



Supporting Information

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A Silver-Catalyzed Intermolecular Amination of Saturated C–H Bonds

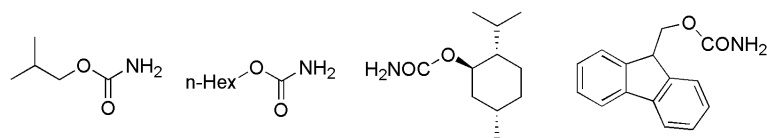
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Department of Chemistry, The University of Chicago, 5735 S. Ellis Avenue
Chicago, IL USA 60637

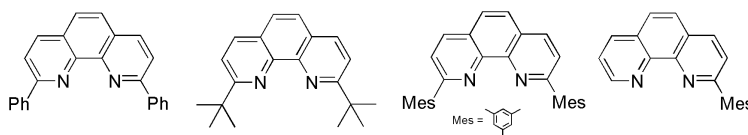
General: All reagents were bought from commercial sources and used as received unless otherwise noted. All reactions were performed under an inert N₂ atmosphere and all solvents were distilled before use unless otherwise noted. Chromatographic purification of products was accomplished by using forced-flow chromatography on EM Science Geduran silica gel 60 (35-75µm). Thin layer chromatography was performed on EM Science silica gel 60 F254 plates (250 µm). Visualization of the developed chromatogram was accomplished by UV lamp and/or by staining with aqueous potassium permanganate/K₂CO₃ solutions, or aqueous ceric ammonium molybdate (CAM) solutions. Nuclear magnetic resonance (NMR) spectra were acquired on Bruker DRX-500/400 operating at 500/400 and 125/100 MHz for ¹H and ¹³C, respectively; residual protio solvent signals were used as internal standard for calibration purpose. Data for ¹H NMR are recorded as follows: chemical shift (ppm), multiplicity (s, singlet; d, doublet; m, multiplet), integration, coupling constant (Hz). Data for ¹³C NMR are reported in terms of chemical shift (d, ppm). High-Resolution Mass was performed by Mass Spectrometry Facility at University of Norte Dame. X-ray diffraction data were collected on a Bruker Platform goniometer with a Charged Coupled Device (CCD) detector (Smart Apex). Structures were solved by direct methods using the SHELXTL (version 5.1) program library (G. Sheldrick, Bruker Analytical X-ray Systems, Madison, WI).

Synthesis of substrates and ligands

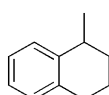
PhI=NNs: The preparation of PhI=NNs was performed based on a previous procedure. Iodobenzene diacetate was added to a mixture of 4-nitrobenzenesulfonamide and potassium hydroxide dissolved in methanol at 0 °C. After reacting at room temperature for 4 h, a cream precipitate formed. The precipitate was filtered, washed with water, and dried at RT in a desiccator. The product, PhI=NNs, was used without any further purification. A yield comparable to the reference was obtained.¹



Carbamates: A portion of trichloroacetyl isocyanate (1/2 equiv) was added dropwise to a CH_2Cl_2 solution of the corresponding alcohol at 0 °C. The mixture was warmed up to room temperature left to react overnight. The solvent was evaporated and 0.1 equiv of K_2CO_3 in methanol was added. The solution was stirred for several hours then quenched by adding aqueous NH_4Cl solution. The mixture was extracted with CH_2Cl_2 and the organic layer was collected, dried, and then column purified. Comparable yields were achieved according to the reference.²



Phenanthroline ligands: 2,9-Disubstituted phenanthroline ligands were synthesized by reacting phenanthroline with a corresponding lithium reagent. Comparable yields were achieved in comparison with the referenced procedure.³



1-Methyl-1,2,3,4-tetrahydronaphthalene was synthesized in three steps from benzenebutanoic acid using a literature procedure.⁴ The benzenebutanoic acid was reacted with methyl lithium in THF at 0 °C under nitrogen atmosphere to yield 5-phenyl-2-pentanone. In the second step, 5-phenyl-2-pentanone was reacted with lithium aluminum hydride in diethyl ether under nitrogen atmosphere for five min, forming 5-phenyl-2-pentanol. In the last step, trifluoromethanesulfonic acid was added to a stirred solution of 5-phenyl-2-pentanol and SiO_2 in methylene chloride. The mixture was left to

react for seven hours at room temperature, yielding 1-methyl-1,2,3,4-tetrahydronaphthalene in yields comparable to the reference.⁴

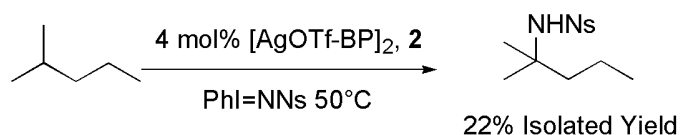
Selected Screening Conditions:

Ligand effects were listed in Table 1. Different metals, silver salts, solvents and additives were tested using the conditions shown in entry 6 of Table 1. Pyridines were found to be a beneficial additive for the intramolecular amidations. No pyridine additives were necessary for the intermolecular aminations.

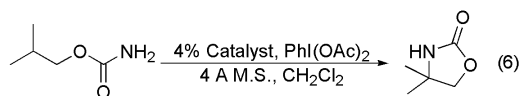
Typical reaction procedure for intermolecular amination (all steps were carried out under nitrogen atmosphere): AgOTf (2%) and 2.4% ligand were mixed in 3 ml CH₂Cl₂ in a tube for 20 min. 1,4-diisopropylbenzene (202 mg) was added, followed by 500 mg 4Å molecular sieves and 100 mg PhI=NNs. The tube was sealed and placed in a 50°C oil bath. Another 2% AgOTf and 2.4% ligand mixed in 1 ml CH₂Cl₂ were added after 2 h. The tube was placed back into the oil bath and then left to react overnight. After overnight reaction the solution typically turned dark-yellow or dark-red. The reaction was filtered through a short silica gel pad and washed multiple times with both CH₂Cl₂ and diethyl ether. The organic solvents were evaporated and the residue was applied to column purification (silica gel, hexanes/EtOAc) to yield 52 mg product (57% yield based on amount of oxidant used). If the catalyst was added in one portion, slightly lower to no change in yield was observed depending on the substrate. PhI=NNs should be freshly made as is not suitable for a long-term storage.

Amination of Linear Aliphatic Hydrocarbons:

Tertiary C–H groups also undergo the amination reactions under the same conditions to afford corresponding products in 20-30% yields. For examples:



SI-Table 1: Selected Results of Ligand Screening



Entry	Ligand	Conversion(%) ^{a,b}
1		44
2		68(58) ^c
3		23
4		trace ^d
5		26
6		>95 ^e

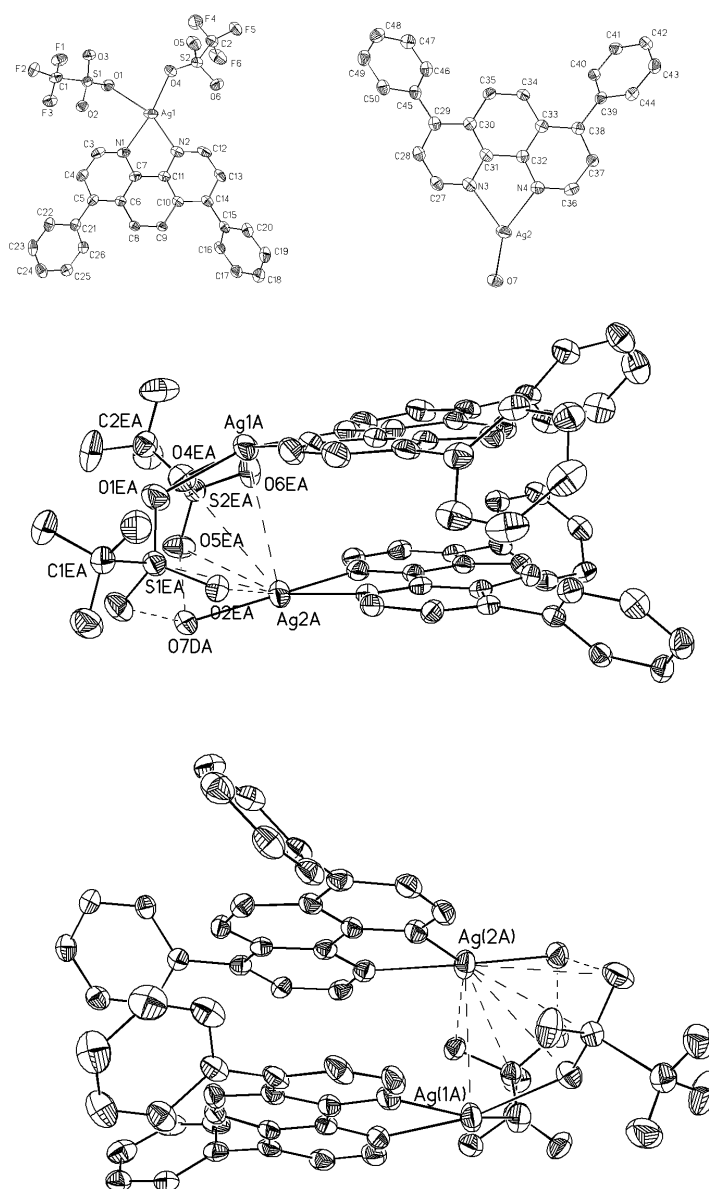
a: All reactions were carried out with 0.5 mmol substrate:0.8mmol PhI(OAc)₂: 500mg 4 A molecular sieves in 4 ml CH₂Cl₂ at 50°C for overnight. 4 % AgOTf was used with metal:ligand=1:1.2 together with 10% 4-*t*BuPyridine, except otherwise specified. b: Proton NMR Conversion. c: without 4-*t*BuPyridine. d: R=*t*Bu, Ph, Methyl. e: with 4% *t*Bu₃Py. 2% AgOTf and bathophenanthroline were added first and then 2% AgOTf and bathophenanthroline were added after 2 hours.

- [1] J. Gullick, D. Ryan, P. McMorn, D. Bethell, F. King, F. Hancock, G. Hutchings, *New J. Chem.* **2004**, 28, 1470-1478.
- [2] C. D. B. Espino, J. Du Bois, *Angew. Chem. Intl. Ed.* **2001**, 40, 598-600.
- [3] C. O. Dietrich-Buchecker, P. A. Marnot, J.P. Sauvage, *Tetrahedron Lett.* **1982**, 23, 5291-5294.
- [4] P. J. Kropp, G. W. Breton, S. L. Craig, S.D. Crawford, W. F. Durland, J. E. Jones Jr., J. S. Raleigh III, *J. Org. Chem.* **1995**, 60, 4146-4152.

Crystal Data

Crystals of **2** were obtained by slow evaporation of a methylene chloride solution at 4 °C. X-ray diffraction data were collected on a Bruker Platform goniometer with a Charged Coupled Device (CCD) detector (Smart Apex). The structure was solved by the direct method using the SHELXTL (version 5.1) program library (G. Sheldrick, Bruker

Analytical X-ray Systems, Madison, WI) and refined. The crystal was washed with hexanes and THF and sent for elemental analysis. [Ag(BP)(THF)]₂(OTf)₂ Found: C 52.89 H 3.19 N 4.81. Calculated: C 52.66 H 3.66 N 4.24.



SI-Figure 1: Crystal Structure of AgOTf and Bathophenanthroline Complexes

Equations of interest:

$$R_{\text{int}} = \frac{\sum |F_o|^2 - \langle F_o^2 \rangle}{\sum |F_o|^2}$$

$$R1 = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|}$$

$$wR2 = [\sum [w (F_o^2 - F_c^2)^2] / \sum [w (F_o^2)^2]]^{1/2} \quad \text{GooF} = S = [\sum [w (F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$$

where: $w = q / \sigma^2 (F_o^2) + (aP)^2 + bP$; $n = \text{number of independent reflections}$;
 q, a, b, P as defined in [1] $p = \text{number of parameters refined}$

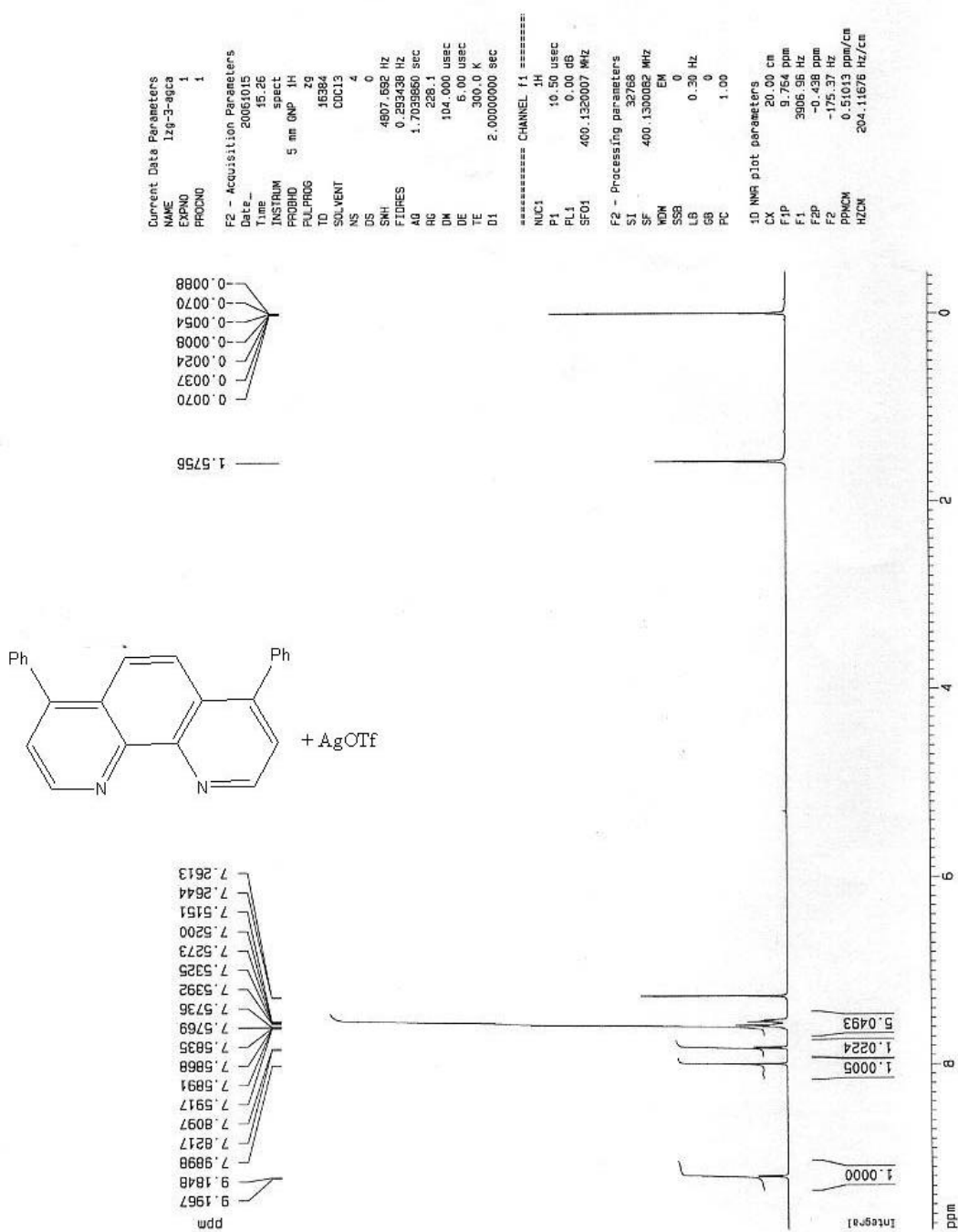
Table 1. Crystal and structure refinement for structure ID.

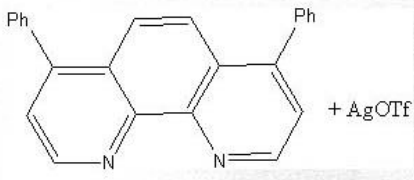
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Empirical formula	$\text{AgC}_{24}\text{H}_{16}\text{ON}_2 + \text{AgC}_{26}\text{H}_{16}\text{F}_6\text{N}_2\text{O}_6\text{S}_2 + 2\text{CH}_2\text{Cl}_2$	
Formula weight	1409.55	
Temperature	103 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space Group	C2/c	
Unit cell dimensions	$a = 39.617(14)$ Å	$a = 90.0^\circ$
	$b = 12.406(4)$ Å	$b = 132.017(4)^\circ$
	$c = 29.020(10)$ Å	$g = 90.0^\circ$
Volume	10597(6) Å ³	
Z	8	
Density (calculated)	1.763 Mg/m ³	
Absorption coefficient	1.100 mm ⁻¹	
F(000)	5608	
Crystal size, color, habit	0.36 x 0.08 x 0.08 mm, clear, fragment	
Theta range for data collection	1.78 – 28.41 °	
Index ranges	-52 ≤ h ≤ 52, -16 ≤ k ≤ 16, -37 ≤ l ≤ 38	
Reflections collected	61,694	
Independent reflections	12,924 (R _{int} = 0.0766)	
Reflections with I > 4σ(F _o)	8,892	
Absorption correction	SADABS based on redundant diffractions	
Max. and min. transmission	1.0, 0.482	
Refinement method	Full-matrix least squares on F ²	
Weighting scheme	$w = q [\sigma^2 (F_o^2) + (aP)^2 + bP]^{-1}$ where: $P = (F_o^2 + 2 F_c^2)/3$, $a = 0.0395$, $b = 0.0$, $q = 1$	
Data / restraints / parameters	12,924 / 0 / 742	
Goodness-of-fit on F ²	0.924	
Final R indices [I > 2 sigma(I)]	R1 = 0.0456, wR2 = 0.0893	
R indices (all data)	R1 = 0.0779, wR2 = 0.0979	

Largest diff. peak and hole

0.980, -0.764 eÅ⁻³

Copy of ¹H NMR and ¹³C NMR of [Ag(BP)OTf]₂ in CDCl₃





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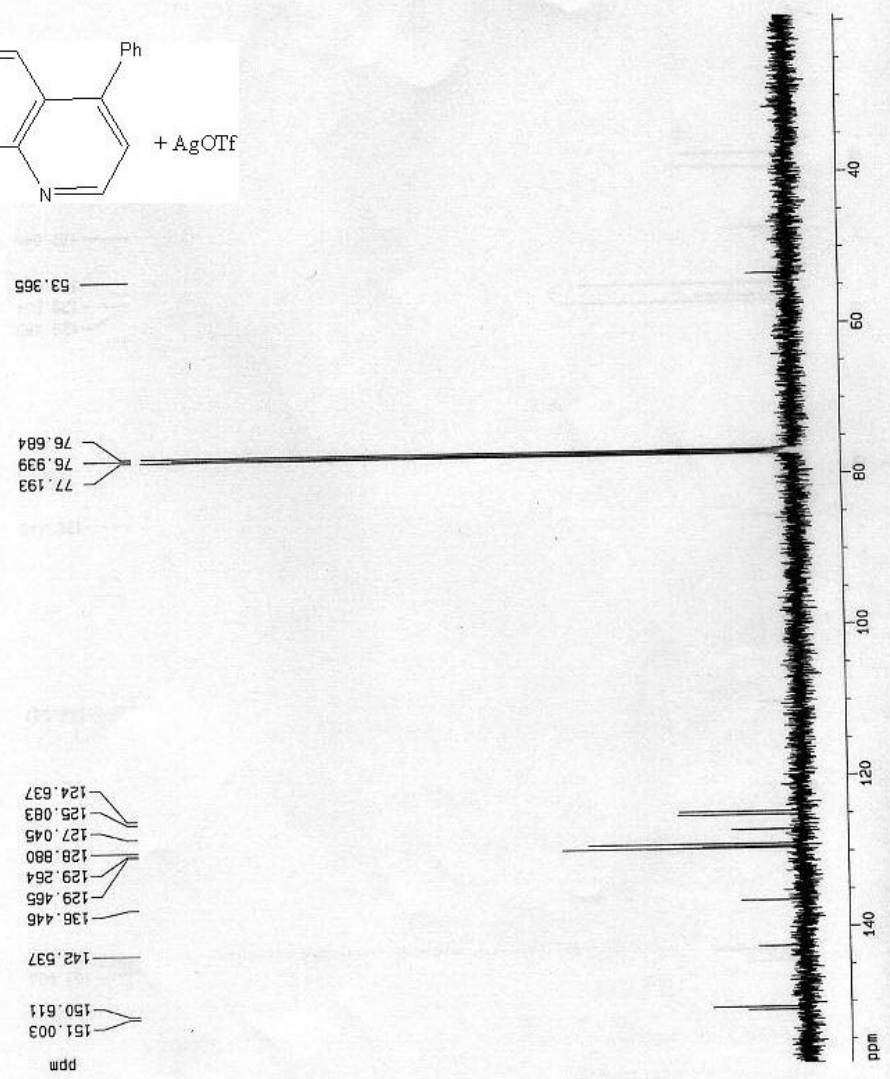
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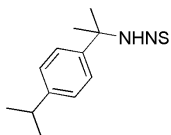
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1D NMR plot parameters
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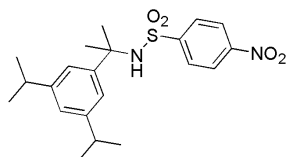


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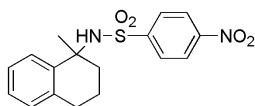
N-[1-(4-Isopropyl-phenyl)-1-methyl-ethyl]-4-nitro-benzenesulfonamide:

IR (% T): ^1H NMR (d, ppm): 8.09-8.07 (dd, 2H, J= 7.2, 2 Hz), 7.66-7.64 (dd, 2H, J=7.2, 2 Hz), 7.10-7.08 (dd, 2H, J=8, 2 Hz), 6.93-6.91 (dd, 2H, J=8, 2 Hz), 5.42 (s(br), 1H), 2.82-2.75 (m, 1H), 1.70 (s, 6H). 1.17-1.15 (6H, d, J= 6.8 Hz) ^{13}C NMR (d, ppm): 149.2, 148.2, 147.7, 140.3, 128.1, 126.0, 125.9, 123.6, 58.5, 33.4, 30.1, 23.8. HRMS(m/z): Found(M-1):361.1216, Calculated: 362.1300.



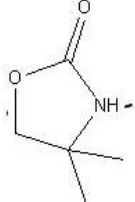
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IR (% T): ^1H NMR (d, ppm): 8.02-8.00 (dd, 2H, J= 8.4, 2 Hz), 7.66-7.64 (dd, 2H, J=8, 2 Hz), 6.83 (s, 2H), 6.80 (s, 1H), 5.38 (s(br), 1H), 2.68-2.61 (m, 1H), 1.64 (s, 6H). 1.09-1.06 (12H, d, J= 6.9 Hz) ^{13}C NMR (d, ppm): 149.3, 148.7, 147.8, 143.1, 128.1, 123.6, 123.4, 121.6, 58.9, 34.1, 30.0, 23.9. HRMS(m/z): Found(M-1): 403.1679, Calculated: 404.1770.



N-(1-Methyl-1,2,3,4-tetrahydro-naphthalen-1-yl)-4-nitro-benzenesulfonamide

^1H NMR (d, ppm): 8.27-8.25 (dd, 2H, J= 7, 2 Hz), 7.91-7.89 (dd, 2H, J= 7, 2 Hz), 7.22-7.00 (m, 4H), 2.71-2.67 (m, 2H), 2.38-2.35 (m, 1H), 1.91-1.57 (m, 6H). ^{13}C NMR (d, ppm): 149.5, 148.6, 138.9, 137.0, 129.3, 128.1, 127.7, 127.0, 126.3, 124.0, 58.9, 37.7, 29.4, 19.3. HRMS: Found(M+1): 347.1045, Calculated: 346.0987.



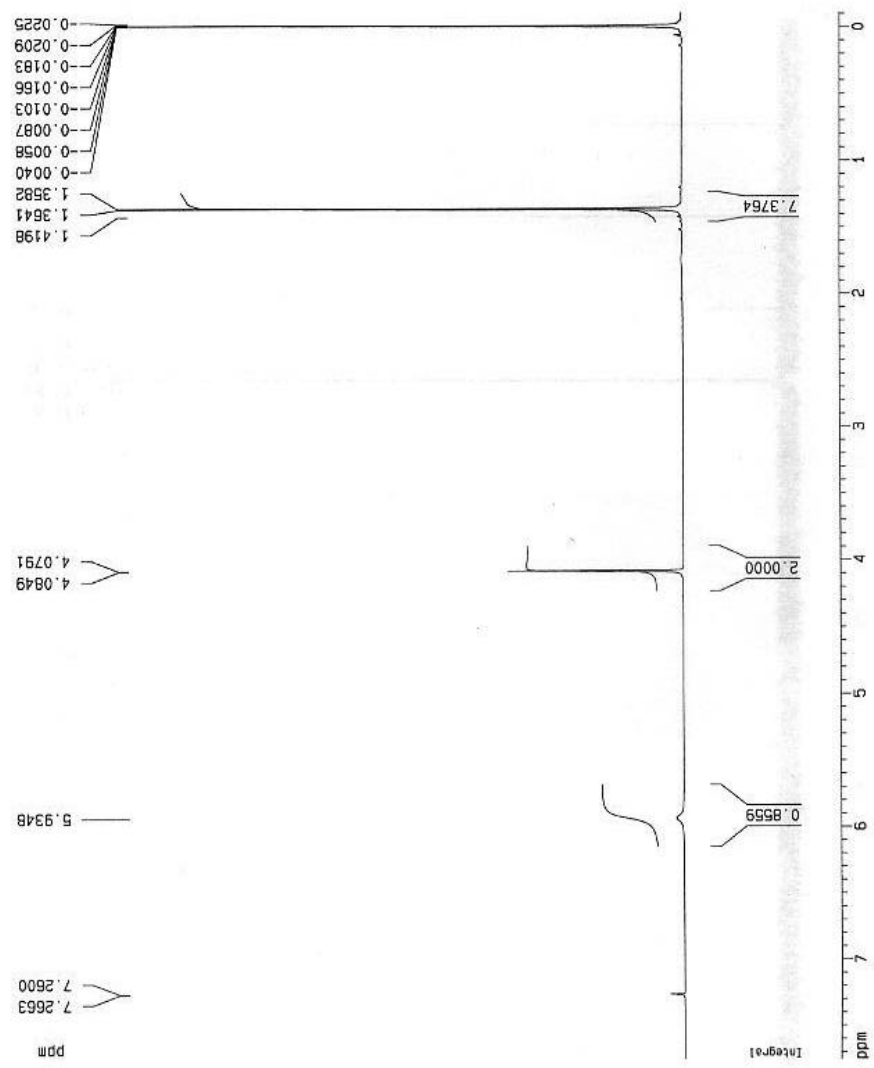
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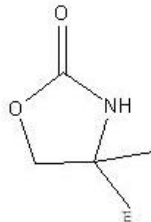
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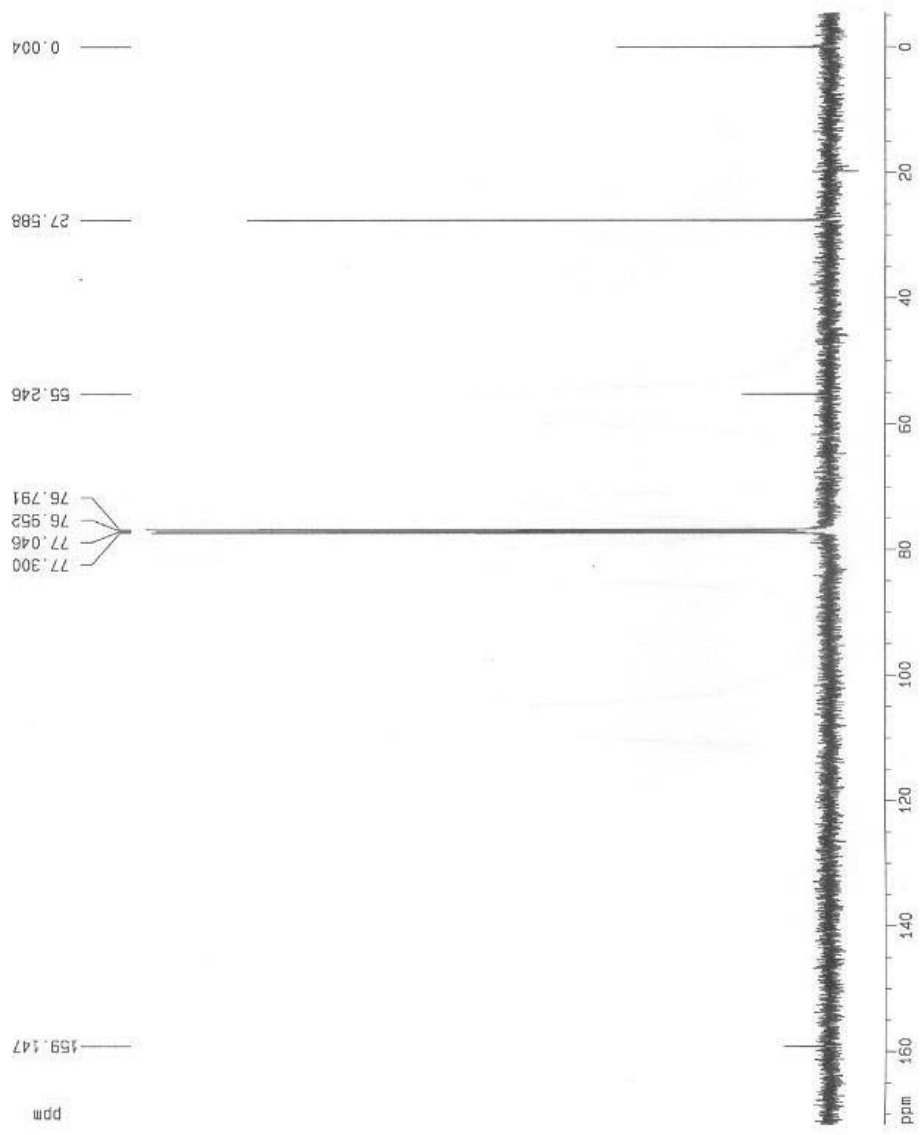
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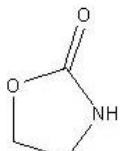
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97/10/17, #5, 122.5 mg





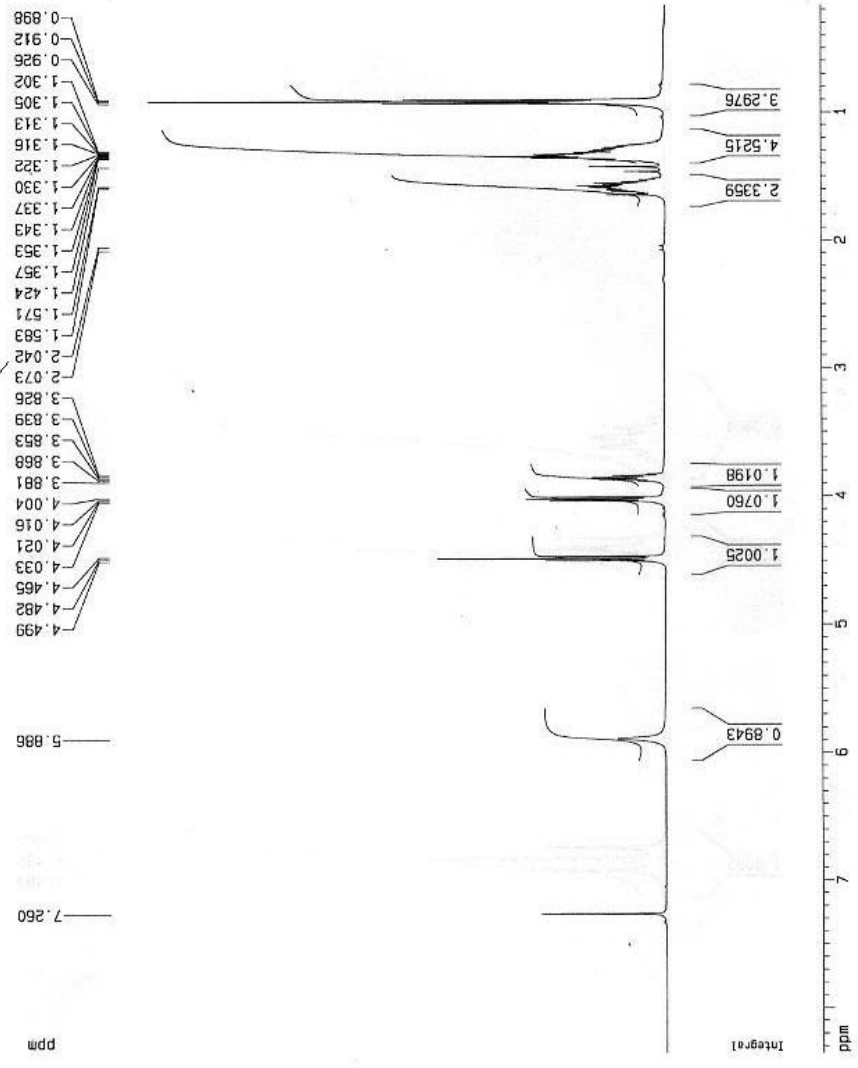
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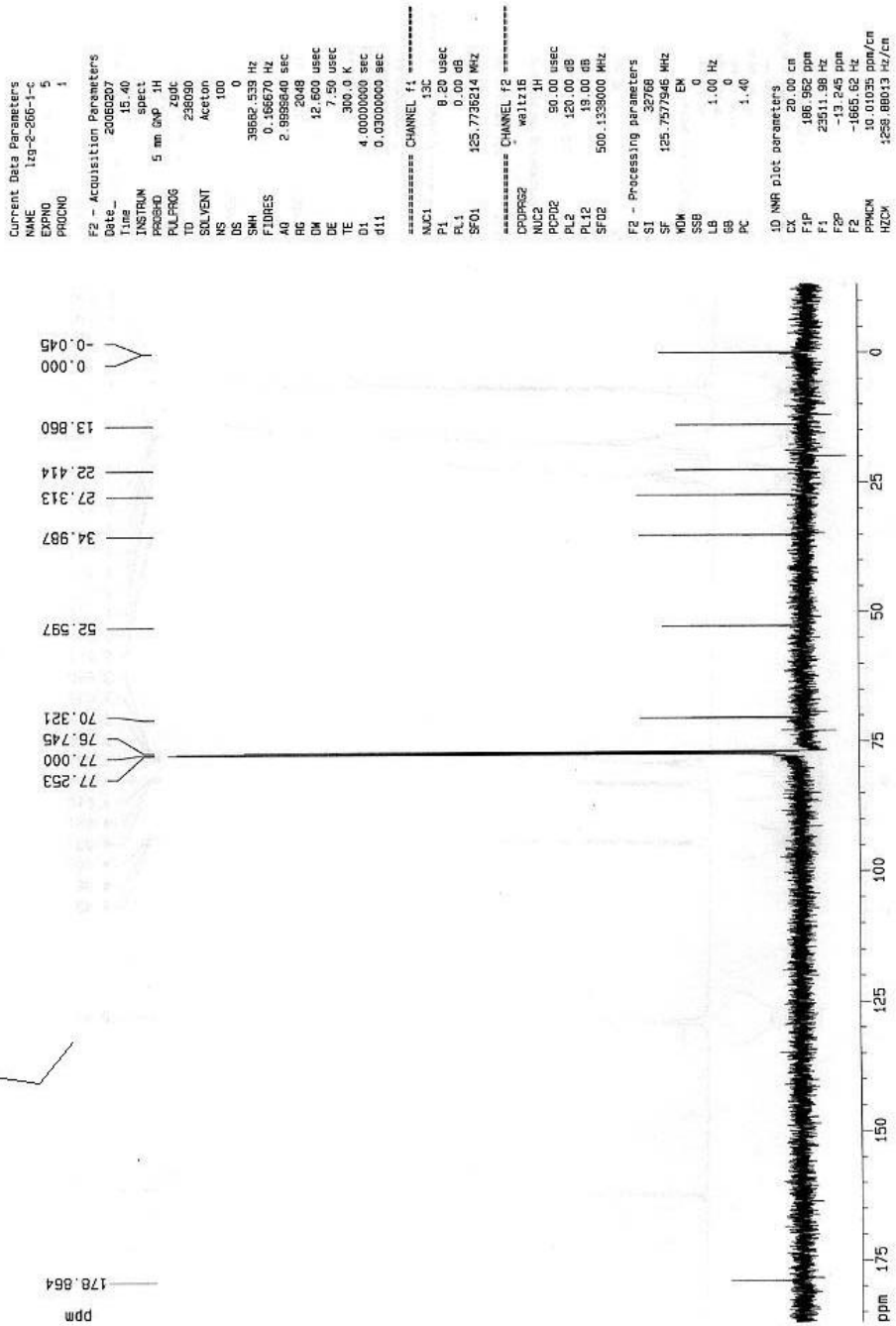
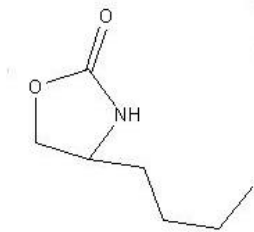
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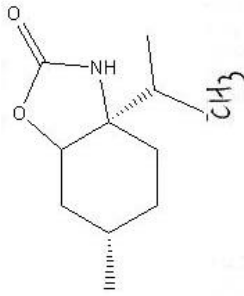
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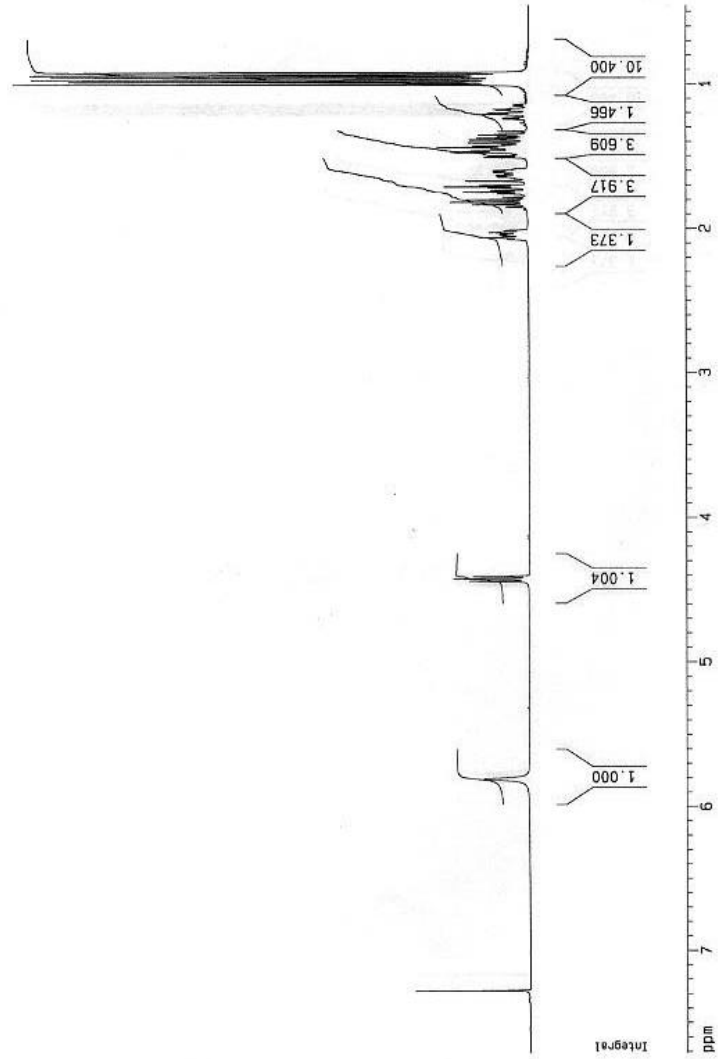
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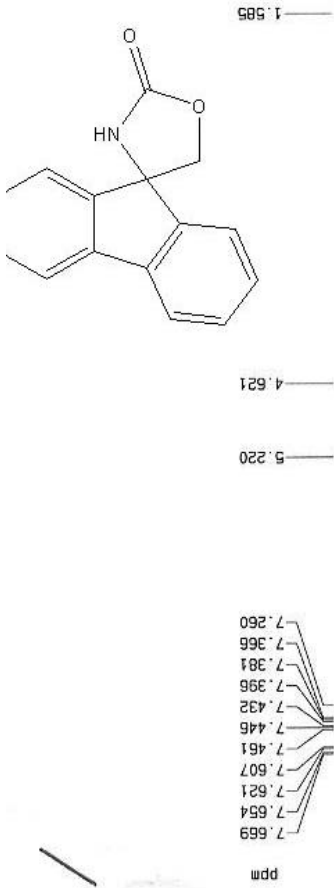
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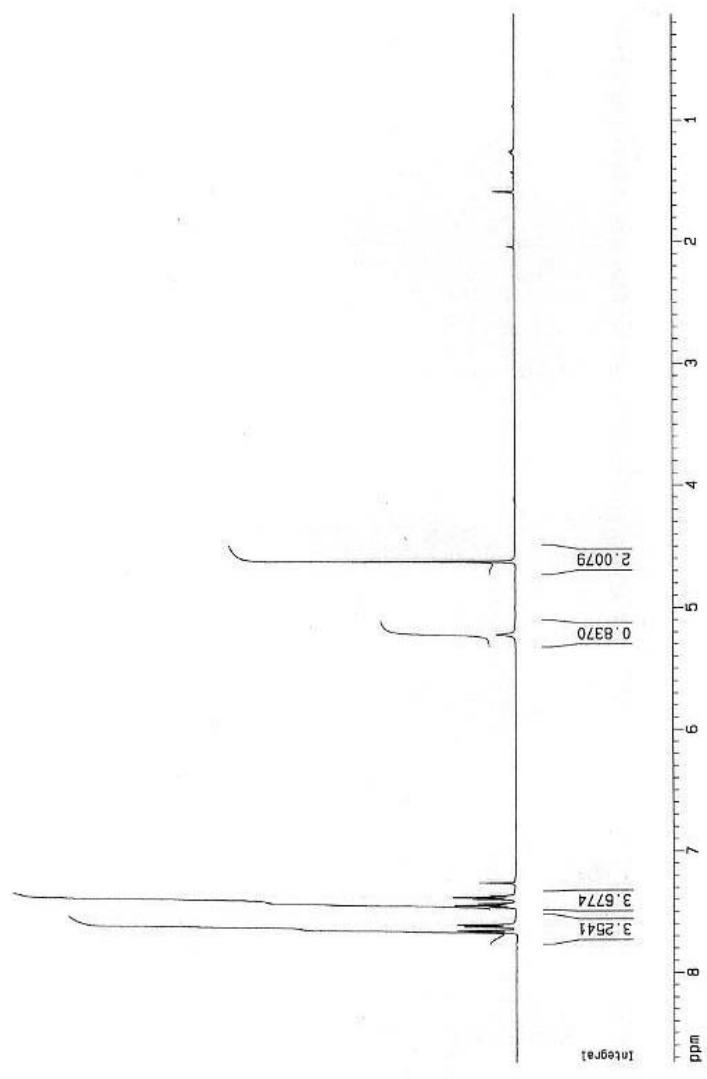
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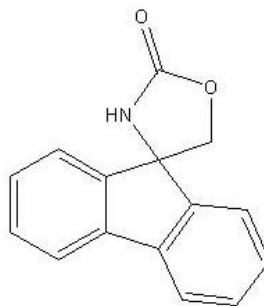
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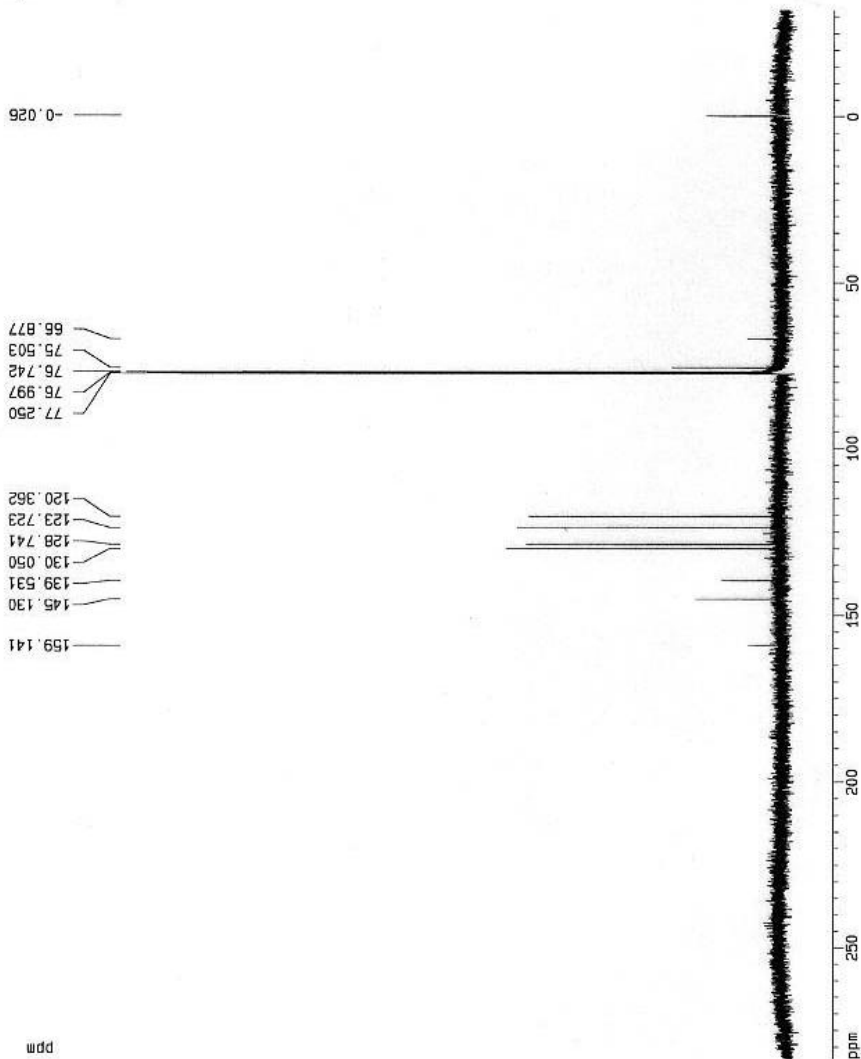
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 D11 0.05000000 sec

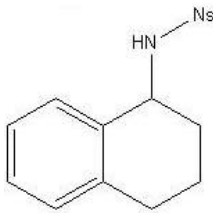
===== CHANNEL f1 =====
 NUC1 13C
 P1 8.20 usec
 PL1 0.00 dB
 SFO1 125.7735214 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 120.00 dB
 PL12 19.00 dB
 SFO2 500.1338000 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577934 MHz
 MDW EH
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 283.568 ppm
 F1 35660.84 Hz
 F2P -31.980 ppm
 F2 -4021.70 Hz
 PPMCN 15.7737 ppm/cm
 HZCN 1984.12671 Hz/cm





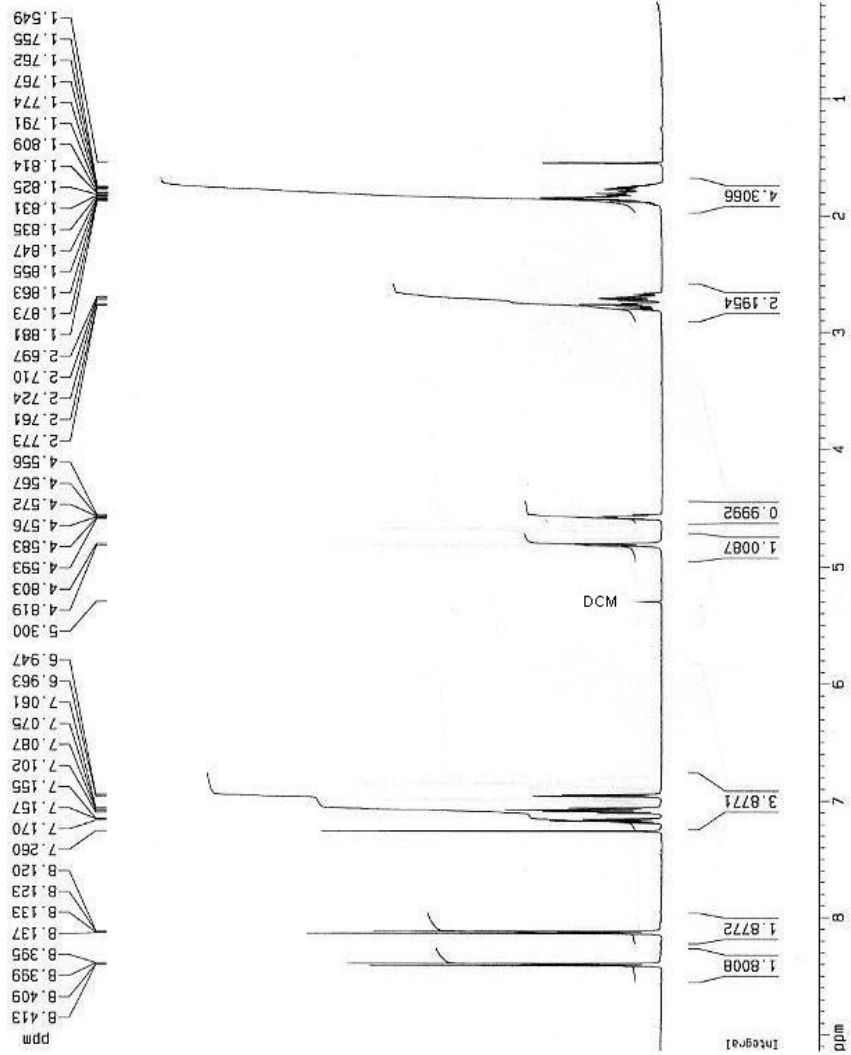
Current Data Parameters
 NAME 12g-2-265-3-p
 EXPNO 5
 PROCNO 1

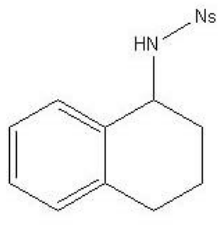
F2 - Acquisition Parameters
 Date_ 20060213
 Time 11.05
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT DMS
 NS 8
 DS 0
 SWH 5209.333 Hz
 FIDRES 0.158946 Hz
 AQ 3.1457779 sec
 RG 128
 DM 96.000 usec
 DE 4.50 usec
 TE 300.0 K
 D1 2.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.00 usec
 PL1 0.00 dB
 SFO1 500.1320116 MHz

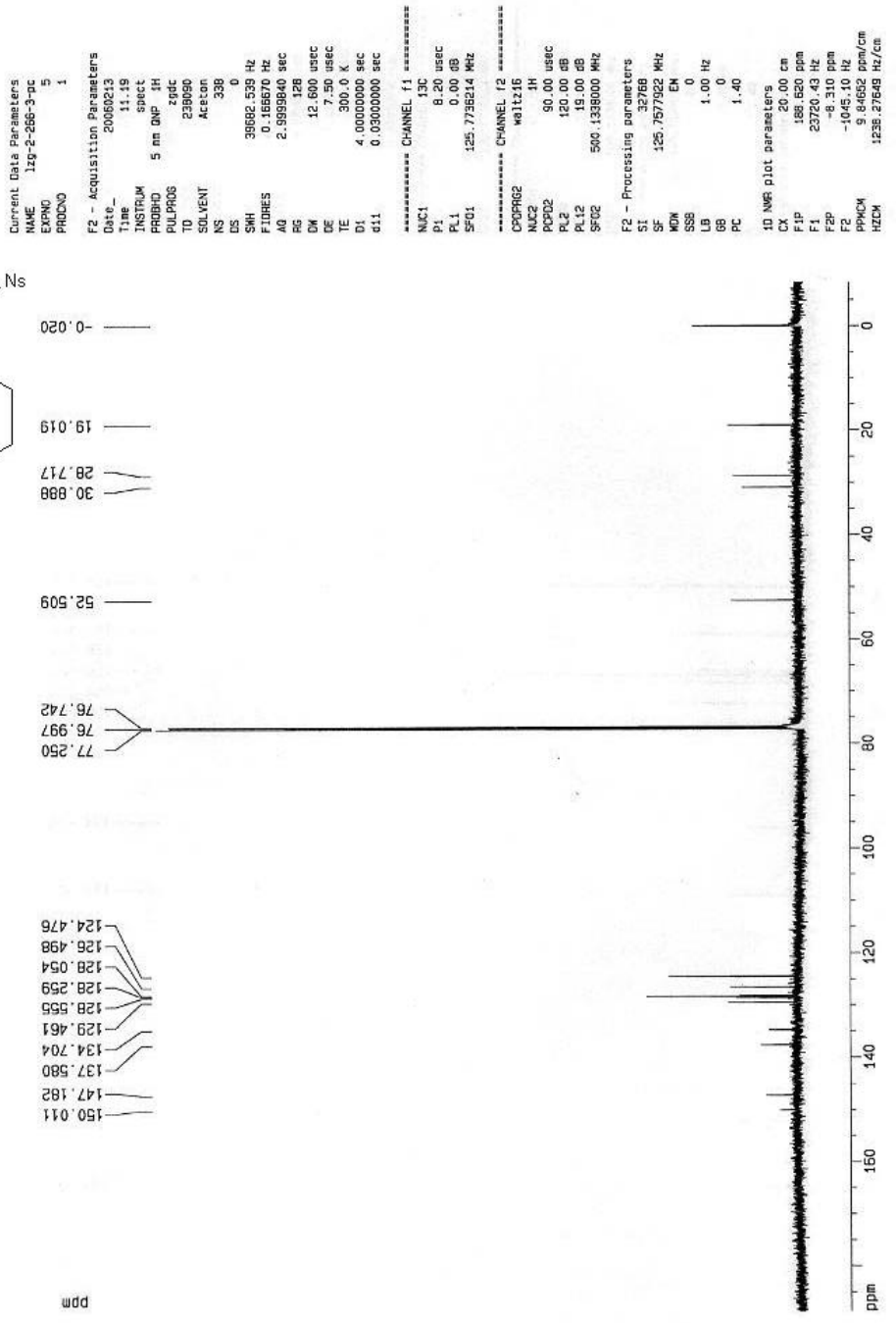
F2 - Processing parameters
 SI 16384
 SF 500.1300132 MHz
 MDW EH
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

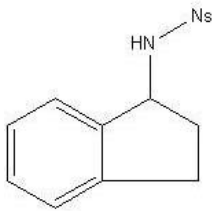
10 NMR plot parameters
 CX 20.00 cm
 F1 9.138 ppm
 F2 4570.25 Hz
 F3 0.173 ppm
 F4 86.69 Hz
 PPMCN 0.44624 ppm/cm
 HZCN 254.18008 Hz/cm





97/10/17, #5, 122.5 mg





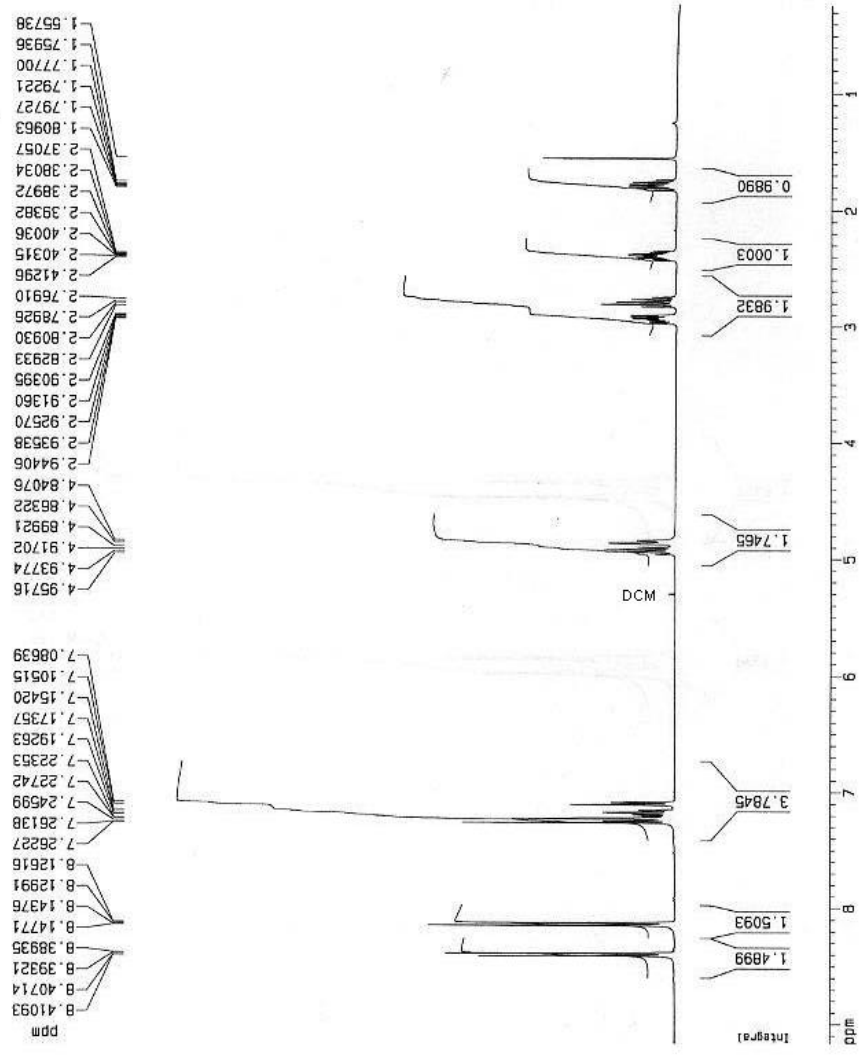
Current Data Parameters
 NAME 129-2-276-2-4
 EXPNO 1
 PROCNO 1

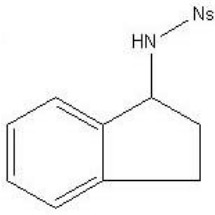
F2 - Acquisition Parameters
 Date_ 20060226
 Time_ 9:59
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 4807.692 Hz
 FIDRES 0.2934438 Hz
 AQ 1.7039860 sec
 RG 161.3
 DM 104.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 10.50 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300092 MHz
 MDK EK
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 9.170 ppm
 F1 3669.16 Hz
 F2P 0.234 ppm
 F2 93.65 Hz
 PPMCM 0.44679 ppm/cm
 HZCM 176.77574 Hz/cm





Current Data Parameters
 NAME 179-2-282-4c
 EXPNO 5
 PROCNO 1

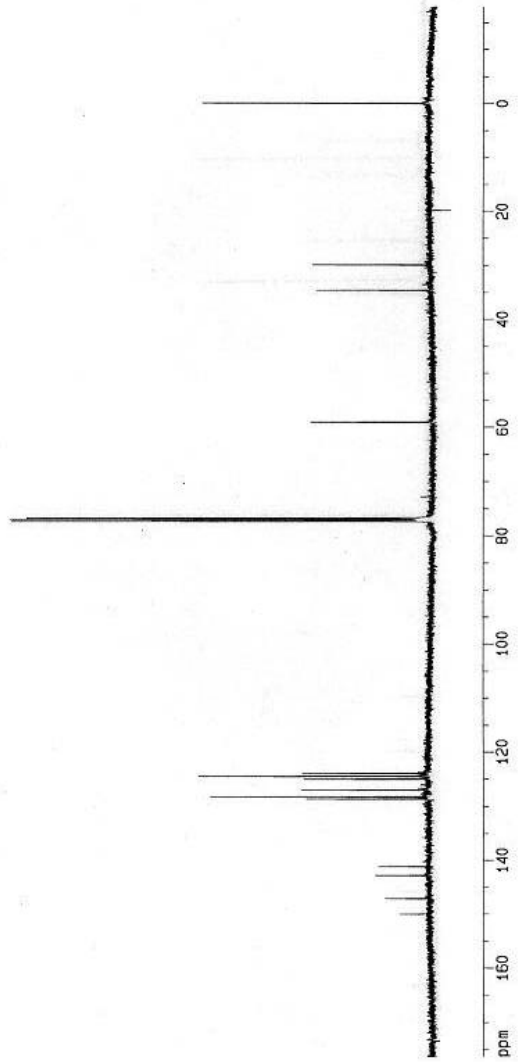
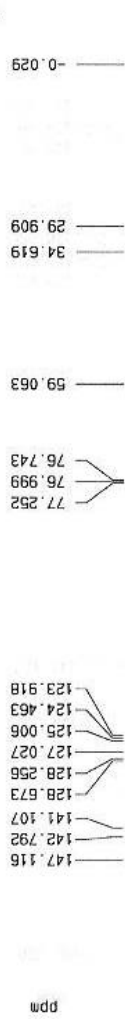
F2 - Acquisition Parameters
 Date_ 20060219
 Time 10.40
 INSTRUM spect
 PROBHD 5 mm QNP 3H
 PULPROG zgpg
 TD 298090
 SOLVENT Aceton
 NS 350
 DS 0
 SWH 39682.539 Hz
 FIDRES 0.166570 Hz
 AQ 2.9999840 sec
 RG 2048
 DM 12.600 usec
 DE 7.50 usec
 TE 300.0 K
 D1 4.00000000 sec
 d11 0.03000000 sec

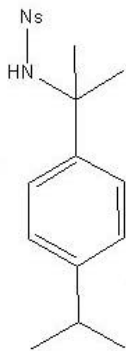
----- CHANNEL f1 -----
 NUC1 13C
 P1 8.20 usec
 PL1 0.00 dB
 SFO1 125.7735214 MHz

----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 P2 50.00 usec
 PL2 120.00 dB
 PL12 19.00 dB
 SFO2 500.1350000 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577955 MHz
 NQW 0
 EQ 0
 LS 1.00 Hz
 LB 0
 GB 0
 PC 1.40

ID NMR plot parameters
 AX 20.00 cm
 F1P 175.593 ppm
 F1 2212.25 Hz
 F2P -17.645 ppm
 F2 -264.48 Hz
 PRGM 9.70795 ppm/cm
 HZCM 1220.63511 Hz/cm





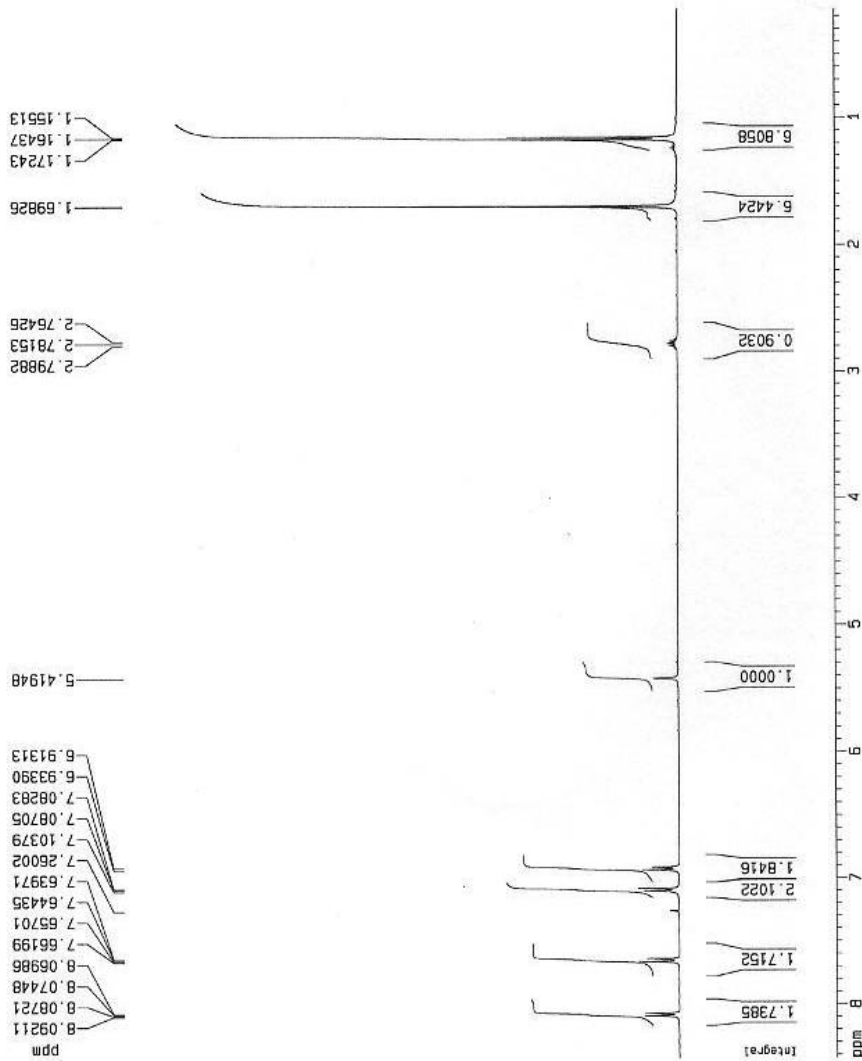
Current Data Parameters
 NAME 1zg-9-50-4
 EXPNO 1
 PROCNO 1

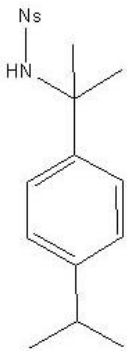
F2 - Acquisition Parameters
 Date_ 2005.015
 Time 15.16
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 4807.692 Hz
 FIDRES 0.293436 Hz
 AQ 1.7039860 sec
 RG 50.6
 DW 104.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.0000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 10.50 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300084 MHz
 MVM EN
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

10 NMR plot parameters
 CX 20.00 cm
 F1P 6.427 ppm
 F1 3371.71 Hz
 F2P 0.142 ppm
 F2 56.77 Hz
 PPMCM 0.41423 ppm/cm
 HZCM 165.74698 Hz/cm





Current Data Parameters
 NAME 12g-3-90-4c
 EXPNO 5
 PROCNO 1

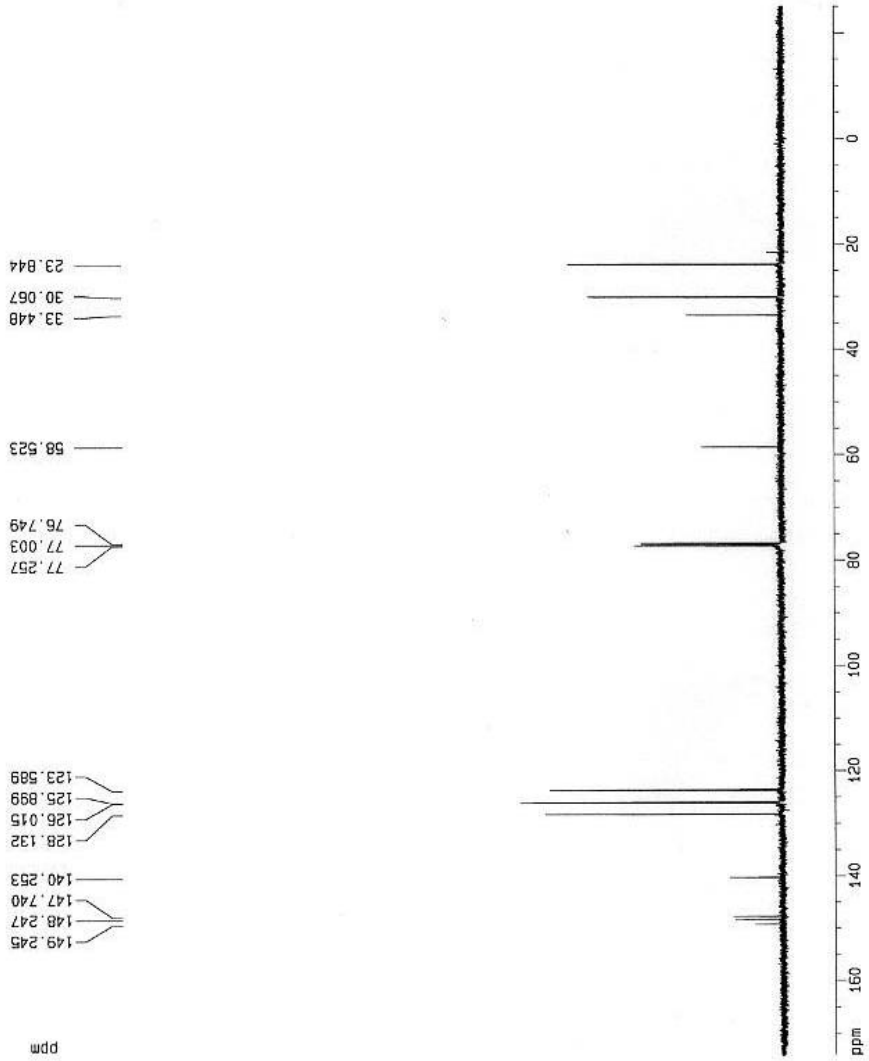
F2 - Acquisition Parameters
 Date_ 20061015
 Time 15.37
 INSTRUM spect
 PROBHD 5 mm DNP 1H
 PULPROG zgpg30
 TD 112780
 SOLVENT CDCl3
 NS 36
 DS 0
 SWH 25602.656 Hz
 FIDRES 0.222226 Hz
 AQ 2.250010 sec
 RG 8192
 DM 19.560 usec
 DE 7.50 usec
 TE 300.0 K
 D1 4.0000000 sec
 d11 0.0300000 sec

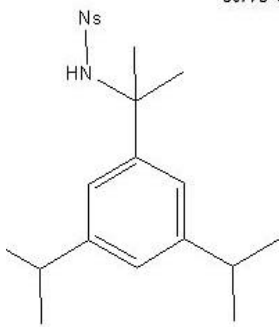
===== CHANNEL f1 =====
 NUC1 13C
 P1 8.00 usec
 PL1 3.00 dB
 SFO1 125.767108 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P2 50.00 usec
 PL2 120.00 dB
 PL12 19.00 dB
 SFO2 500.1338000 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577949 MHz
 NH 64
 SH 0
 SB 1.00 Hz
 LB 0
 GB 0
 PC 1.40

1D NMR plot parameters
 CD 20.50 cm
 F1 174.202 ppm
 F2 2107.721 Hz
 F3 -281.091 ppm
 F4 -3188.449 Hz
 FRACK 9.98465 ppm/cm
 HZCX 1253.13281 Hz/cm





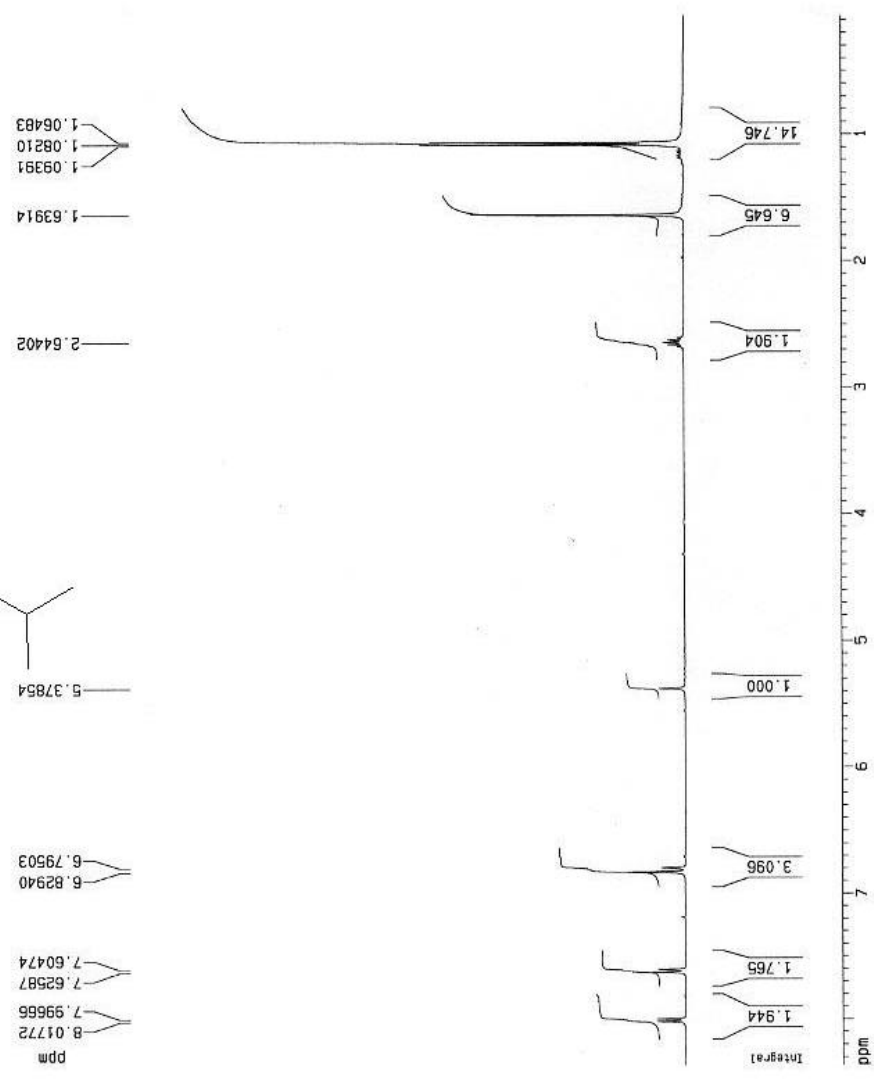
Current Data Parameters
 NAME 1zg-3-90-3
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20061015
 Time 15.10
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 16384
 SOLVENT CDCl3
 NS 4
 DS 0
 SMH 4807.692 Hz
 FT2RES 0.293438 Hz
 AQ 1.7099860 sec
 RG 32
 DM 104.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 10.50 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Process/fg parameters
 SI 32768
 SF 400.1300380 MHz
 NQ 1
 NVM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 8.356 ppm
 F1 3347.32 Hz
 F2P 0.058 ppm
 F2 27.23 Hz
 PPHCM 0.41488 ppm/cm
 HZCM 166.00435 Hz/cm



```

Current Data Parameters
NAME      120-3-30-3c
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20061015
Time     15.34
INSTRUM  spect
PROBHD   5 mm QNP 1H
PULPROG  zgpg30
TD       121208
SOLVENT  CDCl3
NS       145
DS       0
SWH      30303.031 Hz
FIDRES   0.254008 Hz
AQ       1.9969820 sec
RG        2048
Dw       16.500 usec
DE       7.50 usec
TE       300.2 K
D1       3.0000000 sec
d11      0.0300000 sec

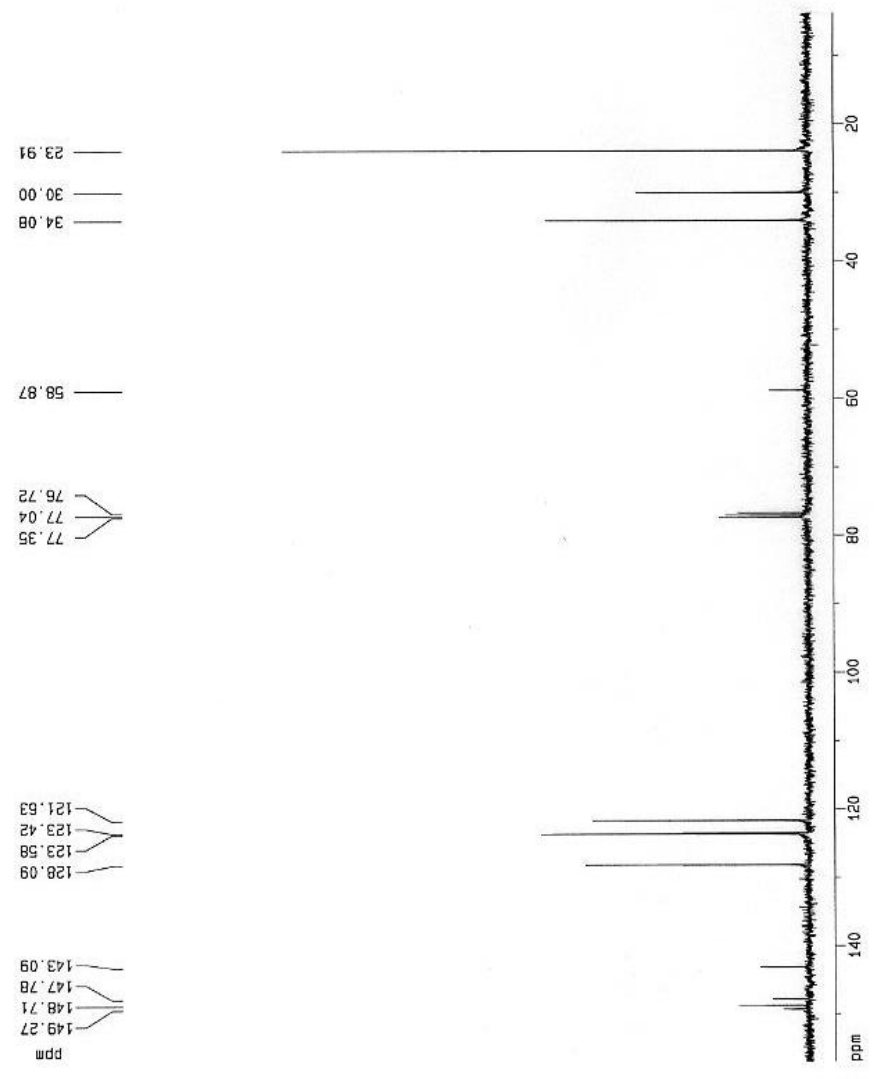
===== CHANNEL f1 =====
NUC1     13C
P1       11.00 usec
PL1     -2.00 dB
SFO1    100.6260697 MHz

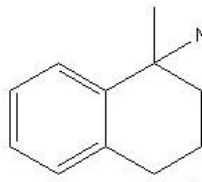
===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2   101.00 usec
PL2     120.00 dB
PL12    19.00 dB
SFO2    400.1324710 MHz

F2 - Processing parameters
SI       16384
SF       500.6127717 MHz
RG       0
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.00

1D NMR plot parameters
CX       20.00 cm
FIP      155.866 ppm
F1       15794.76 Hz
F2       3.814 ppm
F2       383.70 Hz
PPMCH    7.65865 ppm/cm
HZCX     770.55347 Hz/cm

```





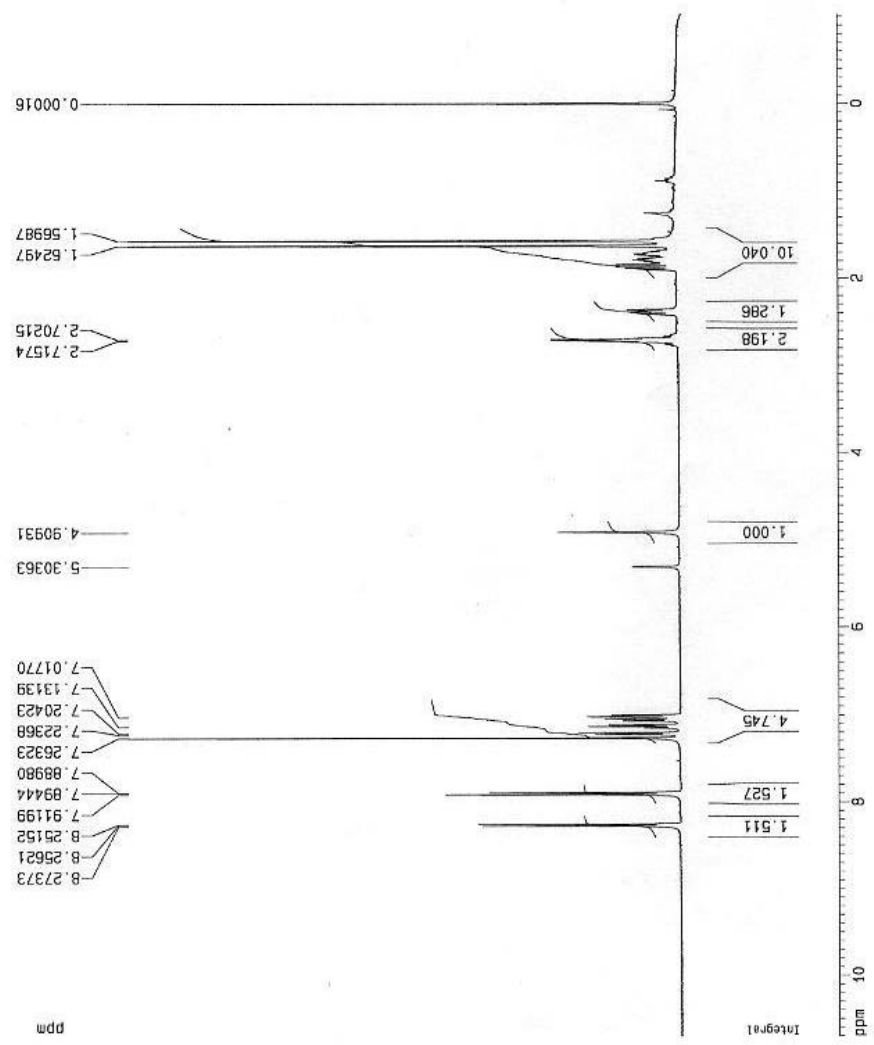
Current
 NAME
 EXPNO
 PROCNO

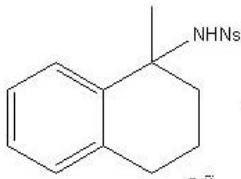
F2 - Acquisition Parameters
 Date_ 20060829
 Time 16:53
 INSTRUM spect
 PROBHD 5 mm GNP 1H
 PULPROG zg
 TO 16364
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 4867.692 Hz
 FIDRES 0.293438 Hz
 AQ 1.7639860 sec
 RG 25.4
 DW 164.000 USEC
 DE 6.00 USEC
 TE 300.0 K
 D1 2.0000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 10.50 USEC
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300073 MHz
 MDK EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1F 10.705 ppm
 F1 4283.41 Hz
 F2F -1.310 ppm
 F2 -524.26 Hz
 PPMCM 0.60077 ppm/cm
 HZCM 240.38454 Hz/cm





Current Data
 NAME 12
 EXPNO 12
 PROCNO 1

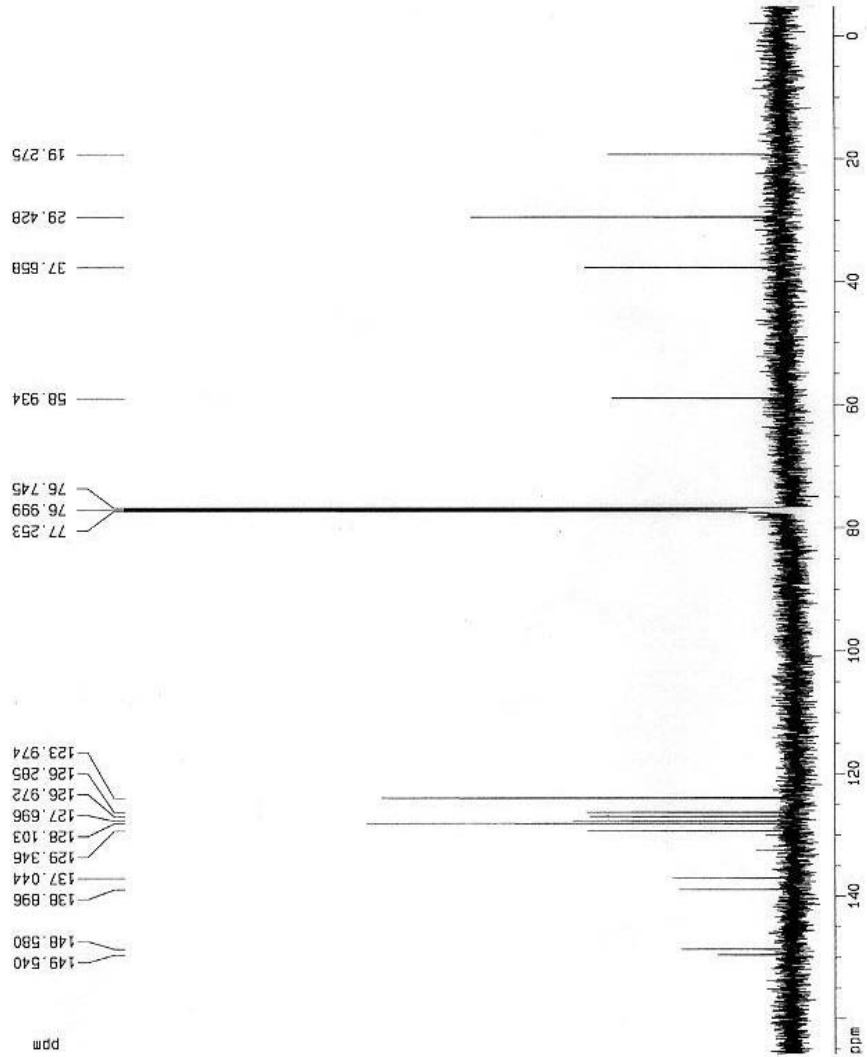
F2 - Acquisition Parameters
 Date_ 20060830
 Time 14.53
 INSTRUM spect
 PROBHD 5 mm DNP 1H
 PULPROG zgpg30
 TD 126360
 SOLVENT CDCl3
 NS 213
 DS 0
 SWH 22727.273 Hz
 FIDRES 0.165671 Hz
 AQ 2.9969700 sec
 RG 8192
 DN 22.000 usec
 DE 7.50 usec
 TE 300.0 K
 D1 4.0000000 sec
 d11 0.0300000 sec

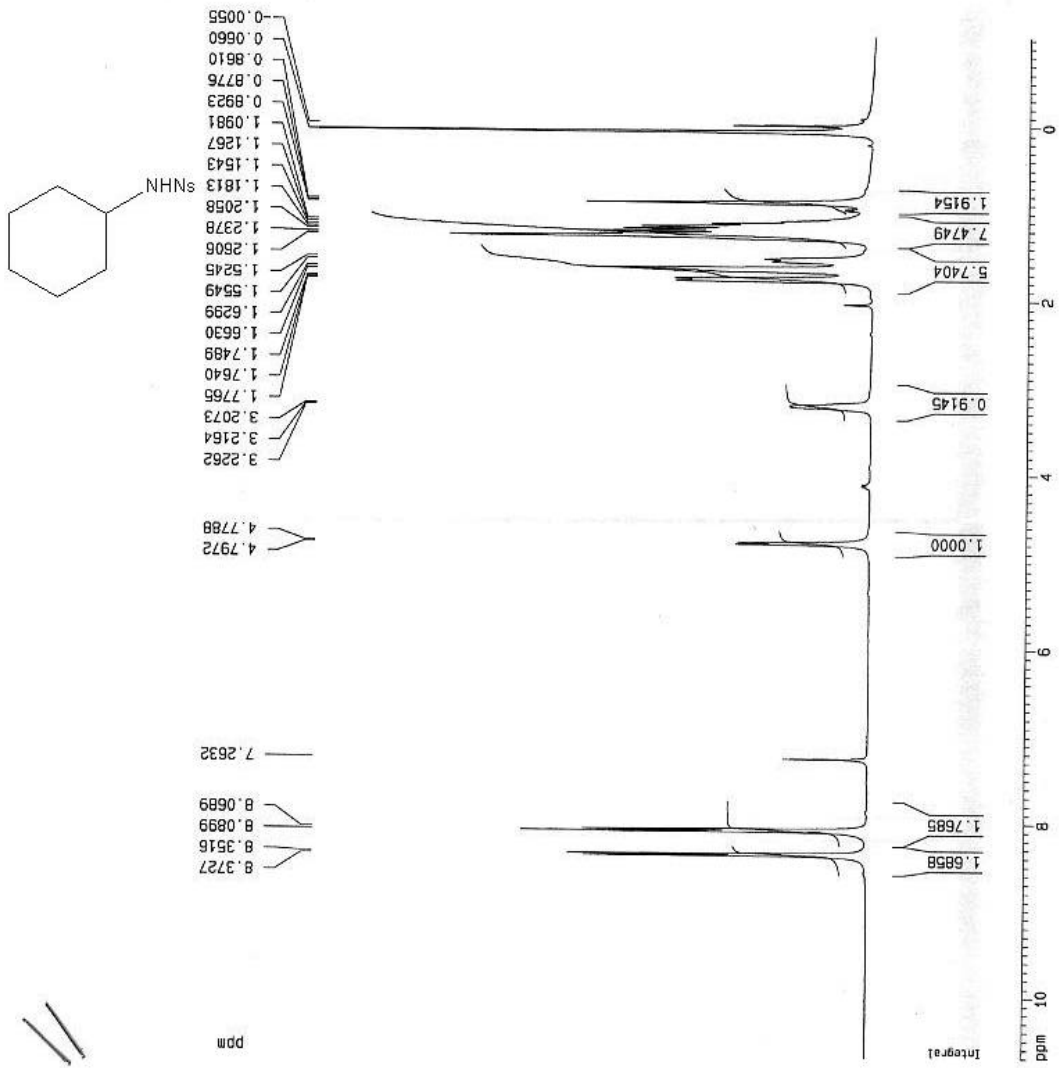
***** CHANNEL f1 *****
 NUC1 13C
 P1 11.50 usec
 PL1 -2.00 dB
 SFO1 125.7677956 MHz

***** CHANNEL f2 *****
 CPDPRG2 meltz16
 NUC2 1H
 PCPD2 50.00 usec
 PL2 120.00 dB
 PL12 19.00 dB
 SFO2 500.1385000 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577945 MHz
 MDN EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 165.761 ppm
 F1 20845.79 Hz
 F2P -4.753 ppm
 F2 -587.73 Hz
 PPMCM 8.52572 ppm/cm
 HZCM 1072.17578 Hz/cm





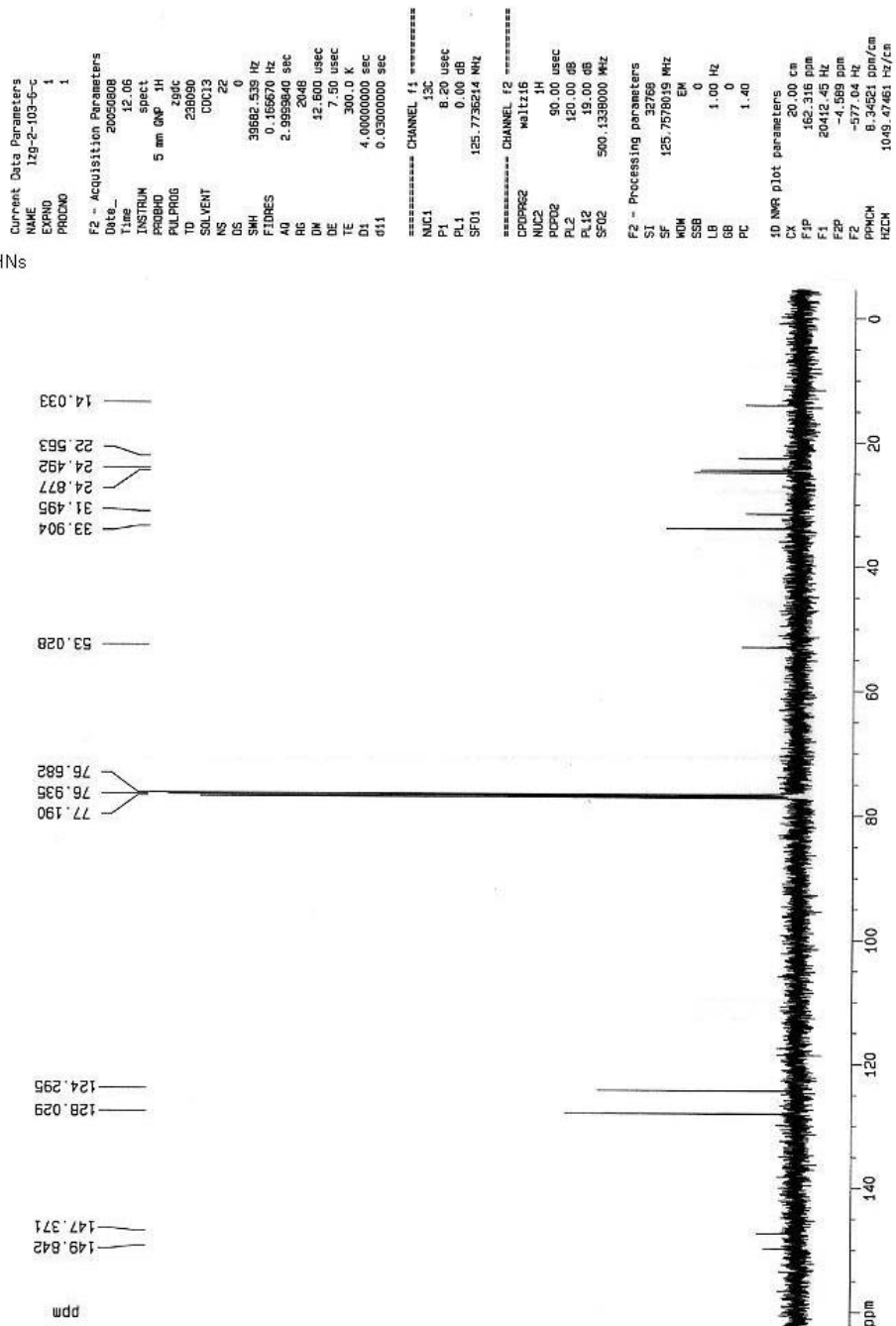
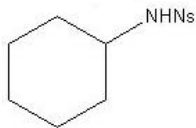
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 EXPNO 1
 PROCNO 1

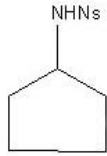
F2 - Acquisition Parameters
 Date_ 20050806
 Time 11.54
 INSTRUM spect
 PROBD 5 mm GNP 5H
 PULPROG zg
 TD 16384
 SOLVENT CDCl3
 NS 4
 DS 0
 SWH 4807.692 Hz
 FIDRES 0.203438 Hz
 AQ 1.7039860 sec
 RG 64
 DM 104.000 usec
 DE 5.00 usec
 TE 300.0 K
 D1 2.0000000 sec

***** CHANNEL f1 *****
 NUC1
 P1 10.50 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300090 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 FJP 10.705 ppm
 F1 4283.41 Hz
 F2P -1.310 ppm
 F2 -524.28 Hz
 PPMCH 0.60077 ppm/cm
 HZCM 240.38454 Hz/cm





Current Data Parameters
 NAME 12p-2-276-1-1
 EXPNO 1
 PROCNO 1

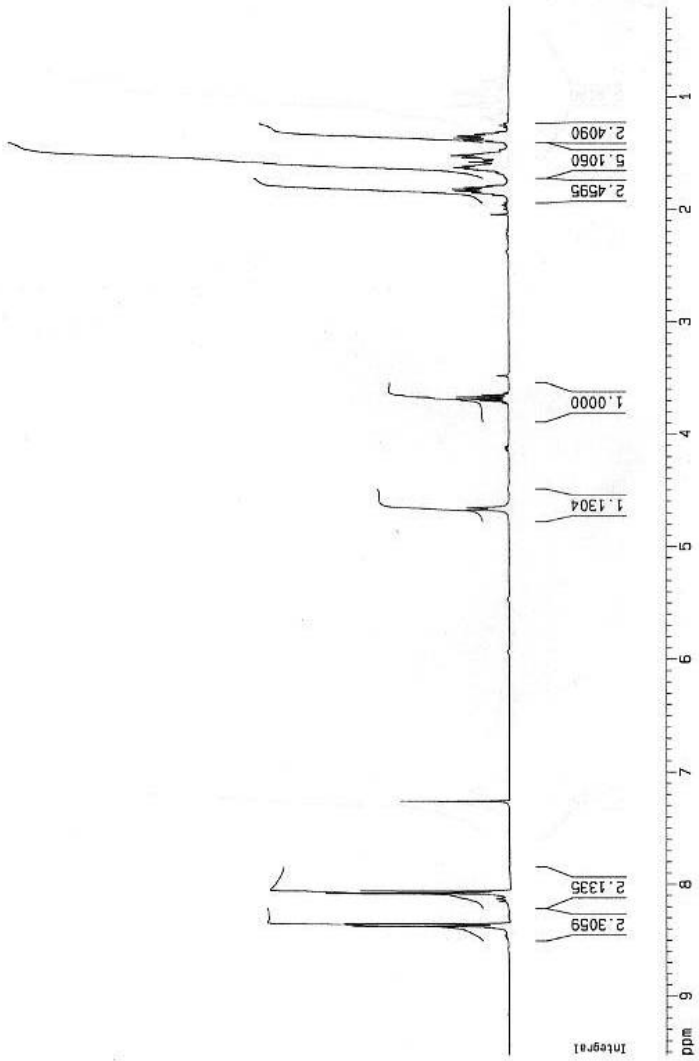
F2 - Acquisition Parameters
 Date_ 20060214
 Time 16.35
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TO 16384
 SOLVENT CDCl3
 NS 8
 DS 0
 SMH 4807.602 Hz
 FIDRES 0.293438 Hz
 AQ 1.7038800 sec
 RG 50.5
 DN 104.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.0000000 sec

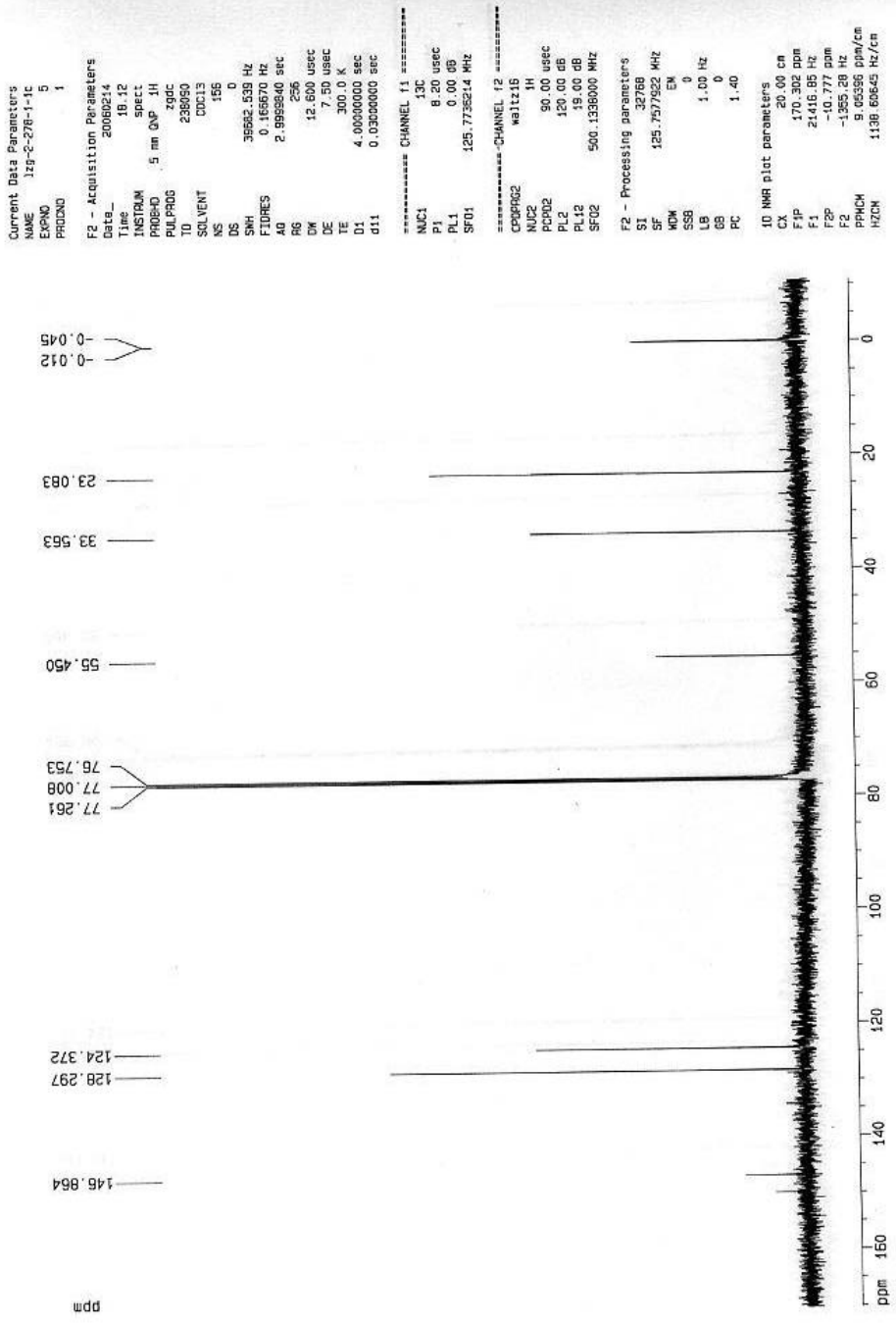
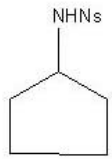
***** CHANNEL f1 *****
 NUC1 1H
 P1 10.50 usec
 PL1 0.00 dB
 SF01 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300104 MHz
 NOK EK
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1p 9.527 ppm
 F1 3612.05 Hz
 F2p 0.206 ppm
 F2 82.31 Hz
 PPMCN 0.46607 ppm/cm
 HZCN 166.48674 Hz/cm

8.3718
 8.3588
 8.0824
 8.06097
 7.25988
 7.25846







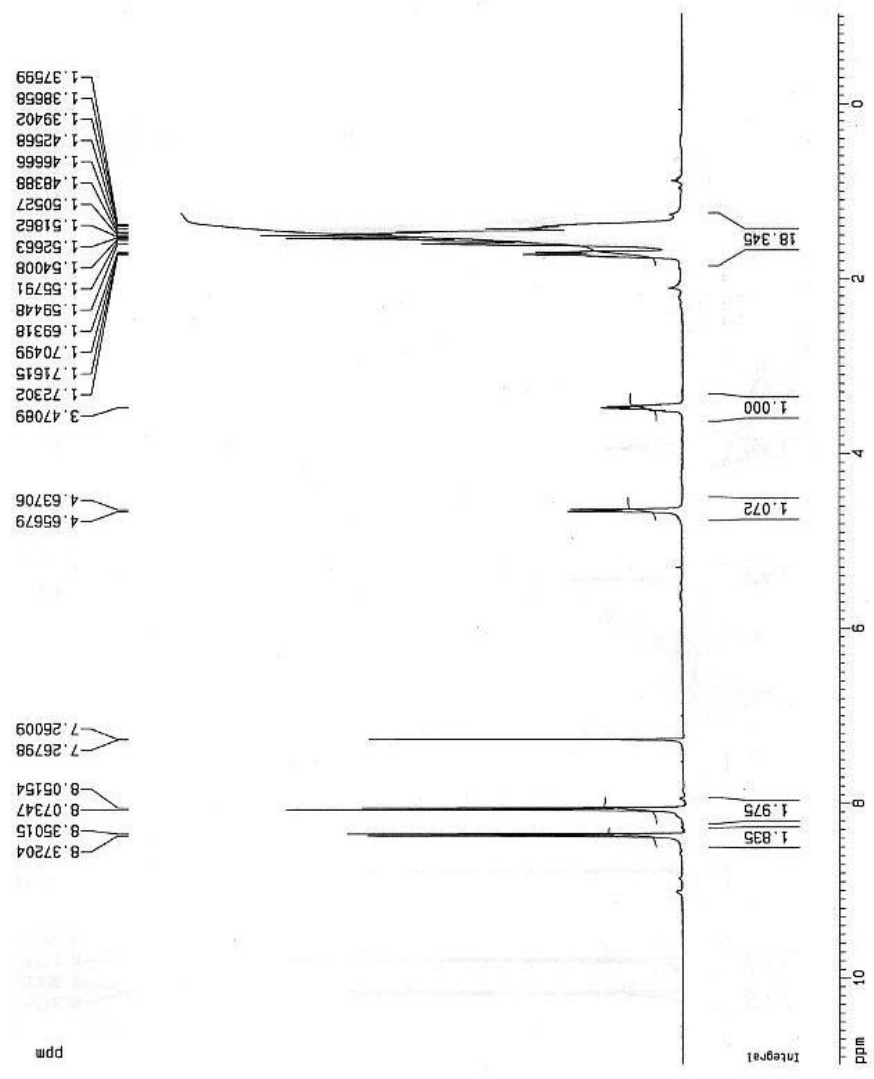
Current Data Parameters
 NAME 1Z5-5-0c
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20090516
 Time 10.29
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TO 16384
 SOLVENT CDCl3
 NS 8
 DS 0
 SMH 4807.692 Hz
 FIDRES 0.233438 Hz
 AQ 1.7039860 sec
 RG 181
 DM 104.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.0000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 10.50 usec
 PL1 0.00 dB
 SFO1 400.1320007 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300038 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F.F 40.983 ppm
 F1 4394.73 Hz
 F2 -1.032 ppm
 F2 -412.96 Hz
 PPMCM 0.60077 ppm/cm
 HZCM 240.38455 Hz/cm





```

Current Data Parameters
NAME      1zg-3-octc
EXPNO     5
PROCNO    1

F2 - Acquisition Parameters
Data_     20060514
Time      11.56
INSTRUM   spect
PROBHD    5 mm DNP 3H
PULPROG   zgpg30
TD         290c
TO         136360
SOLVENT    CDCl3
NS         109
DS         0
SHH        22727.273 Hz
FIDRES     0.166671 Hz
AQ         2.8989700 sec
RG         4096
DM         22.000 usec
DE         7.50 usec
TE         300.0 K
D1         4.00000000 sec
d11        0.03000000 sec

===== CHANNEL f1 =====
NUC1       13C
P1         11.50 usec
PL1        -2.00 dB
SF01       125.7677895 MHz

===== CHANNEL f2 =====
CPOPRG2    mltz16
NUC2        1H
P2          90.00 usec
PL2         120.00 dB
PL12        19.00 dB
SF02        500.1338000 MHz

F2 - Processing parameters
SI          32768
SF          125.7577895 MHz
RG          0
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40

10 NMR plot parameters
CX          20.00 cm
FIP         157.984 ppm
F1          19669.01 Hz
F2P         -3.446 ppm
F2          -433.39 Hz
PFGMCH     8.07202 ppm/cm
HZCN       1015.11981 Hz/cm
  
```

23.034
25.080
27.071
30.291
32.626

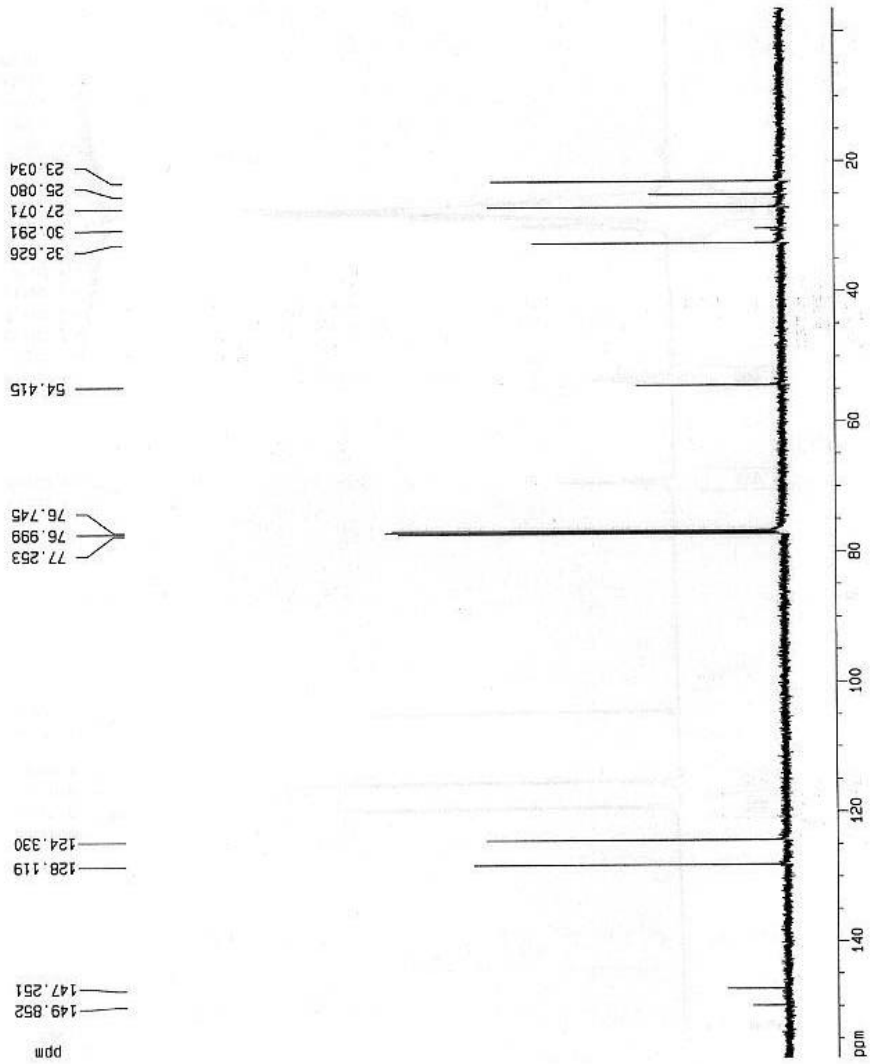
54.415

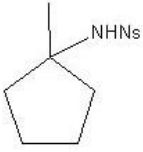
76.745
76.999
77.253

124.330
128.119

147.251
149.852

ppm





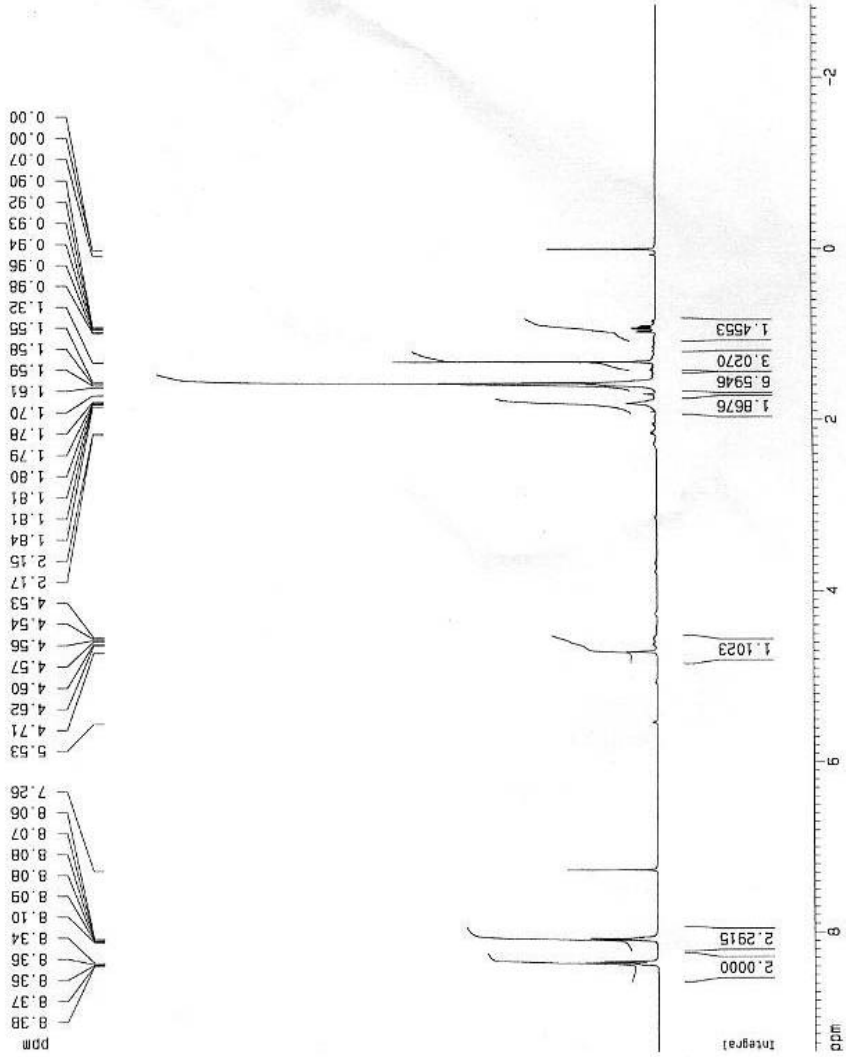
Current Data Parameters
 NAME 125-3-47-22
 EXPNO 5
 PROCNO 1

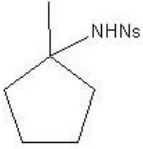
F2 - Acquisition Parameters
 Date_ 20060830
 Time 9.28
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zg
 TD 32768
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 7507.507 Hz
 FIDRES 0.228111 Hz
 AQ 2.1623888 sec
 RG 64
 DW 65.600 usec
 DE 4.50 usec
 TE 300.0 K
 D1 2.0000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 10.00 usec
 PL1 -1.00 dB
 SFO1 500.1320118 MHz

F2 - Processing parameters
 SI 16384
 SF 500.1300121 MHz
 KW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1p 9.400 ppm
 F1 4701.13 Hz
 F2p -2.848 ppm
 F2 -1424.24 Hz
 FFCM 0.61238 ppm/cm
 HZCM 305.26862 Hz/cm





Current Data Parameters
 NAME 120-3-47-4
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060920
 Time 17.04
 INSTRUM spect
 PROCNO 5
 PULPROG zgpg30
 TD 135360
 SOLVENT CDD13
 NS 113
 DS 0
 SWH 22727.273 Hz
 FIDRES 0.166671 Hz
 AQ 2.99883700 sec
 RG 6193
 DW 22.000 usec
 DE 7.50 usec
 TE 300.2 K
 D1 4.00000000 sec
 D11 0.03000000 sec

----- CHANNEL f1 -----
 NUC1 13C
 P1 11.50 usec
 PL1 -2.00 dB
 SFO1 125.767955 MHz

----- CHANNEL f2 -----
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 50.00 usec
 PL2 19.00 dB
 PL12 19.00 dB
 SFO2 500.135000 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577958 MHz
 MDN EN
 SSS 0
 LB 1.00 Hz
 GB 0
 PC 1.40

ID NMR plot parameters
 CA 20.00 cm
 F1P 165.550 ppm
 F1 21989.49 Hz
 F2P -10.757 ppm
 F2 -387.78 Hz
 PPRCM 9.05915 ppm/cm
 RZCM 1136.35353 Hz/cm

