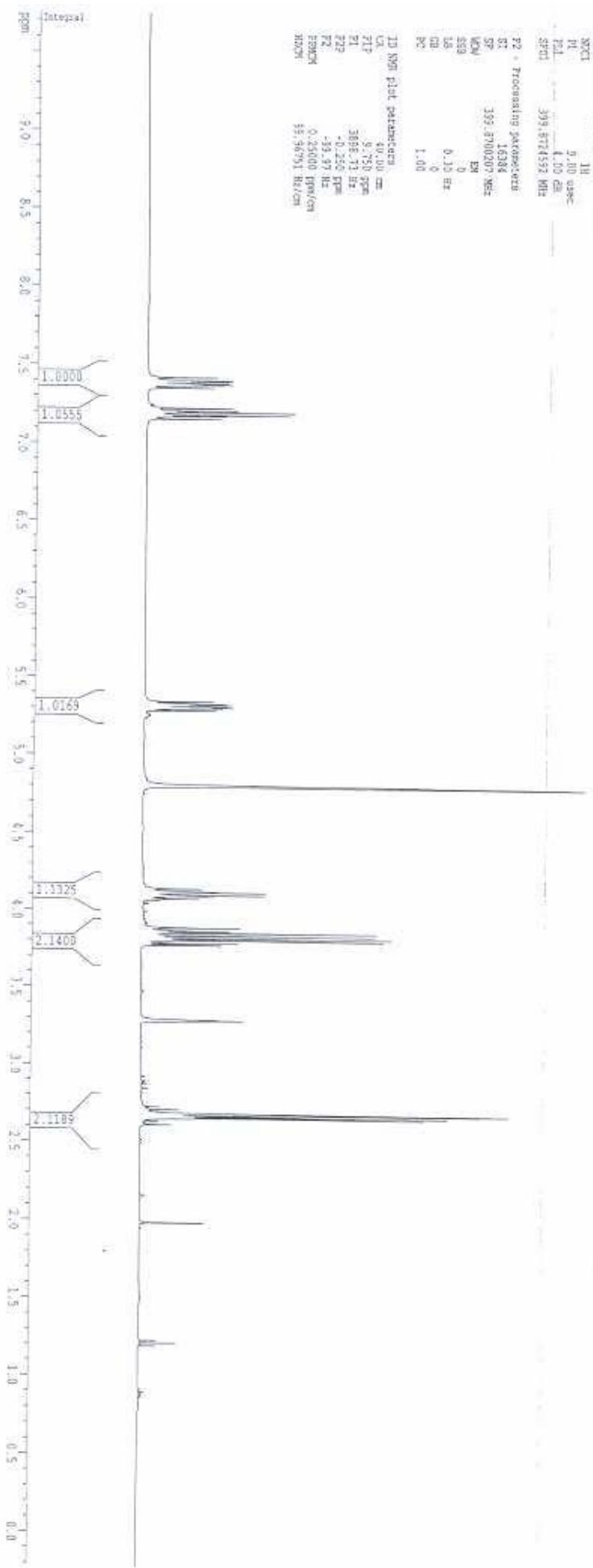
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TEC 100.0 K

TDZ 0

TECDW 0.1000000 sec

==== CHANNEL II =====

H

7.42001
7.40279
7.39852
7.39260
7.38134
7.37542
7.37115
7.35994
7.32566
7.20884
7.19999
7.18332
7.17440
7.15763

D

5.34006
5.32664
5.32077
5.31666
5.30743
5.30322
5.29744
5.28401
4.83017

C

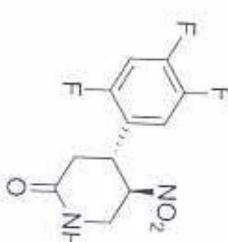
4.12874
4.12030
4.11506
4.09700
4.09154
3.88412
3.86509
3.85211
3.83298
3.81554
3.80223
3.78427
3.77104
3.28985
3.28582
3.28175

N

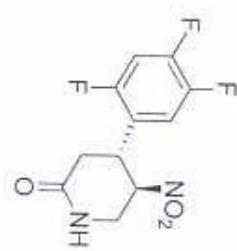
2.68186
2.67784
2.66401
2.65375

O

1.98530



nmr400b c-13 (h-1 decoupled)



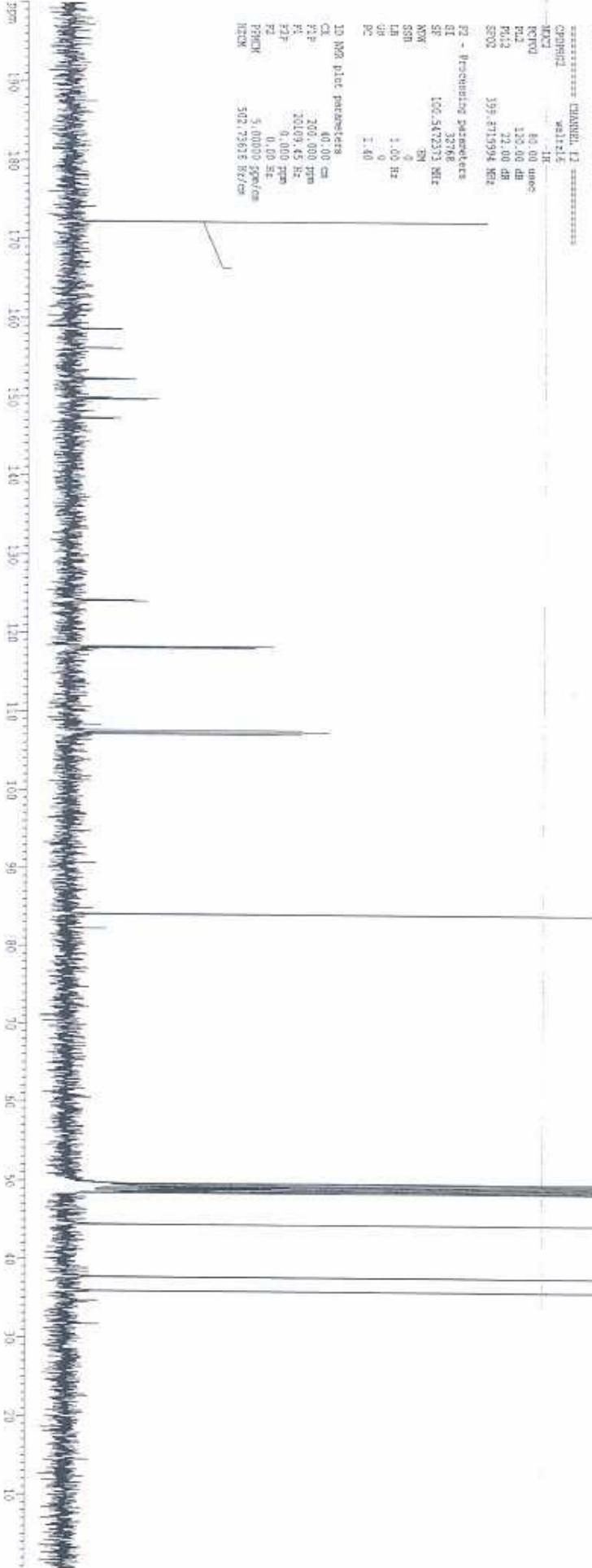
Current Data Parameters
Date: 2005-07-18
ExpID: 2
PROCID: 1

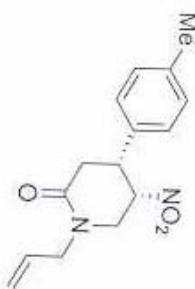
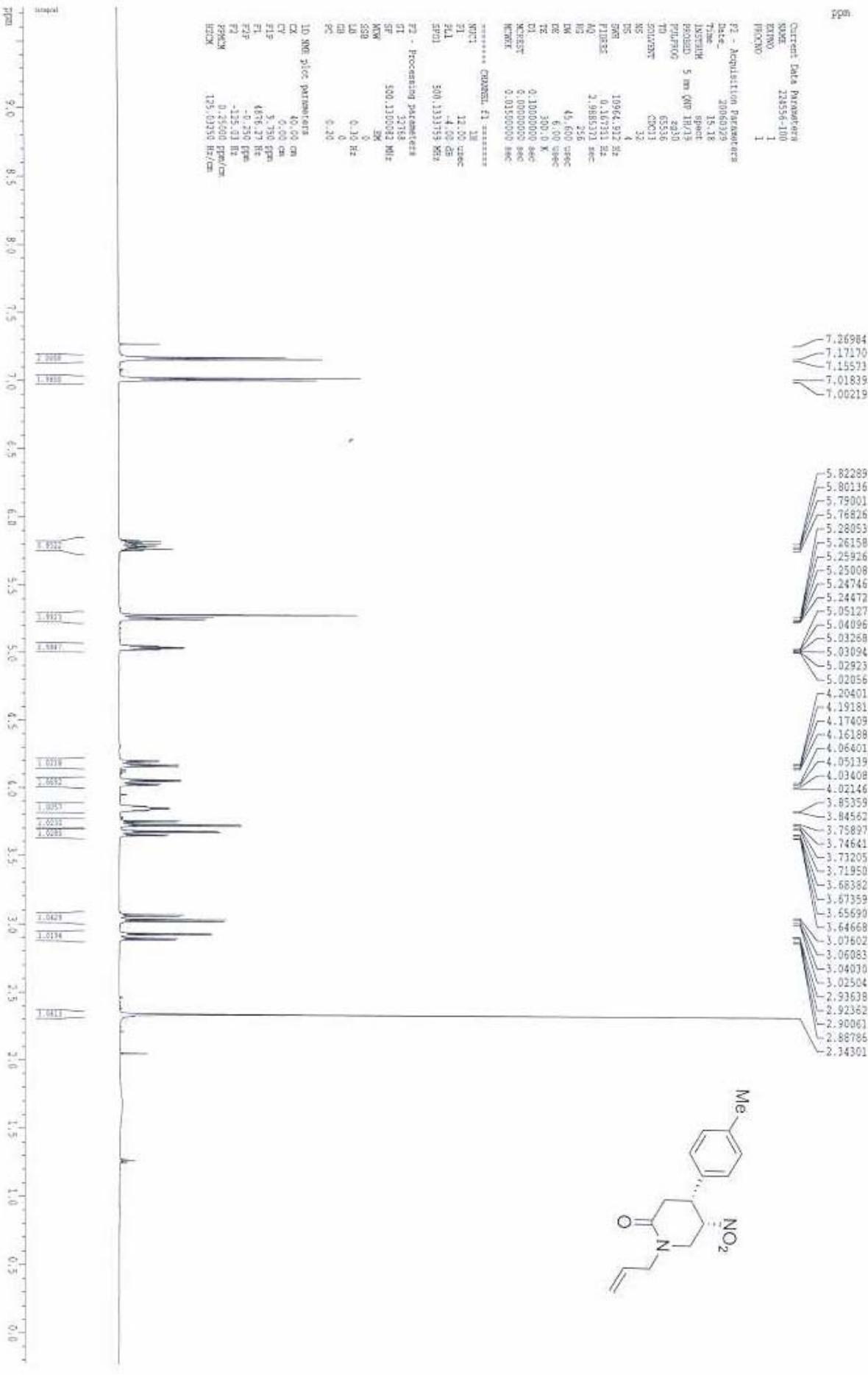
=====
E2 - Acquisition Parameters
E2 Dwell Time: 10.16 sec
INSTRUM: spect
PROBOD: 5 mm QNP 1H
QUBICFG: 65536
SW2FMT: 00013
NS: 598
DS: 4
SW1: 2635.78 Hz
FIDRES: 0.01534 Hz
TD: 1251140 sec
RG: 18192
DW: 19.000 usec
DE: 6.00 usec
TE: 300.0 K
D1: 0.000000 sec
T1: 0.000000 sec
=====
===== CHANNEL E1 =====

MOCV1: 110
P1: 2.00 ussec
T1I: 5.00 dB
SPG1: 190.5580112 MHz
=====
===== CHANNEL T1 =====

CPMG1: 100
P2C1: 60.00 ussec
P2L1: 128.00 dB
P2L2: 21.00 dB
SPG2: 199.8715994 MHz
=====
E2 - Processing parameters
SI: 30798
SF: 100.5472313 MHz
NMW: 30
SSB: 0
LB: 1.00 Hz
QF: 0
PC: 1.00

10.000 ppm
200.000 ppm
P1: 200.00 Hz
P2F: 0.000 ppm
PDMF: 5.00000 sec/sec
NDIM: 502,73613 bytes





Current Data Parameters

NAME 22455-100
EXPNO 2
PROCNO 1

P2 - Acquisition Parameters

Date 20060329
Time 15:24
INSTRUM spect
PROVIDER 5 mm QNP 1H/13C
PULPROG zg30
TD 131072
SOLVENT CDCl3
NS 4
SWH 40972.582 Hz
ETR 0.307637 Hz
AQ 1.625552 sec
RG 8192
TM 12.400 usec
DE 6.00 usec
TE 300.0 °K
D1 0.1000000 sec
T1 0.0300000 sec
TDREFST 0.0000000 sec
MCNMR 0.0300000 sec

===== CHANNEL 1 =====

NUCL 13C
PL 2.50 usec
B1L 0.00 dB
SPOL 125.7705643 MHz

===== CHANNEL 12 =====

NUCL 13C
CPDPRG2 w11t15
PCPD1 80.00 usec
PCPD2 11.50 dB
PL12 11.50 dB
SP02 500.132506 MHz

F1 - Processing parameters

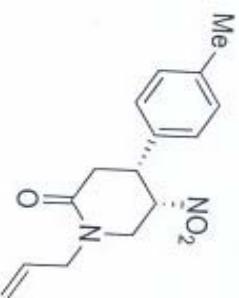
SI 6996
SF 125.7579627 MHz
RMW 8K
SSB 0
LB 1.00 Hz
GB 0
PC 0.20

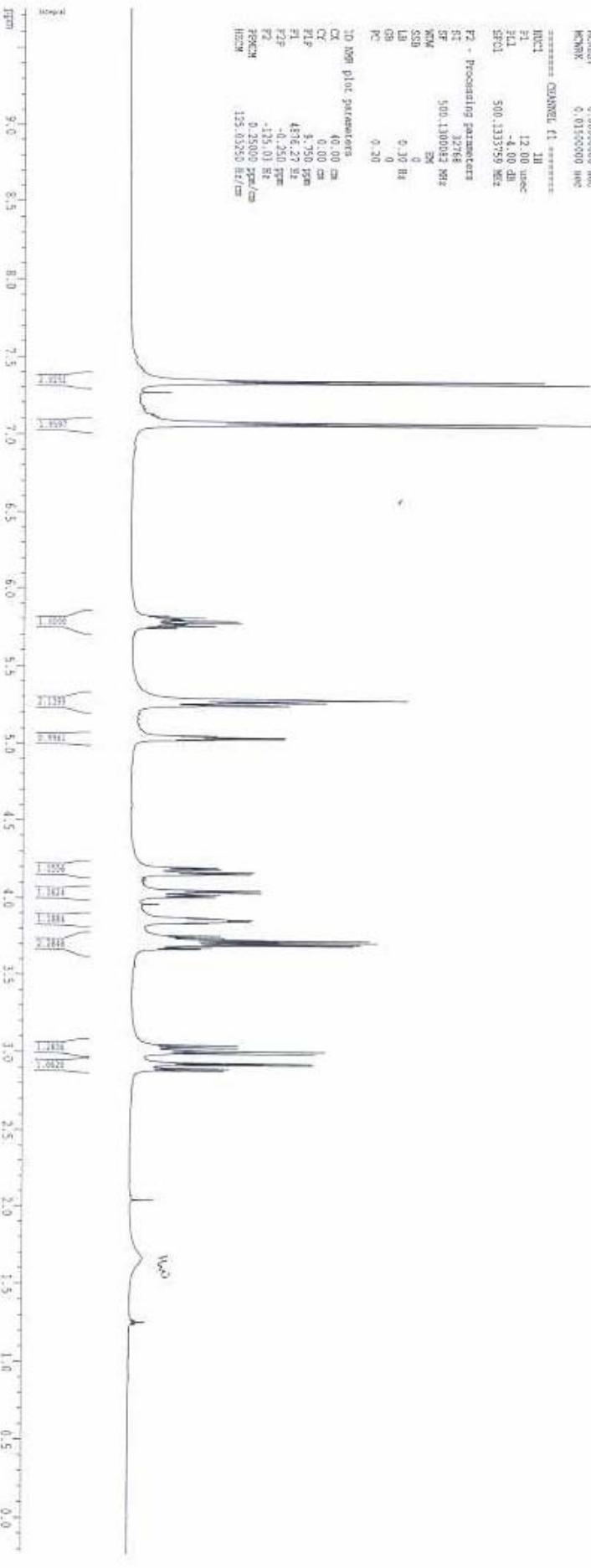
1D WAVE parameters

CK	40.00 cm
CV	0.00 cm
TF	100.000 ppm
TL	3.3893.97 Hz
TP	0.00 ppm
T2	0.00 Hz
TPWEN	4.7500 ppm/cm
ROHM	597.3493 Hz/cm

82.42
77.84
77.59
77.57
77.49
77.41
77.39
77.34
77.23
77.13
77.11
76.98

49.47
46.16
40.96
34.19
21.26





P2 - Acquisition Parameters

DATE	20090118
NAME	1
SCANS	1
TE(MS)	16.12
T1(MS)	1.00
TD	65536
SIMBLT	CW13
SWF	32
AS	4
SWE	10944.912 Hz
FWHM	0.167311 sec
NO	1.988313 sec
SD	125
DM	45.000 usec
D2	6.00 usec
TE	350.0 deg
CL	0.100000 sec
MPSAT	0.000000 sec
NCURR	0.0150000 usec

P2 - Processing parameters

DATA1	1H
NUC1	1H
TE1	12.00 usec
PL1	-4.00 dB
SF1	500.133759 MHz

==== CHANNEL 11 =====

P2 - Processing parameters

ST	32768
SF	500.130043 kHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	0.20

1D NMR parameters

CD	40.00 cm
CY	0.00 cm
CP	5.750 ppm
FI	486.27 Hz
F2P	-0.250 ppm
F2	-175.01 Hz
PPM	0.25000 ppm/cm
HEM	12.6326 Hz/cm

P2 - Acquisition Parameters

DATE	20090118
NAME	1
SCANS	1
TE(MS)	16.12
T1(MS)	1.00
TD	65536
SIMBLT	CW13
SWF	32
AS	4
SWE	10944.912 Hz
FWHM	0.167311 sec
NO	1.988313 sec
SD	125
DM	45.000 usec
D2	6.00 usec
TE	350.0 deg
CL	0.100000 sec
MPSAT	0.000000 sec
NCURR	0.0150000 usec

P2 - Processing parameters

DATA1	1H
NUC1	1H
TE1	12.00 usec
PL1	-4.00 dB
SF1	500.133759 MHz

==== CHANNEL 11 =====

P2 - Processing parameters

ST	32768
SF	500.130043 kHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	0.20

1D NMR parameters

CD	40.00 cm
CY	0.00 cm
CP	5.750 ppm
FI	486.27 Hz
F2P	-0.250 ppm
F2	-175.01 Hz
PPM	0.25000 ppm/cm
HEM	12.6326 Hz/cm

P2 - Acquisition Parameters

DATE	20090118
NAME	1
SCANS	1
TE(MS)	16.12
T1(MS)	1.00
TD	65536
SIMBLT	CW13
SWF	32
AS	4
SWE	10944.912 Hz
FWHM	0.167311 sec
NO	1.988313 sec
SD	125
DM	45.000 usec
D2	6.00 usec
TE	350.0 deg
CL	0.100000 sec
MPSAT	0.000000 sec
NCURR	0.0150000 usec

P2 - Processing parameters

DATA1	1H
NUC1	1H
TE1	12.00 usec
PL1	-4.00 dB
SF1	500.133759 MHz

==== CHANNEL 11 =====

P2 - Processing parameters

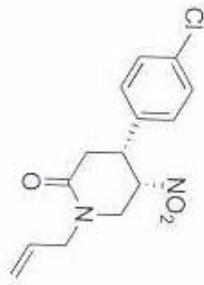
ST	32768
SF	500.130043 kHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	0.20

1D NMR parameters

CD	40.00 cm
CY	0.00 cm
CP	5.750 ppm
FI	486.27 Hz
F2P	-0.250 ppm
F2	-175.01 Hz
PPM	0.25000 ppm/cm
HEM	12.6326 Hz/cm

 -7.34760
 -7.33678
 -7.31866
 -7.26938
 -7.07774
 -7.06084

 -5.81223
 -5.80395
 -5.79985
 -5.79112
 -5.77866
 -5.77017
 -5.76614
 -5.75784
 -5.28670
 -5.28408
 -5.26821
 -5.24900
 -5.05554
 -5.04469
 -5.03628
 -5.02520
 -4.20126
 -4.18907
 -4.17140
 -4.15922
 -4.05604
 -4.04347
 -4.02629
 -4.01368
 -3.86814
 -3.86258
 -3.85475
 -3.84876
 -3.76019
 -3.74805
 -3.73303
 -3.72092
 -3.70926
 -3.69888
 -3.68212
 -3.04795
 -3.03232
 -3.01226
 -2.99666
 -2.93668
 -2.92399
 -2.92101
 -2.88831



ppm 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

Current Data Parameters
NAME: 224556-79
ENSO
H2O2D
FD2 - Acquisition Parameters
Date: 20051208
Time: 15:17
INSTRNMNT: spect
PROBODIM: 5 mm DNP JMR19
TE: 130.0 sec
TD: 131024
SCNTR: 438
DW: 40122.582 Hz
NEXES: 307612
AQ: 1.625552 sec
RG: 8122
DW: 12.400 usec
TE: 6.00 usec
SR: 300.0 Hz
D1: 0.000000 sec
D11: 0.000000 sec
ACQST: 0.000000 sec
MEMR: 0.0150000 sec
===== CHANNEL F1 =====
MSTL: 13C
PR: 2.50 usec
PL: 0.00 dB
SP01: 155.770583 Hz
===== CHANNEL F2 =====
CPSIG02: waltz16
MSTL: 2H
PRFO: 80.00 usec
PLF2: 11.50 dB
PL12: 11.50 dB
SP02: 500.1325096 Hz
F1 - Processing parameters
SI: 65536
SP: 125.757763 Hz
AVER: 200M
SWH: 0
LB: 1.00 Hz
DW: 0
PC: 0.20

11 NOE plot parameters
CX: 40.00 cm
CY: 0.00 cm
F1P: 140.000 ppm
F1I: 2436.40 Hz
F2B: 0.000 ppm
F2I: 0.00 Hz
FINCH: 4.50000 ppm/cm
HE2W: 5.55.50947 Hz/cm

F2 - Acquisition Parameters
Date: 20051208
Time: 15:17
INSTRNMNT: spect
PROBODIM: 5 mm DNP JMR19
TE: 130.0 sec
TD: 131024
SCNTR: 438
DW: 40122.582 Hz
NEXES: 307612
AQ: 1.625552 sec
RG: 8122
DW: 12.400 usec
TE: 6.00 usec
SR: 300.0 Hz
D1: 0.000000 sec
D11: 0.000000 sec
ACQST: 0.000000 sec
MEMR: 0.0150000 sec
===== CHANNEL F1 =====
MSTL: 13C
PR: 2.50 usec
PL: 0.00 dB
SP01: 155.770583 Hz
===== CHANNEL F2 =====
CPSIG02: waltz16
MSTL: 2H
PRFO: 80.00 usec
PLF2: 11.50 dB
PL12: 11.50 dB
SP02: 500.1325096 Hz
F1 - Processing parameters
SI: 65536
SP: 125.757763 Hz
AVER: 200M
SWH: 0
LB: 1.00 Hz
DW: 0
PC: 0.20

11 NOE plot parameters
CX: 40.00 cm
CY: 0.00 cm
F1P: 140.000 ppm
F1I: 2436.40 Hz
F2B: 0.000 ppm
F2I: 0.00 Hz
FINCH: 4.50000 ppm/cm
HE2W: 5.55.50947 Hz/cm

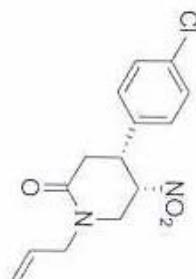
ppm 167.06
134.78
131.65
129.63
129.60
129.56
129.46
129.43
129.22
129.04
128.87
128.50
124.18
121.87
119.40
119.34
119.31

82.26
82.12
77.84
77.80
77.71
77.69
77.67
77.62
77.58
77.49
77.38
77.23
77.15
77.05
76.98
76.92
76.90
72.32
54.09

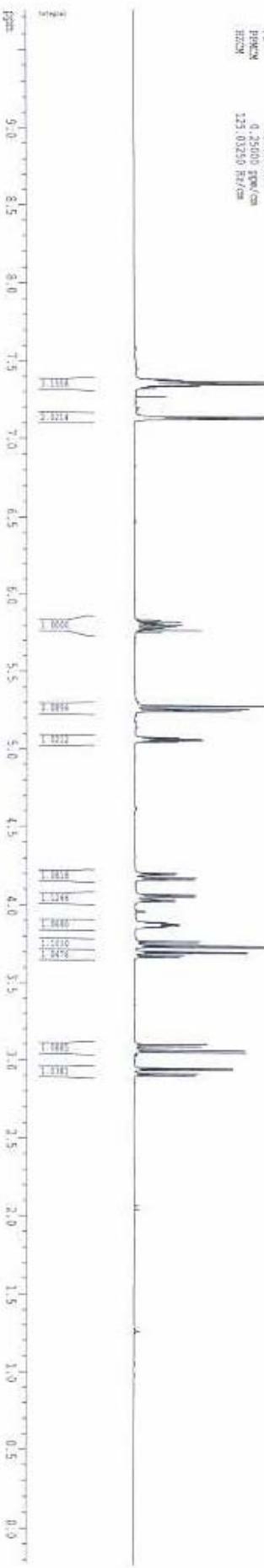
49.47
46.30
46.27
46.18
40.77
40.73
40.68

33.95

7.90

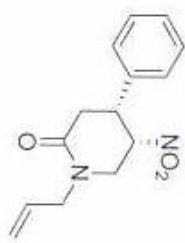


C-13 nmr500c



12.00 usec
-4.00 dB
500.1333759 MHz

7.37906
 7.36714
 7.36405
 7.36104
 7.35192
 7.35091
 7.34720
 7.33905
 7.33703
 7.14211
 7.13855
 7.13523
 7.13020
 7.12695
 7.12591
 7.12428
 7.12328
 5.82484
 5.80102
 5.79222
 5.77018
 5.28576
 5.28325
 5.28075
 5.26432
 5.26176
 5.25033
 5.24761
 5.07852
 5.07031
 5.06752
 5.05909
 5.04864
 4.20805
 4.19583
 4.17813
 4.16590
 4.06309
 4.05658
 4.03930
 4.02668
 3.88256
 3.87471
 3.77366
 3.76153
 3.74662
 3.73439
 3.70634
 3.69615
 3.67935
 3.66898
 3.31062
 3.30904
 3.07025
 3.05459
 2.95292
 2.94018
 2.91720
 2.90447



167.26

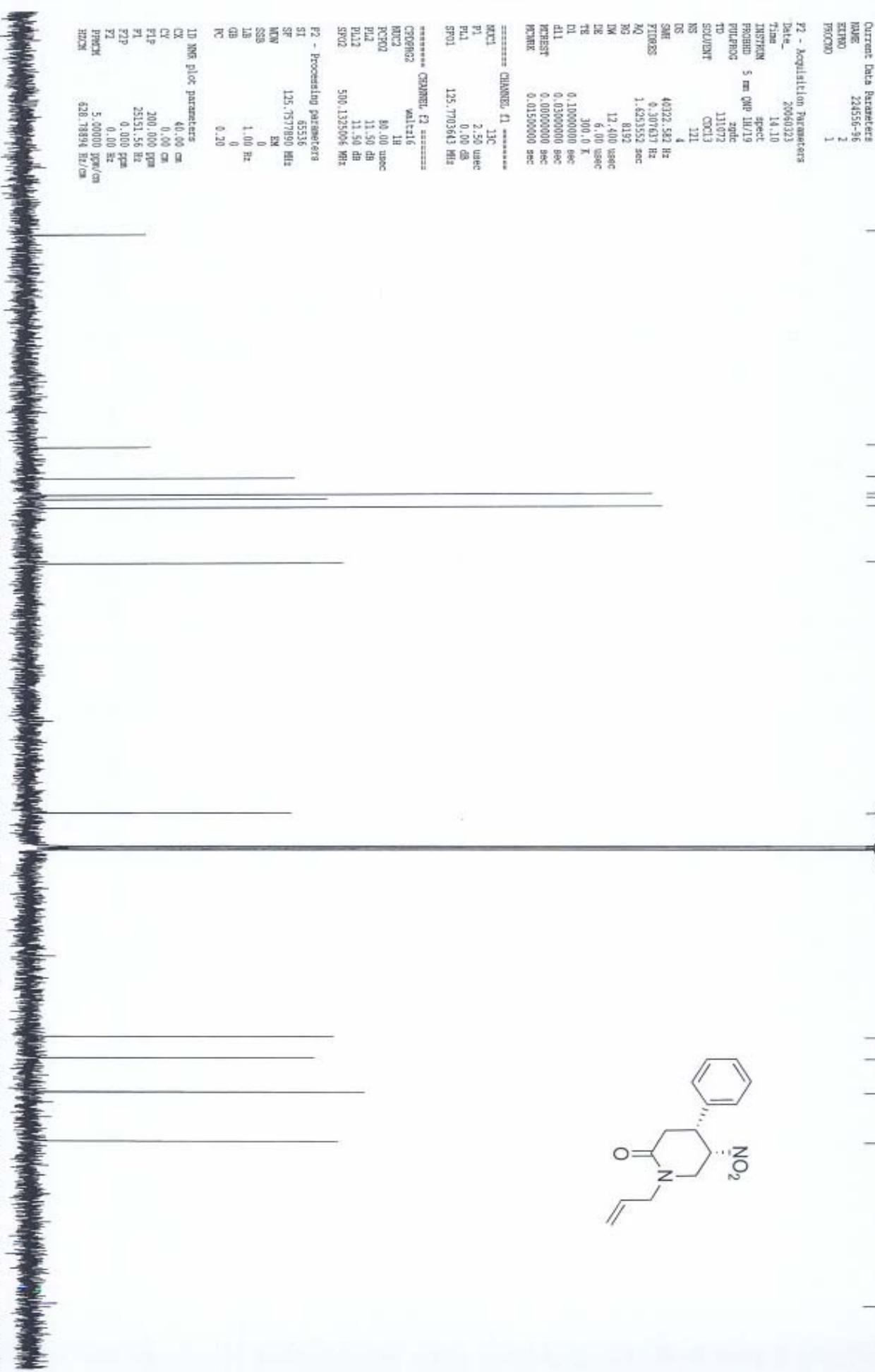
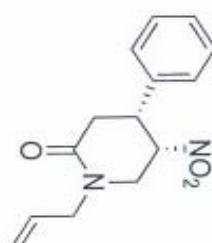
136.10

119.03

82.16
77.38
77.28
77.12
77.03
76.96
76.92
76.78

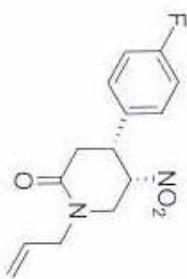
49.25
46.07
41.04
33.81

10.10

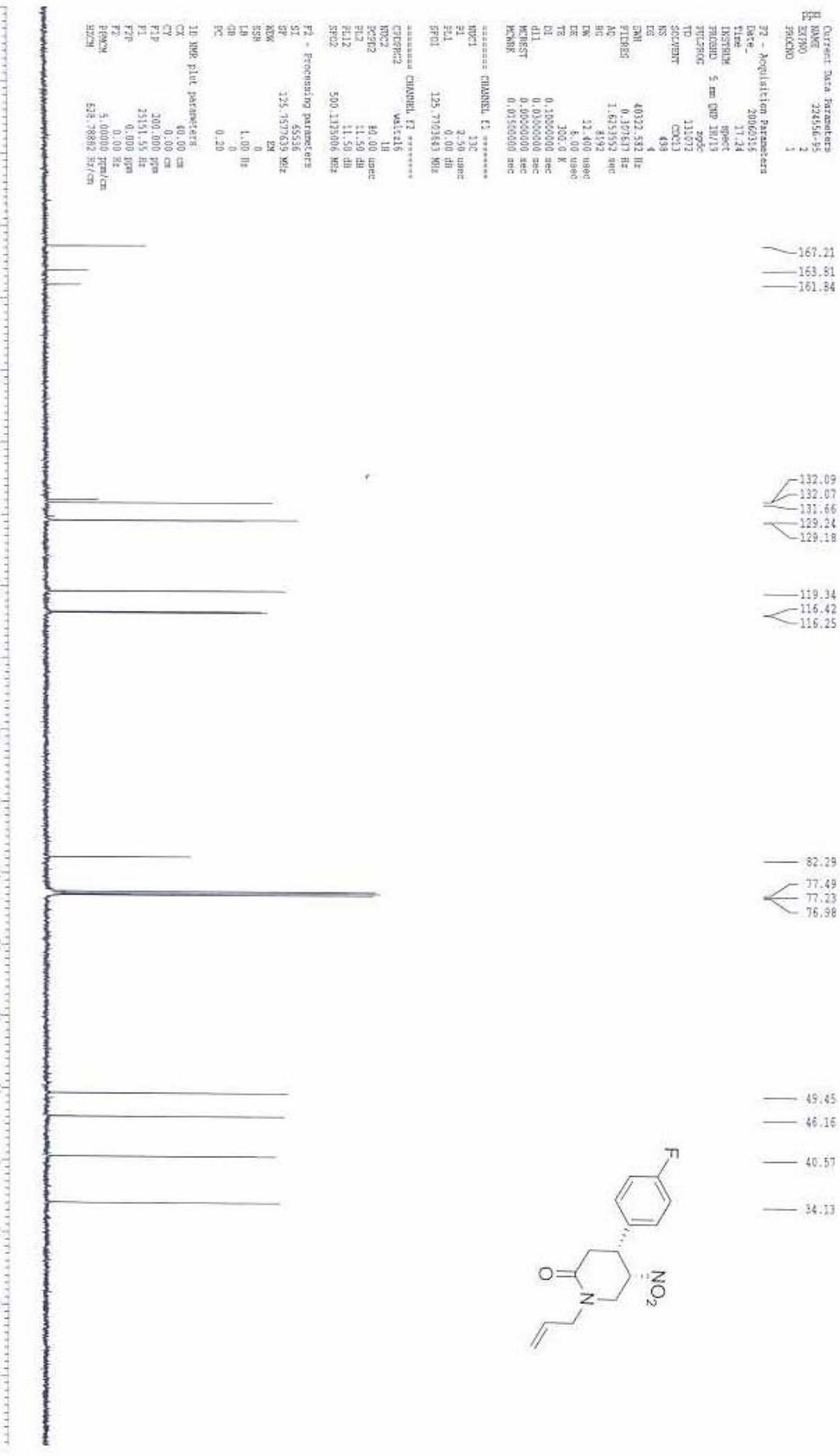


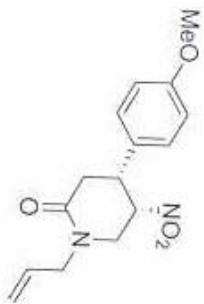
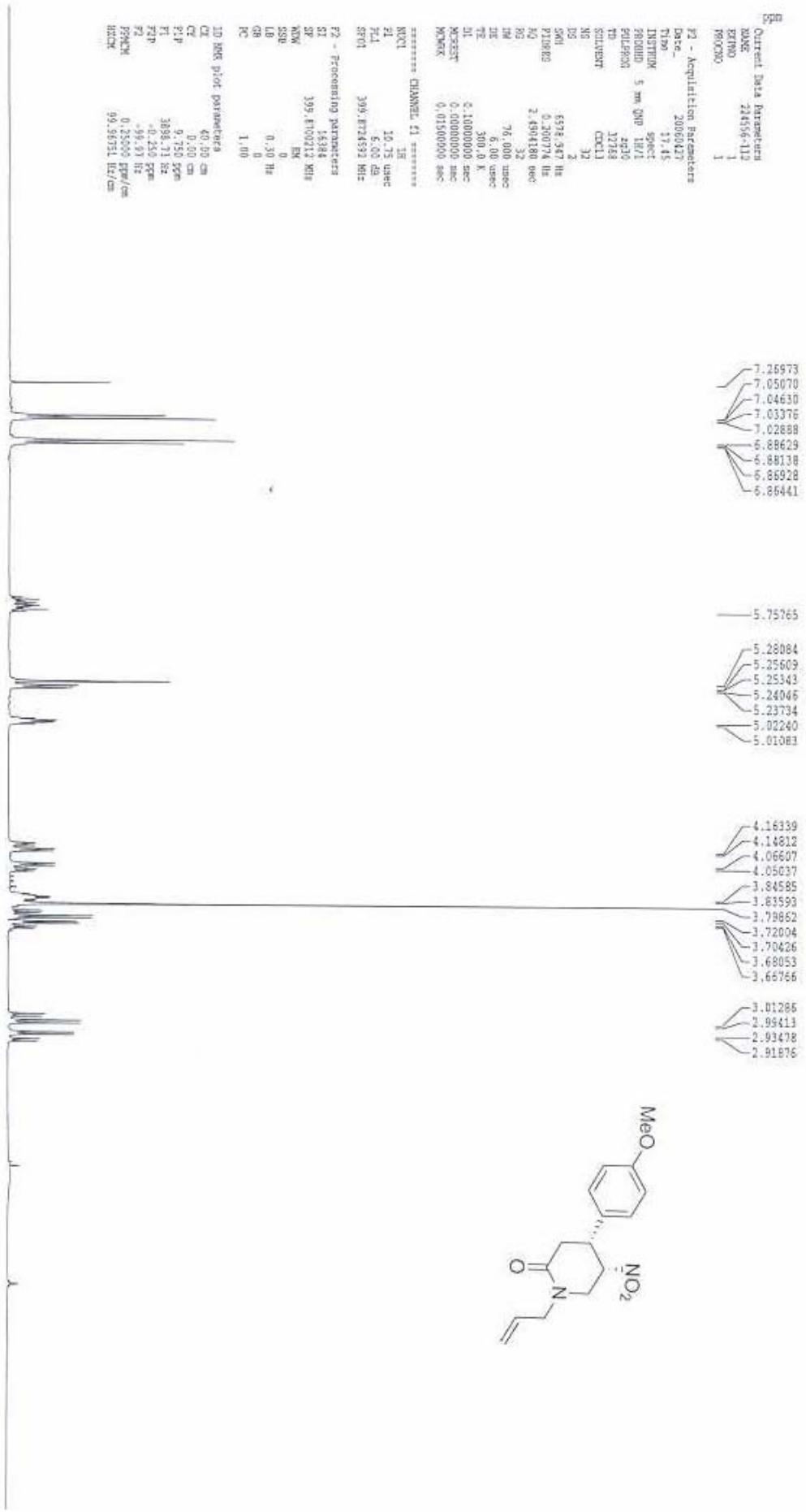


7.12059
7.11808
7.11022
7.10720
7.10283
7.09599
7.09250
7.06724
7.06258
7.05165
7.05035
7.04599
7.03720
7.03292
5.81152
5.79050
5.77787
5.75705
5.28230
5.28049
5.26372
5.26142
5.25928
5.24697
5.24397
5.05238
5.04418
5.04129
5.03318
5.03038
5.02189
4.19911
4.18686
4.16517
4.15697
4.05605
4.03430
4.02612
4.01347
3.87097
3.86307
3.75669
3.74467
3.72956
3.71754
3.70669
3.69630
3.67954
3.05000
3.03432
3.01430
2.99861
2.93731
2.92455
2.90160
2.88885

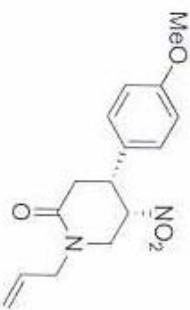
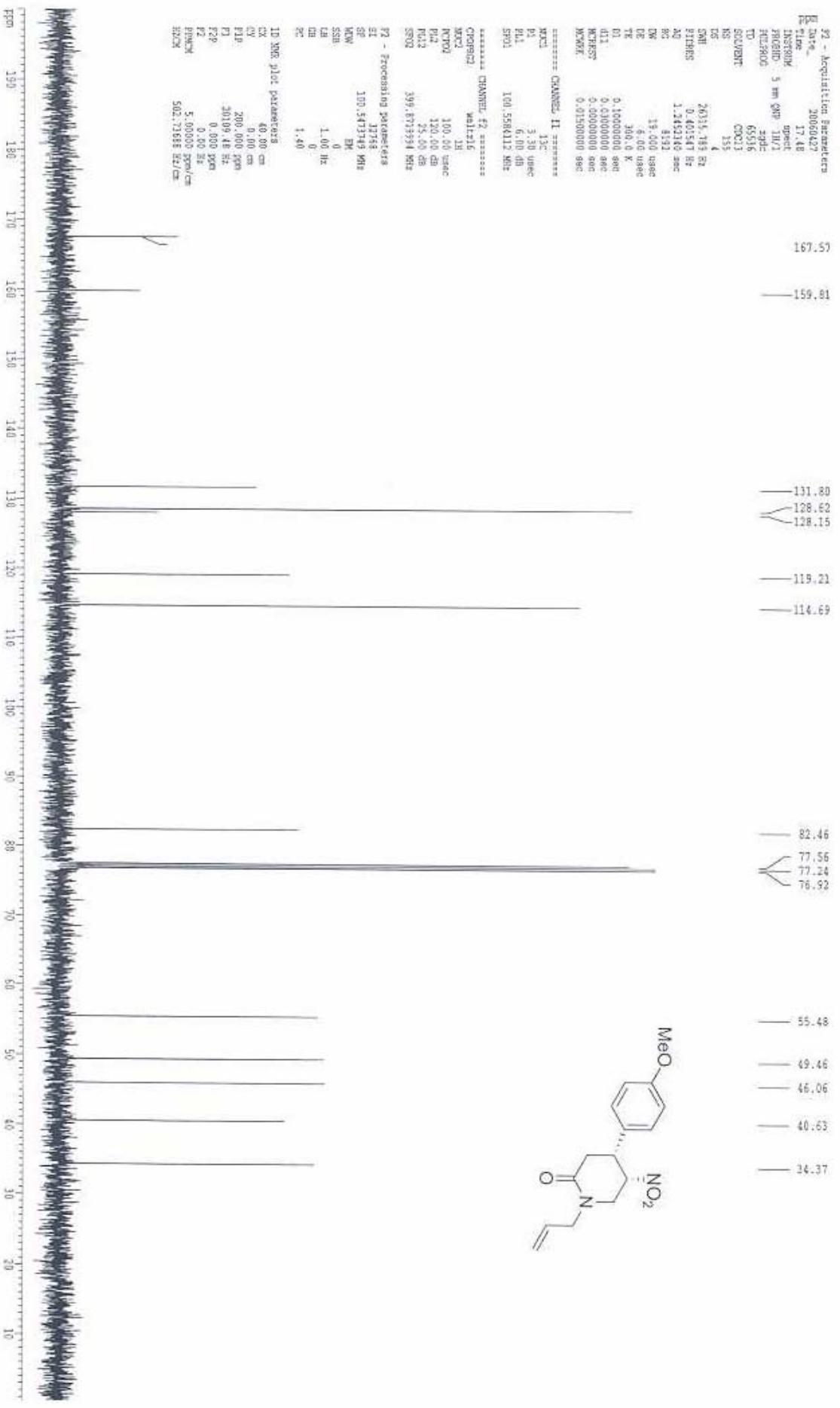


p-Nitroacetan

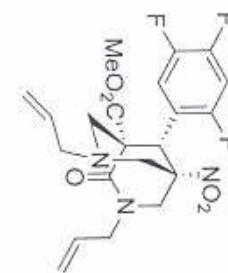
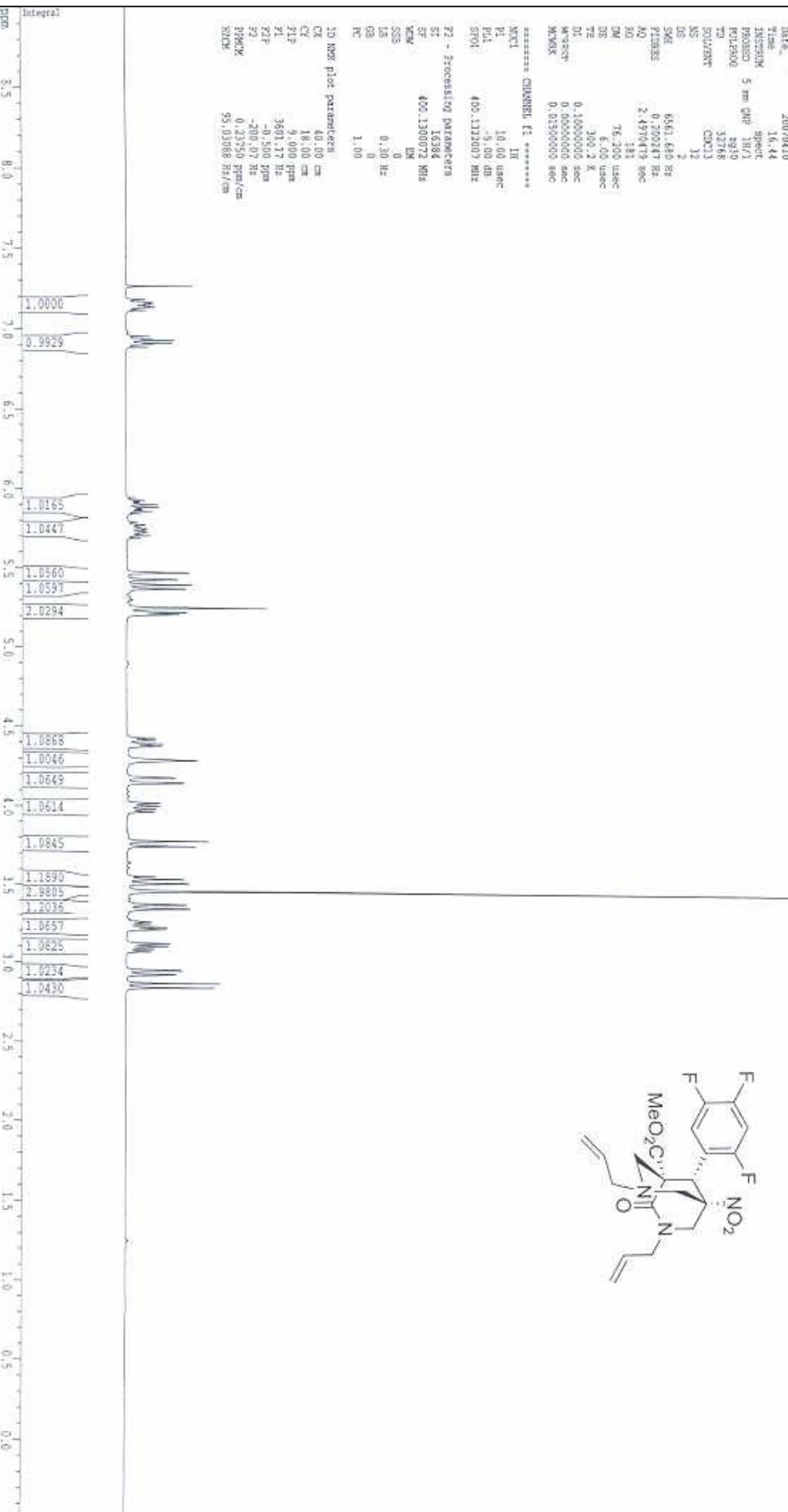


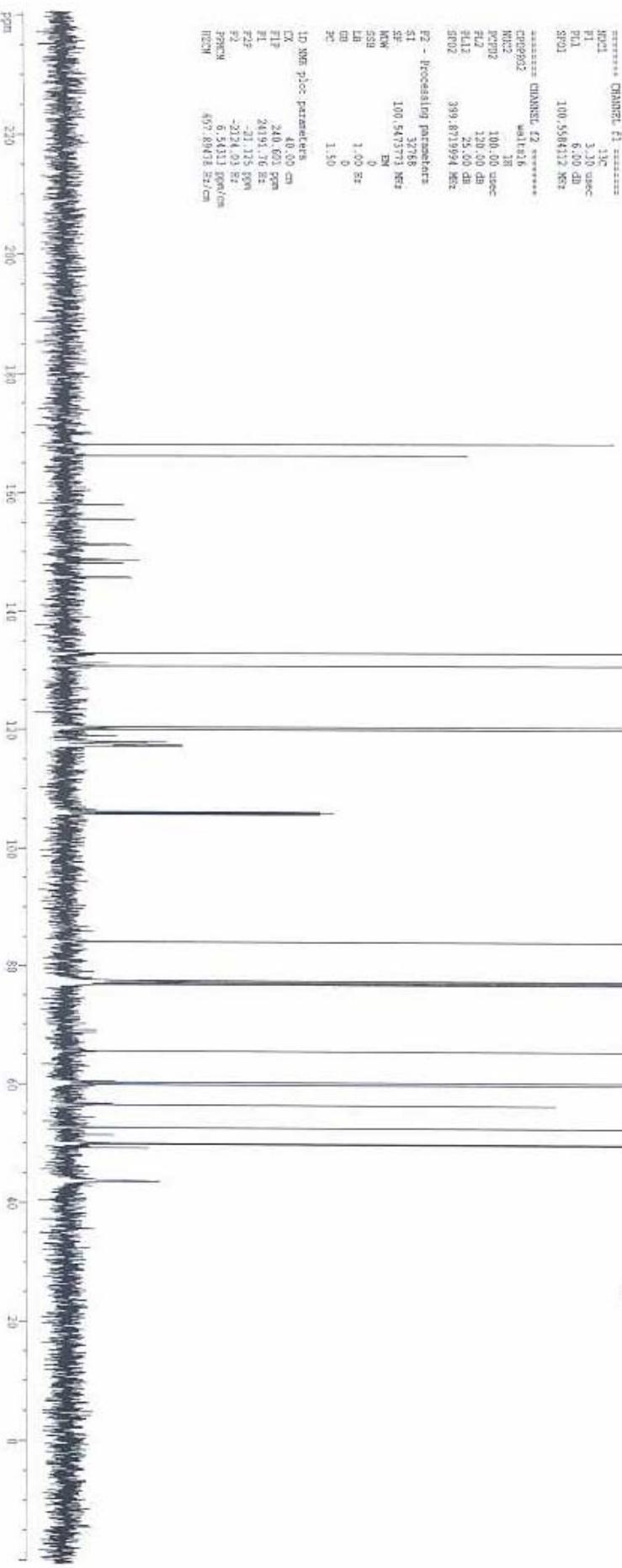


nmr400b c-13 (h-1 decoupled)



Current Data Parameters	
Date...	20070410
Sample	1
PSOCO	1
P2 - Acquisition Parameters	
TE...	16.44
TR...	1.000000
TE/TR	spec
FWHM	5 mm GPP
FWHM0	2.920
TD	3.786
POLYENT	CDC13
NS	12
SC	2
SMR	6547.48 Hz
FLBES	0.020241 Hz
AD	2.47049 sec
SD	1.11
DW	76.200 us
DS	6.00 us
TZ	300.2 sec
DP	0.1000000 sec
DPGTY	0.0000000 sec
MDIMX	0.0100000 sec
***** CHANNEL 1: *****	
MUL	III
PL	10.00 us
PD	5.00 us
SP01	400.112003 MHz
P1 - Processing parameters	
P1	1.63084
SP	400.130072 MHz
WEM	
SSS	0
LB	EM
IS	0.30 Hz
GB	0
PC	1.00
1D FID plot parameters	
CM	40.00 cm
CH	18.00 cm
CP	9.000 ppm
PL	3001.17 Hz
FP	-0.500 ppm
F2	-200.37 Hz
DP00K	0.27500 ppm
DP0K	95.0008 Hz



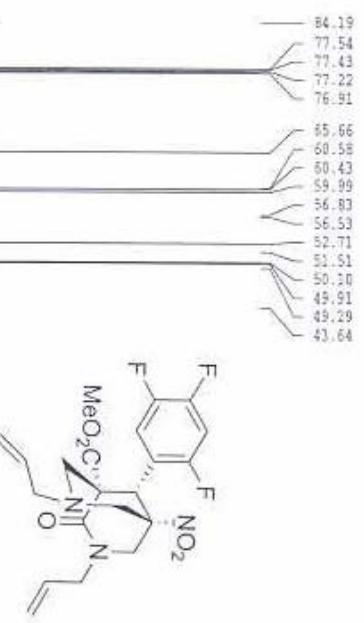


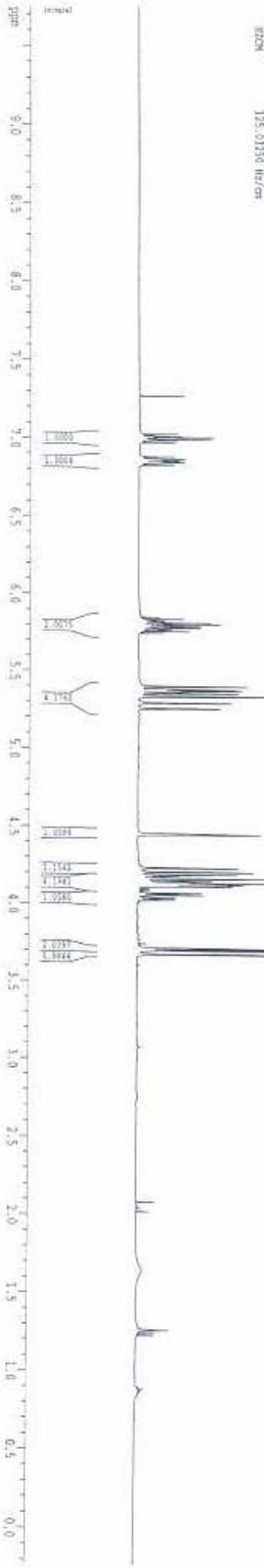
F1 - Acquisition Parameters
 Date_ 20070411
 Time 11:20
 IN520W 5 mm QNP
 BEAKER 1H T1
 T1L750G 65536
 SOLVENT CDCl3
 D5 1351
 D6 1351
 T6 300.0 K
 P61 2615.789 Hz
 P62 0.40547 Hz
 P71 1.245230 sec
 F6 6132
 F7 19,000 usec
 D7 6.00 usec
 T7 0.1100000 sec
 D11 0.0300000 sec
 MESTET 0.0000000 sec
 M62X 0.0150000 sec

******* CHANNEL F1 *******
 NO2C 13C
 PT 3.20 usec
 P1 6.00 dB
 SW1 1.00, 55.84112 MHz

******* CHANNEL F2 *******
 NO2C 1H
 DPPG 100.00 usec
 P2 120.00 dB
 F1,2 25.00 dB
 SW2 339.871994 Hz

F2 - Processing parameters
 ST 37.688
 SP 100.573773 Hz
 MW EN
 NS 0
 SW 1.00 Hz
 OB 0
 PC 1.50

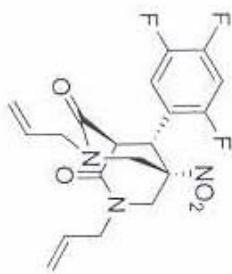


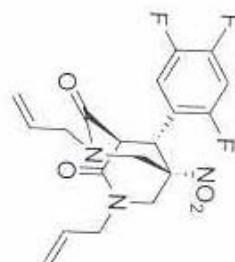
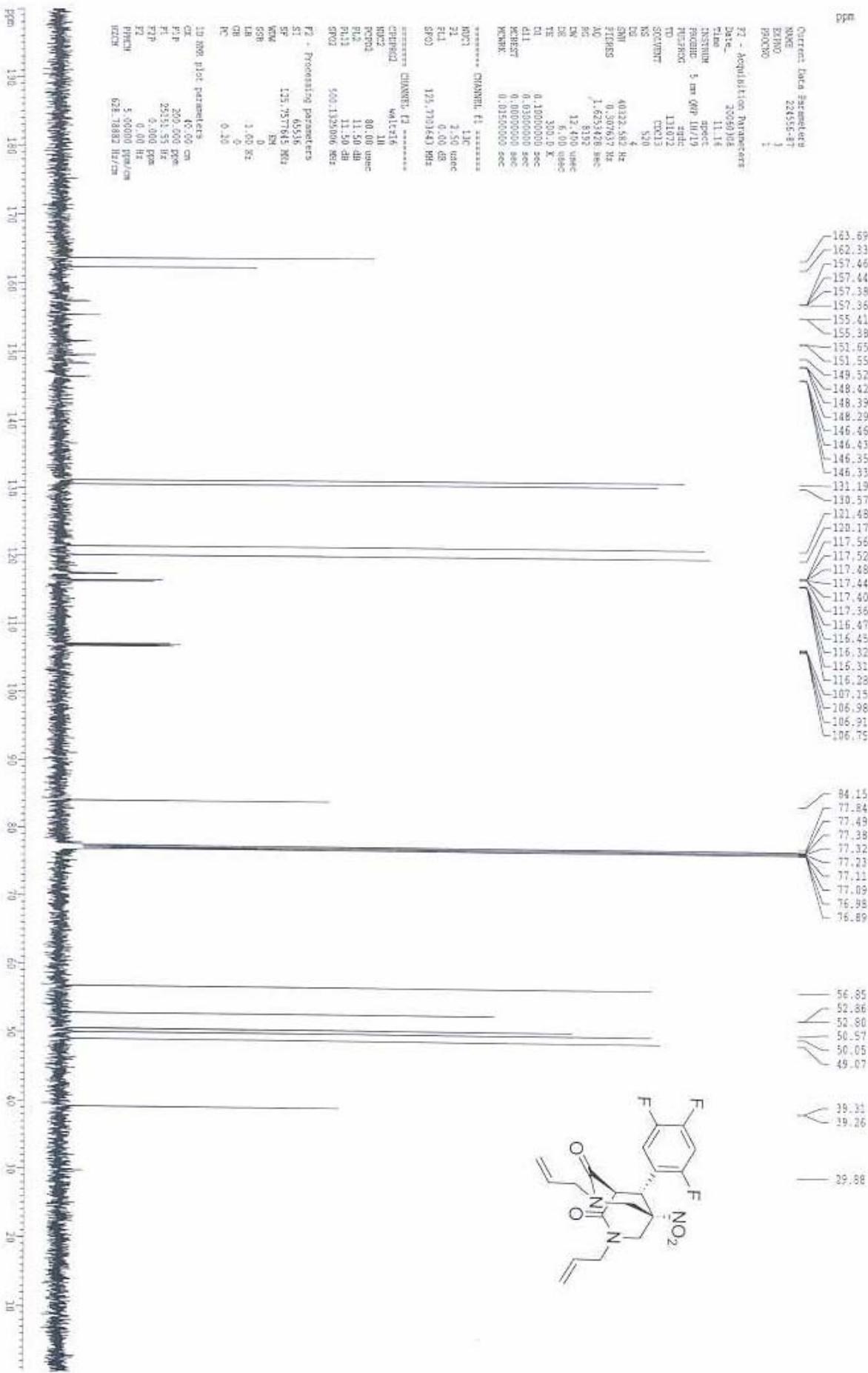


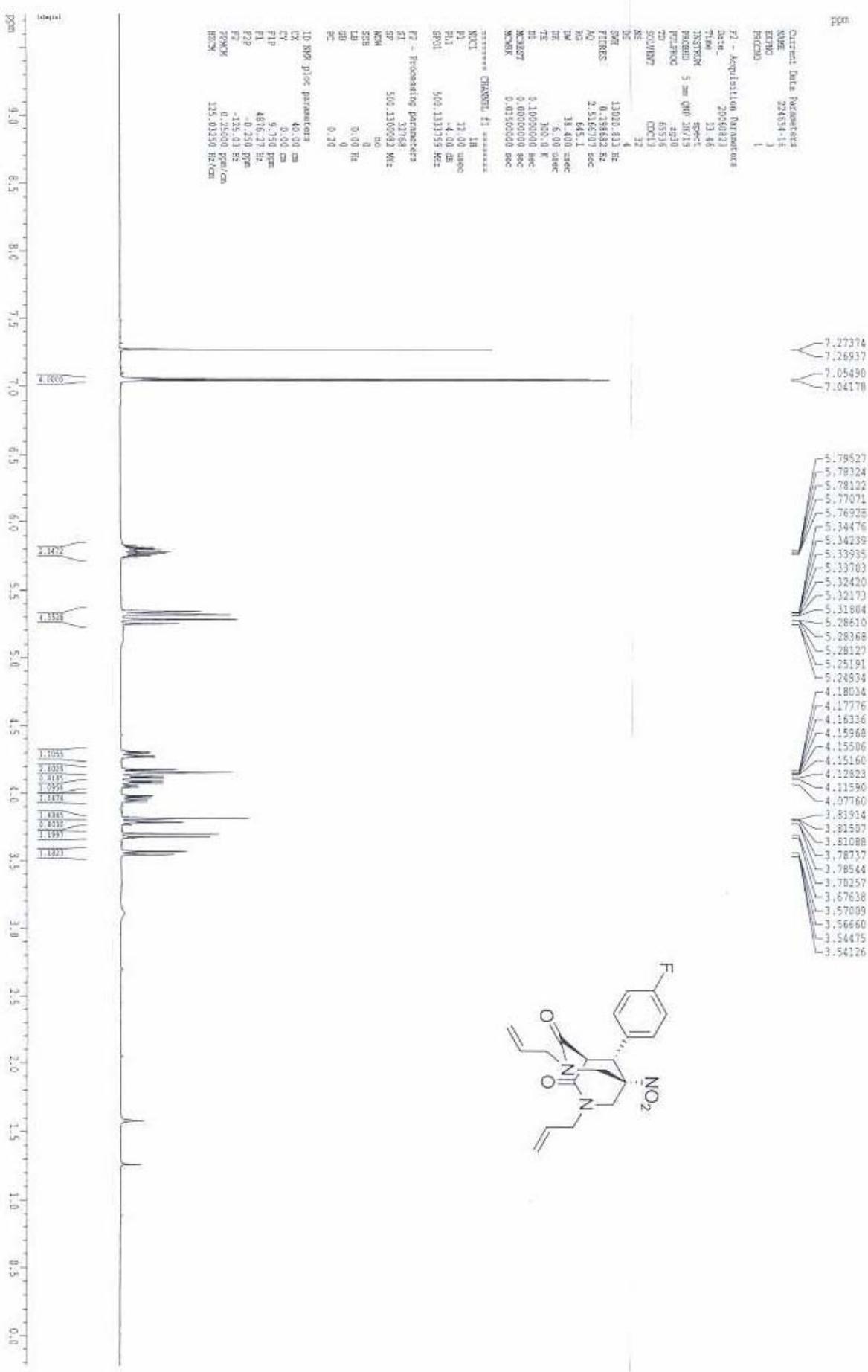
=====
R2 - Acquisition Parameters
Date_ 20060317
Time 16:39
INSTRUM spect
PROBOD 5 mm QNP HBT19
TOMGR32 30720
TD 65536
SWH 6312500 Hz
RWA 456.1
DW 18400 usec
DE 5.00 usec
TP 300.0 sec
TM 0.1000000 sec
AQ 0.0000000 sec
NCYC 0.0150000 sec
=====
QCPMG F1 =====
AQ1 1H
TP1 12.00 usec
PA1 -4.00 deg
SW1 500.133333 MHz
=====
H2 - Processing parameters
SI 22768
SF 500.130092 MHz
WDW no
SSB 0
LB 0.10 Hz
TR 4.00
TC 1.40
PC 0.2088
=====
1D NMR plot parameters
CDX 0.05 cm
P1F 9.150 ppm
FL1 4676.21 Hz
F2P -0.755 ppm
F2Z -175.01 Hz
PPM1 0.15000 Hz/cm
PPM2 125.01250 Hz/cm

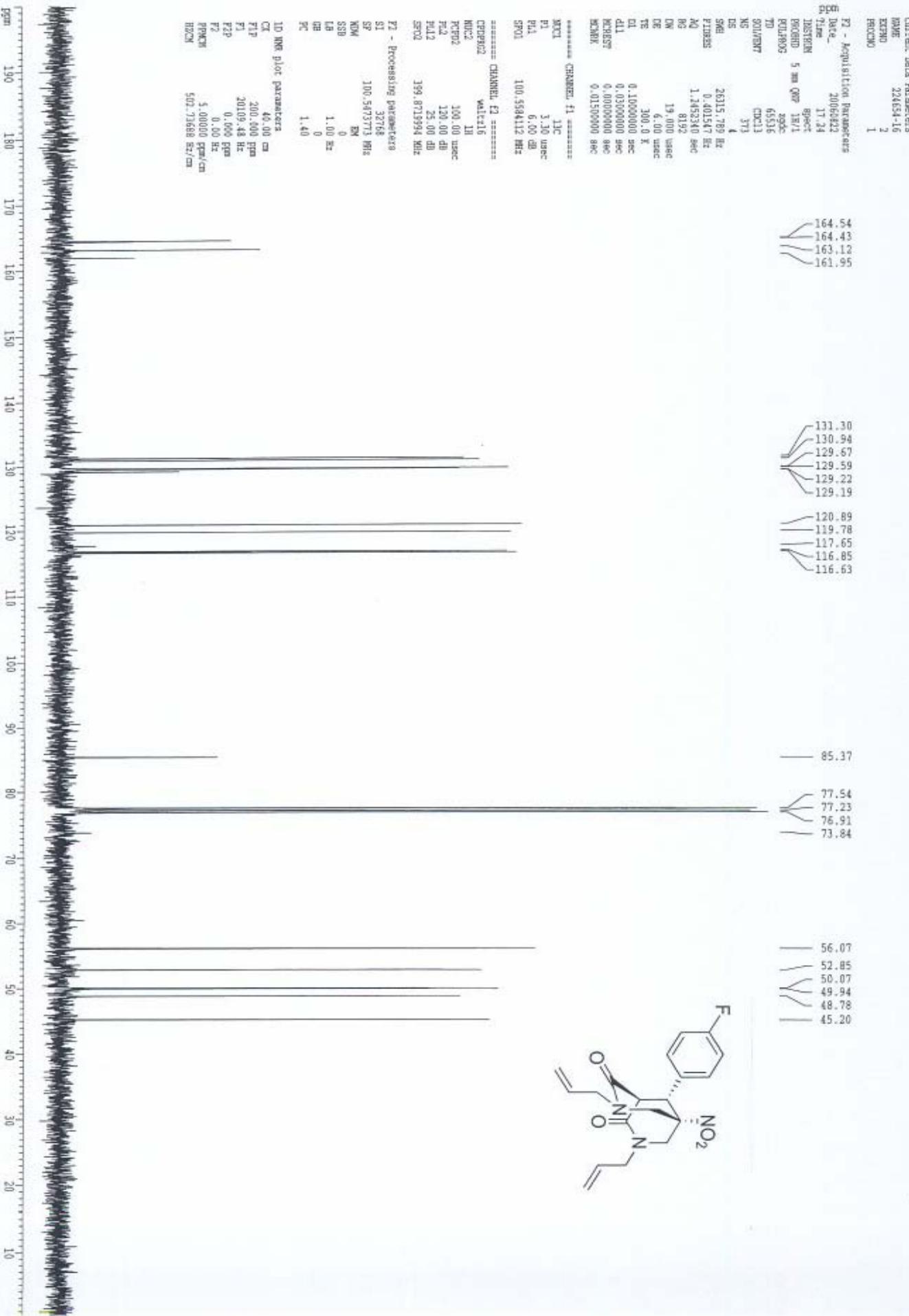
7.00473
6.99149

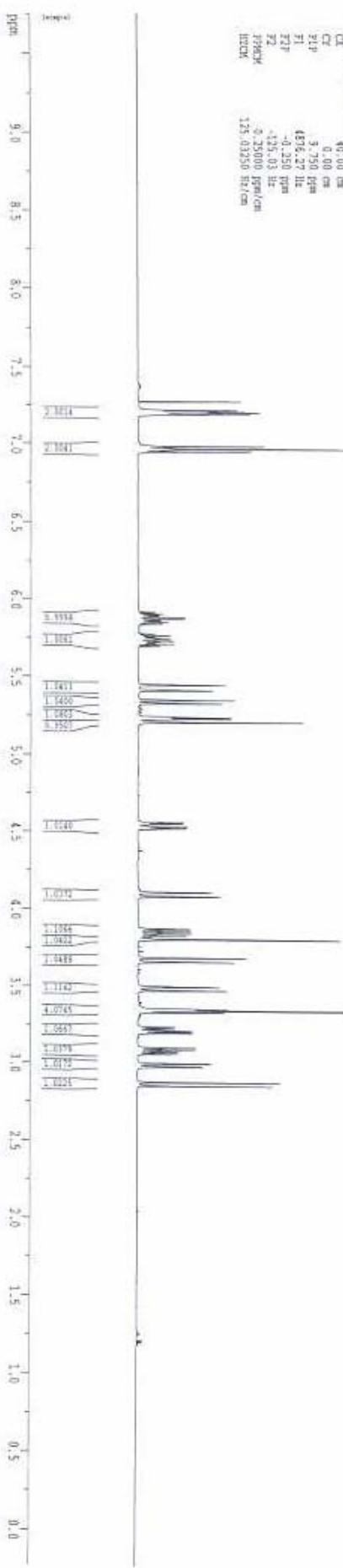
5.81534
5.80170
5.39761
5.37781
5.36940
5.36720
5.35338
5.35220
5.33317
5.29175
5.28941
5.25752
5.25519
4.44954
4.23400
4.23183
4.20850
4.20846
4.19086
4.16428
4.14984
4.13703
4.12871
4.12627
4.12329
4.11530
3.71877
3.71425
3.70319
3.67784











Current Data Parameters
NAME: 22654-138
STIM: 1
PRONO: 1

P1 - Acquisition Parameters
Date: 2007/11/13
Time: 16:24
INSTRUM: spect
PROBOD: 5 mm QPP 1H/13
PULPROG: 2010
TD: 6576
SOLVENT: CDCl3
NS: 32
DS: 4
SW1: 14500.813 Hz
SW2: 9198682 Hz
A2: 2.516787 sec
R2: 181
D8: 38.400 usec
D9: 6.00 usec
TE: 300.0 °K
D1: 0.1000000 sec
MEST: 0.0000000 sec
NMOM: 0.0150000 sec

***** CHANNELS: F1 *****
NUCL: 1H
PL: 10.75 usec
HL: -4.00 dB
SP: 3.001331759 MHz

P2 - Processing parameters
ST: 3.788
SF: 1300208 MHz
NM: 00
SSB: 0
LB: 0.00 Hz
GB: 0
RC: 0.20

13 NMR Plot parameters
CX: 40.00 cm
CY: 0.00 cm
P1: 3.750 Hz
F1: 483.27 Hz
T1: -0.200 ppm
F2: -423.03 Hz
P2W: 0.25000 ppm/cm
H2CM: 125.03250 Hz/cm

P3 -
Current Data Parameters
NAME: 22654-138
STIM: 1
PRONO: 1

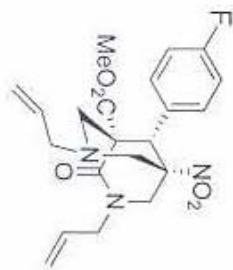
P1 - Acquisition Parameters
Date: 2007/11/13
Time: 16:24
INSTRUM: spect
PROBOD: 5 mm QPP 1H/13
PULPROG: 2010
TD: 6576
SOLVENT: CDCl3
NS: 32
DS: 4
SW1: 14500.813 Hz
SW2: 9198682 Hz
A2: 2.516787 sec
R2: 181
D8: 38.400 usec
D9: 6.00 usec
TE: 300.0 °K
D1: 0.1000000 sec
MEST: 0.0000000 sec
NMOM: 0.0150000 sec

***** CHANNELS: F1 *****
NUCL: 1H
PL: 10.75 usec
HL: -4.00 dB
SP: 3.001331759 MHz

P4 -
Current Data Parameters
NAME: 22654-138
STIM: 1
PRONO: 1

P1 - Acquisition Parameters
Date: 2007/11/13
Time: 16:24
INSTRUM: spect
PROBOD: 5 mm QPP 1H/13
PULPROG: 2010
TD: 6576
SOLVENT: CDCl3
NS: 32
DS: 4
SW1: 14500.813 Hz
SW2: 9198682 Hz
A2: 2.516787 sec
R2: 181
D8: 38.400 usec
D9: 6.00 usec
TE: 300.0 °K
D1: 0.1000000 sec
MEST: 0.0000000 sec
NMOM: 0.0150000 sec

***** CHANNELS: F1 *****
NUCL: 1H
PL: 10.75 usec
HL: -4.00 dB
SP: 3.001331759 MHz



ppm

Current Data Parameters
NAME: 22654-138
EPRID: 2
PROB: 1

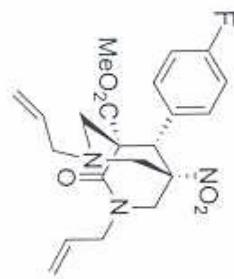
F2 - Acquisition Parameters
DATE: 20070413
TIME: 16:32
INSTRUM: spect
PROBOD: 5 mm QNP 1W13
PULPROG: zqzdc
TD: 131024
SCANS: 1024
NS: 32
SW1: 40322.582 Hz
PT1: 0.01611 Hz
AQ: 1.625552 sec
RG: 8192
DW: 17.400 usec
DE: 6.00 usec
TE: 300.0 K
D1: 0.1000000 sec
D11: 0.0100000 sec
NOESYD1: 0.0000000 sec
NOESYD11: 0.0100000 sec
SCAMM: 0.600

***** CHANNEL f1 *****
NO1: 13C
P1: 7.50 usec
T1: 0.00 dB
SP1: 175.7703643 Hz

***** CHANNEL f2 *****
CYTRIM2: waltz16
NUC2: 1H
TOPQ: 80.00 usec
TQD: 1.50 dB
F2A: 11.50 dB
F2B: 11.50 dB
SSPQ: 500.132056 MHz

F2 - Processing parameters
SI: 65536
SF: 115.7571845 MHz
RM: 0
SSB: 0
LB: 1.00 Hz
GS: 1.00
T: 1.40

1D ACP plot parameters:
CX: 0.00 cm
CY: 0.00 cm
F1P: 265.589 ppm
F1I: 32761.04 Hz
F2P: -60.128 ppm
F2I: -7581.54 Hz
PPM9: 8.01592 ppm/cm
HECM: 1008.04491 Hz/cm



ppm

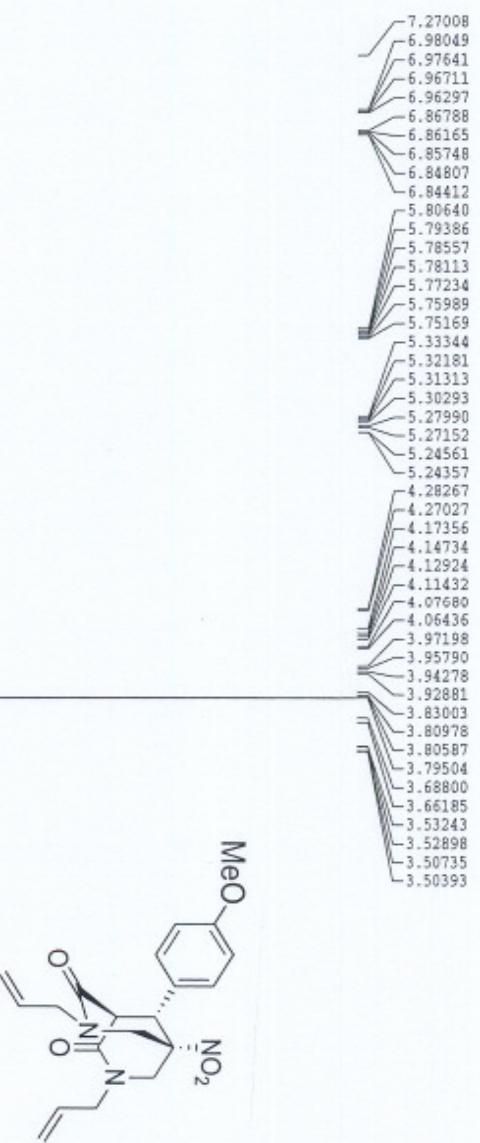
240 220 200 180 160 140 120 100 80 60 40 20 0 -20 -40

ppm

Current Data Parameters
NAME 22456-136A
EXPO 1
PROCNO 1

P2 - Acquisition Parameters

Date_ 20060904
Time 14:56
INSTRUM spect
PROBID 5 mm QNP WB19
RJMAGN 2510
TD 65536
SOLVENT CDCl3
NS 33
DS 4
SWH 13020.813 Hz
PI0ES 0.198662 Hz
AQ 2.5166707 sec
RG 322.5
DW 38.400 usec
DE 6.00 usec
TE 300.0 K
OL 0.1000000 SEC
D1 0.0000000 SEC
DWEST 0.0000000 SEC
NDWAV 0.0150000 SEC



-7.27008
-6.98049
-6.97641
-6.96711
-6.96297
-6.86788
-6.86165
-6.85748
-6.84807
-6.84412
-5.80640
-5.79386
-5.78557
-5.78113
-5.77234
-5.75989
-5.75169
-5.33344
-5.32181
-5.31313
-5.30293
-5.27990
-5.27152
-5.24561
-5.24357
-4.28267
-4.27027
-4.17356
-4.14734
-4.12924
-4.11432
-4.07680
-4.06436
-3.97198
-3.95790
-3.94278
-3.92881
-3.83003
-3.80978
-3.80587
-3.79504
-3.68800
-3.66185
-3.53243
-3.52898
-3.50735
-3.50393

==== CHANNEL f1 ======
MCHL 1H
PL 12.00 usec
PUL 4.00 dB
SP01 500.133759 MHz

P2 - Processing parameters

ST 32768
SP 500.1330082 MHz
MW no
SSB 0
LB 0.00 Hz
GS 0
PC 0.20

1D NMR plot parameters

CY 40.00 cm
CP 0.00 cm
F1P 9.750 ppm
F1 4876.27 Hz
F2P -0.250 ppm
F2 -125.03 Hz
PRSW 0.20000 ppm/cm
DCX 125.03250 Hz/cm

ppm 10.0 9.5 9.0 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 0.0

ppm

Current Data Parameters
 DME 224554.sx
 EXPD 1
 TROCO 1

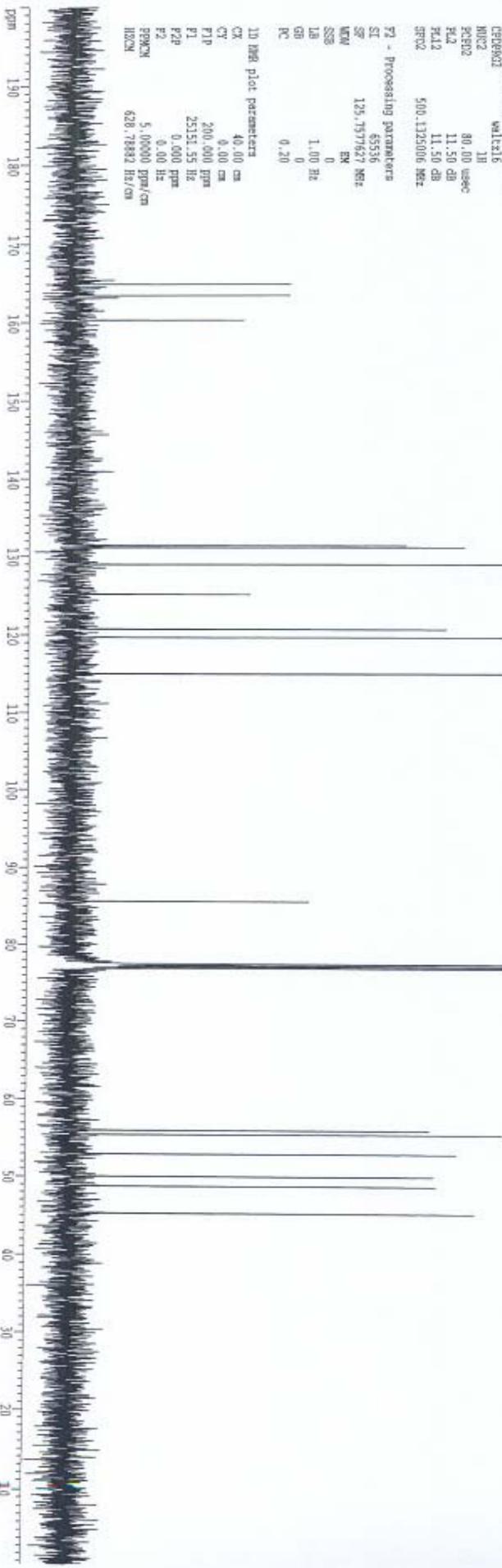
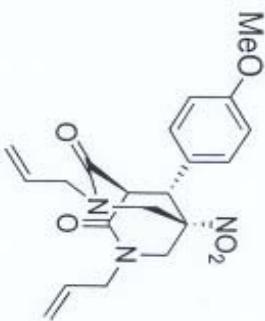


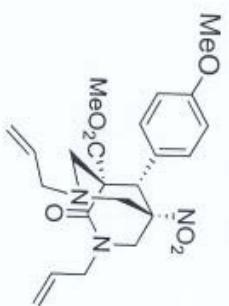
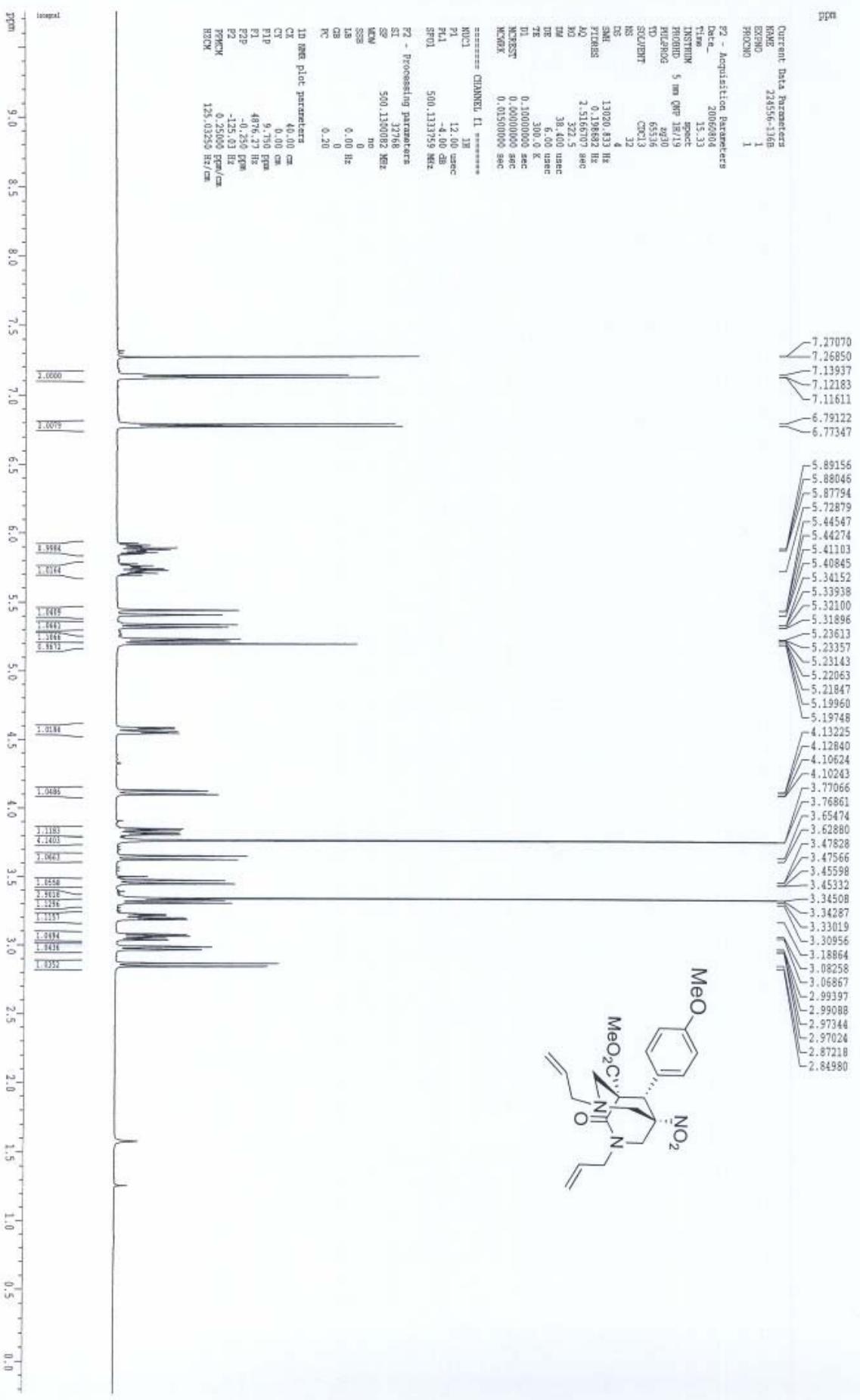
164.93
163.48
163.17
160.31

131.43
131.17
128.97
125.14
120.73
119.70
115.01

85.65
77.61
77.49
77.44
77.39
77.36
77.23
77.16
77.08
76.98
76.90
76.89

56.04
55.50
52.99
50.09
50.05
48.80
45.29





Current Data Parameters

NAME 221556-13B

EXNO 2

PROCNO 1

P2 - Acquisition Parameters

Date_ 20060804

Time 15.41

INSTRUM

spacet

ISSQUID 5 mm QNP

TR 15000

TD 8000

TOFSPG 131.072

SOLVENT DPC13

NS 219

DS 4

TE 4032.562

SR 0.307637

PTIMES 8e

AQ 1.625582

NUC1S 8192

SWH 12.400

DE 6.00

TEC 360.0

DW 0.10000000

D1 2.50

NUC2 0.03000000

D2 0.00

NUC3 0.00000000

D3 0.01500000

NUC4 0.00000000

D4 0.00000000

NUC5 0.00000000

D5 0.00000000

NUC6 0.00000000

D6 0.00000000

NUC7 0.00000000

D7 0.00000000

NUC8 0.00000000

D8 0.00000000

NUC9 0.00000000

D9 0.00000000

NUC10 0.00000000

D10 0.00000000

NUC11 0.00000000

D11 0.00000000

NUC12 0.00000000

D12 0.00000000

NUC13 0.00000000

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NUC14 0.00000000

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NUC15 0.00000000

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NUC16 0.00000000

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NUC17 0.00000000

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NUC18 0.00000000

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NUC19 0.00000000

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NUC40 0.00000000

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NUC41 0.00000000

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NUC42 0.00000000

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NUC90 0.00000000

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NUC100 0.00000000

D100 0.00000000

NUC101 0.00000000

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NUC103 0.00000000

D103 0.00000000

NUC104 0.00000000

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NUC105 0.00000000

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NUC106 0.00000000

D106 0.00000000

NUC107 0.00000000

D107 0.00000000

NUC108 0.00000000

D108 0.00000000

NUC109 0.00000000

D109 0.00000000

NUC110 0.00000000

D110 0.00000000

NUC111 0.00000000

D111 0.00000000

NUC112 0.00000000

D112 0.00000000

NUC113 0.00000000

D113 0.00000000

NUC114 0.00000000

D114 0.00000000

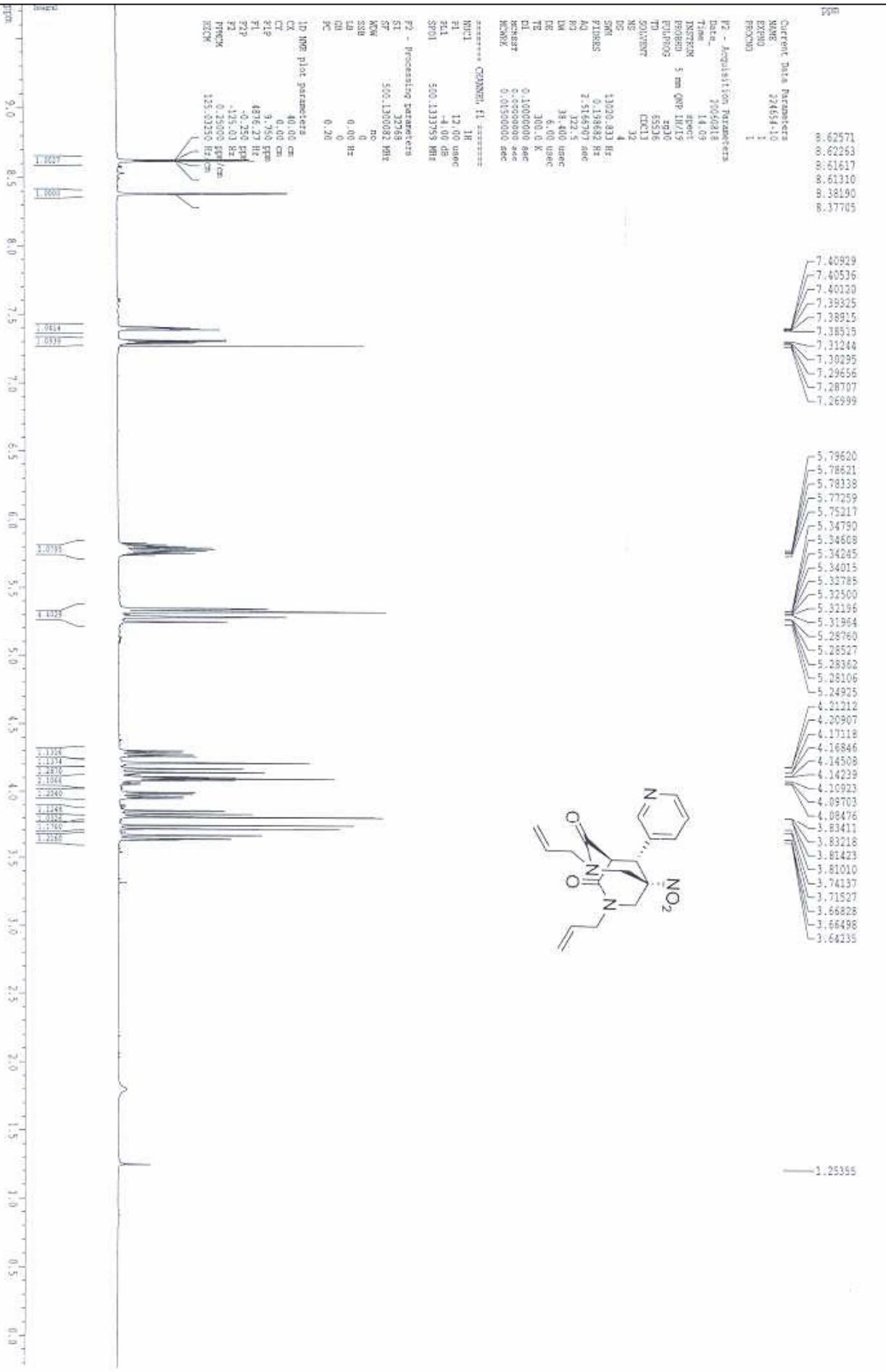
NUC115 0.00000000

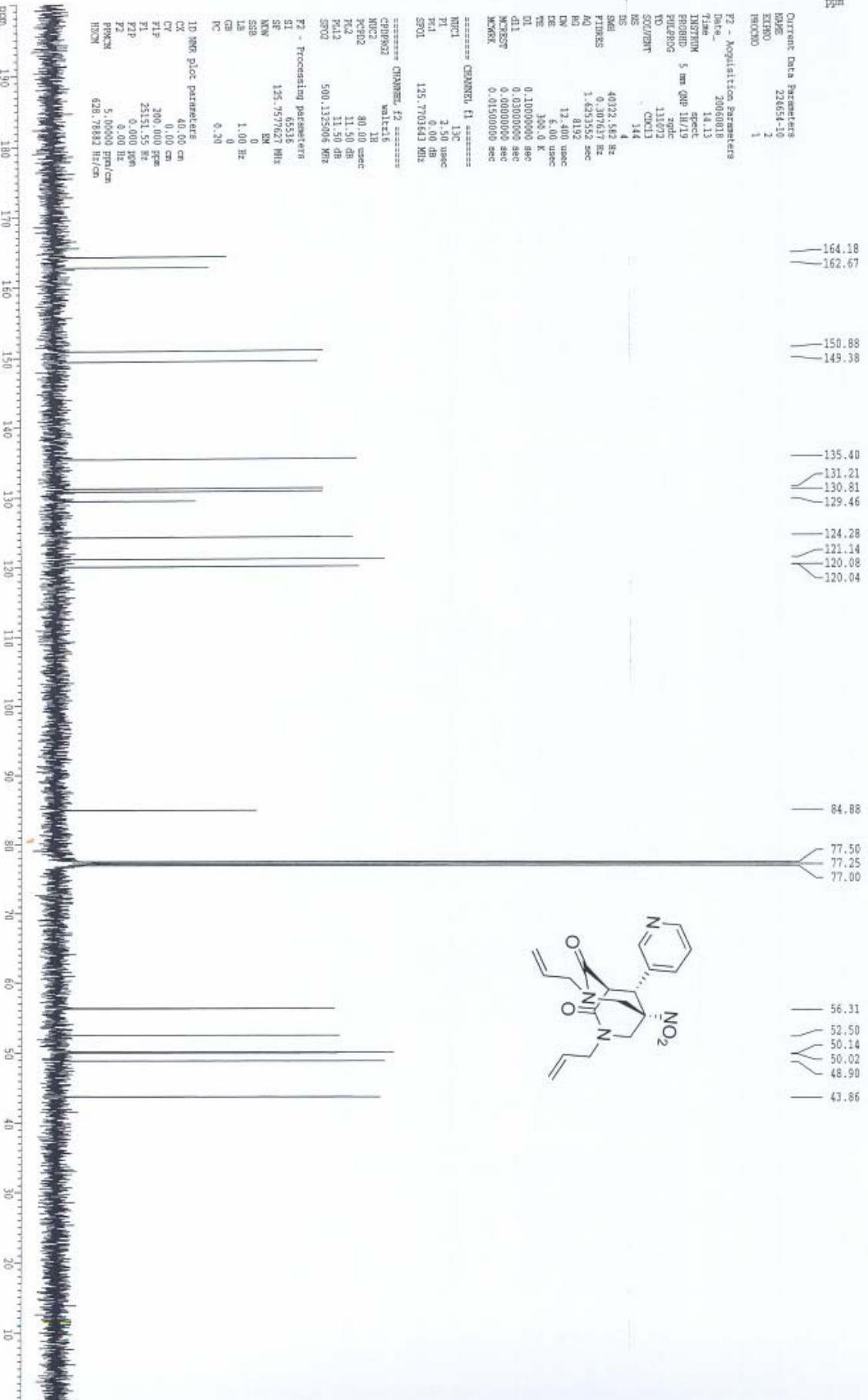
D115 0.00000000

NUC116 0.00000000

D116 0.00000000

NUC117 0.00000000







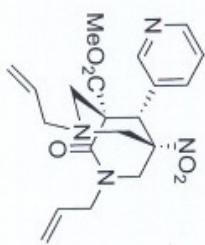
=====
Current Data Parameters
NAME 22454-1
EXEND 1
PRCDNO 1

F2 - Acquisition Parameters
Date_ 20050318
Time_ 14:17
DURATION 5 sec QPP IR19
PULPROG zg10
TD 65536
SOLVENT CDCl3
NS 17
TE 13000.833 Hz
SH 0.13962 Hz
SWR 2.5166707 sec
AQ 322.5
DM 38.400 usec
IR 6.00 usec
TE 300.0 K
EL 0.1000000 sec
DW 0.0000000 sec
NODST 0.0130000 sec
MEANBK 0.0

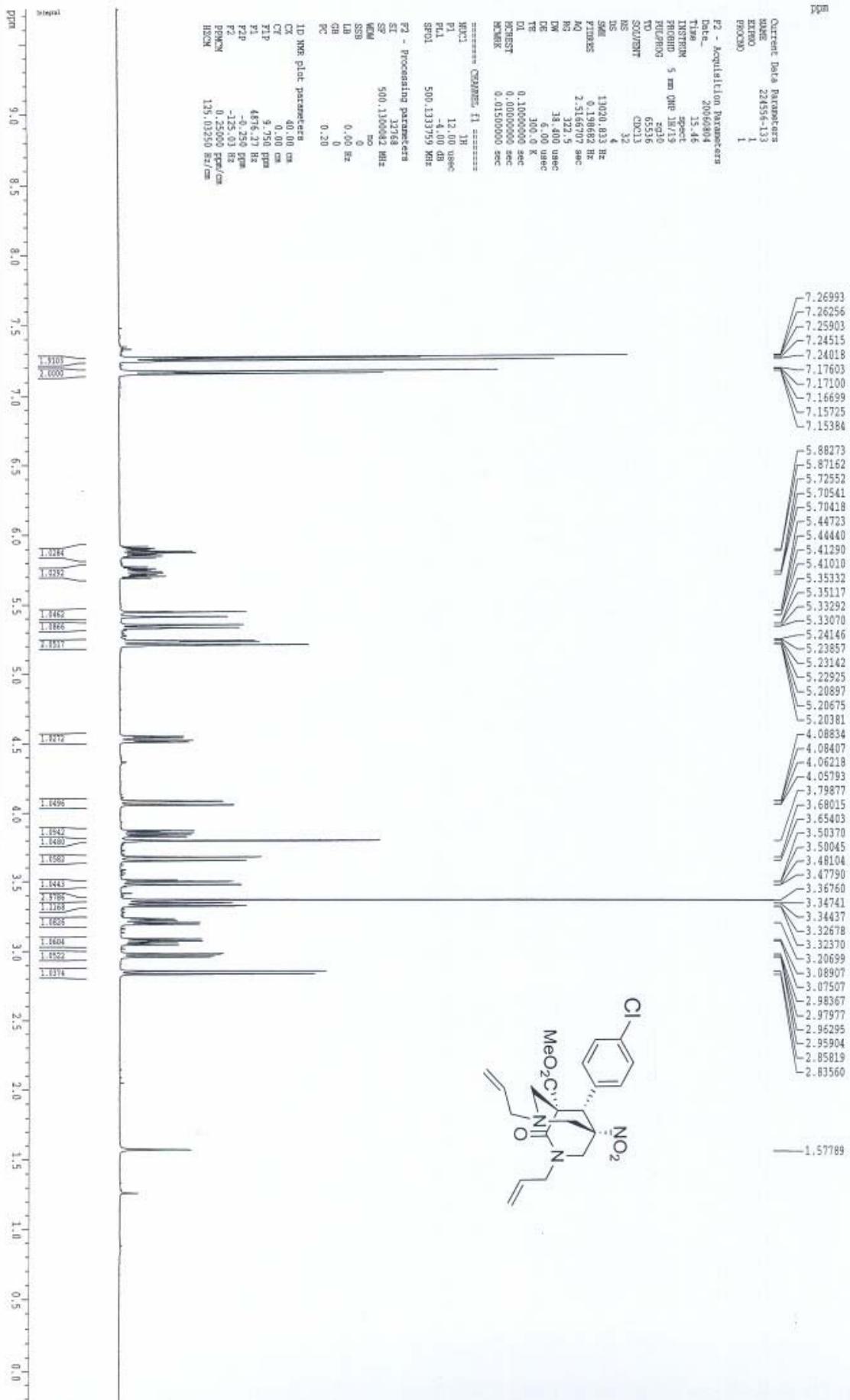
===== CHANNEL F1 =====
MCL1 1H
PI 12.00 usec
P1 4.00 dB
SPOL 500.1331759 MHz

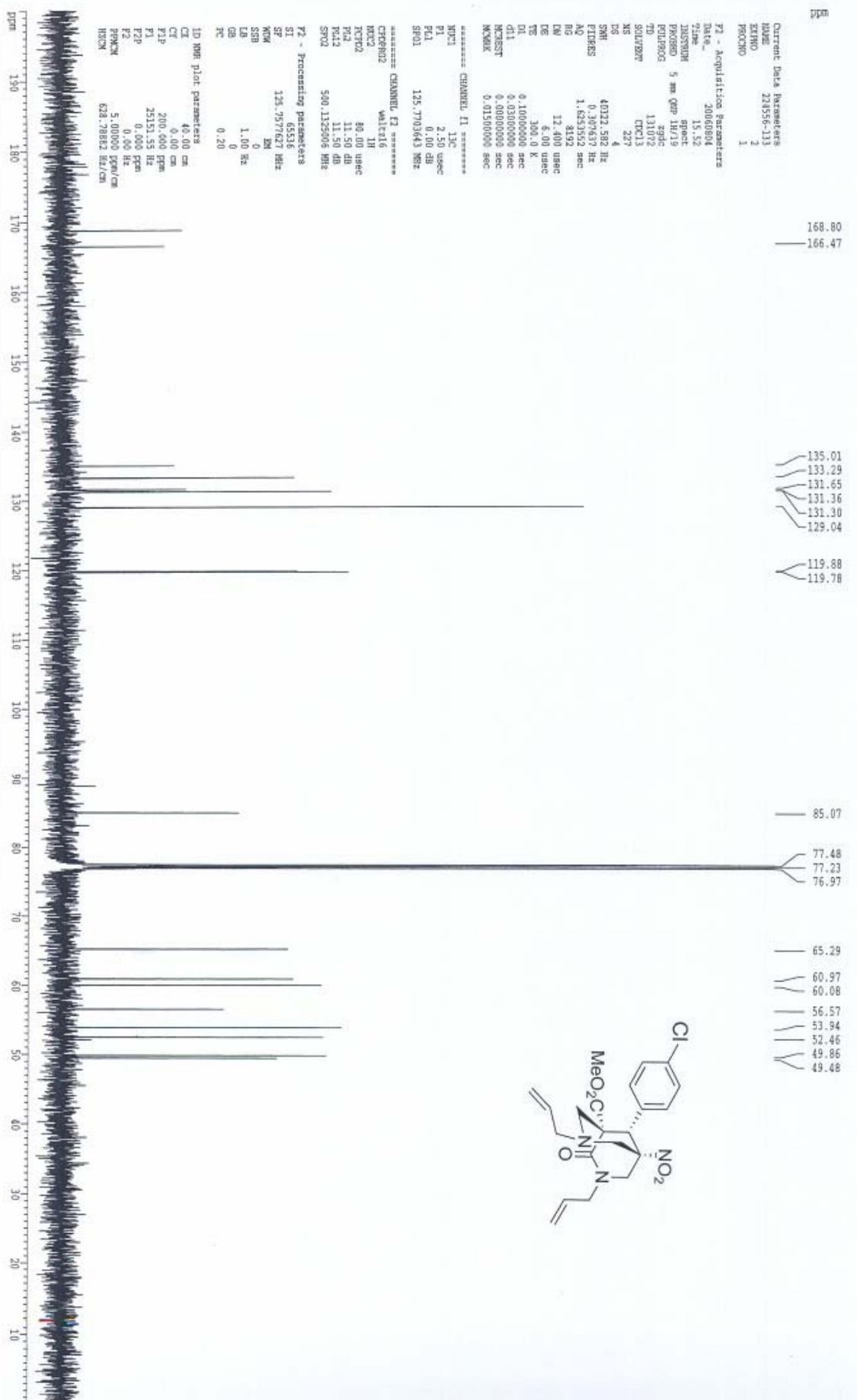
F2 - Processing parameters
SF 37768
SS 500.1330002 MHz
KOM no
SSB 0
LB 0.00 Hz
GB 0
PC 0.20

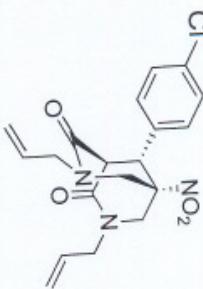
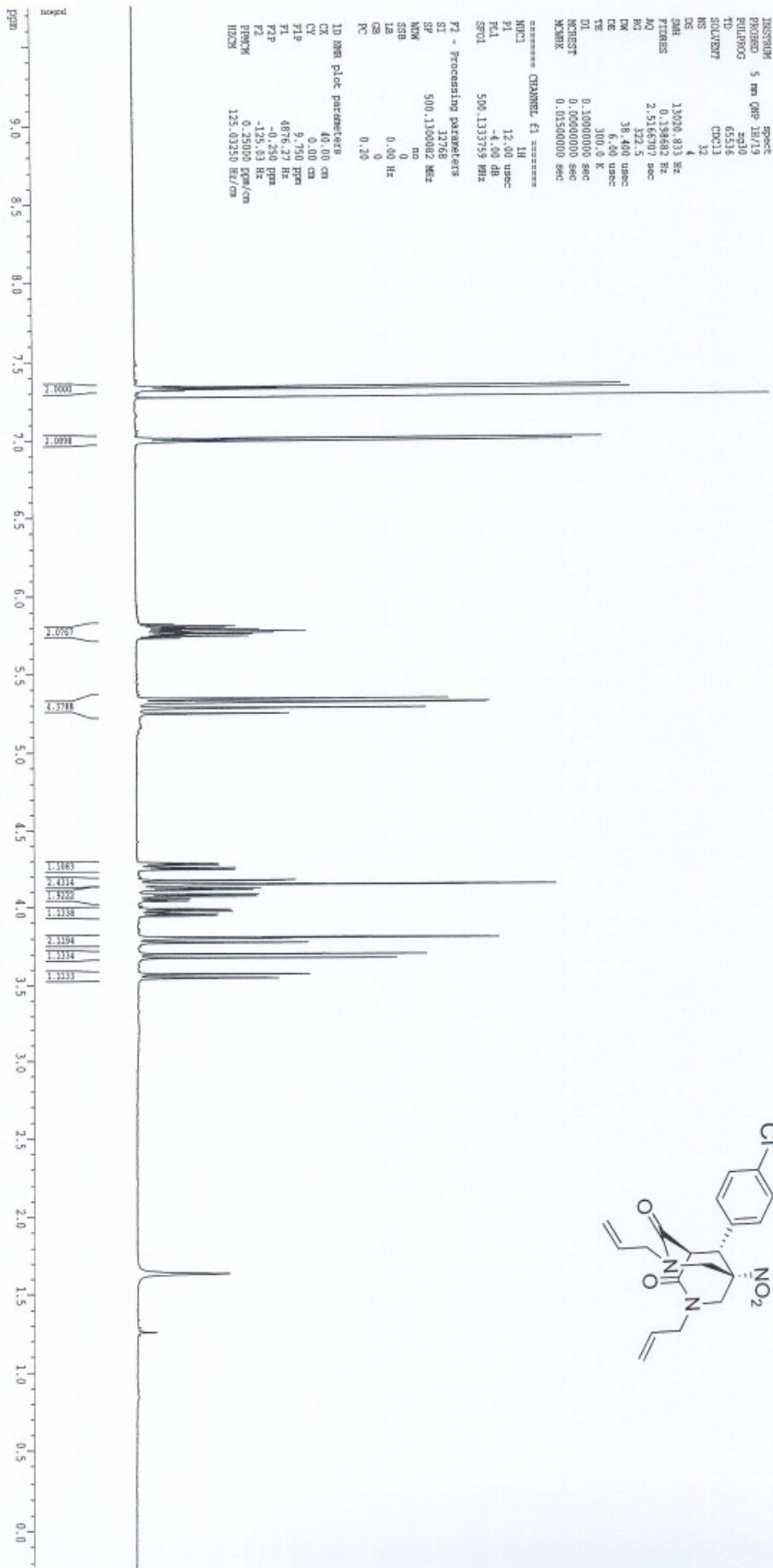
1D NMR plot parameters
CX 40.00 cm
CY 0.00 cm
P1P 9.750 ppm
F1 4876.27 Hz
F2P -0.250 ppm
F2Z -125.93 Hz
PPCM 0.25000 Hz/cm
SSBM



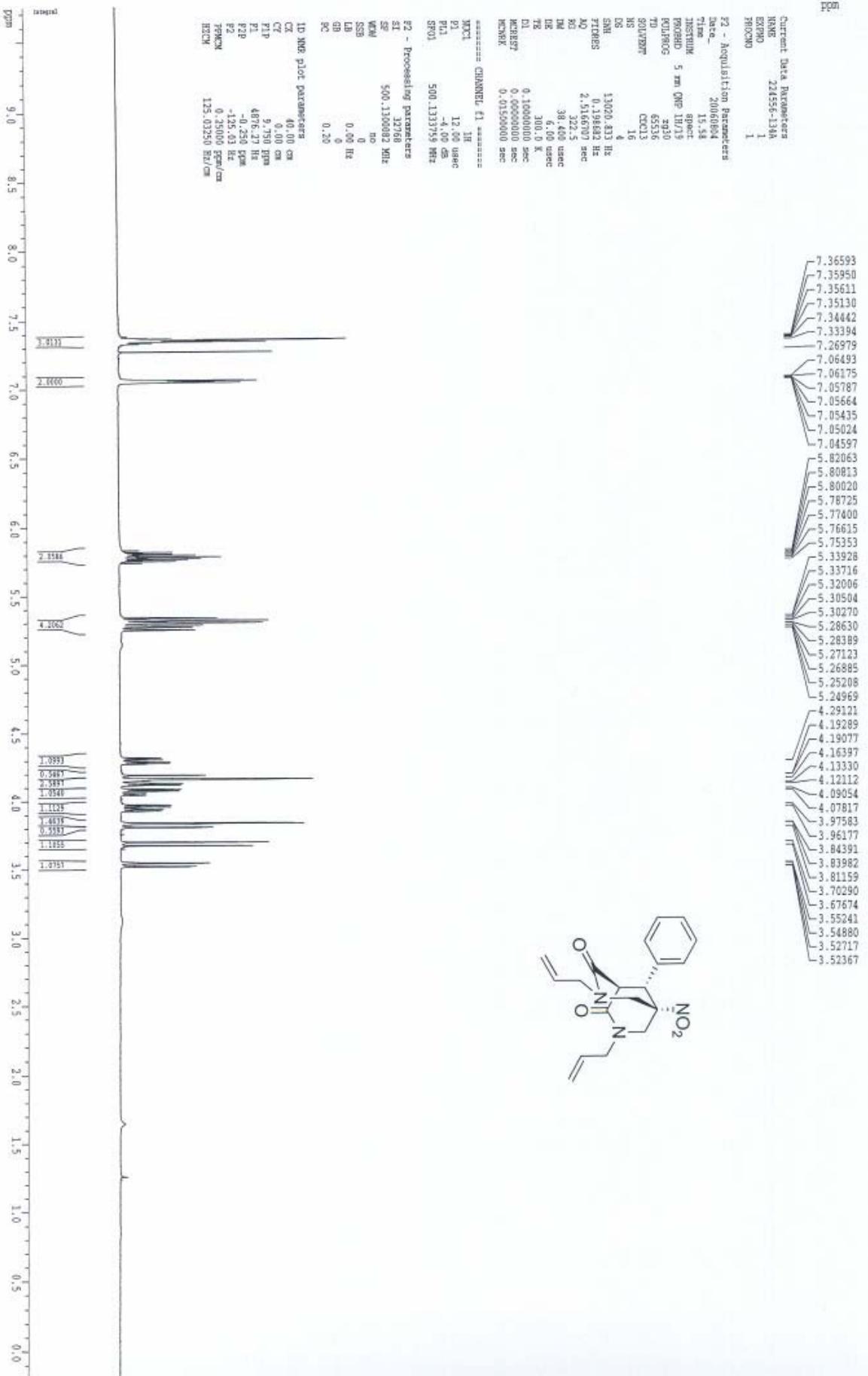


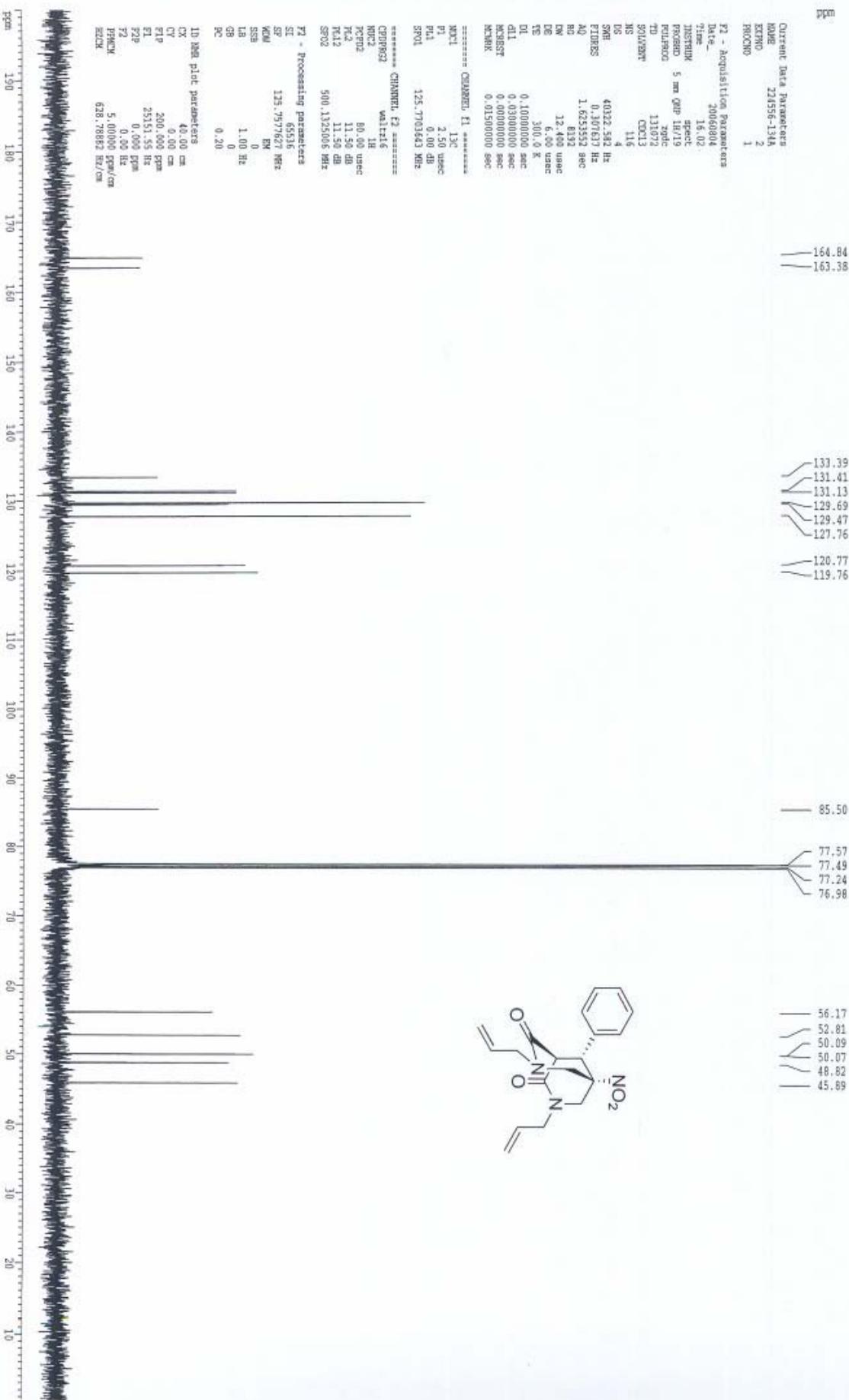


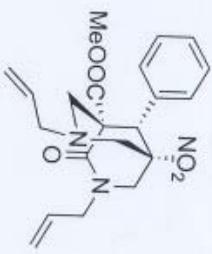
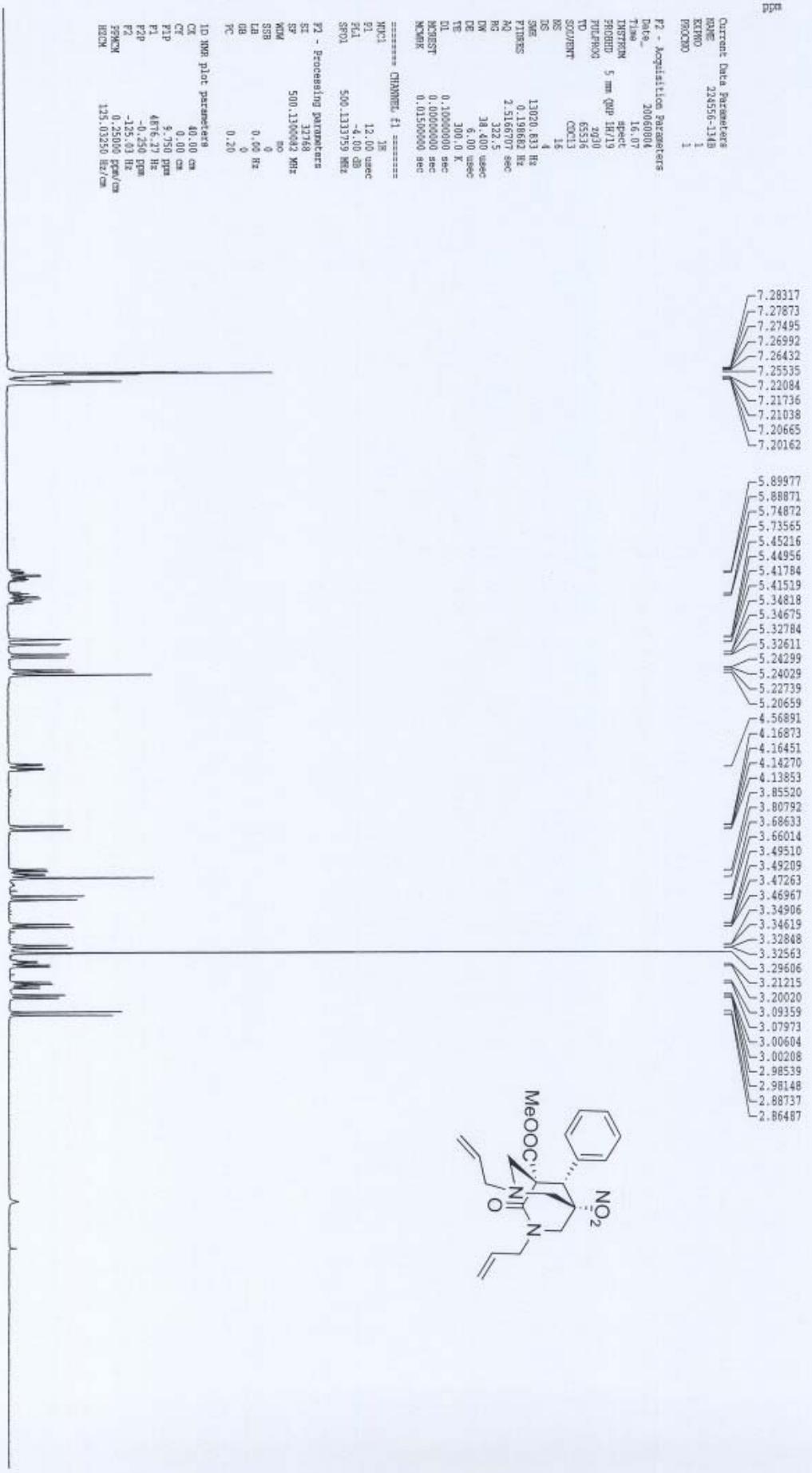
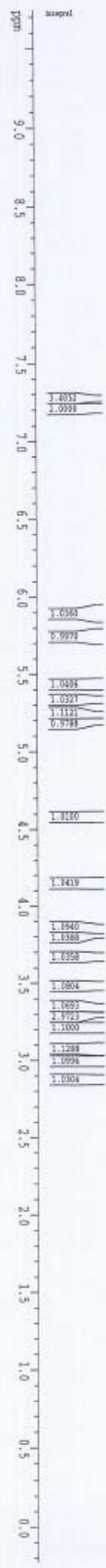


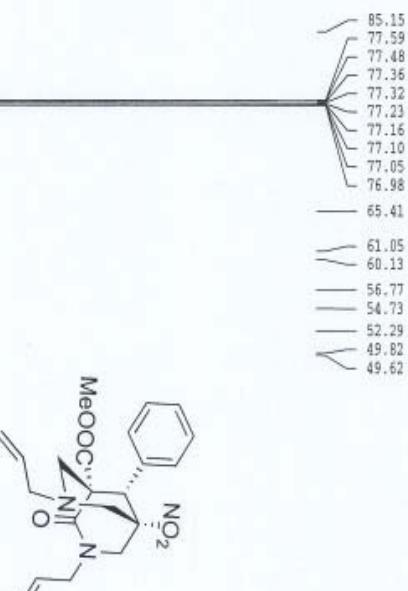
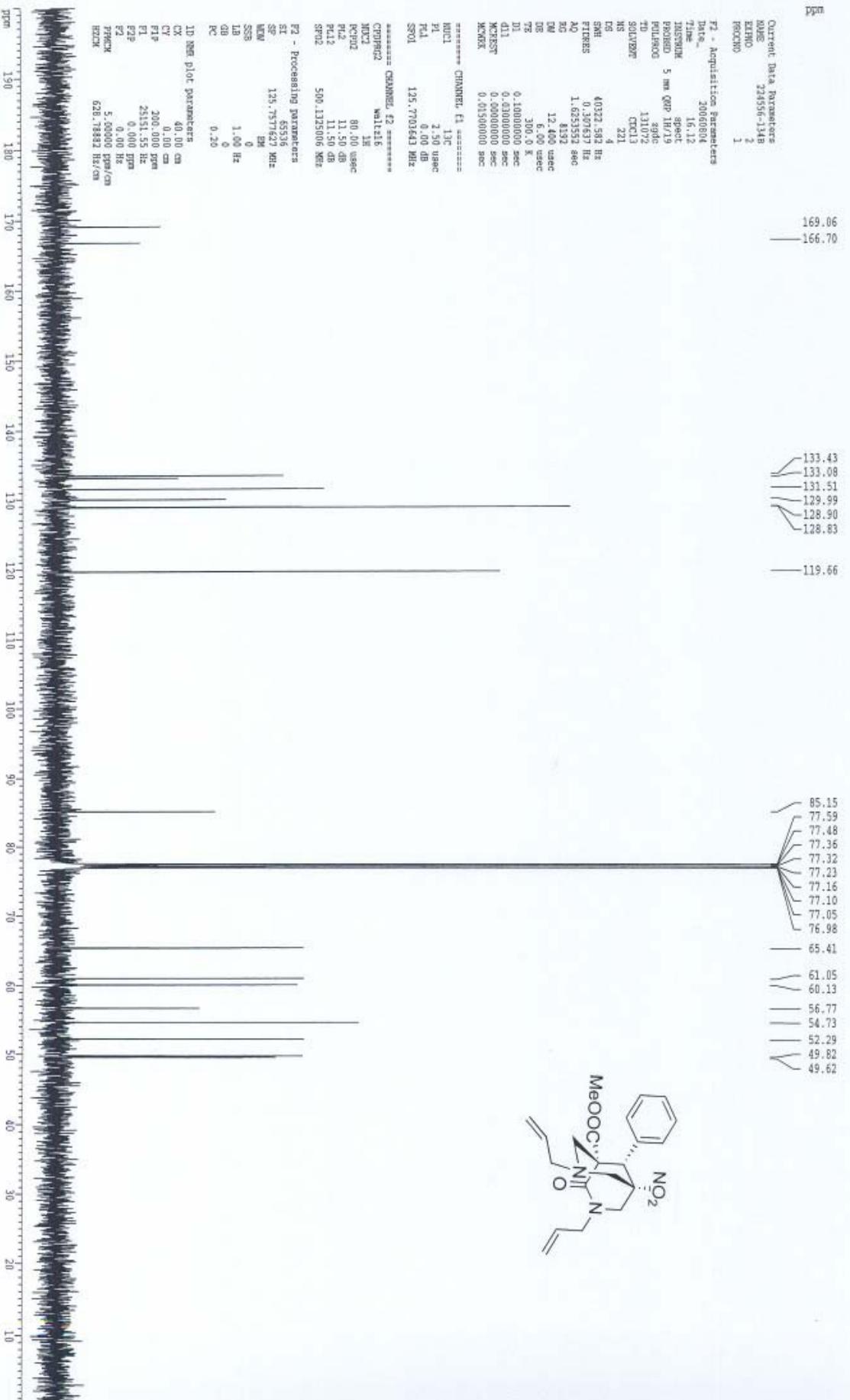


— 1.63845





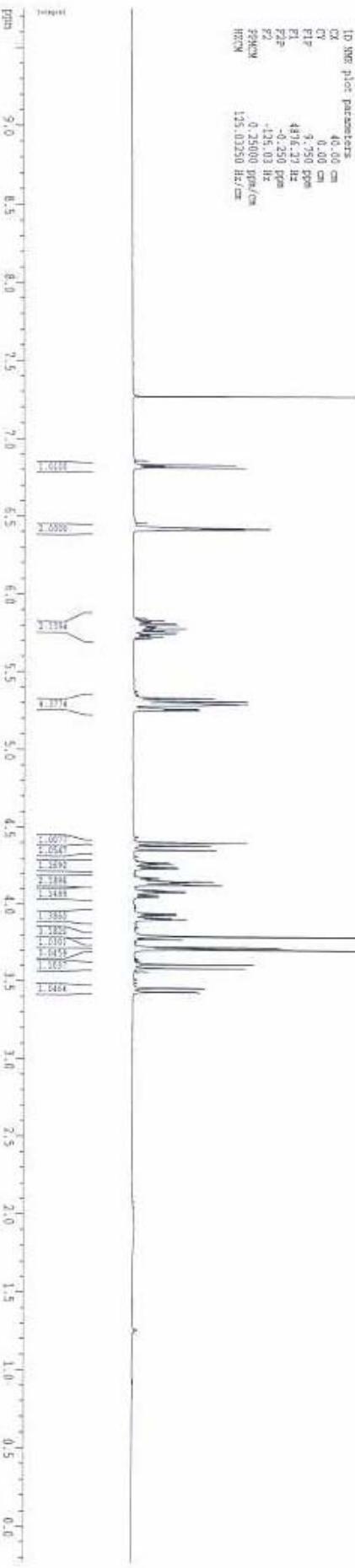
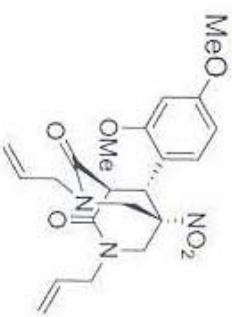
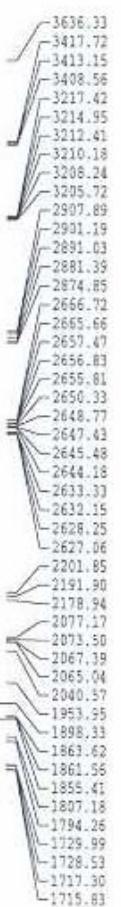


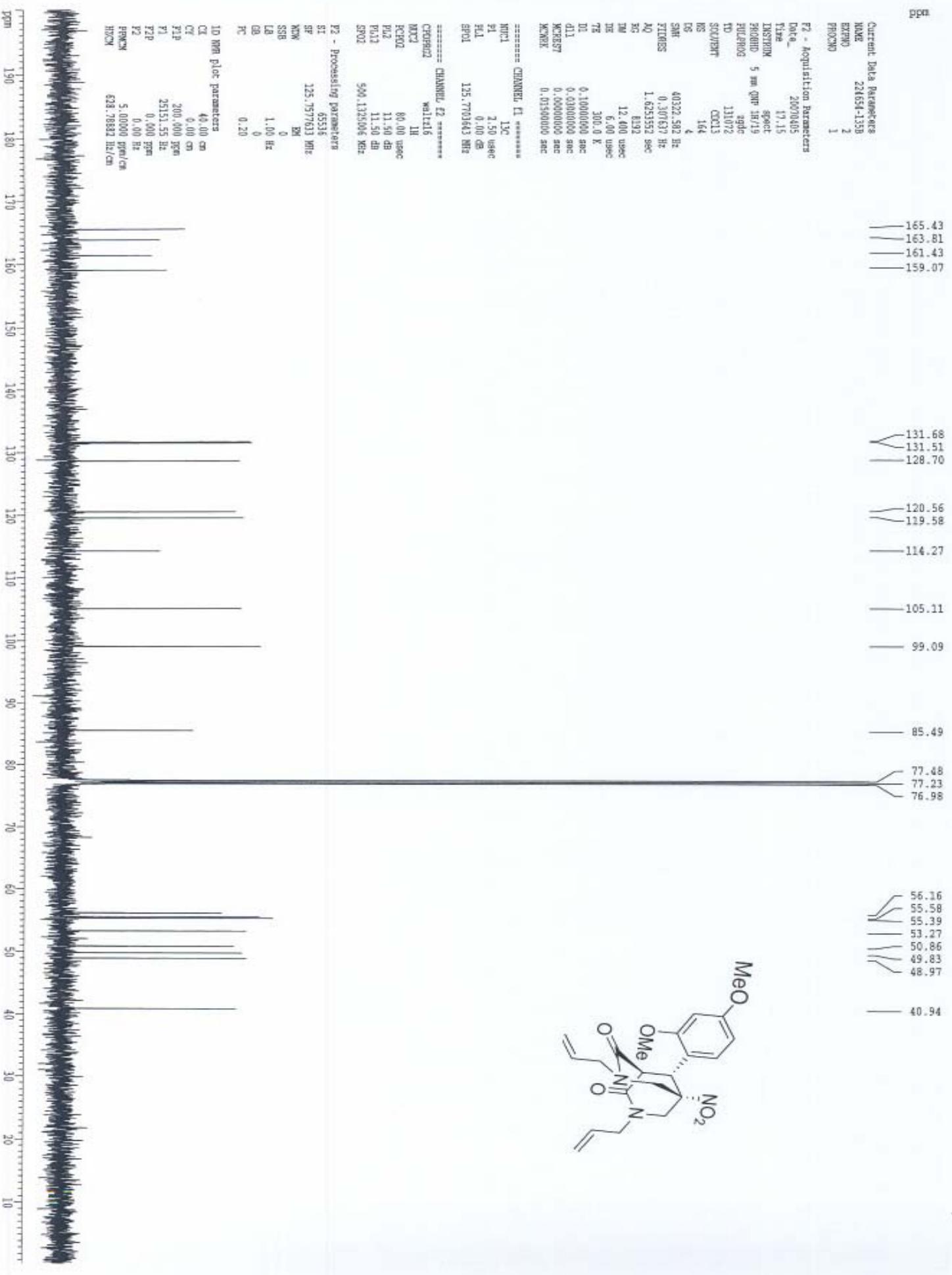


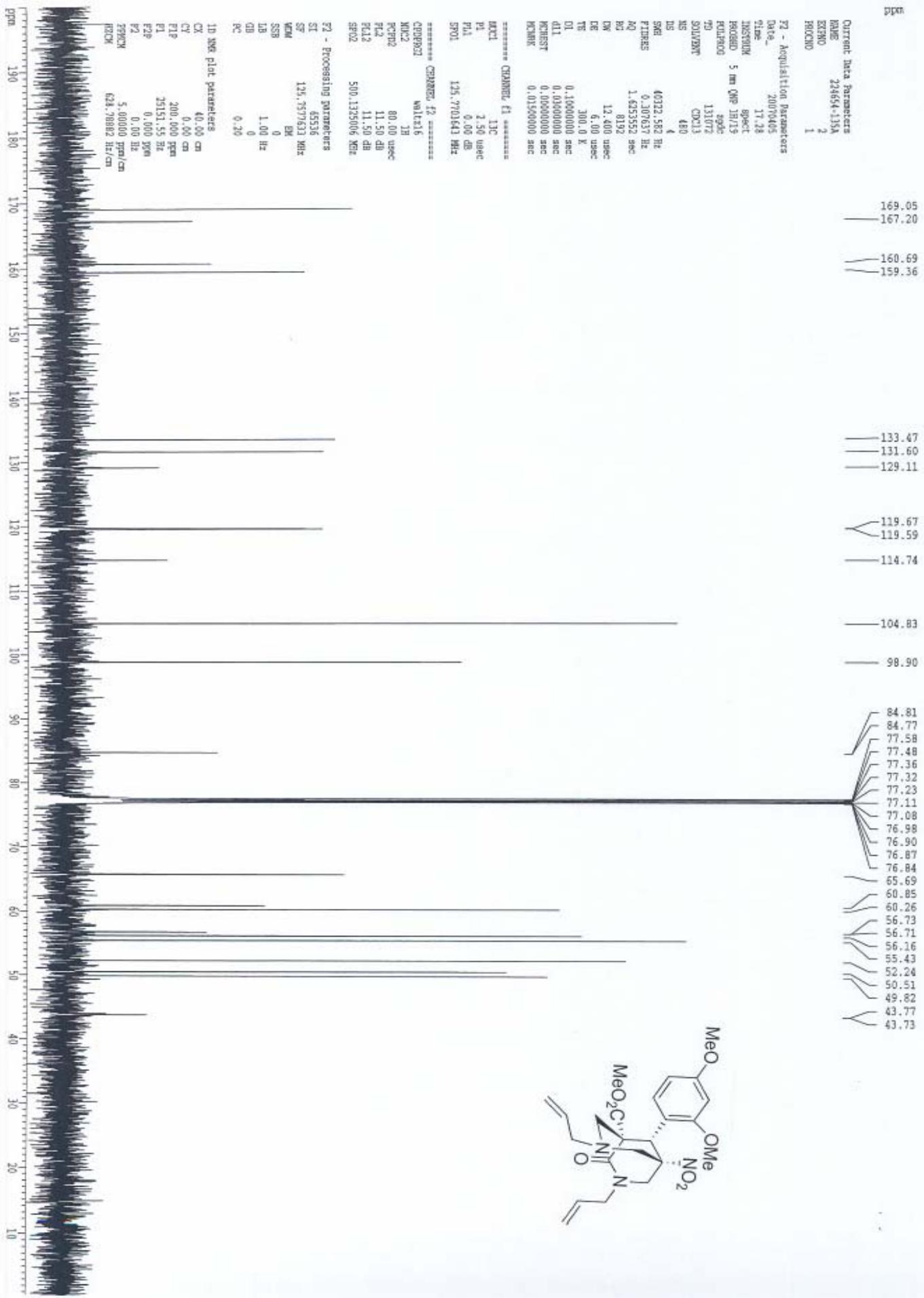
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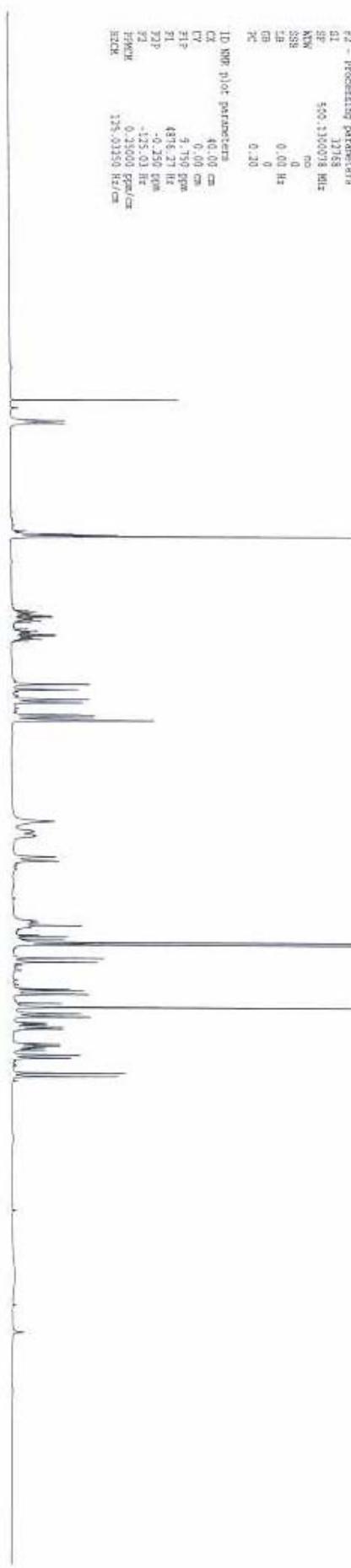
Current Data Parameters
RMS 224654-135B
STD 1
TRIGC 1

P2 - Acquisition Parameters	
	Daten-
TIME	17.07
NUSTEM	
PRETRIM	5 ms JDE 18.13
PULPROF	2010
TD	65536
SUNDET	
NS	32
SCALING	4
SME	
FIDRES	0.15625 Hz
AQ	2.548301 sec
R5	405.4
R4	33.400 user
R3	5.40 user
PS	300.0 K
MEAST	0.1000000 sec
NODIM	0.0500000 sec
NOHWH	









PL - Acquisition parameters

Date_ 20010405
Time_ 11:21
INSTRUM_ Bruker
PROBOD_ 3 mm COP ZH19
TE_ 200.000
TM_ 65.036
TD_ 327687
DW13_ 0.0013

SW1 - Frequency

1H: 400.00000 Hz
13C: 100.00000 Hz
15N: 300.00000 Hz

TE: 0.1000000 sec

SWFID: 0.0000000 sec

NUC1: 1H

NUC2: 13C

NUC3: 15N

NUC4: NOE

NUC5: DPPG

NUC6: T1

NUC7: T2

NUC8: T3

NUC9: T4

NUC10: T5

NUC11: T6

NUC12: T7

NUC13: T8

NUC14: T9

NUC15: T10

NUC16: T11

NUC17: T12

NUC18: T13

NUC19: T14

NUC20: T15

NUC21: T16

NUC22: T17

NUC23: T18

NUC24: T19

NUC25: T20

NUC26: T21

NUC27: T22

NUC28: T23

NUC29: T24

NUC30: T25

NUC31: T26

NUC32: T27

NUC33: T28

NUC34: T29

NUC35: T30

SW2 - Decoupling F1 parameters

1H: 10.75 unec
13C: -4.00 dB

15N: 500.1111753 MHz

SW3 - Processing parameters

SI: 32768

SF: 1100078 Hz

WDW: no

SSB: 0.0

LB: 0.00 Hz

RR1: 0.0

RR2: 0.10

RR3: 0.0

RR4: 0.0

RR5: 0.0

RR6: 0.0

RR7: 0.0

RR8: 0.0

RR9: 0.0

RR10: 0.0

RR11: 0.0

RR12: 0.0

RR13: 0.0

RR14: 0.0

RR15: 0.0

RR16: 0.0

RR17: 0.0

RR18: 0.0

RR19: 0.0

RR20: 0.0

RR21: 0.0

RR22: 0.0

RR23: 0.0

RR24: 0.0

RR25: 0.0

RR26: 0.0

RR27: 0.0

RR28: 0.0

RR29: 0.0

RR30: 0.0

SW4 - 1D NMR plot parameters

CD1: 40.00 cm

CD2: 0.00 cm

CD3: 9.750 ppm

CD4: 4316.77 Hz

CD5: -0.250 ppm

CD6: 125.03 Hz

CD7: 0.20000 ppm/cm

CD8: 175.0310 Hz/cm

CD9: 0.0

CD10: 0.0

CD11: 0.0

CD12: 0.0

CD13: 0.0

CD14: 0.0

CD15: 0.0

CD16: 0.0

CD17: 0.0

CD18: 0.0

CD19: 0.0

CD20: 0.0

CD21: 0.0

CD22: 0.0

CD23: 0.0

CD24: 0.0

CD25: 0.0

CD26: 0.0

CD27: 0.0

CD28: 0.0

CD29: 0.0

CD30: 0.0

CD31: 0.0

CD32: 0.0

CD33: 0.0

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