

**Type 2 Intramolecular *N*-acylazo Diels-Alder Reaction: Regio- and Stereoselective Synthesis of Bridgehead Bicyclic 1,2-Diazines**

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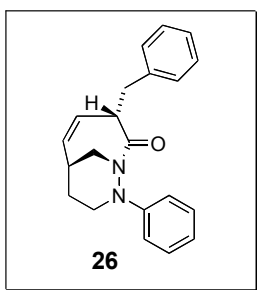
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## I. General Information and Materials.

Reagents were purchased and used without further purification. Solvents were either used as purchased or dried prior to use according to common methods of distillation where appropriate. All reactions were run in oven-dried glassware under a dry nitrogen atmosphere and were stirred magnetically, unless noted otherwise. Reactions were monitored by TLC plates precoated with silica gel, and spots were visualized by UV or by anisaldehyde stain. The crude reaction mixtures were purified by column chromatography on silica gel 60A° (200-400 mesh). Combined organic extracts were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. Solvents were removed by concentration under reduced pressure using a rotor-evaporator. Infrared (IR) spectra were recorded,  $\nu_{max}$  in cm<sup>-1</sup>. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded at 500 MHz and 125 MHz respectively. Chemical shifts are reported in parts per million (ppm,  $\delta$ ) relative to tetramethylsilane (TMS) as internal reference. The observed multiplicity of the absorptions are abbreviated as s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, app d = apparent d, app t = apparent t, and br = broad signal.

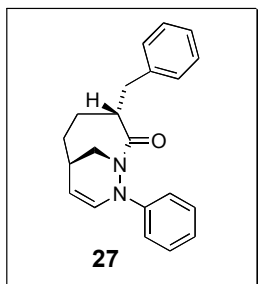
## II. Characterization of 26 and 27.



### 3-Benzyl-9-phenyl-1,9-diaza-bicyclo[4.3.1]dec-4-en-2-one (26).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.30-7.25 (m, 7H), 7.89(d,  $J$  = 7.7 Hz, 2H), 6.82(t,  $J$  = 6.0 Hz, 1H), 5.64(br s, 2H), 4.14(d,  $J$  = 14.7 Hz, 1H), 4.03(t,  $J$  = 3.8 Hz, 1H), 3.74(dd,  $J$  = 4.0, 14.5 Hz, 1H), 3.48(t,  $J$  = 14.5 Hz, 2H), 3.36(dd,  $J$  = 6.2, 14.2 Hz, 1H), 2.84(dd,  $J$  = 8.7, 14.2 Hz, 1H), 2.42(br s, 1H), 2.06(m, 1H), 1.48(d,  $J$  = 13.5 Hz, 1H), <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$

176.2, 146.9, 139.8, 132.8, 129.6, 129.5, 129.4, 128.7, 126.5, 119.4, 113.5, 46.8, 44.4, 42.5, 36.6, 33.5, 26.5; HRMS (ES)  $m/z$  calcd. for  $C_{21}H_{22}N_2O$   $[M + H]^+$  319.1810 found 319.1810.



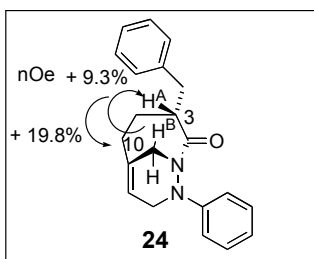
**3-Benzyl-9-phenyl-1,9-diaza-bicyclo[4.3.1]dec-7-en-2-one (27).**

$^1H$  NMR (500 MHz,  $CDCl_3$ )  $\delta$  7.33 (m, 6H), 7.00(d,  $J = 7.7$  Hz, 1H), 6.92 (d,  $J = 6.0$  Hz, 3H), 4.92 (t,  $J = 5.2$  Hz, 1H), 4.12 (d,  $J = 14.6$  Hz, 1H), 3.38 (dddd,  $J = 13.7, 13.7, 4.3, 4.3$  Hz, 2H), 3.10(m, 1H), 2.62(dd,  $J = 7.9, 14.1$  Hz, 1H), 2.37(br s 1H), 1.86(d,  $J = 13.0$  Hz, 1H), 1.77 (d,  $J = 12.7$  Hz, 1H), 1.70-1.60 (m, 3H);  $^{13}C$  NMR(125MHz,  $CDCl_3$ )  $\delta$  181.7, 144.4, 140.3, 131.5, 129.3, 129.0, 128.4, 126.2, 120.9, 114.6, 107.3, 50.2, 45.0, 37.8, 33.2, 31.6, 27.7; HRMS (ES)  $m/z$  calcd. for  $C_{21}H_{22}N_2O$   $[M + H]^+$  319.1810 found 319.1810.

### III. Stereochemical Proofs of Cycloadducts

#### A. Bridgehead bicyclic 1,2-diazine **24**.

The peaks in the  $^1H$  NMR spectra were assigned using  $^1H/^{13}C$  HMQC,  $^1H$  NMR chemical shifts. The determination of the relative stereochemistry of cycloadduct was achieved through nOe spectroscopy.

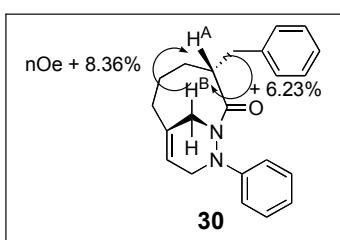


$H^A$  irradiated:  $H^B$  (19.8 %)

$H^B$  irradiated:  $H^A$  (9.3 %)

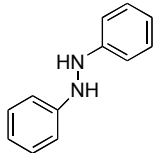
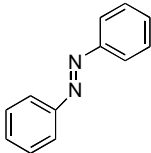
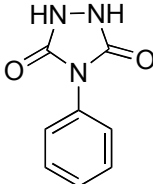
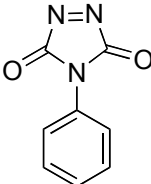
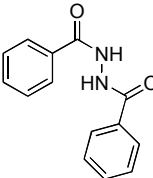
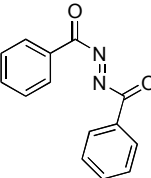
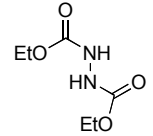
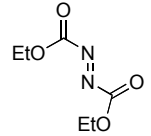
B. Bridgehead bicyclic 1,2-diazine **30**.

The peaks in the  $^1\text{H}$  NMR spectra were assigned using  $^1\text{H}/^{13}\text{C}$  HMQC,  $^1\text{H}$  NMR chemical shifts. The determination of the relative stereochemistry of cycloadduct was achieved through nOe spectroscopy.



H<sup>A</sup> irradiated: H<sup>B</sup> (6.23 %)

H<sup>B</sup> irradiated: H<sup>A</sup> (8.36 %)

entry	substrate	solvent <sup>a</sup>	time	product	yield (%)
1		CH <sub>2</sub> Cl <sub>2</sub>	30 min		88 <sup>a-c</sup>
2		acetone	30 min		100 <sup>a,d</sup>
3		CH <sub>2</sub> Cl <sub>2</sub>	24 h		N. R. <sup>e</sup>
4		acetone	24 h		N. R. <sup>e</sup>

<sup>a</sup> Standard reaction conditions: To a cooled (0 °C) solution of a hydrazide or hydrazine in was added *n*-Bu<sub>4</sub>NIO<sub>4</sub> (1.3 equiv). The reaction mixture was stirred at room temperature (TLC monitoring) and washed with 2 portions of sat Na<sub>2</sub>SO<sub>3</sub>. The organic layers were combined, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated *in vacuo*.

<sup>b</sup> SiO<sub>2</sub> was added to the reaction mixture and stirred for 5 min. Then the reaction mixture was filtered and the CH<sub>2</sub>Cl<sub>2</sub> was removed. Azobenzene was isolated without further purification.

<sup>c</sup>The <sup>1</sup>H NMR and <sup>13</sup>C NMR spectrum of the product were compared with commercially available material.

<sup>d</sup>The % yield for the oxidation of 4-Phenyl urazole was determined by the crude <sup>1</sup>H NMR of the reaction mixture, which only showed 4-Phenyl-1,2,4-triazoline-3,5-dione and tetra-*n*-butyl-ammonium. See <sup>1</sup>H NMR and <sup>13</sup>C NMR. The formation of 4-Phenyl-1,2,4-triazoline-3,5-dione was also conformed by trapping the product with cyclohexadiene.

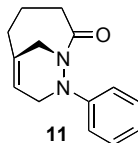
<sup>e</sup> No reaction was observed .

#### IV. X-ray Crystallography Data

X-ray Data Collection, Structure Solution and Refinement for kjs11.

A colorless crystal of approximate dimensions 0.27 x 0.36 x 0.41 mm was mounted on a glass fiber and transferred to a Bruker CCD platform diffractometer. The SMART<sup>1</sup> program package was used to determine the unit-cell parameters and for data collection (20 sec/frame scan time for a sphere of diffraction data). The raw frame data was processed using SAINT<sup>2</sup> and SADABS<sup>3</sup> to yield the reflection data file. Subsequent calculations were carried out using the SHELXTL<sup>4</sup> program. The diffraction symmetry was  $2/m$  and the systematic absences were consistent with the centrosymmetric monoclinic space group  $P2_1/c$  which was later determined to be correct.

The structure was solved by direct methods and refined on  $F^2$  by full-matrix least-squares techniques. The analytical scattering factors<sup>5</sup> for neutral atoms were used throughout the analysis. Hydrogen atoms were located from a difference-Fourier map and refined ( $x,y,z$  and  $U_{iso}$ ). At convergence,  $wR2 = 0.0905$  and  $Goof = 1.023$  for 219 variables refined against 2851 data. As a comparison for refinement on  $F$ ,  $R1 = 0.0347$  for those 2510 data with  $I > 2.0(I)$ .



## References.

1. SMART Software Users Guide, Version 5.1, Bruker Analytical X-Ray Systems, Inc.; Madison, WI 1999.
2. SAINT Software Users Guide, Version 6.0, Bruker Analytical X-Ray Systems, Inc.; Madison, WI 1999.
3. Sheldrick, G. M. SADABS, Version 2.03, Bruker Analytical X-Ray Systems, Inc.; Madison, WI 2000.
4. Sheldrick, G. M. SHELXTL Version 5.10, Bruker Analytical X-Ray Systems, Inc.; Madison, WI 1999.
5. International Tables for X-Ray Crystallography 1992, Vol. C., Dordrecht: Kluwer Academic Publishers.

## Definitions:

$$wR2 = \frac{[\sum w(F_o^2 - F_c^2)^2]}{[\sum w(F_o^2)^2]}^{1/2}$$

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

Goof = S =  $\frac{[\sum w(F_o^2 - F_c^2)^2]}{(n-p)}^{1/2}$  where n is the number of reflections and p is the total number of parameters refined.

The thermal ellipsoid plot is shown at the 50% probability level.

Table 1. Crystal data and structure refinement for kjs11.

Identification code	kjs11 (Chun Chow)
Empirical formula	C <sub>14</sub> H <sub>16</sub> N <sub>2</sub> O
Formula weight	228.29
Temperature	163(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic

Space group	$P2_1/c$	
Unit cell dimensions	$a = 9.1592(4) \text{ \AA}$	$\alpha = 90^\circ$ .
	$b = 10.4770(4) \text{ \AA}$	$\beta = 90.8100(10)^\circ$ .
	$c = 12.3420(5) \text{ \AA}$	$\gamma = 90^\circ$ .
Volume	$1184.23(8) \text{ \AA}^3$	
Z	4	
Density (calculated)	$1.280 \text{ Mg/m}^3$	
Absorption coefficient	$0.082 \text{ mm}^{-1}$	
F(000)	488	
Crystal size	$0.41 \times 0.36 \times 0.27 \text{ mm}^3$	
Theta range for data collection	$2.22$ to $28.27^\circ$ .	
Index ranges	$-12 \ h \ 11, -13 \ k \ 13, -16 \ l \ 15$	
Reflections collected	12061	
Independent reflections	2851 [R(int) = 0.0224]	
Completeness to theta = $28.27^\circ$	97.0 %	
Absorption correction	None	
Max. and min. transmission	0.9782 and 0.9672	
Refinement method	Full-matrix least-squares on $F^2$	
Data / restraints / parameters	2851 / 0 / 219	
Goodness-of-fit on $F^2$	1.023	
Final R indices [ $I > 2\sigma(I)$ ]	$R1 = 0.0347, wR2 = 0.0861$	
R indices (all data)	$R1 = 0.0401, wR2 = 0.0905$	
Extinction coefficient	$0.011(2)$	
Largest diff. peak and hole	$0.261$ and $-0.157 \text{ e.\AA}^{-3}$	



Table 2. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for kjs11.  $U(\text{eq})$  is defined as one third of the trace of the orthogonalized  $U^{\text{ij}}$  tensor.

	x	y	z	U(eq)
O(1)	4064(1)	9474(1)	2239(1)	30(1)
N(1)	5809(1)	8006(1)	1823(1)	22(1)
C(2)	4378(1)	8373(1)	2036(1)	22(1)
C(3)	3223(1)	7324(1)	2000(1)	26(1)
C(4)	2591(1)	7128(1)	844(1)	28(1)
C(5)	3640(1)	6540(1)	13(1)	33(1)
C(6)	5103(1)	7169(1)	136(1)	27(1)
C(7)	5530(1)	8252(1)	-329(1)	27(1)
C(8)	6798(1)	8953(1)	179(1)	26(1)
N(9)	6680(1)	9003(1)	1396(1)	22(1)
C(10)	5949(1)	6840(1)	1153(1)	27(1)
C(11)	7986(1)	9209(1)	1992(1)	23(1)
C(12)	9116(1)	9937(1)	1552(1)	32(1)
C(13)	10369(1)	10192(1)	2162(1)	37(1)
C(14)	10529(1)	9730(1)	3205(1)	36(1)
C(15)	9412(1)	9017(1)	3647(1)	34(1)
C(16)	8145(1)	8758(1)	3053(1)	27(1)

Table 3. Bond lengths [Å] and angles [°] for kjs11.

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O(1)-C(2)	1.2163(12)
N(1)-C(2)	1.3941(13)
N(1)-N(9)	1.4207(11)
N(1)-C(10)	1.4819(13)
C(2)-C(3)	1.5258(13)
C(3)-C(4)	1.5462(14)
C(4)-C(5)	1.5433(16)
C(5)-C(6)	1.4987(16)
C(6)-C(7)	1.3339(15)
C(6)-C(10)	1.5048(15)
C(7)-C(8)	1.5027(15)
C(8)-N(9)	1.5077(12)
N(9)-C(11)	1.4119(13)
C(11)-C(16)	1.3983(14)
C(11)-C(12)	1.4013(14)
C(12)-C(13)	1.3897(17)
C(13)-C(14)	1.3815(19)
C(14)-C(15)	1.3850(17)
C(15)-C(16)	1.3899(15)

C(2)-N(1)-N(9)	113.69(8)
C(2)-N(1)-C(10)	114.88(8)
N(9)-N(1)-C(10)	110.22(8)
O(1)-C(2)-N(1)	121.73(9)
O(1)-C(2)-C(3)	121.53(9)
N(1)-C(2)-C(3)	116.74(9)
C(2)-C(3)-C(4)	111.87(8)
C(5)-C(4)-C(3)	115.97(9)
C(6)-C(5)-C(4)	108.75(8)
C(7)-C(6)-C(5)	126.71(10)
C(7)-C(6)-C(10)	113.77(10)
C(5)-C(6)-C(10)	115.66(9)
C(6)-C(7)-C(8)	117.75(10)
C(7)-C(8)-N(9)	111.55(8)
C(11)-N(9)-N(1)	113.23(7)
C(11)-N(9)-C(8)	116.91(8)
N(1)-N(9)-C(8)	113.09(8)
N(1)-C(10)-C(6)	103.26(8)
C(16)-C(11)-C(12)	118.63(10)
C(16)-C(11)-N(9)	120.87(9)
C(12)-C(11)-N(9)	120.39(9)
C(13)-C(12)-C(11)	120.24(11)
C(14)-C(13)-C(12)	120.89(11)
C(13)-C(14)-C(15)	119.12(11)
C(14)-C(15)-C(16)	120.91(11)
C(15)-C(16)-C(11)	120.20(10)

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Table 4. Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for kjs11. The anisotropic displacement factor exponent takes the form:  $-2\pi^2 [ h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12} ]$

	U <sup>11</sup>	U <sup>22</sup>	U <sup>33</sup>	U <sup>23</sup>	U <sup>13</sup>	U <sup>12</sup>
O(1)	29(1)	28(1)	34(1)	-11(1)	10(1)	-5(1)
N(1)	24(1)	21(1)	20(1)	4(1)	0(1)	-3(1)
C(2)	25(1)	26(1)	14(1)	-2(1)	1(1)	-5(1)
C(3)	27(1)	27(1)	22(1)	-1(1)	1(1)	-8(1)
C(4)	30(1)	27(1)	27(1)	0(1)	-5(1)	-7(1)
C(5)	46(1)	28(1)	24(1)	-6(1)	-2(1)	-7(1)
C(6)	37(1)	23(1)	22(1)	-6(1)	5(1)	3(1)
C(7)	36(1)	28(1)	18(1)	-1(1)	3(1)	3(1)
C(8)	31(1)	26(1)	22(1)	5(1)	7(1)	2(1)
N(9)	22(1)	23(1)	21(1)	5(1)	3(1)	-2(1)
C(10)	31(1)	19(1)	31(1)	3(1)	3(1)	4(1)
C(11)	20(1)	19(1)	29(1)	0(1)	3(1)	3(1)
C(12)	26(1)	32(1)	38(1)	6(1)	6(1)	-2(1)
C(13)	23(1)	35(1)	54(1)	2(1)	7(1)	-5(1)
C(14)	22(1)	33(1)	52(1)	-6(1)	-5(1)	-1(1)
C(15)	31(1)	33(1)	36(1)	0(1)	-6(1)	-2(1)
C(16)	25(1)	27(1)	29(1)	1(1)	-1(1)	-4(1)

Table 5. Hydrogen coordinates ( $\times 10^4$ ) and isotropic displacement parameters ( $\text{\AA}^2 \times 10^{-3}$ ) for kjs11.

	x	y	z	U(eq)
H(3A)	3618(15)	6515(13)	2296(11)	34(3)
H(3B)	2429(14)	7611(13)	2459(11)	31(3)
H(4A)	2234(14)	7988(13)	574(11)	31(3)
H(4B)	1715(15)	6577(14)	896(11)	37(3)
H(5A)	3717(16)	5593(14)	162(12)	42(4)
H(5B)	3211(16)	6661(14)	-722(13)	43(4)
H(7)	5003(15)	8665(13)	-922(11)	35(3)
H(8A)	6852(13)	9833(12)	-98(10)	26(3)
H(8B)	7747(15)	8514(13)	2(11)	33(3)
H(10A)	7005(15)	6724(12)	1019(10)	30(3)
H(10B)	5589(14)	6104(13)	1537(11)	31(3)
H(12)	9011(15)	10287(14)	831(12)	41(4)
H(13)	11142(17)	10694(15)	1848(13)	49(4)
H(14)	11417(17)	9894(14)	3628(12)	43(4)
H(15)	9508(17)	8694(15)	4381(13)	47(4)
H(16)	7370(14)	8264(13)	3361(11)	31(3)

Table 6. Torsion angles [°] for kjs11.

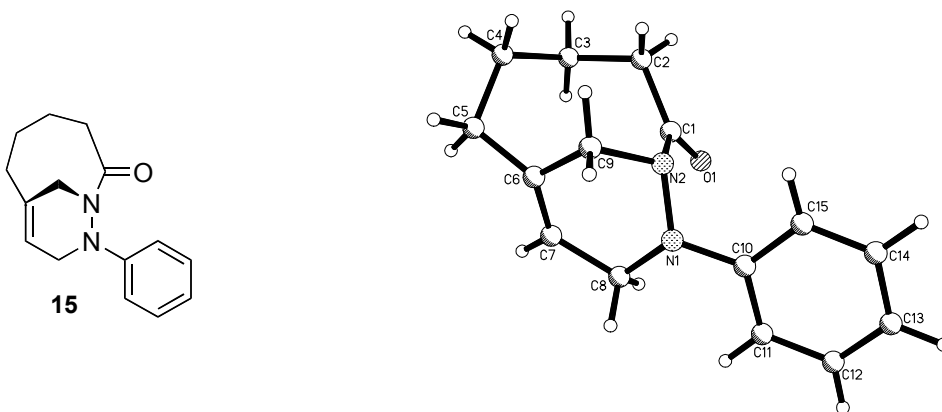
N(9)-N(1)-C(2)-O(1)	-26.60(13)
C(10)-N(1)-C(2)-O(1)	-154.89(9)
N(9)-N(1)-C(2)-C(3)	153.40(8)
C(10)-N(1)-C(2)-C(3)	25.11(12)
O(1)-C(2)-C(3)-C(4)	92.58(12)
N(1)-C(2)-C(3)-C(4)	-87.42(11)
C(2)-C(3)-C(4)-C(5)	69.28(12)
C(3)-C(4)-C(5)-C(6)	-43.72(13)
C(4)-C(5)-C(6)-C(7)	-85.75(13)
C(4)-C(5)-C(6)-C(10)	70.58(12)
C(5)-C(6)-C(7)-C(8)	159.07(10)
C(10)-C(6)-C(7)-C(8)	2.35(14)
C(6)-C(7)-C(8)-N(9)	-43.65(13)
C(2)-N(1)-N(9)-C(11)	125.89(9)
C(10)-N(1)-N(9)-C(11)	-103.47(9)
C(2)-N(1)-N(9)-C(8)	-98.18(9)
C(10)-N(1)-N(9)-C(8)	32.46(11)
C(7)-C(8)-N(9)-C(11)	158.03(9)
C(7)-C(8)-N(9)-N(1)	23.82(11)
C(2)-N(1)-C(10)-C(6)	57.32(10)
N(9)-N(1)-C(10)-C(6)	-72.69(10)
C(7)-C(6)-C(10)-N(1)	53.84(11)
C(5)-C(6)-C(10)-N(1)	-105.58(10)
N(1)-N(9)-C(11)-C(16)	-18.69(13)
C(8)-N(9)-C(11)-C(16)	-152.84(9)
N(1)-N(9)-C(11)-C(12)	165.24(9)
C(8)-N(9)-C(11)-C(12)	31.09(13)

C(16)-C(11)-C(12)-C(13)	0.56(16)
N(9)-C(11)-C(12)-C(13)	176.72(10)
C(11)-C(12)-C(13)-C(14)	0.28(18)
C(12)-C(13)-C(14)-C(15)	-0.73(19)
C(13)-C(14)-C(15)-C(16)	0.34(18)
C(14)-C(15)-C(16)-C(11)	0.51(17)
C(12)-C(11)-C(16)-C(15)	-0.95(16)
N(9)-C(11)-C(16)-C(15)	-177.09(10)

X-ray Data Collection, Structure Solution and Refinement for kjs15.

A colorless crystal of approximate dimensions 0.27 x 0.42 x 0.48 mm was mounted on a glass fiber and transferred to a Bruker CCD platform diffractometer. The SMART<sup>1</sup> program package was used to determine the unit-cell parameters and for data collection (20 sec/frame scan time for a sphere of diffraction data). The raw frame data was processed using SAINT<sup>2</sup> and SADABS<sup>3</sup> to yield the reflection data file. Subsequent calculations were carried out using the SHELXTL<sup>4</sup> program. The diffraction symmetry was  $2/m$  and the systematic absences were consistent with the centrosymmetric monoclinic space group  $P2_1/c$  that was later determined to be correct.

The structure was solved by direct methods and refined on  $F^2$  by full-matrix least-squares techniques. The analytical scattering factors<sup>5</sup> for neutral atoms were used throughout the analysis. Hydrogen atoms were located from a difference-Fourier map and refined ( $x, y, z$  and  $U_{iso}$ ). At convergence,  $wR2 = 0.0949$  and  $Goof = 1.035$  for 235 variables refined against 2775 data (0.78Å). As a comparison for refinement on  $F$ ,  $R1 = 0.0356$  for those 2427 data with  $I > 2.0(I)$ .





## References.

1. SMART Software Users Guide, Version 5.1, Bruker Analytical X-Ray Systems, Inc.; Madison, WI 1999.
2. SAINT Software Users Guide, Version 6.0, Bruker Analytical X-Ray Systems, Inc.; Madison, WI 1999.
3. Sheldrick, G. M. SADABS, Version 2.10, Bruker Analytical X-Ray Systems, Inc.; Madison, WI 2002.
4. Sheldrick, G. M. SHELXTL Version 6.12, Bruker Analytical X-Ray Systems, Inc.; Madison, WI 2001.
5. International Tables for X-Ray Crystallography 1992, Vol. C., Dordrecht: Kluwer Academic Publishers.

## Definitions:

$$wR2 = \left[ \frac{[\sum w(F_o^2 - F_c^2)^2]}{[\sum w(F_o^2)^2]} \right]^{1/2}$$

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

Goof = S =  $\left[ \frac{[\sum w(F_o^2 - F_c^2)^2]}{(n-p)} \right]^{1/2}$  where n is the number of reflections and p is the total number of parameters refined.

The thermal ellipsoid plot is shown at the 50% probability level.

Table 1. Crystal data and structure refinement for kjs15.

Identification code	kjs15 (Claudia Molina)	
Empirical formula	C <sub>15</sub> H <sub>18</sub> N <sub>2</sub> O	
Formula weight	242.31	
Temperature	163(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2 <sub>1</sub> /c	
Unit cell dimensions	a = 8.9659(8) Å	α = 90°.
	b = 13.1890(12) Å	β = 100.930(2)°.
	c = 10.8474(10) Å	γ = 90°.
Volume	1259.4(2) Å <sup>3</sup>	
Z	4	
Density (calculated)	1.278 Mg/m <sup>3</sup>	
Absorption coefficient	0.081 mm <sup>-1</sup>	
F(000)	520	
Crystal color	colorless	
Crystal size	0.48 x 0.42 x 0.27 mm <sup>3</sup>	
Theta range for data collection	2.31 to 27.10°	
Index ranges	-11 ≤ h ≤ 11, -16 ≤ k ≤ 16, -13 ≤ l ≤ 13	
Reflections collected	12777	
Independent reflections	2775 [R(int) = 0.0242]	
Completeness to theta = 27.10°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9784 and 0.9621	
Refinement method	Full-matrix least-squares on F <sup>2</sup>	
Data / restraints / parameters	2775 / 0 / 235	
Goodness-of-fit on F <sup>2</sup>	1.035	

Final R indices [ $I > 2\sigma(I)$  = 2427 data]

R1 = 0.0356, wR2 = 0.0891

R indices (all data; 0.78Å)

R1 = 0.0418, wR2 = 0.0949

Largest diff. peak and hole

0.295 and -0.161 e.Å<sup>-3</sup>

Table 2. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for kjs15.  $U(\text{eq})$  is defined as one third of the trace of the orthogonalized  $U^{ij}$  tensor.

---

	x	y	z	$U(\text{eq})$
O(1)	9663(1)	2310(1)	9652(1)	28(1)
N(1)	7063(1)	1507(1)	8464(1)	24(1)
N(2)	8153(1)	1881(1)	7786(1)	20(1)
C(1)	9551(1)	2144(1)	8532(1)	20(1)
C(2)	10895(1)	2254(1)	7875(1)	22(1)
C(3)	11898(1)	1291(1)	7898(1)	25(1)
C(4)	11472(1)	543(1)	6804(1)	26(1)
C(5)	10257(1)	-259(1)	6937(1)	27(1)
C(6)	8949(1)	205(1)	7418(1)	22(1)
C(7)	8577(1)	-48(1)	8510(1)	24(1)
C(8)	7485(1)	548(1)	9124(1)	25(1)
C(9)	8241(1)	1155(1)	6771(1)	21(1)
C(10)	6183(1)	2229(1)	8939(1)	24(1)
C(11)	5432(1)	1990(1)	9922(1)	29(1)
C(12)	4445(1)	2693(1)	10304(1)	34(1)
C(13)	4199(1)	3630(1)	9735(1)	34(1)
C(14)	4967(1)	3877(1)	8774(1)	31(1)
C(15)	5953(1)	3192(1)	8384(1)	26(1)

---

Table 3. Bond lengths [Å] and angles [°] for kjs15.

---

O(1)-C(1)	1.2198(13)
N(1)-C(10)	1.3965(14)
N(1)-N(2)	1.4185(12)
N(1)-C(8)	1.4669(15)
N(2)-C(1)	1.4013(14)
N(2)-C(9)	1.4725(14)
C(1)-C(2)	1.5191(15)
C(2)-C(3)	1.5534(16)
C(3)-C(4)	1.5348(17)
C(4)-C(5)	1.5441(17)
C(5)-C(6)	1.5010(16)
C(6)-C(7)	1.3327(16)
C(6)-C(9)	1.5156(15)
C(7)-C(8)	1.5062(16)
C(10)-C(11)	1.4010(16)
C(10)-C(15)	1.4038(17)
C(11)-C(12)	1.3973(18)
C(12)-C(13)	1.381(2)
C(13)-C(14)	1.3913(19)
C(14)-C(15)	1.3857(17)

C(10)-N(1)-N(2)	116.52(9)
C(10)-N(1)-C(8)	121.27(9)
N(2)-N(1)-C(8)	114.46(9)
C(1)-N(2)-N(1)	114.57(8)
C(1)-N(2)-C(9)	115.09(8)
N(1)-N(2)-C(9)	107.50(8)
O(1)-C(1)-N(2)	121.01(10)
O(1)-C(1)-C(2)	121.89(10)
N(2)-C(1)-C(2)	117.05(9)
C(1)-C(2)-C(3)	115.02(9)
C(4)-C(3)-C(2)	117.03(10)
C(3)-C(4)-C(5)	116.42(10)
C(6)-C(5)-C(4)	111.41(9)
C(7)-C(6)-C(5)	123.41(11)
C(7)-C(6)-C(9)	117.71(10)
C(5)-C(6)-C(9)	117.55(10)
C(6)-C(7)-C(8)	123.76(10)
N(1)-C(8)-C(7)	111.29(9)
N(2)-C(9)-C(6)	105.60(8)
N(1)-C(10)-C(11)	120.93(11)
N(1)-C(10)-C(15)	120.41(10)
C(11)-C(10)-C(15)	118.52(11)
C(12)-C(11)-C(10)	119.95(13)
C(13)-C(12)-C(11)	121.14(12)
C(12)-C(13)-C(14)	119.03(12)
C(15)-C(14)-C(13)	120.71(13)
C(14)-C(15)-C(10)	120.61(12)

---

Table 4. Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for kjs15. The anisotropic displacement factor exponent takes the form:  $-2\pi^2 [ h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12} ]$

	U <sup>11</sup>	U <sup>22</sup>	U <sup>33</sup>	U <sup>23</sup>	U <sup>13</sup>	U <sup>12</sup>
O(1)	34(1)	29(1)	19(1)	-4(1)	5(1)	-2(1)
N(1)	24(1)	26(1)	24(1)	4(1)	10(1)	1(1)
N(2)	21(1)	22(1)	18(1)	1(1)	7(1)	0(1)
C(1)	26(1)	15(1)	19(1)	0(1)	5(1)	1(1)
C(2)	23(1)	21(1)	21(1)	0(1)	3(1)	-4(1)
C(3)	21(1)	26(1)	27(1)	4(1)	4(1)	0(1)
C(4)	29(1)	25(1)	28(1)	1(1)	12(1)	4(1)
C(5)	33(1)	21(1)	26(1)	-3(1)	7(1)	1(1)
C(6)	24(1)	19(1)	21(1)	-3(1)	2(1)	-4(1)
C(7)	26(1)	20(1)	25(1)	2(1)	3(1)	-3(1)
C(8)	27(1)	27(1)	23(1)	5(1)	8(1)	-2(1)
C(9)	23(1)	24(1)	17(1)	0(1)	2(1)	-1(1)
C(10)	19(1)	32(1)	20(1)	-4(1)	3(1)	-1(1)
C(11)	25(1)	40(1)	23(1)	-2(1)	5(1)	-3(1)
C(12)	24(1)	56(1)	25(1)	-11(1)	9(1)	-4(1)
C(13)	22(1)	47(1)	33(1)	-15(1)	4(1)	3(1)
C(14)	25(1)	35(1)	32(1)	-8(1)	1(1)	4(1)
C(15)	23(1)	32(1)	24(1)	-3(1)	5(1)	1(1)

Table 5. Hydrogen coordinates ( $\times 10^4$ ) and isotropic displacement parameters ( $\text{\AA}^2 \times 10^{-3}$ ) for kjs15.

	x	y	z	U(eq)
H(2A)	10587(15)	2485(10)	7012(13)	24(3)
H(2B)	11510(17)	2765(11)	8323(13)	29(4)
H(3A)	12921(17)	1542(11)	7882(13)	29(3)
H(3B)	11954(15)	938(11)	8720(13)	27(3)
H(4A)	11165(16)	947(11)	6022(13)	29(3)
H(4B)	12401(17)	173(11)	6700(13)	32(4)
H(5A)	10708(16)	-792(11)	7538(13)	29(3)
H(5B)	9931(16)	-581(11)	6109(14)	32(4)
H(7A)	9060(16)	-609(11)	8998(13)	30(4)
H(8A)	7947(16)	661(11)	10041(13)	30(4)
H(8B)	6519(17)	164(11)	9100(13)	32(4)
H(9A)	8853(15)	1450(10)	6192(12)	21(3)
H(9B)	7189(16)	1052(10)	6304(13)	27(3)
H(11)	5572(16)	1331(12)	10307(14)	33(4)
H(12)	3932(18)	2510(12)	10976(15)	41(4)
H(13)	3502(19)	4119(12)	10020(15)	40(4)
H(14)	4788(18)	4570(13)	8376(15)	40(4)
H(15)	6480(16)	3338(11)	7717(14)	30(4)



Table 6. Torsion angles [°] for kjs15.

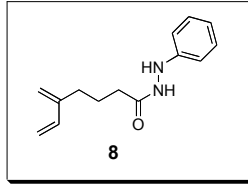
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C(10)-N(1)-N(2)-C(1)	85.64(11)
C(8)-N(1)-N(2)-C(1)	-64.17(12)
C(10)-N(1)-N(2)-C(9)	-145.08(9)
C(8)-N(1)-N(2)-C(9)	65.11(11)
N(1)-N(2)-C(1)-O(1)	-19.72(14)
C(9)-N(2)-C(1)-O(1)	-145.12(10)
N(1)-N(2)-C(1)-C(2)	162.68(9)
C(9)-N(2)-C(1)-C(2)	37.28(13)
O(1)-C(1)-C(2)-C(3)	87.69(13)
N(2)-C(1)-C(2)-C(3)	-94.74(12)
C(1)-C(2)-C(3)-C(4)	90.43(12)
C(2)-C(3)-C(4)-C(5)	-85.86(13)
C(3)-C(4)-C(5)-C(6)	44.34(14)
C(4)-C(5)-C(6)-C(7)	-117.05(12)
C(4)-C(5)-C(6)-C(9)	49.43(13)
C(5)-C(6)-C(7)-C(8)	167.17(10)
C(9)-C(6)-C(7)-C(8)	0.71(16)
C(10)-N(1)-C(8)-C(7)	-173.46(10)
N(2)-N(1)-C(8)-C(7)	-25.22(13)
C(6)-C(7)-C(8)-N(1)	-8.07(16)
C(1)-N(2)-C(9)-C(6)	60.66(11)
N(1)-N(2)-C(9)-C(6)	-68.32(10)
C(7)-C(6)-C(9)-N(2)	36.61(13)
C(5)-C(6)-C(9)-N(2)	-130.66(10)
N(2)-N(1)-C(10)-C(11)	-160.04(10)
C(8)-N(1)-C(10)-C(11)	-12.43(16)

N(2)-N(1)-C(10)-C(15)	24.20(15)
C(8)-N(1)-C(10)-C(15)	171.81(10)
N(1)-C(10)-C(11)-C(12)	-174.03(11)
C(15)-C(10)-C(11)-C(12)	1.80(17)
C(10)-C(11)-C(12)-C(13)	-0.38(18)
C(11)-C(12)-C(13)-C(14)	-0.85(19)
C(12)-C(13)-C(14)-C(15)	0.62(19)
C(13)-C(14)-C(15)-C(10)	0.85(18)
N(1)-C(10)-C(15)-C(14)	173.82(11)
C(11)-C(10)-C(15)-C(14)	-2.04(17)

---

1H spectrum



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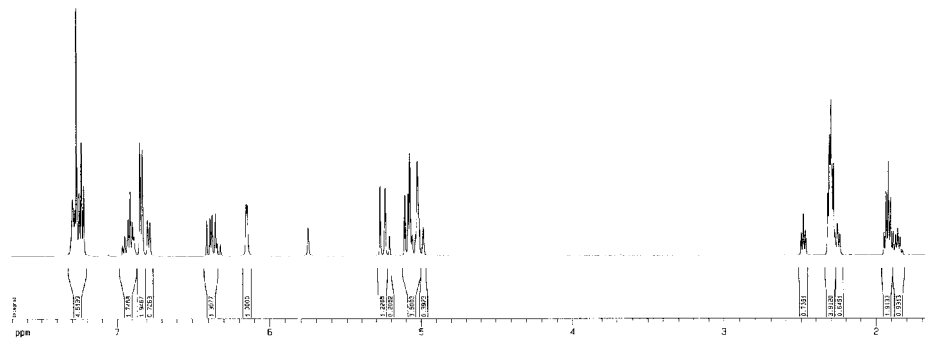
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 DS 4  
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 FIDRES 0.402088 Hz  
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 RG 3160.0  
 CW 16.500 uSFC  
 DE 4.50 uSFC  
 TE 295.0 K  
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 D11 0.03000000 sec

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 PL1 0.00 dB  
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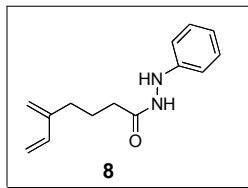
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 SF 125.7602478 MHz  
 WF 0  
 WD 0  
 LB 1.00 Hz  
 GB 0  
 PC 2.00

ID parameters  
 CX 20.80 cm  
 CY 15.85 cm  
 FIP 181.090 dB  
 P1 2414.48 Hz  
 F2P 6.351 dB  
 F2 796.863 Hz  
 FWHM 8.45312 dB/c  
 ZON 1022.48230 Hz/cm



13C spectrum with 1H decoupling



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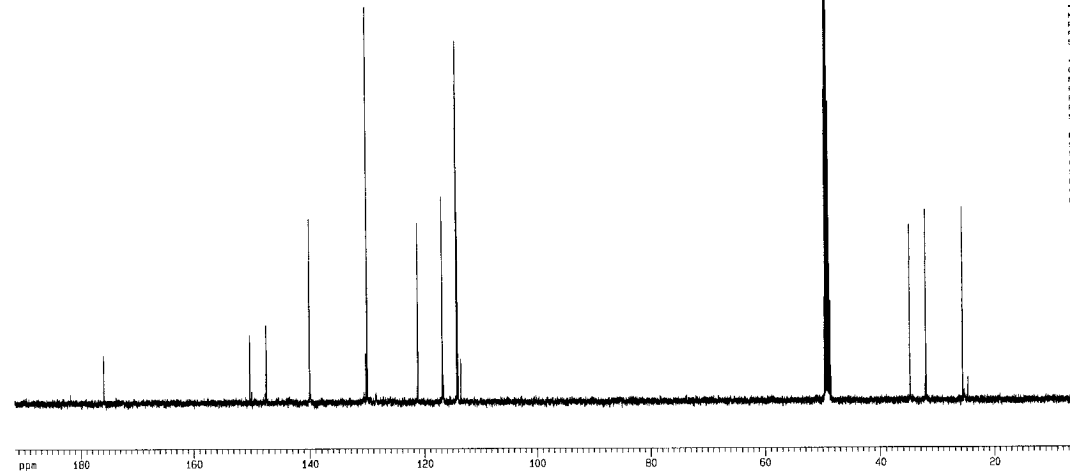
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 SOLVENT CDCl3  
 NS 4  
 DS 4  
 SWH 30303.031 Hz  
 FIDRES 0.402088 Hz  
 AQ 1.0019248 sec  
 RG 3160.0  
 CW 16.500 uSFC  
 DE 4.50 uSFC  
 TE 295.0 K  
 D1 0.25000000 sec  
 D11 0.03000000 sec

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 PL1 0.00 dB  
 SFO1 125.760248 MHz

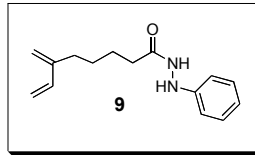
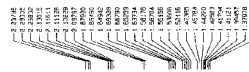
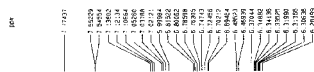
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 PL2 -1.00 dB  
 PL12 14.40 dB  
 SFO2 500.230013 MHz

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 SF 125.7602478 MHz  
 WF 0  
 WD 0  
 LB 1.00 Hz  
 GB 0  
 PC 2.00

ID parameters  
 CX 20.80 cm  
 CY 15.85 cm  
 FIP 181.090 dB  
 P1 2414.48 Hz  
 F2P 6.351 dB  
 F2 796.863 Hz  
 FWHM 8.45312 dB/c  
 ZON 1022.48230 Hz/cm



<sup>1</sup>H spectrum



```

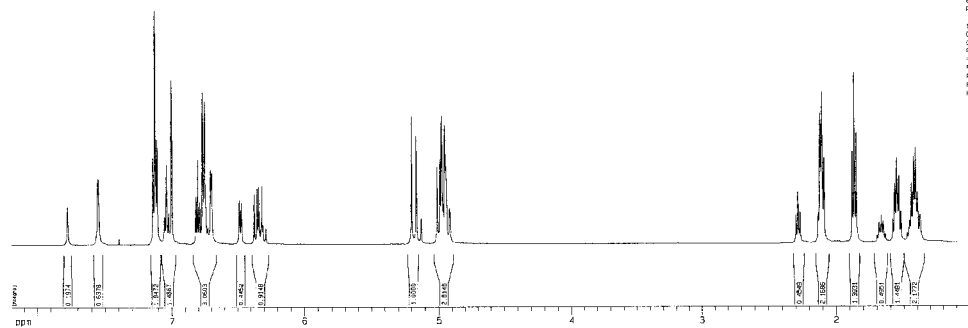
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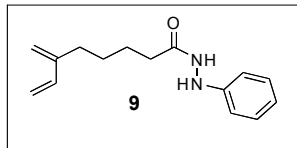
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SSB           0
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BR            0
PC            4.00

1D NMR plot parameters
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CY            2.00 cm
FIDP         6.137 umic
F1           406.13 Hz
F2           1.511 umic
F3           500.136 Hz
P1MACH       0.26287 umic/cm
P2MACH       157.58388 umic/cm
    
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with <sup>1</sup>H decoupling



```

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NAME          hydrate2pac
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PROCNO       1

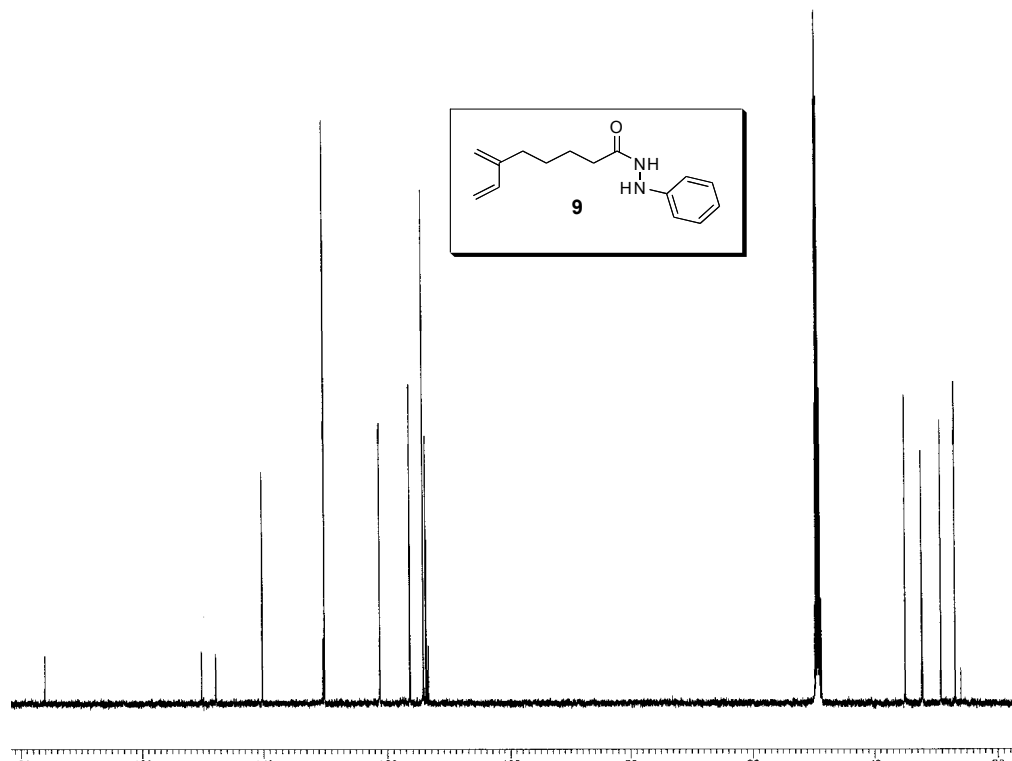
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SWH           8063.031 Hz
FIDRES       0.462688 Hz
AQ           1.0813040 sec
RG           13064
DM           16.500 umic
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D11          0.0300000 sec
MORPHEST     0.0000000 sec
MEMBER       6.0150000 sec

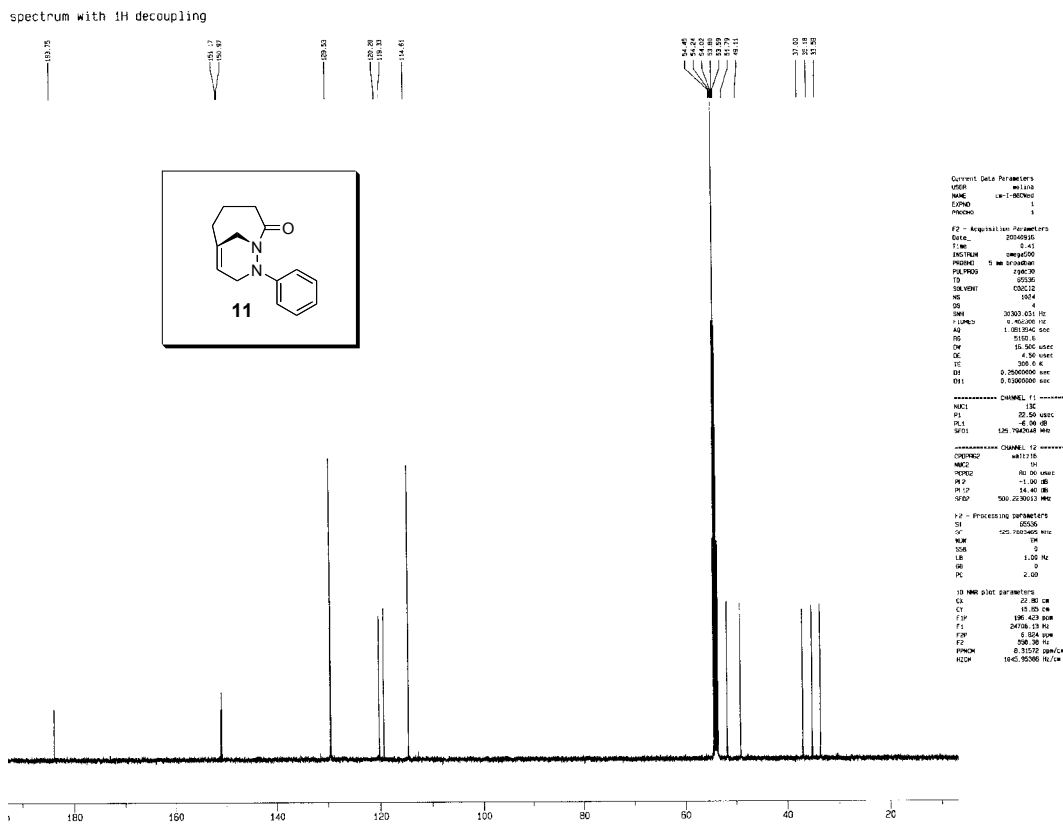
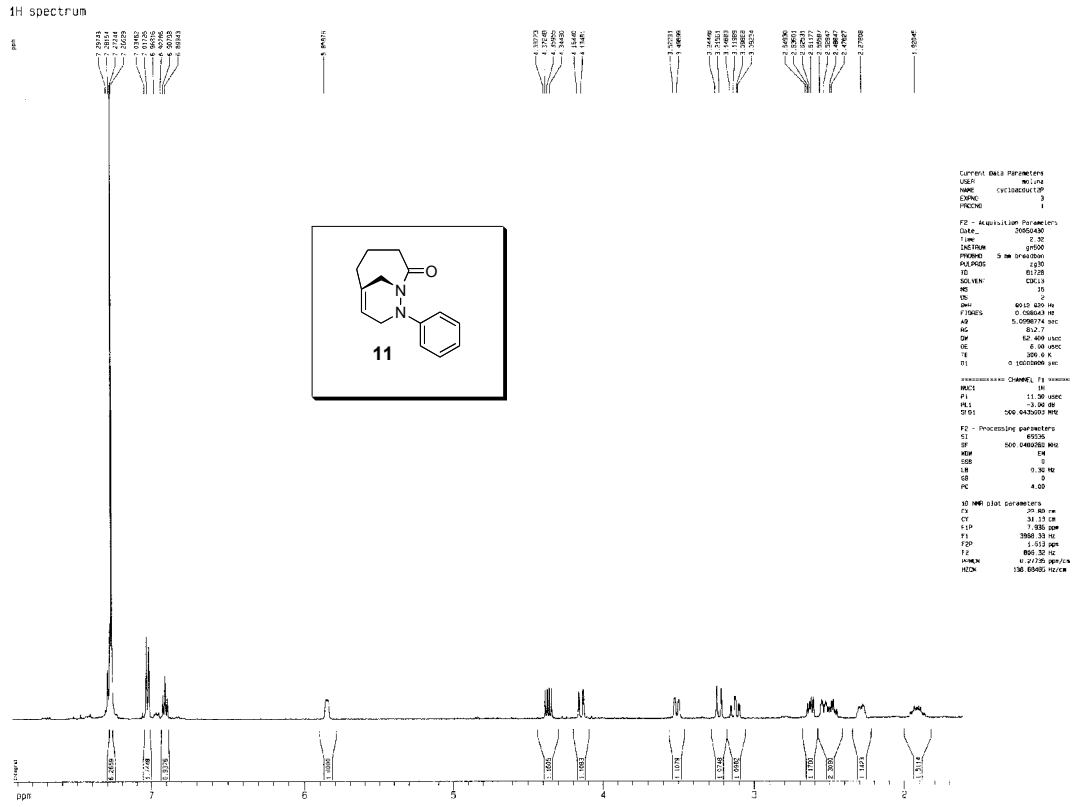
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SFO1         125.7617406 MHz

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PCPD2        80.00 umic
PL12         -1.00 dB
PL12         -1.00 dB
PL12         -1.00 dB
SFO2         500.1362611 MHz

F2 - Processing parameters
SI            800.0
SF           125.7777142 MHz
WDW           EM
SSB           0
GB            1.00 Hz
BR            0
PC            2.00

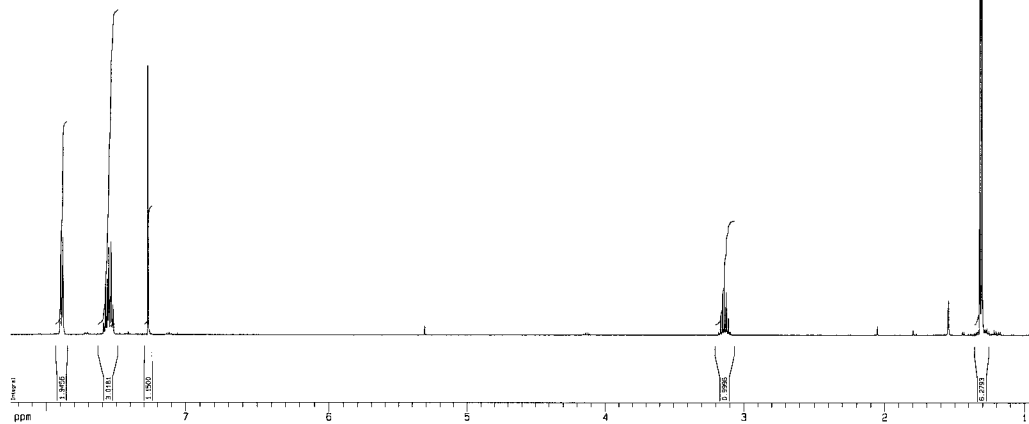
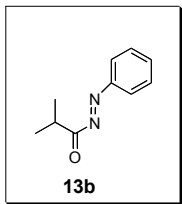
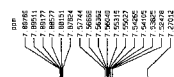
1D NMR plot parameters
CX            26.80 cm
CY            10.05 cm
FIDP         200.5848 umic
F1           25166.11 Hz
F2           7.291 umic
F3           515.50 Hz
P1MACH       8.45285 umic/cm
P2MACH       1663.55774 Hz/cm
    
```







<sup>1</sup>H spectrum



```

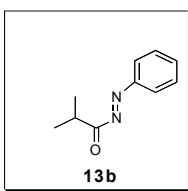
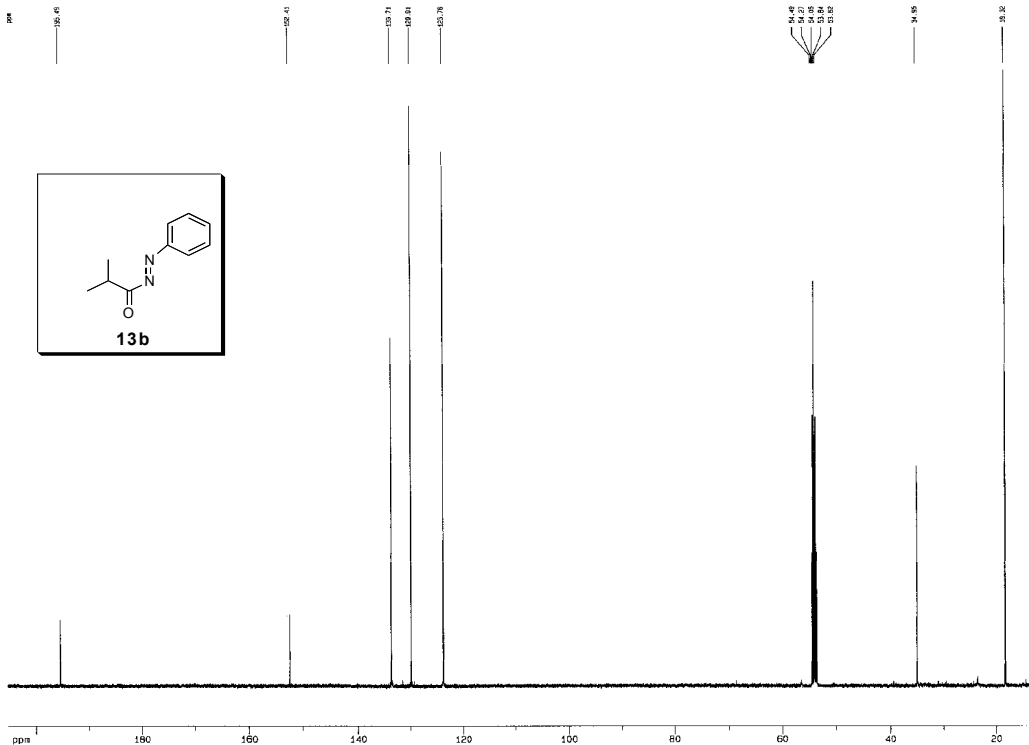
Current Data Parameters
=====
USER          mol11a
NAME          13b011020PM
EXPNO         1
PROCNO       1

F2 - Acquisition Parameters
=====
Date_         20050424
Time          21.47
INSTRUM      spect000
PROBHD       5 mm cryoProbe
PULPROG      zg30
TD           65536
SOLVENT      CDCl3
NS           2
DS           4
SWH          8012.800 Hz
FIDRES      0.398643 Hz
AQ          5.0098774 sec
RG          400.4
EQ          0.00 sec
EM          12.400 usec
TE          298.2 K
D1          0.1000000 sec
d11         0.0000000 sec
d12         0.0000000 sec
d13         0.0000000 sec
d14         0.0000000 sec
d15         0.0000000 sec
d16         0.0000000 sec
d17         0.0000000 sec
d18         0.0000000 sec
d19         0.0000000 sec
d20         0.0000000 sec
===== CHANNEL f1 =====
NUC1         1H
P1           9.25 usec
PL1         -1.00 dB
SFO1        500.261515 MHz

F2 - Processing parameters
=====
SI           32768
SF          500.261515 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           4.00

1D NMR plot parameters
=====
CA           32.80 cm
CY           15.00 cm
FIDRES      0.398643 Hz
F1          4100.85 Hz
F2          0.310 usec
F3          400.40 Hz
PRNHZ      0.3027 usec/cm
PCHW       151.0034 Hz/cm
    
```

<sup>13</sup>C spectrum with <sup>1</sup>H decoupling



```

Current Data Parameters
=====
USER          mol11a
NAME          13b011020PM
EXPNO         2
PROCNO       1

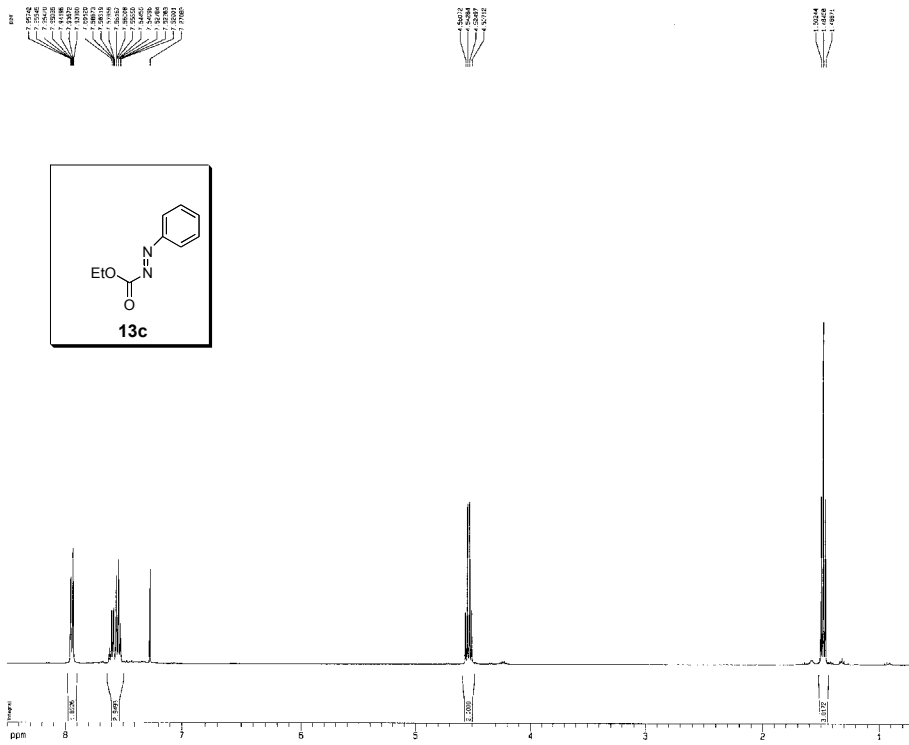
F2 - Acquisition Parameters
=====
Date_         20050424
Time          16.31
INSTRUM      spect000
PROBHD       5 mm cryoProbe
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           2
DS           4
SWH          30362.031 Hz
FIDRES      0.462220 Hz
AQ          0.756488 sec
RG          11288.2
EQ          0.00 sec
EM          16.500 usec
TE          298.2 K
D1          0.2000000 sec
d11         0.0000000 sec
d12         0.0000000 sec
d13         0.0000000 sec
d14         0.0000000 sec
d15         0.0000000 sec
d16         0.0000000 sec
d17         0.0000000 sec
d18         0.0000000 sec
d19         0.0000000 sec
d20         0.0000000 sec
===== CHANNEL f1 =====
NUC1         13C
P1          13.00 usec
PL1         -1.00 dB
SFO1        125.764558 MHz

===== CHANNEL f2 =====
NAME        waltz16
PULPROG     waltz16
TD           65536
SFO2        100.626055 MHz
P12         11.00 usec
PL12        23.54 dB
SFO3        500.261515 MHz

F2 - Processing parameters
=====
SI           32768
SF          125.764558 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           2.00

1D NMR plot parameters
=====
CA           32.80 cm
CY           15.00 cm
FIDRES      0.462220 Hz
F1          5002.000 MHz
F2          100.626055 MHz
F3          500.261515 MHz
PRNHZ      0.37375 usec/cm
PCHW       1101.4650 Hz/cm
    
```

<sup>1</sup>H spectrum



```

Current Data Parameters
NAME      molina
EXPNO    1
PROCNO   1
PULPROG  zgpg30
SOLVENT  CDCl3
SI        65536
SF        500.136091 MHz
AQ        0.462388 Hz
RG        655.36
WDW        EM
SSB        0
LB        0.30 Hz
GB        0
PC        2.00

===== CHANNEL f1 =====
NUC1      13C
P1        12.50 usec
PL1       0.00 dB
SFO1     125.760313 MHz

P2 - Acquisition Parameters
Date_    20041210
Time     22.00
INSTRUM  omega900
PROBHD   5 mm QNP 1H
PULPROG  zgpg30
SOLVENT  CDCl3
SI        65536
SF        500.136091 MHz
AQ        0.462388 Hz
RG        655.36
WDW        EM
SSB        0
LB        0.30 Hz
GB        0
PC        2.00

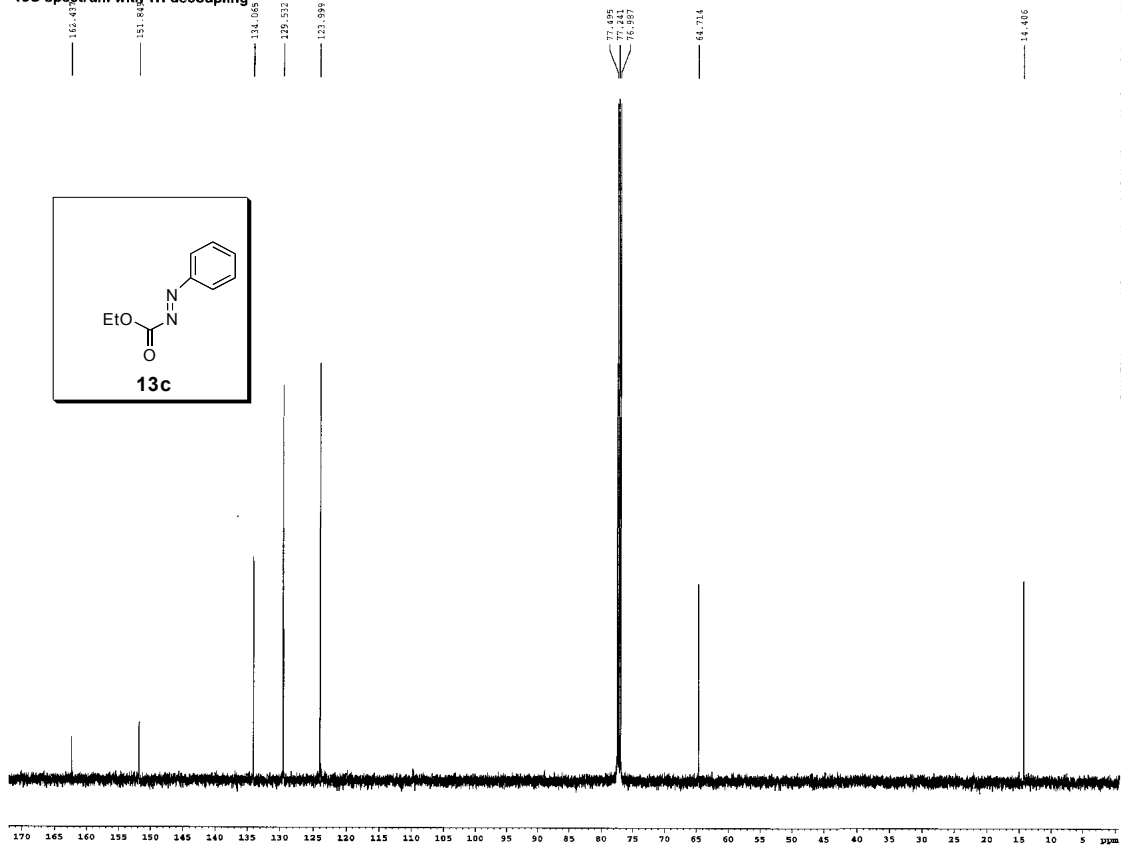
P2 - Processing parameters
SI        65536
SF        125.760313 MHz
WDW        EM
SSB        0
LB        0.30 Hz
GB        0
PC        2.00

===== CHANNEL f2 =====
NUC2      1H
P1        12.50 usec
PL1       0.00 dB
SFO1     500.136091 MHz

P2 - Acquisition Parameters
Date_    20041210
Time     22.00
INSTRUM  omega900
PROBHD   5 mm QNP 1H
PULPROG  zgpg30
SOLVENT  CDCl3
SI        65536
SF        500.136091 MHz
AQ        0.462388 Hz
RG        655.36
WDW        EM
SSB        0
LB        0.30 Hz
GB        0
PC        2.00

P2 - Processing parameters
SI        65536
SF        125.760313 MHz
WDW        EM
SSB        0
LB        0.30 Hz
GB        0
PC        2.00
    
```

<sup>13</sup>C spectrum with <sup>1</sup>H decoupling



```

Current Data Parameters
NAME      molina
EXPNO    1
PROCNO   1
PULPROG  zgpg30
SOLVENT  CDCl3
SI        65536
SF        500.136091 MHz
AQ        0.462388 Hz
RG        655.36
WDW        EM
SSB        0
LB        0.30 Hz
GB        0
PC        2.00

===== CHANNEL f1 =====
NUC1      13C
P1        12.50 usec
PL1       0.00 dB
SFO1     125.760313 MHz

P2 - Acquisition Parameters
Date_    20041210
Time     22.00
INSTRUM  omega900
PROBHD   5 mm QNP 1H
PULPROG  zgpg30
SOLVENT  CDCl3
SI        65536
SF        500.136091 MHz
AQ        0.462388 Hz
RG        655.36
WDW        EM
SSB        0
LB        0.30 Hz
GB        0
PC        2.00

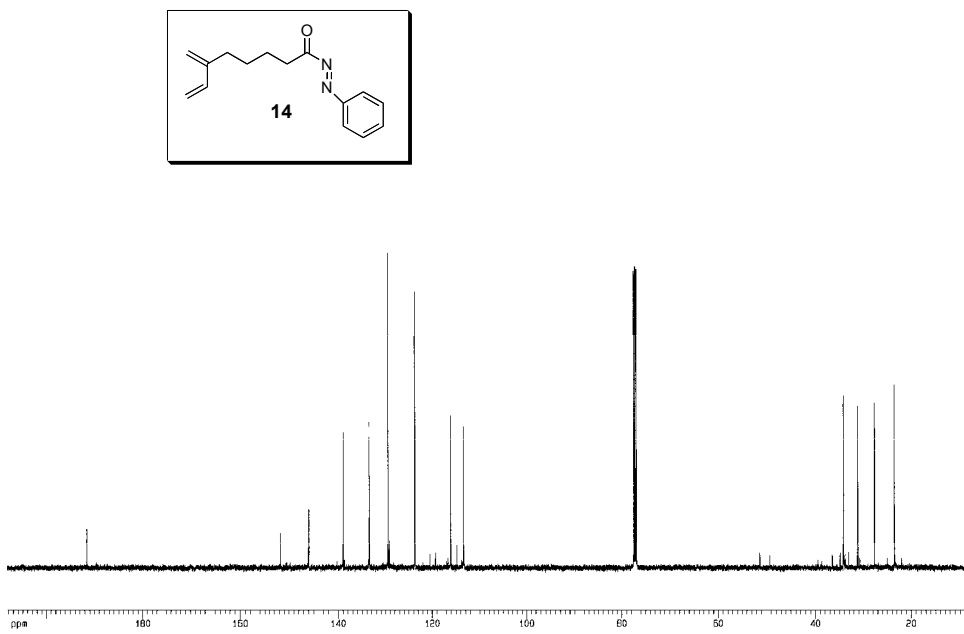
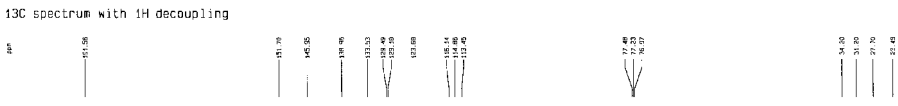
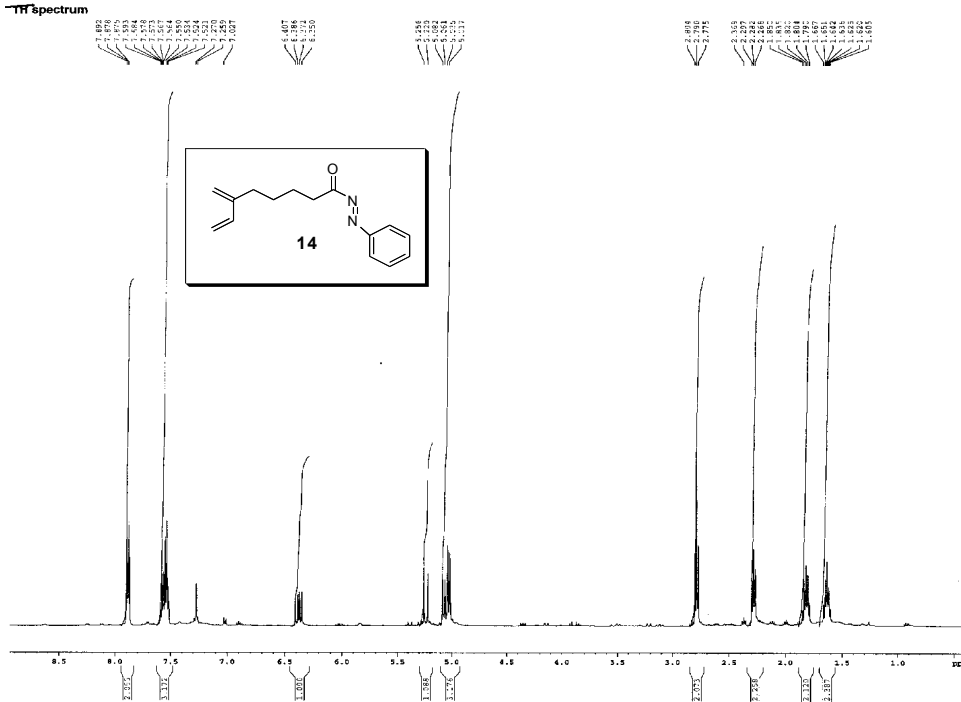
P2 - Processing parameters
SI        65536
SF        125.760313 MHz
WDW        EM
SSB        0
LB        0.30 Hz
GB        0
PC        2.00

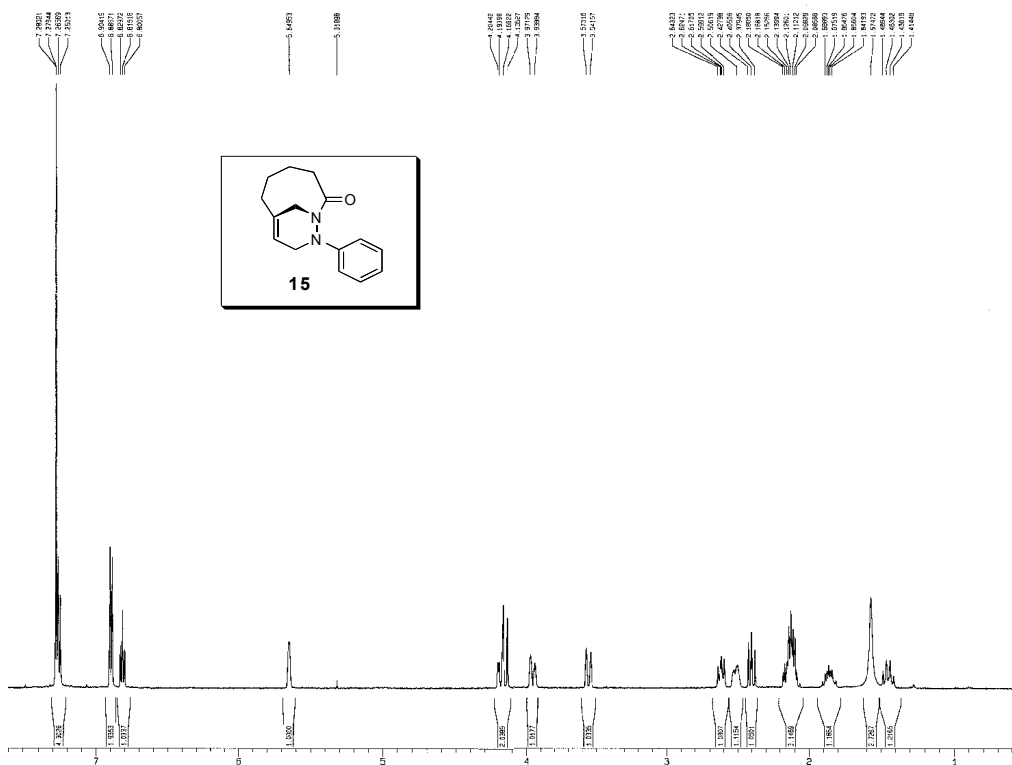
===== CHANNEL f2 =====
NUC2      1H
P1        12.50 usec
PL1       0.00 dB
SFO1     500.136091 MHz

P2 - Acquisition Parameters
Date_    20041210
Time     22.00
INSTRUM  omega900
PROBHD   5 mm QNP 1H
PULPROG  zgpg30
SOLVENT  CDCl3
SI        65536
SF        500.136091 MHz
AQ        0.462388 Hz
RG        655.36
WDW        EM
SSB        0
LB        0.30 Hz
GB        0
PC        2.00

P2 - Processing parameters
SI        65536
SF        125.760313 MHz
WDW        EM
SSB        0
LB        0.30 Hz
GB        0
PC        2.00
    
```







```

Current Data Parameters
USER           molina
NAME           CML1M1195.D1
EXPNO          1
PROCNO         1

F2 - Acquisition Parameters
Date_          20060115
Time           14.41
INSTRUM       spect
PROBHD        5 mm vnmrzbh-1
PULPROG       zgpg30
TD             65536
SOLVENT       CDCl3
AQ             0.291365
RG             655
WDW            EM
SSB            0
GB             0
PC            1.00
SFO            500.135200 MHz
FIDRES        0.000443 Hz
AQRES        0.000311 Hz
GBRES        0.00122
EN           652.000 usec
EC           16.00 usec
TE           298.0 K
D1           0.1000000 sec
d11          0.0000000 sec
MORPH        0.0000000 sec
RGRES        0.0000000 sec

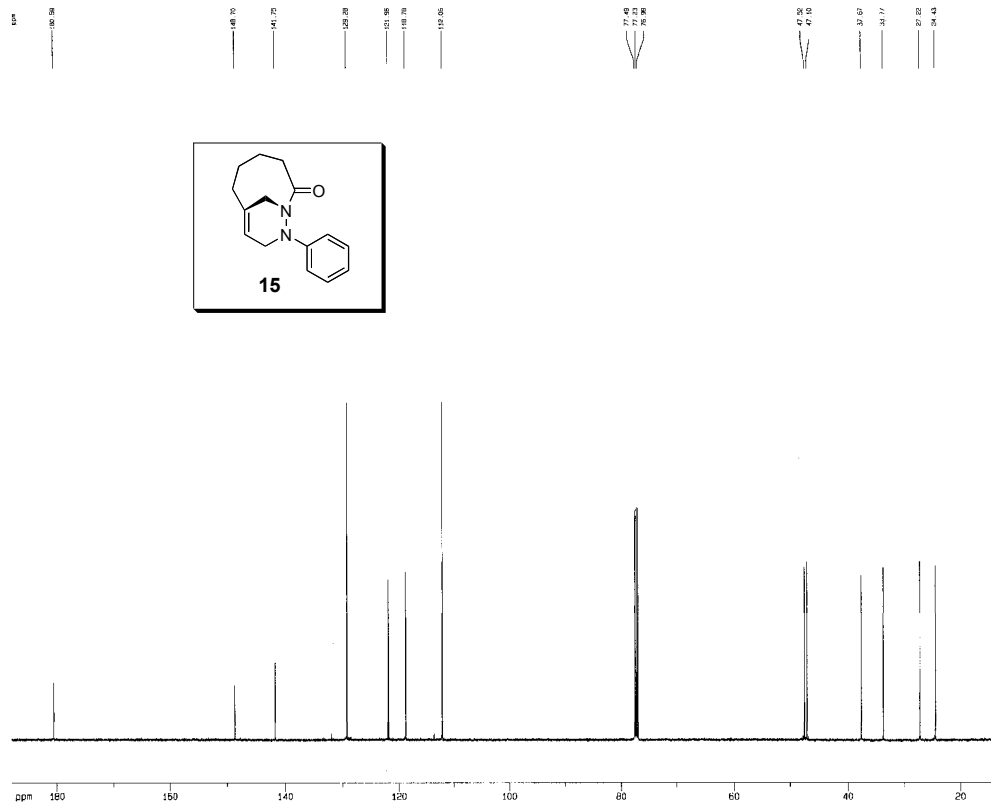
***** CHANNEL f1 *****
NUC1          13
P1           15.00 usec
PL1          -2.00 dB
SFO1         499.9489890 MHz

F2 - Processing parameters
SI           32768
SF           499.9489890 MHz
WDW          EM
SSB          0
GB           0.00 usec
PC           1.00
SFO          500.135200 MHz

ID NMR plot parameters
CX           62.00 cm
CY           7.00 cm
FID         0.055330 cm
F2          0.055330 cm
F3          0.055330 cm
F4          0.055330 cm
F5          0.055330 cm
F6          0.055330 cm
F7          0.055330 cm
F8          0.055330 cm
F9          0.055330 cm
F10         0.055330 cm
F11         0.055330 cm
F12         0.055330 cm
F13         0.055330 cm
F14         0.055330 cm
F15         0.055330 cm

```

13C spectrum with 1H decoupling



```

Current Data Parameters
USER           molina
NAME           CML1M1195.D1
EXPNO          1
PROCNO         1

F2 - Acquisition Parameters
Date_          20060115
Time           15.33
INSTRUM       spect
PROBHD        5 mm vnmrzbh-1
PULPROG       zgpg30
TD             65536
SOLVENT       CDCl3
AQ             0.291365
RG             655
WDW            EM
SSB            0
GB             0
PC            1.00
SFO            500.135200 MHz
FIDRES        0.000443 Hz
AQRES        0.000311 Hz
GBRES        0.00122
EN           652.000 usec
EC           16.00 usec
TE           298.0 K
D1           0.2000000 sec
d11          0.0000000 sec
MORPH        0.0000000 sec
RGRES        0.0000000 sec

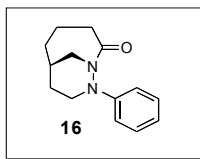
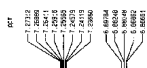
***** CHANNEL f1 *****
NUC1          13
P1           15.00 usec
PL1          -1.00 dB
SFO1         125.7645444 MHz

***** CHANNEL f2 *****
NAME         wa1115
NUC2         13
P2           10.00 usec
PL2          23.54 dB
SFO2         509.8225911 MHz

F2 - Processing parameters
SI           32768
SF           509.8225911 MHz
WDW          EM
SSB          0
GB           0.00 usec
PC           1.00
SFO          509.8225911 MHz

ID NMR plot parameters
CX           62.00 cm
CY           7.00 cm
FID         0.055330 cm
F2          0.055330 cm
F3          0.055330 cm
F4          0.055330 cm
F5          0.055330 cm
F6          0.055330 cm
F7          0.055330 cm
F8          0.055330 cm
F9          0.055330 cm
F10         0.055330 cm
F11         0.055330 cm
F12         0.055330 cm
F13         0.055330 cm
F14         0.055330 cm
F15         0.055330 cm

```

**<sup>1</sup>H spectrum**


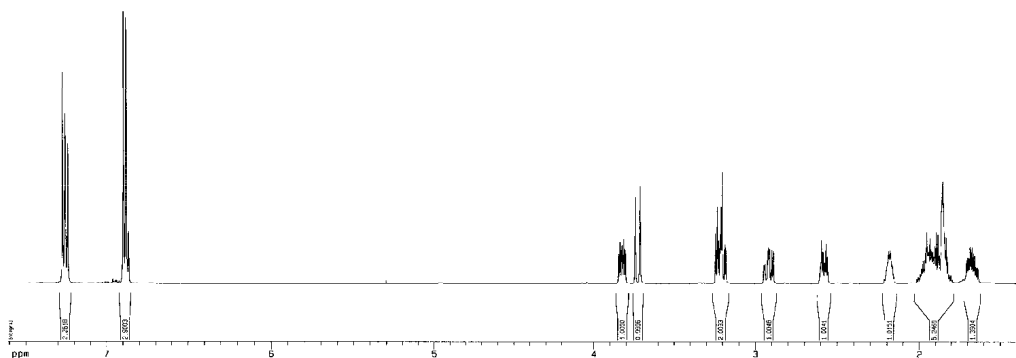
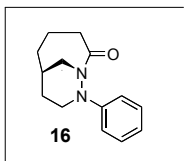
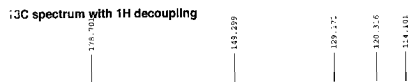
```

Current Data Parameters
USER          molina
NAME         CHD2m11131008
EXPNO       1
PROCNO      1

F2 - Acquisition Parameters
Date_       20060718
Time        14.30
INSTRUM     spect
PROBHD      5 mm 31pWaltz
PULPROG     zgpg30
PC          600
ID          81578
DELTA       0.020
RG          512
AQ          0.0313
AS          0
DS          2
DPR         642.000 Hz
SFORES      0.000000 Hz
AQ          5.9999776 sec
RG          99.0
DM          64.000 usec
DE          6.50 usec
TE          298.0 K
EI          0.0000000 sec
RESOLV     0.0000000 sec
NAME        ***** CHANNEL f1 *****
NUC1        13
P1          12.00 usec
PL1        0.00 dB
SFO1       125.7622448 MHz

F2 - Processing parameters
SI          65536
SF          500.2250247 MHz
WDW         EM
SSB         0
LB          0
GB          0
PC          4.00

3D sub-pilz parameters
CK          22.00 usec
CY          7.00 usec
PAF         7.000 usec
FB          3800.00 Hz
FOP         1.270 usec
PD          675.70 usec
RHO         0.27654 usec/turn
RPN         125.1307812 MHz
  
```


**<sup>13</sup>C spectrum with 1H decoupling**


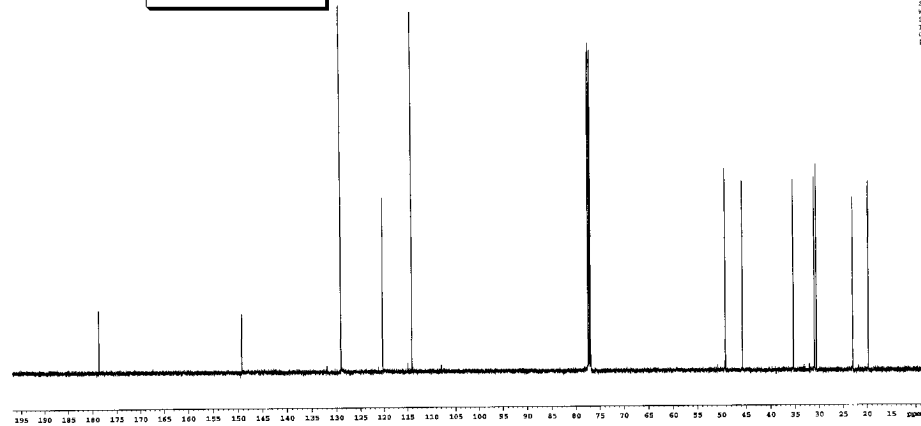
```

Current Data Parameters
USER          molina
NAME         CHD2m11131008
EXPNO       1
PROCNO      1

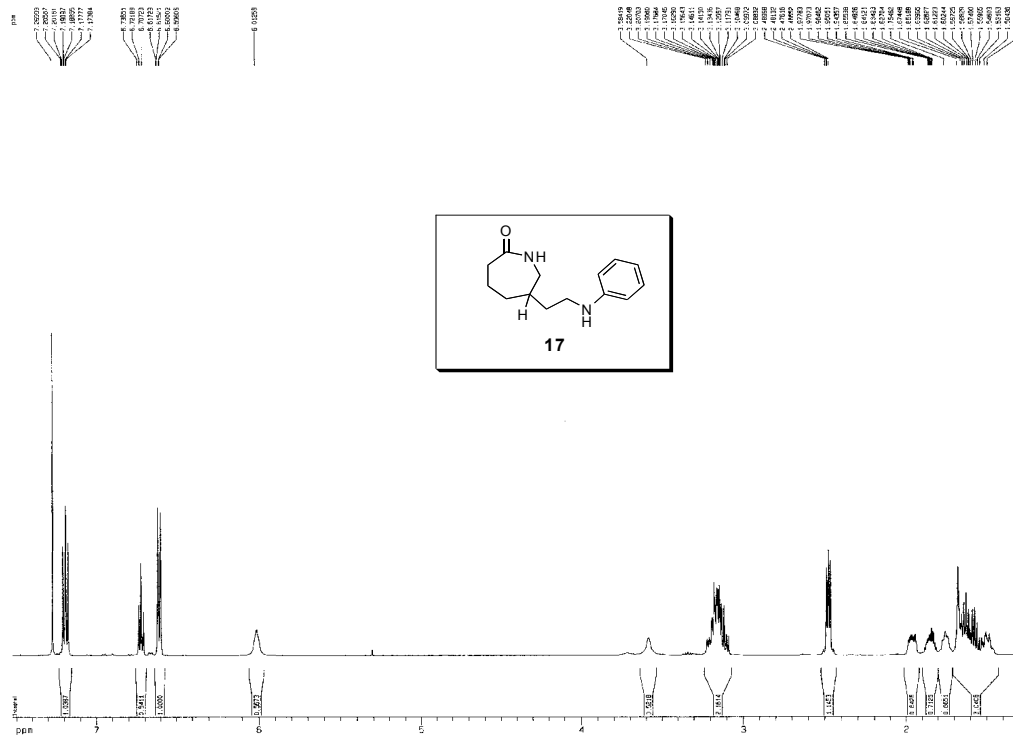
F2 - Acquisition Parameters
Date_       20060718
Time        14.45
INSTRUM     spect
PROBHD      5 mm 31pWaltz
PULPROG     zgpg30
PC          600
ID          81578
DELTA       0.020
RG          512
AQ          0.0313
AS          0
DS          2
DPR         642.000 Hz
SFORES      0.000000 Hz
AQ          5.9999776 sec
RG          99.0
DM          64.000 usec
DE          6.50 usec
TE          298.0 K
EI          0.0000000 sec
RESOLV     0.0000000 sec
NAME        ***** CHANNEL f1 *****
NUC1        13
P1          12.00 usec
PL1        0.00 dB
SFO1       125.7622448 MHz

***** CHANNEL f2 *****
NAME2       13
NUC2        13
P2          1.00 usec
PL2        0.00 dB
SFO2       101.6261815 MHz

F2 - Processing parameters
SI          65536
SF          125.7622448 MHz
WDW         EM
SSB         0
LB          0
GB          0
PC          4.00
  
```



1H spectrum



```

Current Data Parameters
USER          molina
NAME         LLMAN134113
EXPNO        2
PROCNO       1

F2 - Acquisition Parameters
Date_        20070212
Time         17.38
INSTRUM      spect
PROBHD       5 mm CPIC1 1H-
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           4
DS           4
SWH          30618.024 Hz
FIDRES      0.462666 Hz
AQ          1.0766025 sec
RG          1156.0
WDW          EM
SSB          0.000000 sec
LB           3.00 Hz
GB           0.000000 sec
TE           298.2 K
DZ           0.0000000 sec
SFO1         0.0000000 sec
SFO2         0.0000000 sec
SFO3         0.0150000 sec

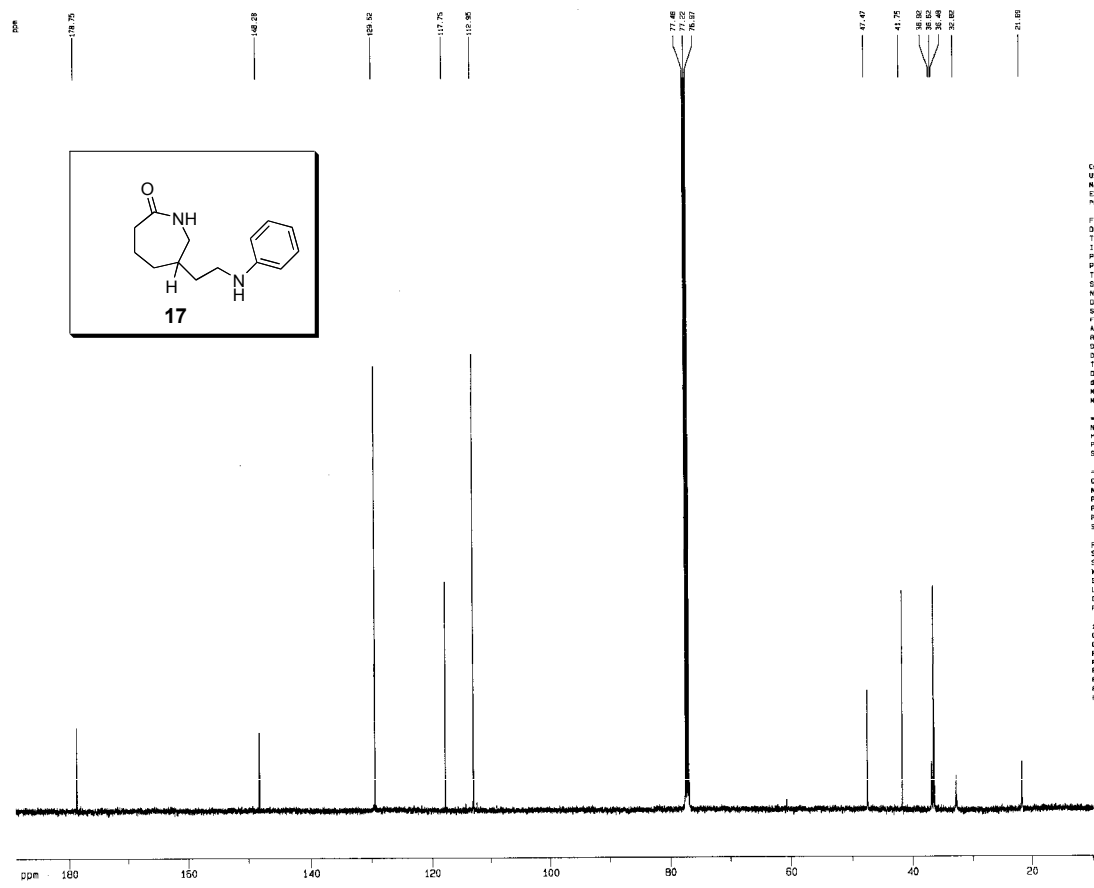
***** CHANNEL f1 *****
NUC1          1H
P1            12.00 usec
PL1          -1.00 dB
SFO1         500.1364515 MHz

F2 - Processing parameters
SI            65536
SF           500.1364515 MHz
WDW          EM
SSB          0.000000 sec
LB           3.00 Hz
GB           0.000000 sec
TE           298.2 K
DZ           0.0000000 sec
SFO1         0.0000000 sec
SFO2         0.0000000 sec
SFO3         0.0150000 sec

IS MRB DQ1 parameters
SI            65536
SF           500.1364515 MHz
WDW          EM
SSB          0.000000 sec
LB           3.00 Hz
GB           0.000000 sec
TE           298.2 K
DZ           0.0000000 sec
SFO1         0.0000000 sec
SFO2         0.0000000 sec
SFO3         0.0150000 sec

```

13C spectrum with 1H decoupling



```

Current Data Parameters
USER          molina
NAME         LLMAN134113
EXPNO        2
PROCNO       1

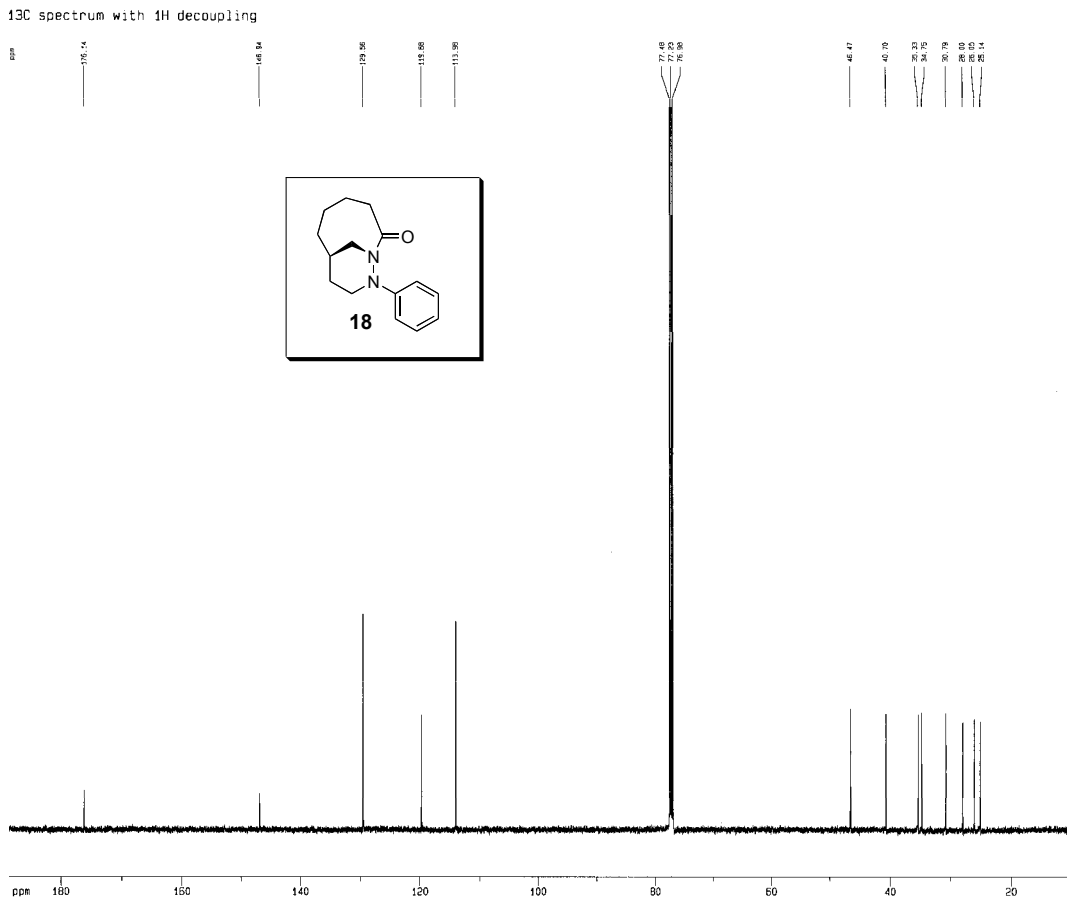
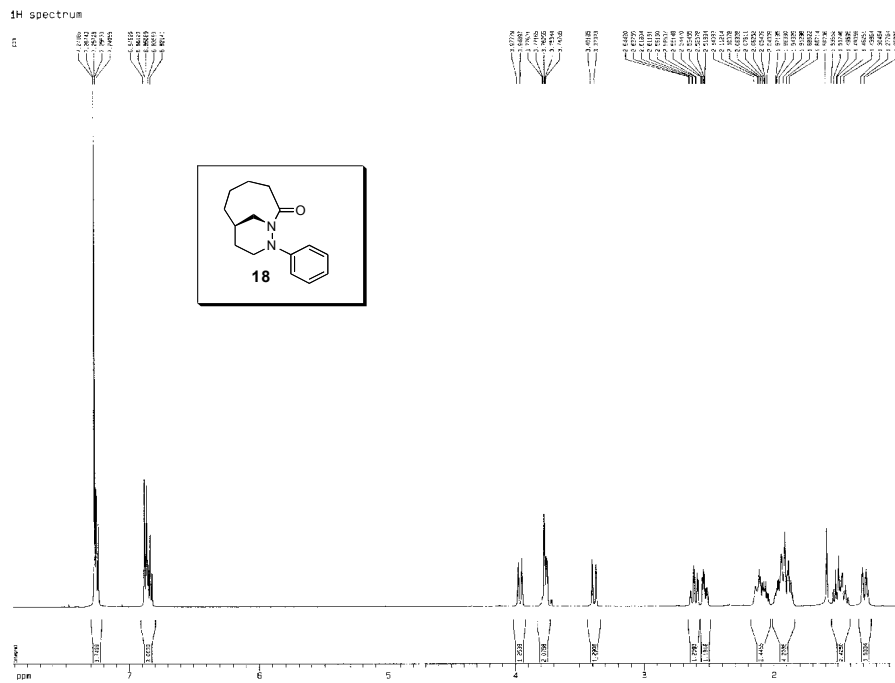
F2 - Acquisition Parameters
Date_        20070212
Time         18.24
INSTRUM      spect
PROBHD       5 mm CPIC1 1H-
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           4
DS           4
SWH          30618.024 Hz
FIDRES      0.462666 Hz
AQ          1.0766025 sec
RG          1156.0
WDW          EM
SSB          0.000000 sec
LB           3.00 Hz
GB           0.000000 sec
TE           298.2 K
DZ           0.0000000 sec
SFO1         0.0000000 sec
SFO2         0.0000000 sec
SFO3         0.0150000 sec

***** CHANNEL f1 *****
NUC1          13C
P1            12.00 usec
PL1          -1.00 dB
SFO1         125.7604515 MHz

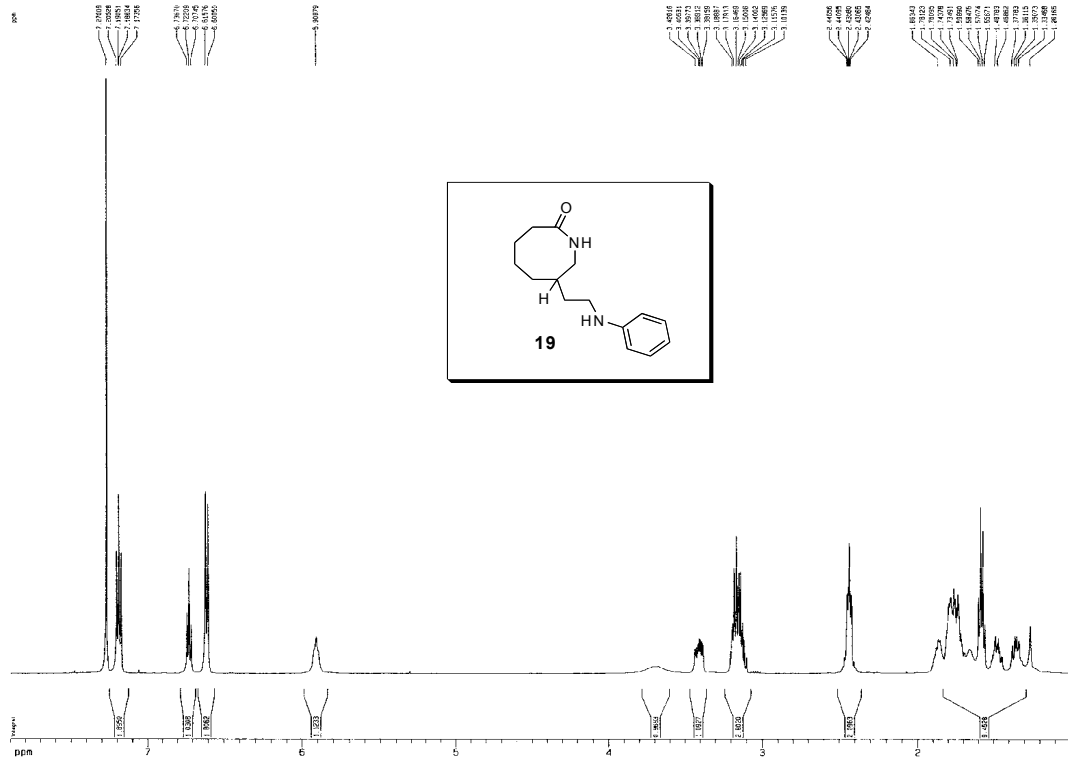
F2 - Processing parameters
SI            65536
SF           125.7604515 MHz
WDW          EM
SSB          0.000000 sec
LB           1.00 Hz
GB           0.000000 sec
TE           298.2 K
DZ           0.0000000 sec
SFO1         0.0000000 sec
SFO2         0.0000000 sec
SFO3         0.0150000 sec

IS MRB DQ1 parameters
SI            65536
SF           125.7604515 MHz
WDW          EM
SSB          0.000000 sec
LB           1.00 Hz
GB           0.000000 sec
TE           298.2 K
DZ           0.0000000 sec
SFO1         0.0000000 sec
SFO2         0.0000000 sec
SFO3         0.0150000 sec

```



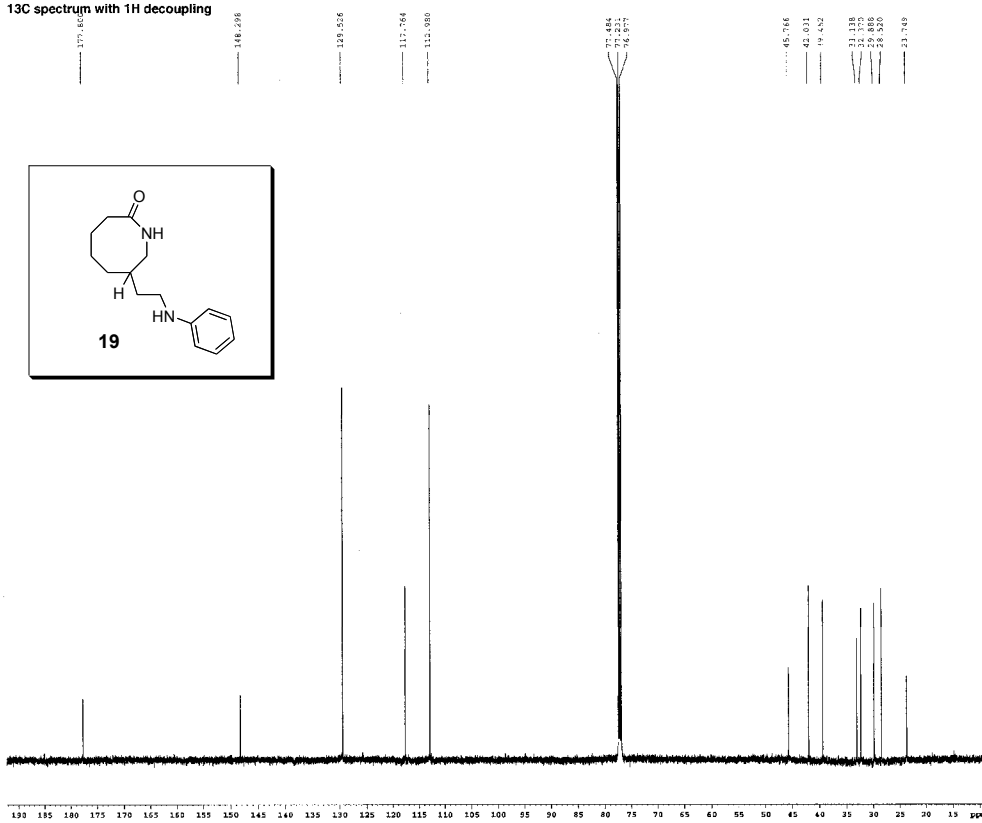
1H spectrum



```

Current Data Parameters
=====
NAME      Molina
EXPNO    13
PROCNO   1
F2 - Acquisition Parameters
Date_    20051212
Time     14.30
INSTRUM  spect
PROBHD   5 mm CPDPC 1H
PULPROG  zgpg30
NUC1      13C
SOLVENT  CDCl3
NS        1024
DS        4
SWH       30763.031 H
FIDRES    0.463222 H
AQ        1.0794479 s
RG         8192
DSB       16.500 u
DE        288.0 K
TE        300.2 K
D1        0.25000000 s
d11       0.00000000 s
MCREST    0.00000000 s
MORPH    0.01660000 s
===== CHANNEL f1 =====
NUC1      13C
P1        15.00 u
PL1       -1.00 d
SFO1     125.7643516 MHz
===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2      13C
PCPD2    100.00 v
PL2       21.60 d
PL12     21.24 d
SFO2     500.2250111 MHz
F2 - Processing parameters
SI        65236
SF        125.764006 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        2.00
ID non dist parameters
CA        20.00 Hz
CY        15.00 um
C1        7.000 um
F1        3951.74 Hz
CF1       0.003 ppm
F2        476.89 Hz
PC1MW    0.00000000 W
PC2MW    100.00000000 W
    
```

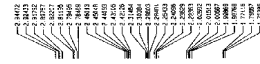
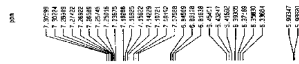
13C spectrum with 1H decoupling



```

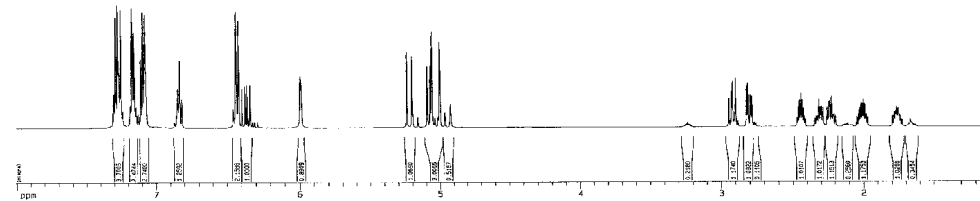
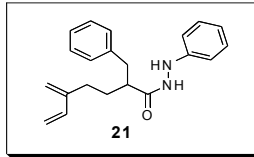
Current Data Parameters
=====
NAME      Molina
EXPNO    13
PROCNO   1
F2 - Acquisition Parameters
Date_    20051212
Time     14.50
INSTRUM  spect
PROBHD   5 mm CPDPC 1H
PULPROG  zgpg30
NUC1      13C
SOLVENT  CDCl3
NS        1024
DS        4
SWH       30763.031 H
FIDRES    0.463222 H
AQ        1.0794479 s
RG         8192
DSB       16.500 u
DE        288.0 K
TE        300.2 K
D1        0.25000000 s
d11       0.00000000 s
MCREST    0.00000000 s
MORPH    0.01660000 s
===== CHANNEL f1 =====
NUC1      13C
P1        15.00 u
PL1       -1.00 d
SFO1     125.7643516 MHz
===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2      13C
PCPD2    100.00 v
PL2       21.60 d
PL12     21.24 d
SFO2     500.2250111 MHz
F2 - Processing parameter
SI        65236
SF        125.764006 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        2.00
    
```

1H spectrum

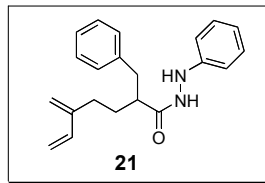
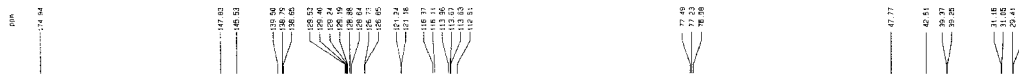


```

Current Data Parameters
NAME 1
NAME CLM4111111111111111
PROCNO 1
F2 - Acquisition Parameters
Date_ 06/13/2012
Time 15:14
INSTRUM cryo500
PROBHD 5 mm QNP1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2
DS 4
SWH 3013.231 Hz
FIDRES 0.463202 Hz
AQ 1.0764610 sec
RG 15004
AQ 16.500 usec
DE 2.00 usec
TE 298.2 K
C1 0.2000000 sec
d11 0.0300000 sec
d12 0.0500000 sec
d13 0.0350000 sec
===== CHANNEL f1 =====
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.764546 MHz
===== CHANNEL f2 =====
CPROG2 waltz16
NUC2 1H
P2 190.00 usec
PL2 1.00 dB
PL12 23.54 dB
SFO2 500.1360911 MHz
F3 - Processing parameters
SI 32768
SF 125.764546 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
ID 000 plot parameters
CP 20.00 cm
CY 9.11 cm
F3 180.000 ppm
F4 22546.47 Hz
F5 19.644 ppm
F6 2476.54 Hz
GAIN 1.00000000
HDCN 864.63300 Hz
  
```

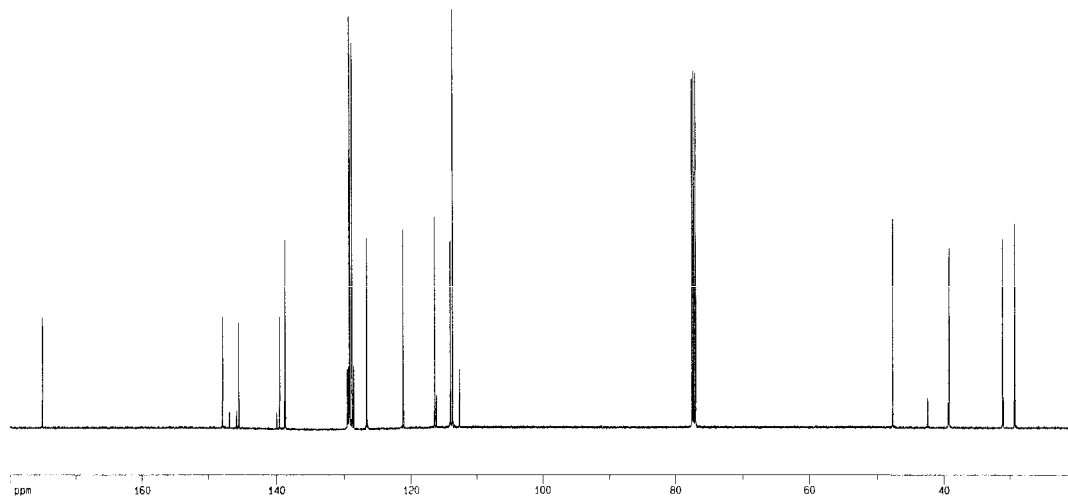


13C spectrum with 1H decoupling

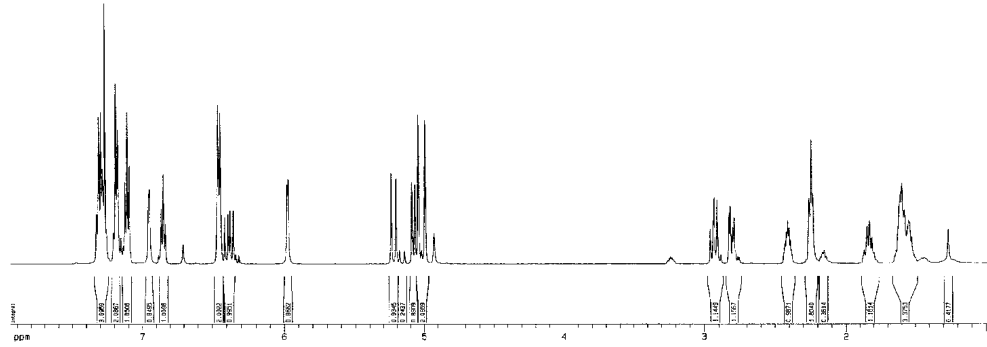
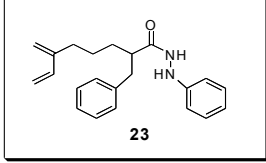


```

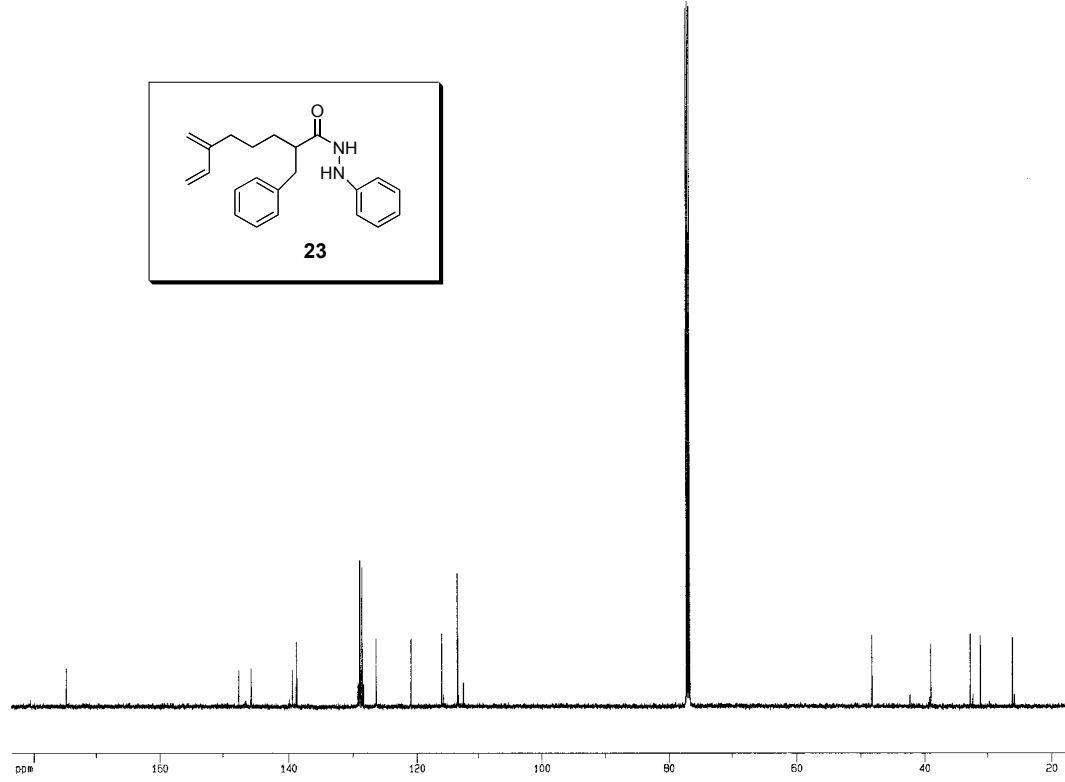
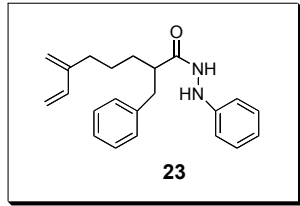
Current Data Parameters
NAME 1
NAME CLM4111111111111111
PROCNO 1
F2 - Acquisition Parameters
Date_ 06/13/2012
Time 15:14
INSTRUM cryo500
PROBHD 5 mm QNP1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2
DS 4
SWH 3013.231 Hz
FIDRES 0.463202 Hz
AQ 1.0764610 sec
RG 15004
AQ 16.500 usec
DE 2.00 usec
TE 298.2 K
C1 0.2000000 sec
d11 0.0300000 sec
d12 0.0500000 sec
d13 0.0350000 sec
===== CHANNEL f1 =====
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.764546 MHz
===== CHANNEL f2 =====
CPROG2 waltz16
NUC2 1H
P2 190.00 usec
PL2 1.00 dB
PL12 23.54 dB
SFO2 500.1360911 MHz
F3 - Processing parameters
SI 32768
SF 125.764546 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
ID 000 plot parameters
CP 20.00 cm
CY 9.11 cm
F3 180.000 ppm
F4 22546.47 Hz
F5 19.644 ppm
F6 2476.54 Hz
GAIN 1.00000000
HDCN 864.63300 Hz
  
```



<sup>1</sup>H spectrum



<sup>13</sup>C spectrum with <sup>1</sup>H decoupling



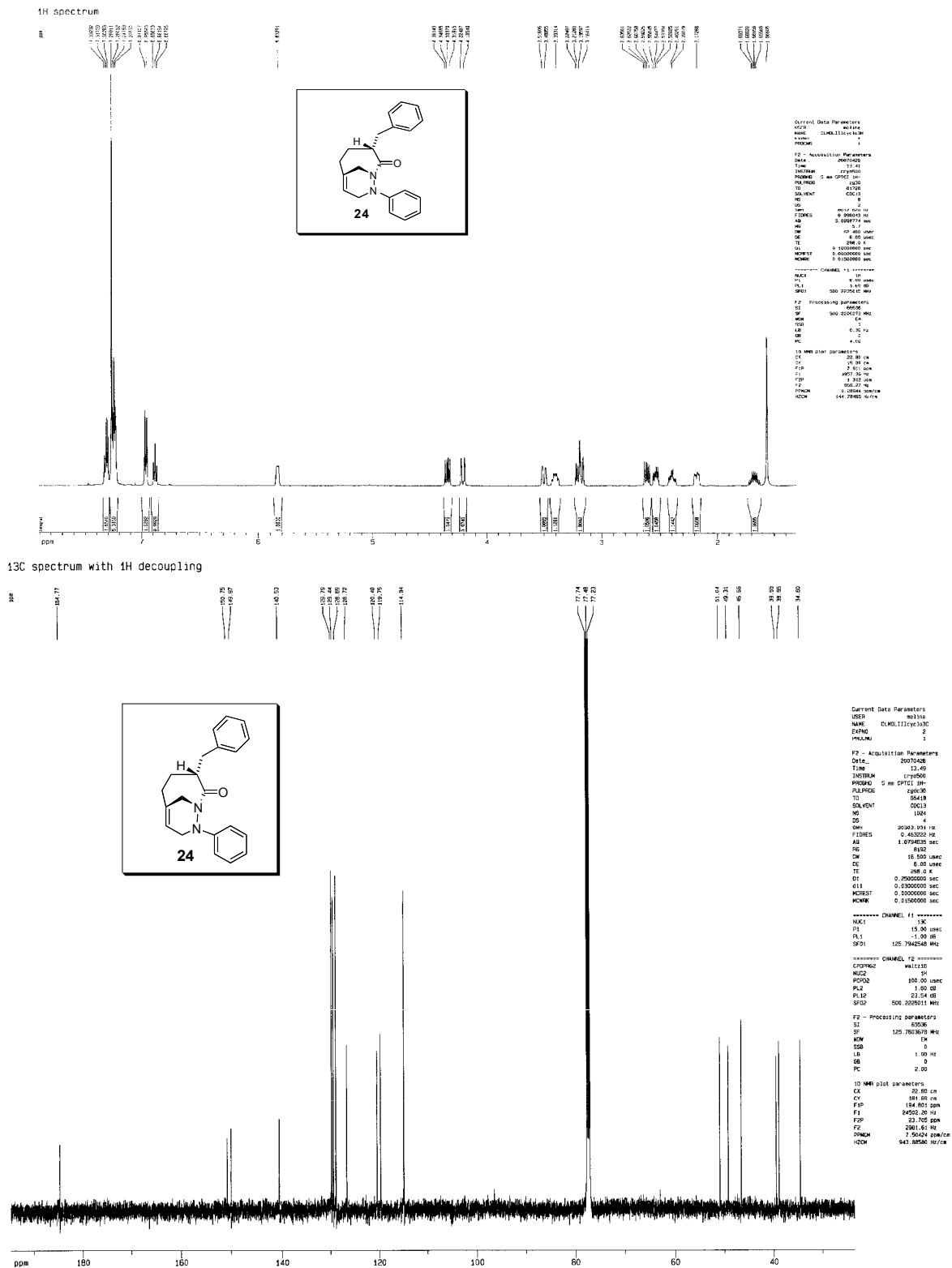
```

Current Data Parameters
USER          molina
NAME         CLMCL1411E1P
EXPNO        1
PROCNO       1
F2 - Acquisition Parameters
Date_        20070208
Time         16:31
INSTRUM      crys000
PROBHD      5 mm CP131 3H-
PULPROG     zgpg30
AQ         10.00
RG          655.00
WDW          EM
SSB          0
LB           0.00
GB           0.00
PC           0.00
RE          16.10
TE           300.2
D1           0.00000000
D2           0.00000000
D3           0.00000000
DELTA        0.00000000
===== CHANNEL f1 =====
NUC1         13
P1           8.00
PL1          1.00
SFO1        100.6283594
F2 - Processing parameters
SI           65536
SF           500.1360520
WDW          EM
SSB          0
LB           0.00
GB           0.00
PC           4.00
SC NMR list parameters
CX           00.00
CY           0.00
F1           7.500
F2           200.136
F3           474.50
G1           0.00000000
G2           0.00000000
G3           0.00000000
HOLD
===== CHANNEL f2 =====
NUC2         13
P2           15.00
PL2          -1.00
SFO2        125.7643540
===== CHANNEL f3 =====
NUC3         13
P3           100.00
PL3          1.00
SFO3        500.1360520
F2 - Processing parameters
SI           65536
SF           125.7643540
WDW          EM
SSB          0
LB           1.00
GB           0.00
PC           2.00
SC NMR list parameters
CX           00.00
CY           00.00
F1           133.628
F2           220.98
F3           17.040
G1           2199.47
G2           1.28186
G3           0.00000000
HOLD
    
```

```

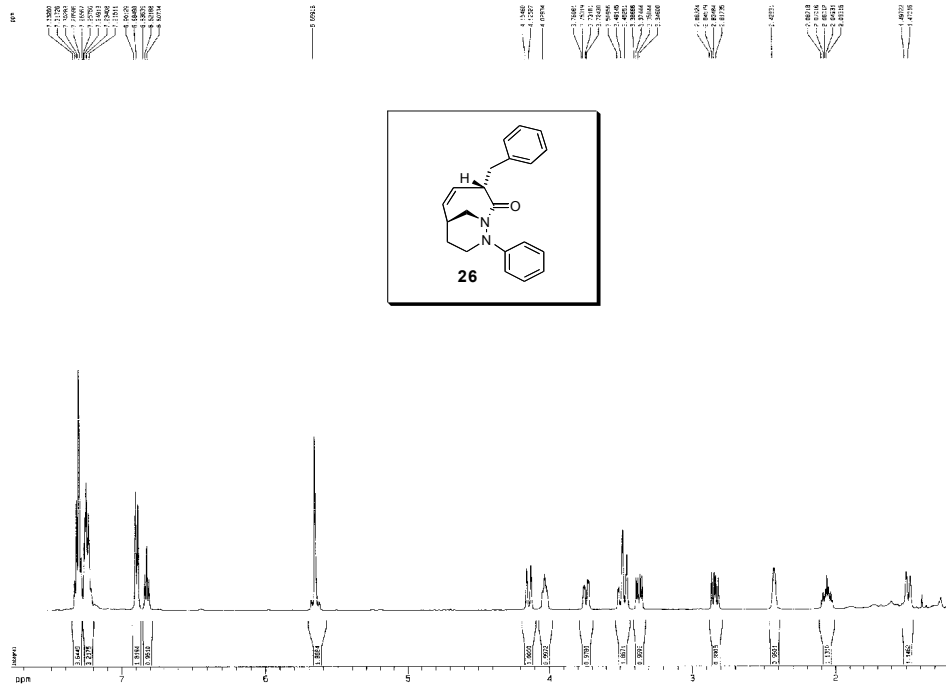
Current Data Parameters
USER          molina
NAME         CLMCL1411E2C
EXPNO        2
PROCNO       1
F2 - Acquisition Parameters
Date_        20070208
Time         16:25
INSTRUM      crys000
PROBHD      5 mm CP131 3H-
PULPROG     zgpg30
AQ         10.00
RG          655.00
WDW          EM
SSB          0
LB           0.00
GB           0.00
PC           0.00
RE          16.10
TE           300.2
D1           0.00000000
D2           0.00000000
D3           0.00000000
DELTA        0.00000000
===== CHANNEL f1 =====
NUC1         13
P1           15.00
PL1          -1.00
SFO1        125.7643540
===== CHANNEL f2 =====
NUC2         13
P2           100.00
PL2          1.00
SFO2        500.1360520
F2 - Processing parameters
SI           65536
SF           125.7643540
WDW          EM
SSB          0
LB           1.00
GB           0.00
PC           2.00
SC NMR list parameters
CX           00.00
CY           00.00
F1           133.628
F2           220.98
F3           17.040
G1           2199.47
G2           1.28186
G3           0.00000000
HOLD
    
```







1H spectrum



```

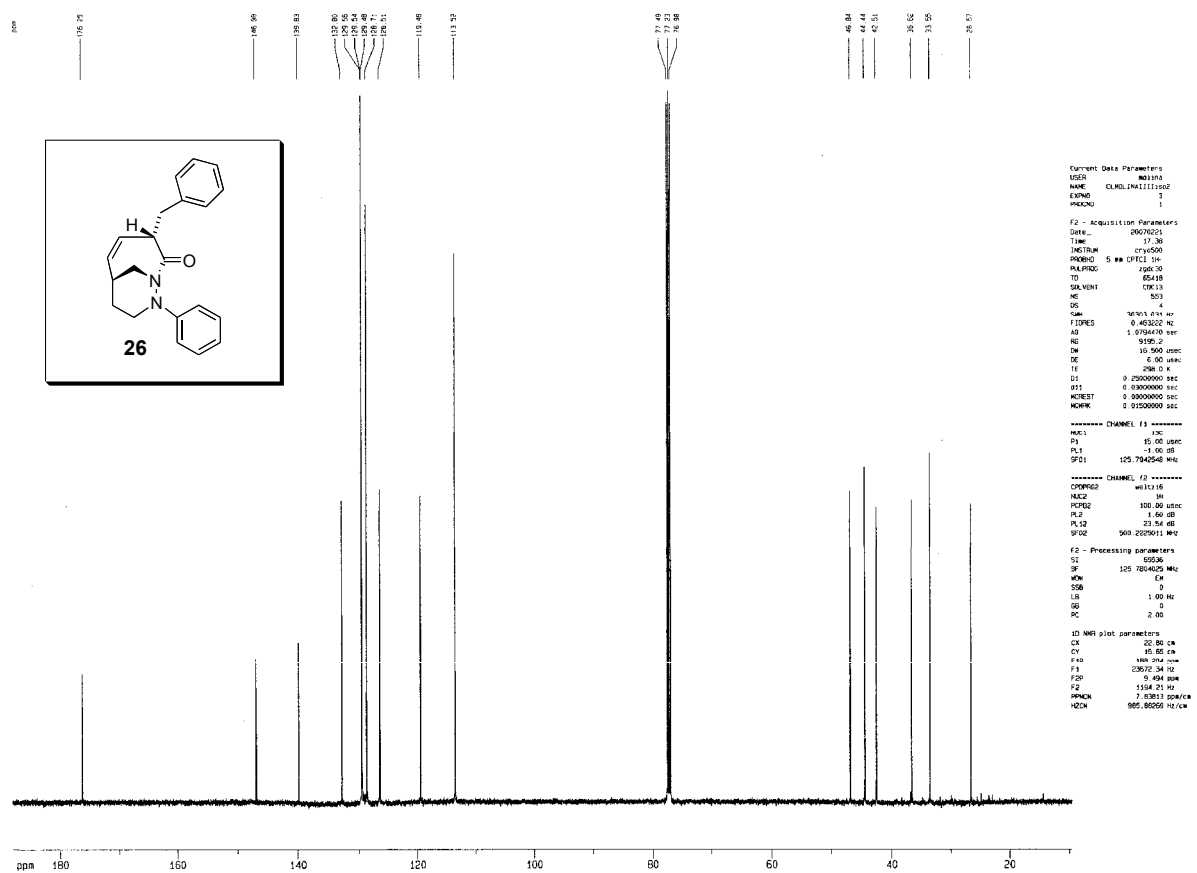
Current Data Parameters
=====
USER      molina
NAME      CMLM14111146
EXPNO    3
PROCNO   1
F2 - Acquisition Parameters
=====
DATE_    09/25/05
TIME     16:37
INSTRUM  crys500
PROBHD   5 mm CPXI 1H
PULPROG  zgpg30
TD       65536
SOLVENT  DMS-D6
NS       640
DS       4
SWH      6012.800 MHz
FIDRES   0.00041 Hz
AQ       0.0001716 sec
RG       64
DE       4.00 mm
TE       300.2 K
SI       0.0000000 sec
SFO      500.1360990 MHz
MORNET   0.0000000 sec
MORPH    144.46983 pulpr1

===== CHANNEL f1 =====
NUC1     13C
P1       8.00 usec
PL1      0.00 dB
SFO1     125.7614540 MHz

F2 - Processing parameters
=====
SI       65536
SF       500.1360990 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
MC       2.00

SD MRB parameters
=====
CX       22.80 cm
CY       10.40 cm
FWD      100.00 usec
PL1      1.60 dB
PL2      33.56 dB
SFO2     500.1360991 MHz
    
```

13C spectrum with 1H decoupling



```

Current Data Parameters
=====
USER      molina
NAME      CMLM14111146
EXPNO    3
PROCNO   1
F2 - Acquisition Parameters
=====
DATE_    09/25/05
TIME     17:38
INSTRUM  crys500
PROBHD   5 mm CPXI 1H
PULPROG  zgpg30
TD       65536
SOLVENT  DMS-D6
NS       640
DS       4
SWH      6012.800 MHz
FIDRES   0.000222 Hz
AQ       0.0001716 sec
RG       64
DE       4.00 mm
TE       300.2 K
SI       0.0000000 sec
SFO      500.1360990 MHz
MORNET   0.0000000 sec
MORPH    144.46983 pulpr1

===== CHANNEL f1 =====
NUC1     13C
P1       8.00 usec
PL1      -1.00 dB
SFO1     125.7614540 MHz

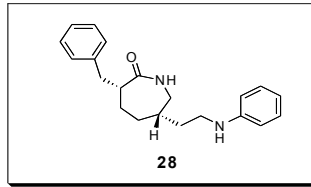
===== CHANNEL f2 =====
NAME     wa11116
NUC2     1H
PROBHD   5 mm
PL2      1.60 dB
PL12     33.56 dB
SFO2     500.1360991 MHz

F2 - Processing parameters
=====
SI       65536
SF       125.7614540 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
MC       2.00

SD MRB parameters
=====
CX       22.80 cm
CY       10.40 cm
FWD      100.00 usec
PL1      1.60 dB
PL2      33.56 dB
SFO2     500.1360991 MHz
MORNET   0.0000000 sec
MORPH    144.46983 pulpr1
    
```

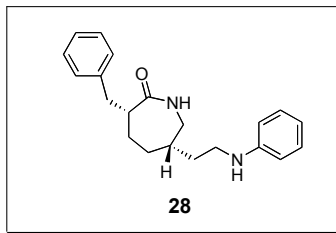
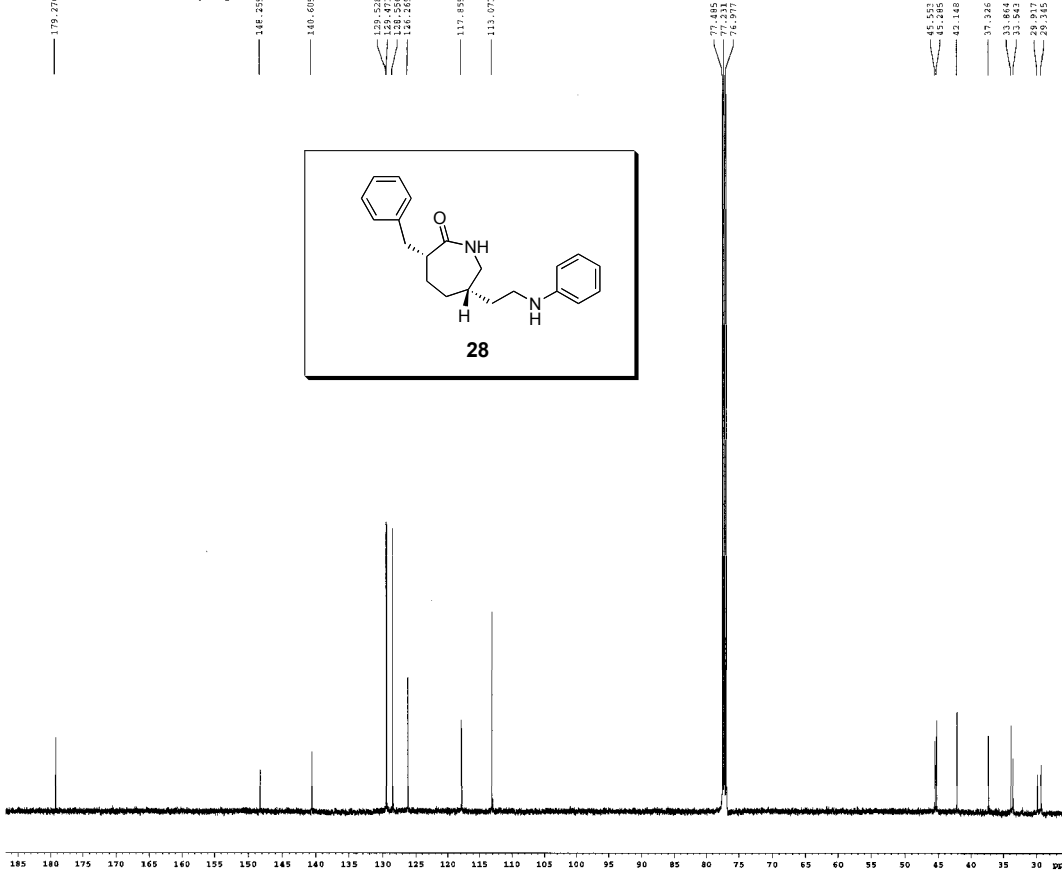


<sup>1</sup>H spectrum



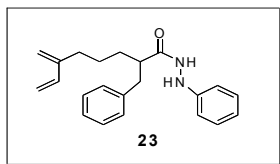
Current Data Parameters  
USER molina  
NAME C:\MOLINA\11M11C  
EXPNO 1  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 06/03/09  
Time 16:14  
INSTRUM crys500  
PROBHD 5 mm CPXI 1H-  
PULPROG zgpg30  
SOLVENT CDCl3  
NS 4  
DS 4  
SWH 30303.011 Hz  
FIDRES 0.463222 Hz  
AQ 1.0794470 sec  
RG 3135.2  
FW 16.500 Hz  
DE 6.00 Hz  
TE 279.2 K  
D1 0.25000000 sec  
d11 0.01000000 sec  
MCHST 0.00000000 sec  
MCHSK 0.01500000 sec  
===== CHANNEL f1 =====  
NUC1 13C  
P1 15.00 Hz  
PL1 -1.00 dB  
SFO1 125.7842548 MHz  
===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 100.00 Hz  
PL2 1.60 dB  
PL12 23.54 dB  
SFO2 500.1325011 MHz  
F2 - Processing parameters  
SI 65336  
SF 125.780406 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 2.00

<sup>13</sup>C spectrum with <sup>1</sup>H decoupling

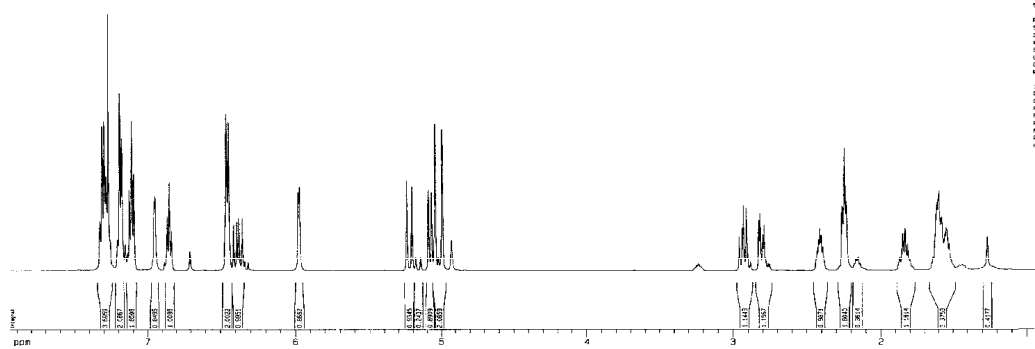


Current Data Parameters  
USER molina  
NAME C:\MOLINA\11M11C  
EXPNO 1  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 06/03/09  
Time 16:14  
INSTRUM crys500  
PROBHD 5 mm CPXI 1H-  
PULPROG zgpg30  
SOLVENT CDCl3  
NS 4  
DS 4  
SWH 30303.011 Hz  
FIDRES 0.463222 Hz  
AQ 1.0794470 sec  
RG 3135.2  
FW 16.500 Hz  
DE 6.00 Hz  
TE 279.2 K  
D1 0.25000000 sec  
d11 0.01000000 sec  
MCHST 0.00000000 sec  
MCHSK 0.01500000 sec  
===== CHANNEL f1 =====  
NUC1 13C  
P1 15.00 Hz  
PL1 -1.00 dB  
SFO1 125.7842548 MHz  
===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 100.00 Hz  
PL2 1.60 dB  
PL12 23.54 dB  
SFO2 500.1325011 MHz  
F2 - Processing parameters  
SI 65336  
SF 125.780406 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 2.00

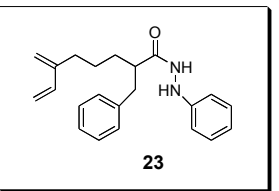
<sup>1</sup>H spectrum



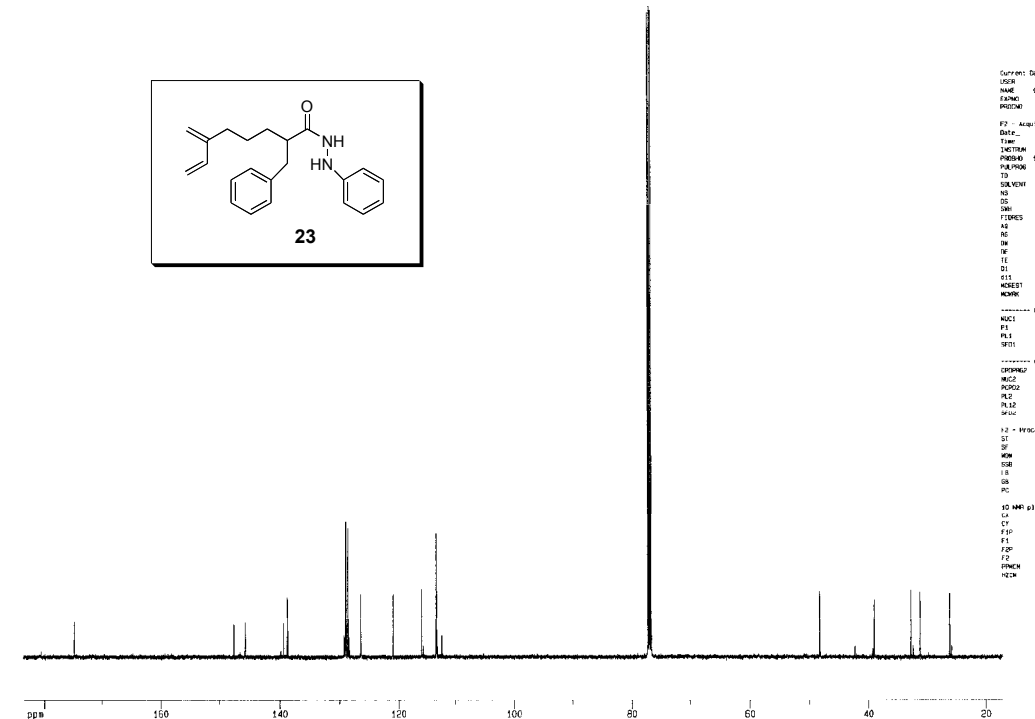
```
Current Data Parameters  
USER          sb416a  
NAME          CUMOLINA1121P  
EXPNO         1  
PROCNO        1  
F2 - Acquisition Parameters  
Date_         09/29/20  
Time          14:29  
INSTRUM       spect  
PROBHD        5 mm CPCLP 1H  
PULPROG       zgpg30  
TD            65536  
SOLVENT       CDCl3  
NS            2  
DS            4  
SWH           6032.833 Hz  
FIDRES       0.000629 Hz  
AQ           0.2568774 sec  
RG           655  
WM           62.430 umc  
SC           8.00 umc  
TE           298.2 K  
D1           0.1000000 sec  
d11          0.0000000 sec  
DELTA        0.0000000 sec  
AQCHY        0.0000000 sec  
----- CHANNEL f1 -----  
NUC1          1H  
P1            9.40 umc  
PL1           1.80 dB  
SFO1         500.201993 MHz  
F2 - Processing parameters  
SI            65536  
SF           500.2060923 MHz  
WDW          EM  
SSB          0  
LB           0.30 Hz  
GB           0  
PC           4.00  
IB WAlt plot parameters  
LA            62.50 umc  
CF            85.00 umc  
FID          163.853 umc  
F1           23698.23 Hz  
F2           2188.47 Hz  
SFO          517.04221 MHz/cm  
H2O          517.04221 MHz/cm
```



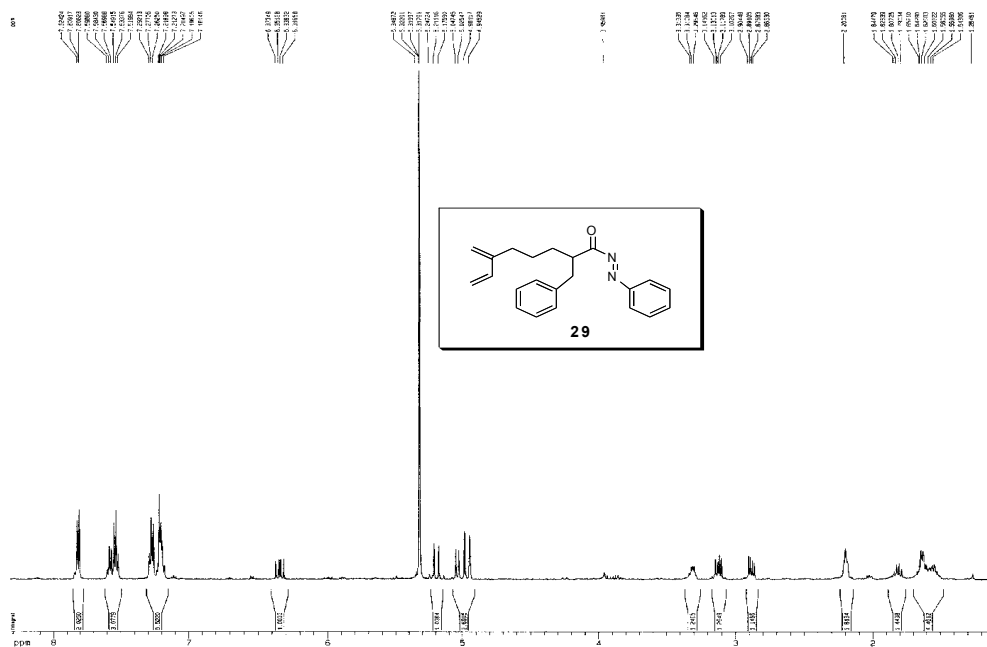
<sup>13</sup>C spectrum with <sup>1</sup>H decoupling



```
Current Data Parameters  
USER          sb416a  
NAME          CUMOLINA1120C2  
EXPNO         2  
PROCNO        1  
F2 - Acquisition Parameters  
Date_         09/29/20  
Time          14:25  
INSTRUM       spect  
PROBHD        5 mm CPCLP 1H  
PULPROG       zgpg30  
TD            65536  
SOLVENT       CDCl3  
NS            2  
DS            4  
SWH           30683.031 Hz  
FIDRES       0.463202 Hz  
AQ           1.1578445 sec  
RG           13004  
WM           86.060 umc  
SC           8.00 umc  
TE           298.2 K  
D1           0.2500000 sec  
d11          0.0000000 sec  
DELTA        0.0000000 sec  
AQCHY        0.0000000 sec  
----- CHANNEL f1 -----  
NUC1          13C  
P1            20.00 umc  
PL1           -1.00 dB  
SFO1         125.7604350 MHz  
----- CHANNEL f2 -----  
CPROG        zgpg30  
NUC2          1H  
PROG2        150.00 umc  
PL2           1.50 dB  
PL12         23.04 dB  
SFO2         500.2060931 MHz  
F2 - Processing parameters  
SI            65536  
SF           125.7604320 MHz  
WDW          EM  
SSB          0  
LB           1.00 Hz  
GB           0  
PC           2.00  
IB WAlt plot parameters  
LA            62.50 umc  
CF            85.00 umc  
FID          163.853 umc  
F1           23698.23 Hz  
F2           2188.47 Hz  
SFO          517.04221 MHz/cm  
H2O          517.04221 MHz/cm
```

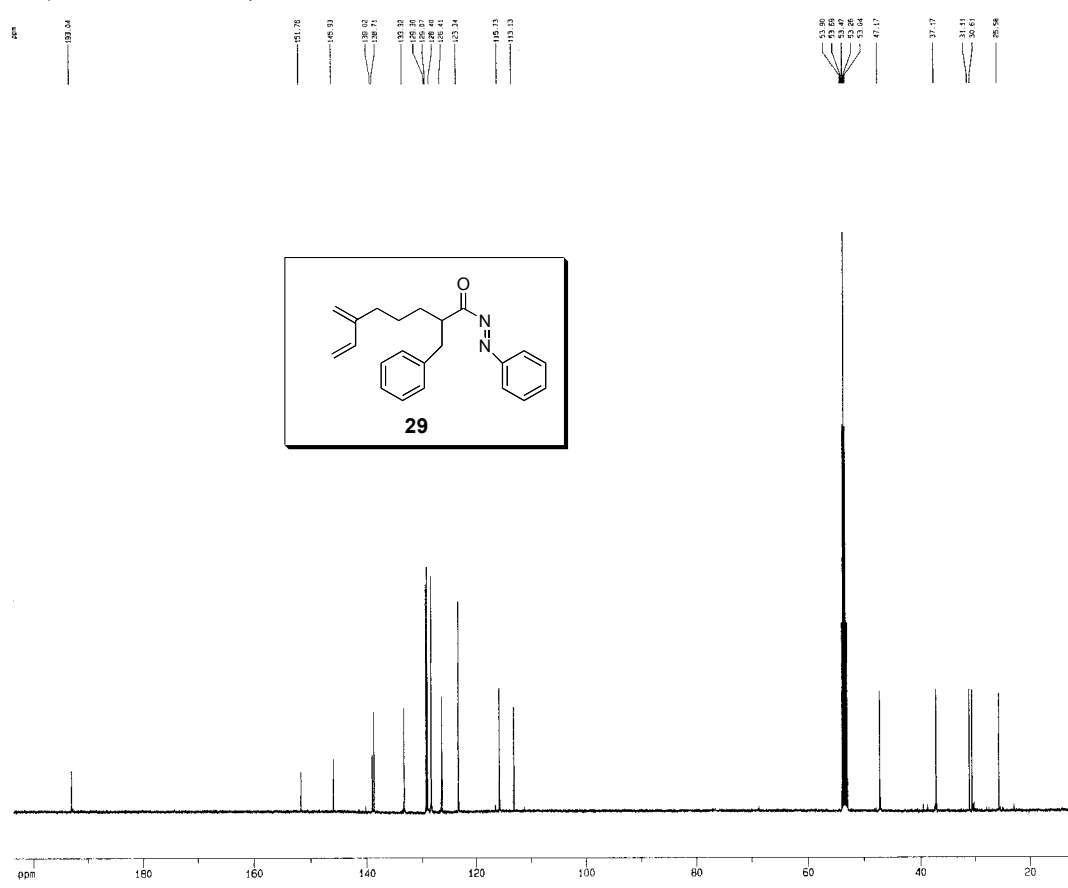


<sup>1</sup>H spectrum



```
Current Data Parameters
USER molina
NAME CML11140304
EXPNO 2
PROCNO 1
----- Acquisition Parameters
Date_ 00070405
Time 00 33
INSTRUM spect
PROBHD 5 mm CPDPR1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 4
SWH 400.1463140 MHz
FIDRES 0.0001000 Hz
AQ 0.0001274 sec
RG 655.5
DE 6.00 usec
TE 300.2 K
SI 1
SFO 100.6261260 MHz
NUC1 13
NUC2 13
PC 4.00
----- CHANNEL f1 -----
NUC1 13
P1 15.00 dB
PL1 -1.00 dB
SFO1 100.6261260 MHz
----- Processing parameters
SI 65536
SF 400.1463124 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
----- 1D NMR data parameters
SI 65536
SF 400.1463124 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
```

<sup>13</sup>C spectrum with <sup>1</sup>H decoupling

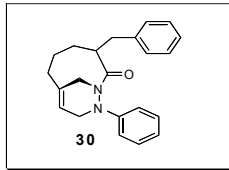
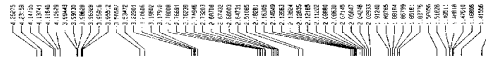


```
Current Data Parameters
USER molina
NAME CML11140304
EXPNO 2
PROCNO 1
----- Acquisition Parameters
Date_ 00070405
Time 08 14
INSTRUM spect
PROBHD 5 mm CPDPR1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 4
SWH 125.7641450 MHz
FIDRES 0.0001000 Hz
AQ 0.0001274 sec
RG 655.5
DE 6.00 usec
TE 300.2 K
SI 1
SFO 125.7641450 MHz
NUC1 13
NUC2 13
PC 2.00
----- CHANNEL f1 -----
NUC1 13
P1 15.00 dB
PL1 -1.00 dB
SFO1 100.6261260 MHz
----- CHANNEL f2 -----
PROBHD spect
NUC1 13
PULPROG zgpg30
P2 100.00 dB
PL2 1.00 dB
PL3 23.36 dB
SFO2 125.7641450 MHz
----- Processing parameters
SI 65536
SF 125.7641450 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
----- 1D NMR data parameters
SI 65536
SF 125.7641450 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
```

1H spectrum



10.0000



```

Current Data Parameters
USER          Molina
NAME         CUMULNALII-2C192
EXPNO        1
PROCNO       1

F2 - Acquisition Parameters
Date_        20070109
Time         10.08
INSTRUM      cryo500
PROBHD       5 mm CPYX1 1H-
PULPROG      zgpg30
SI           62516
SOLVENT      CDCl3
DS           4
SWH          30393.011 Hz
FIDRES       0.463222 Hz
AQ           1.0784470 sec
RG           14594.5
DW           16.500 usec
DE           6.00 usec
TE           298.0 K
D1           0.25000000 sec
d11          0.02000000 sec
MCREST       0.00000000 sec
MCMW         0.01000000 sec

===== CHANNEL f2 =====
NUC1          13C
P1            15.00 usec
PL1           1.00 dB
SFO1          125.7845648 MHz

===== CHANNEL f1 =====
NAME2         waltz16
NUC2          1H
NUC1P2        100 MHz
PL2           1.60 dB
PL12          21.54 dB
SFO2          500.225011 MHz

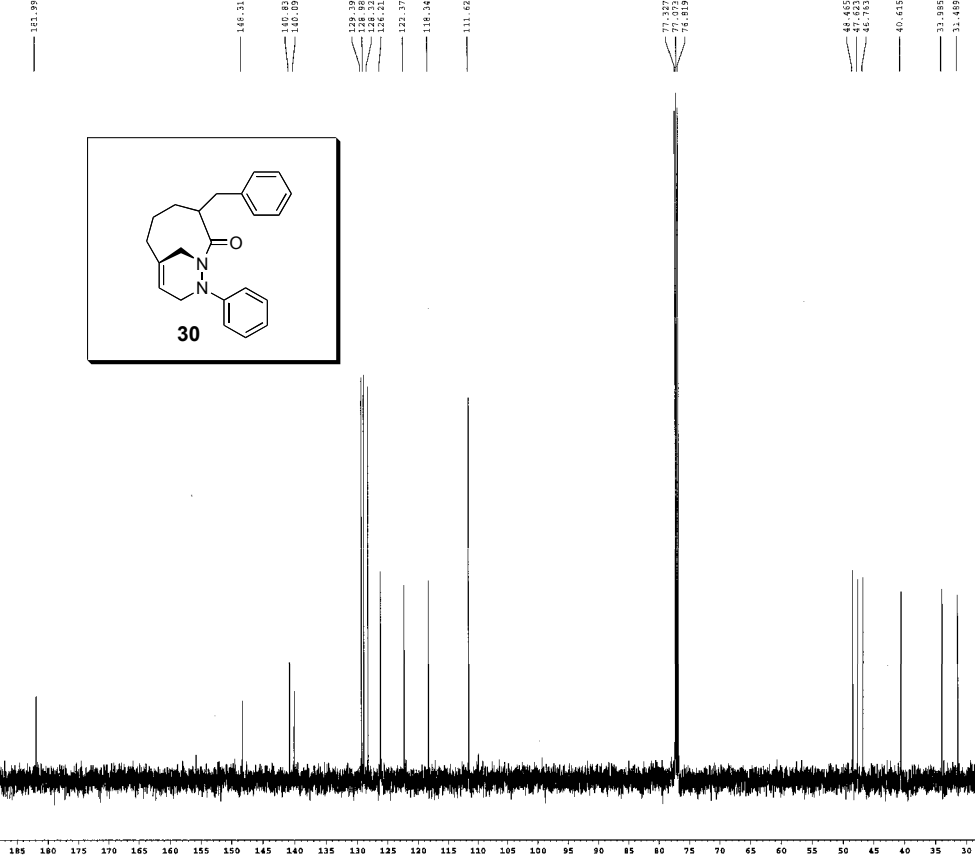
F2 - Processing parameters
SI            65536
SF            125.7804300 MHz
WDW           RM
SSB           0
LB            1.00 Hz
GB            0
PC            2.00

===== CHANNEL f1 =====
NUC1          1H
P1            12.00 usec
PL1           0.00 dB
SFO1          500.1364500 MHz

F2 - Processing parameters
SI            65536
SF            500.1364500 MHz
WDW           RM
SSB           0
LB            1.00 Hz
GB            0
PC            2.00

===== CHANNEL f2 =====
NUC1          13C
P1            15.00 usec
PL1           1.00 dB
SFO1          125.7845648 MHz
    
```

13C spectrum with 1H decoupling



```

Current Data Parameters
USER          Molina
NAME         CUMULNALII-2C192
EXPNO        1
PROCNO       1

F2 - Acquisition Parameters
Date_        20070109
Time         10.08
INSTRUM      cryo500
PROBHD       5 mm CPYX1 1H-
PULPROG      zgpg30
SI           62516
SOLVENT      CDCl3
DS           4
SWH          30393.011 Hz
FIDRES       0.463222 Hz
AQ           1.0784470 sec
RG           14594.5
DW           16.500 usec
DE           6.00 usec
TE           298.0 K
D1           0.25000000 sec
d11          0.02000000 sec
MCREST       0.00000000 sec
MCMW         0.01000000 sec

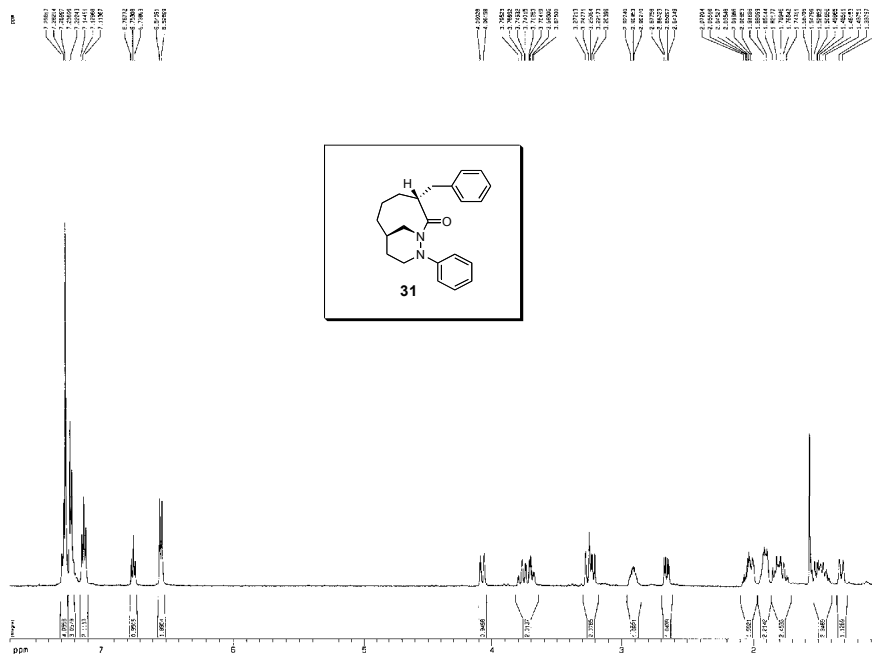
===== CHANNEL E1 =====
NUC1          13C
P1            15.00 usec
PL1           1.00 dB
SFO1          125.7845648 MHz

===== CHANNEL f2 =====
NAME2         waltz16
NUC2          1H
NUC1P2        100 MHz
PL2           1.60 dB
PL12          21.54 dB
SFO2          500.225011 MHz

F2 - Processing parameters
SI            65536
SF            125.7804300 MHz
WDW           RM
SSB           0
LB            1.00 Hz
GB            0
PC            2.00
    
```

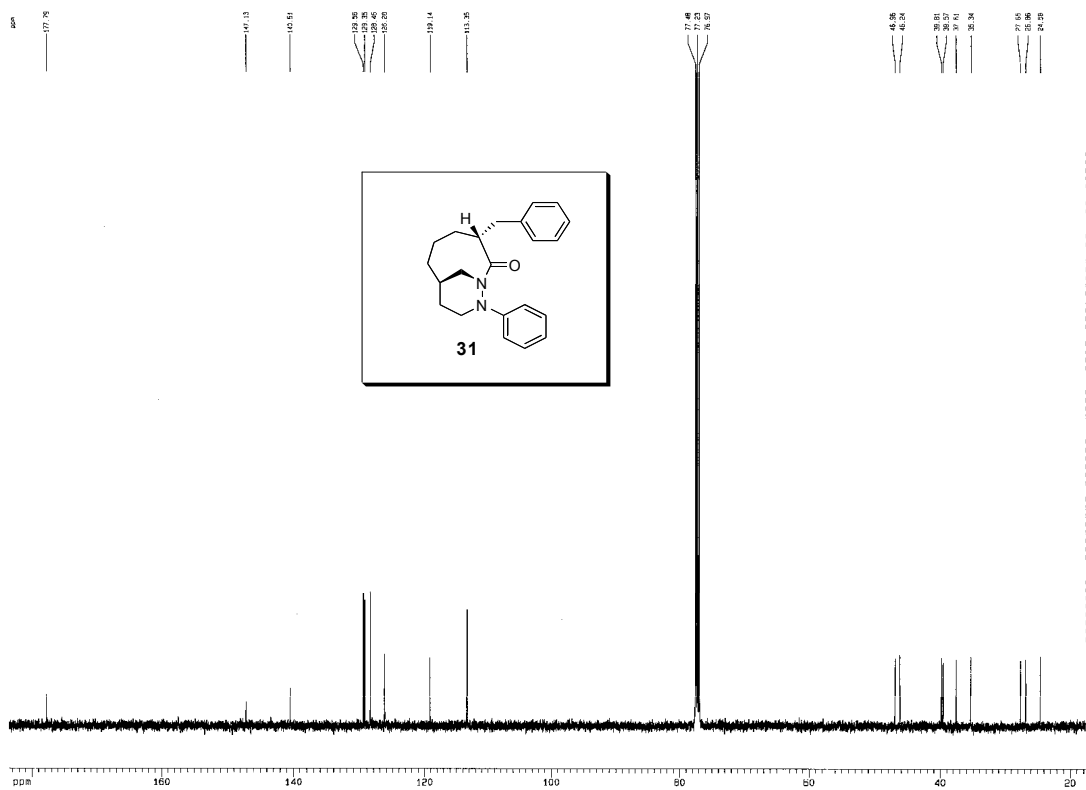


<sup>1</sup>H spectrum



```
Current Data Parameters
NAME CLMELNIAIIIC23
PROBHD 5 mm QNP1H1
PULPROG zgpg30
PROCNO 1
SOLVENT CDCl3
NS 8
DS 4
SWH 12.500 MHz
F2 100.628 MHz
AQ 1.000000 sec
RG 655.36
AQ 1.000000 sec
RG 655.36
PC 0.100000 sec
RG 655.36
PC 0.100000 sec
----- CHANNEL f1 -----
NUC1 13C
P1 12.00 usec
PL1 0.00 dB
SFO1 100.628360 MHz
F2 - Processing parameters
SI 65536
SF 125.762500 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.00
SFO MHz list parameters
CH 120.480 Hz
CP 37.499 Hz
F1 163.199 MHz
F2 157.448 MHz
F3 2219.80 Hz
PRINCP 7.278536351 Hz
HZMW 915.42524 Hz/c
```

<sup>13</sup>C spectrum with <sup>1</sup>H decoupling



```
Current Data Parameters
NAME CLMELNIAIIIC23
PROBHD 5 mm QNP1H1
PULPROG zgpg30
PROCNO 1
SOLVENT CDCl3
NS 8
DS 4
SWH 30.631013 MHz
F2 125.762500 MHz
AQ 1.000000 sec
RG 655.36
AQ 1.000000 sec
RG 655.36
PC 0.100000 sec
RG 655.36
PC 0.100000 sec
----- CHANNEL f1 -----
NUC1 13C
P1 12.00 usec
PL1 0.00 dB
SFO1 125.762500 MHz
F2 - Processing parameters
SI 65536
SF 125.762500 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.00
SFO MHz list parameters
CH 120.480 Hz
CP 37.499 Hz
F1 163.199 MHz
F2 157.448 MHz
F3 2219.80 Hz
PRINCP 7.278536351 Hz
HZMW 915.42524 Hz/c
```

