SUPPORTING INFORMATION

Highly Chemoselective Addition of Amines

to Epoxides in Water

Najmodin Azizi and Mohammad R. Saidi*

Department of Chemistry, Sharif University of Technology,

P. O. Box 11465-9516 Tehran- Iran

E-mail: Saidi@sharif.edu

Contents:	Page number
Experimental Section	S1-S2
¹ H and ¹³ C NMR spectra for products	S3-S38

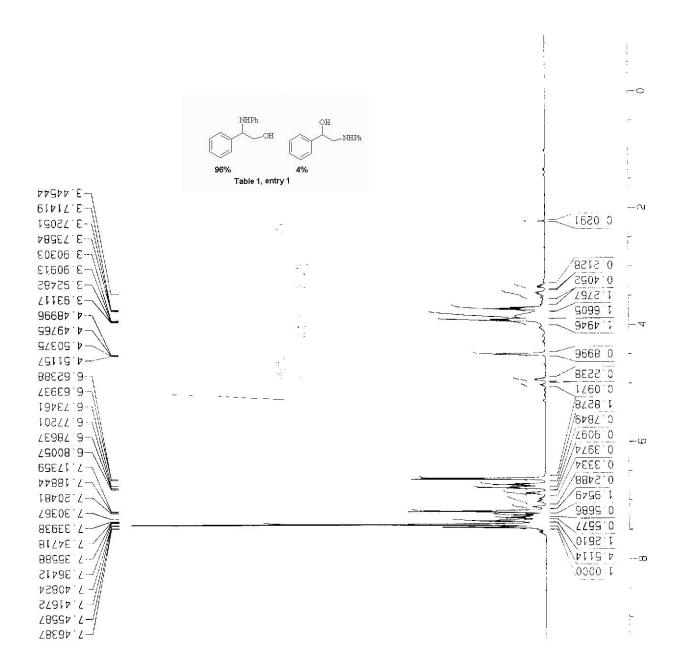
Experimental Section

General. All chemicals were purchased and used without any further purification. NMR spectra were recorded at 500 MHz for proton and at 125.7 MHz for carbon nuclei in (CDCl₃ + CCl₄) or (C_6D_6 + CCl₄). The products were purified by column chromatography carried out on silica gel (230-400 mesh), and by using ethyl acetate/ petroleum ether mixtures. Reactions were carried out room temperature, all the epoxides used are known and all amines employed are commercially available. All β -amino alcohols obtained are known compound.

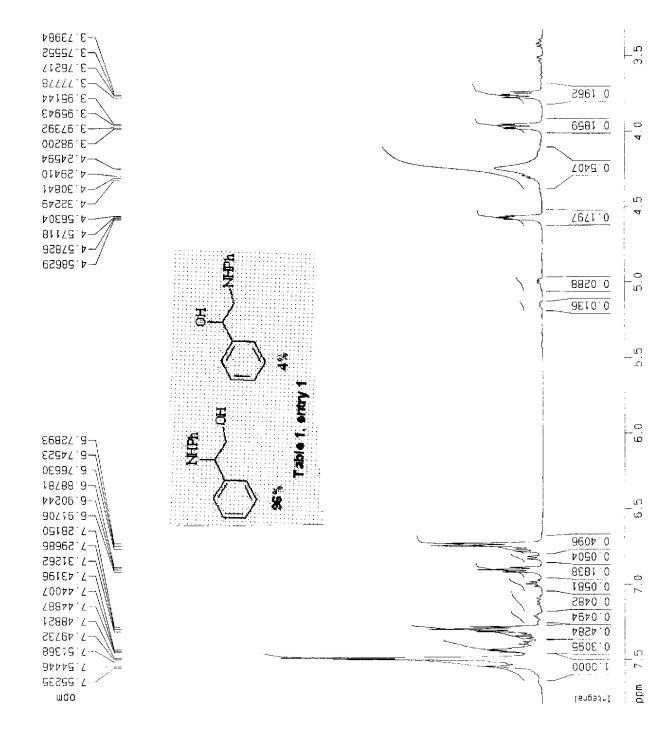
General procedure for the synthesis of β -amino alcohols:

In the test tube equipped were introduces 5 mmol epoxides and 2 ml of water. The amine (6mmol) was added in one portion and the test tube was kept at 0 °C and warmed to room temperature under vigorous stirring for 14 h for styrene oxides (Table 1) and 5-24 h for other epoxides (Table 2). If no participation occurred, water (2 ml) was added and the aqueous mixture was extracted with 10 ml of diethyl ether or ethyl acetate and dried over anhydrous Na₂SO₄, and solvent was removed under reduce pressure to give the β -amino alcohols in the pure state. The crude product was analyzed by ¹H and ¹³C NMR. For the compound Table **1**, Entry **3**, **6**, **8** and Table **2** entry **23**, **25**, further purification was carried out by short column chromatography on silica gel (ethyl acetate/petroleum ether). All compounds were characterized on the basis of their spectroscopic data (IR, NMR) and by comparison with those reported in the literature.¹⁻¹¹

	ers sec	HX AB AB AB AB AB AB AB AB AB	LT Z HMHZ	ст ст ррт А2 ррт Н2 Рг/ст Н2
.a Parameters Azizi 342		40.3 48.400 6.50 300.0 5.0000000 5.0000000 11 11 9.30 500.1330865	ssing paramete 32768 500.1300000 6 0 0 1.00 1.00	t parameters 18.00 11.00 10.706 5354.41 -0.952 -0.952 -180.93 0.64820 0.64820 324.18417
Current Dat NAME EXPNO PROCNO	F2 - Acquis Date - Acquis INSTAUM PROBHD PULPROG TD TD SCLVENT SSLVENT SSUVENT SSUVENT SSUVENT SSUVENT SSUVENT SSUPENT	86 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	- Proce SF WDW SSB SSB CSB CB CB CB CB	10 NMA plo CX CY F1P F1 F2 F2 P9MCM H2CM

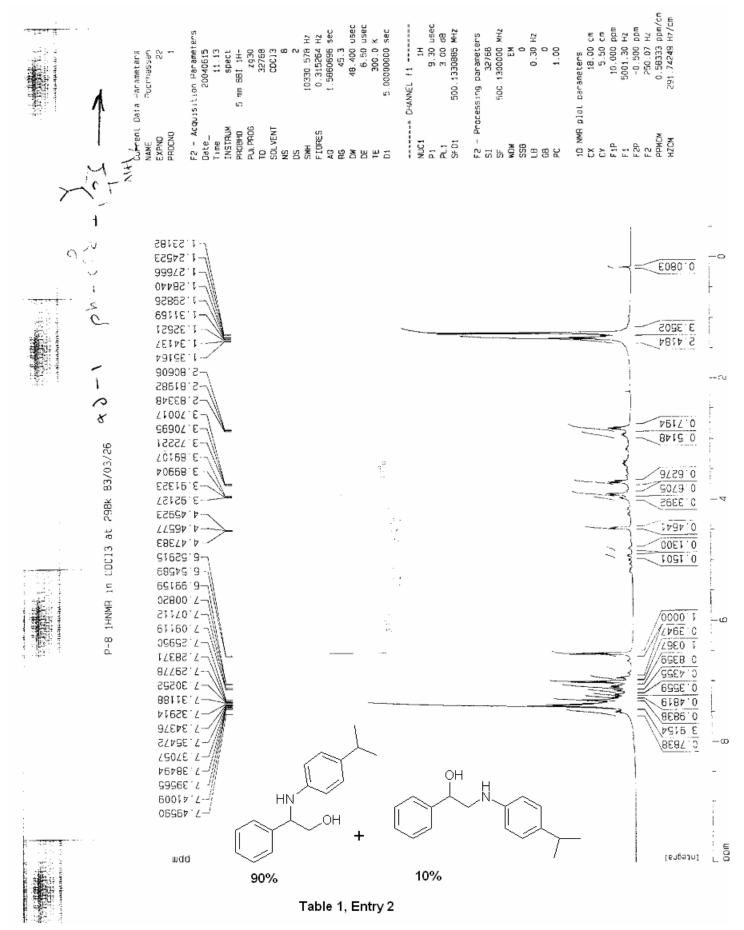


0.24837 ppm/cm 124.21862 Hz/cm 1H 9.30 USEC 3.00 dB 500.1330885 MHz 10 NMA plot parameters CX 18.00 cm CY 7.06 cm F1P 7.792 ppm F1 3.321 ppm F2P 150.99 H2 F2P 1500.99 H2 48.400 usec 6.50 usec 1.5860696 sec 12.7 300.0 K 5.00000000 sec F2 Acquisition Parameters Date____20040412 10330.578 Hz 0.315264 Hz 500.1300000 MHz 0.30 Hz CHANNEL f1 ===== - Processing parameters spect 5 mm BBI 1H-BB zg30 20040412 11.12 1.00 32768 CDC13 32768 AZIZI ດນ ы Current Data Parameters 302 œ ł TD SOLVENT NS DS SWH FIDRES INSTRUM PUL PROG **PROBHD** PROCNO EXPNO F 2 PPMCM HZCM Time NUC1 NAME P1 PL1 SF01 R SSB SSB SI -H H H S HO HO HO



5 1HNMR in CDC13 at 298K 83/01/24

NO



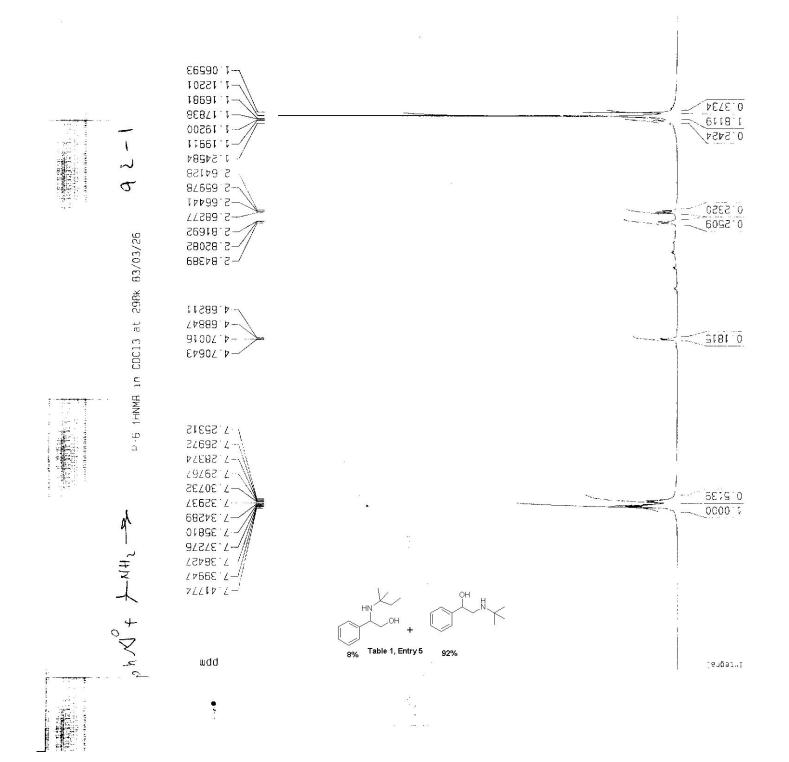
10 NNR plot parameters CX 70.00 cm CV 10.96 cm F1P 227.586 ppm F1 28620.75 Hz F2P -25.064 ppm F2 -3151.98 Hz PPMCM 12.63251 ppm/cm HZCM 1588.63647 Hz/cm Maitz16 Maitz16 11 3.00 dB 22.00 dB 22.00 dB 22.00 dB 13C 10.80 usec 0.00 dB 125.7703143 MHz 40000.000 Hz 0.610352 Hz 0.8192655 sec 1824.6 12.500 usec 6.50 usec 300.0 K F2 - Processing parameters SI 32768 SF 125.757390 MH2 MDW EM SSB 0 H2 C 1.00 H2 C H2 C H2 C 1.40 10.00000000 sec 0.03000000 sec 0.00002000 sec ----- CHANNEL f2 -----F2 - Acquisition Parameters CHANNEL f1 ===== 11.14 5 mm BBI 11-29930 65536 65536 00013 71 71 Current Data Parameters NAME Azizi EXPNO 367 20040528 Time INSTRUM CPDPRG2 рновно PHOCNO NUC2 PCPD2 PL2 PL13 PL13 SFD2 SFD2 Date PL1 SF01 SF01 24.761 24.889 \circ £30.8S 25.162 ₽70.EE ₽18'EG Þ87.82 ¢0£'19 62.575 010.03 168.73 20 71.260 882.SY 110.77 591.87 78.420 ¢£0'76 600.211 160.711 067.911 -156.609 100 156.764 156.834 869.751 127.734 157.967 128.072 618.8S1 128.639 128.803 129.254 150 985.951-138.529 138.529 Ð 10% 138.569 141.623 143.481 146 377 Table 1, entry 2 Ð 200 Z 8 шdd

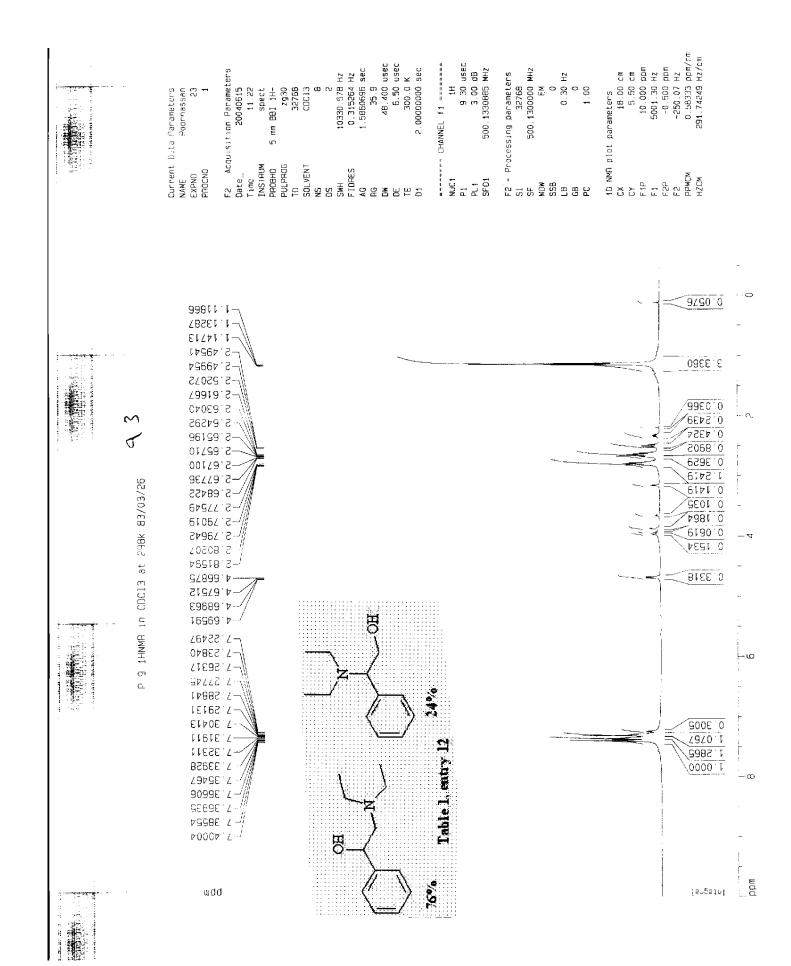
19-P-8 13CNMH in CDC13 at 296K 83/04/08

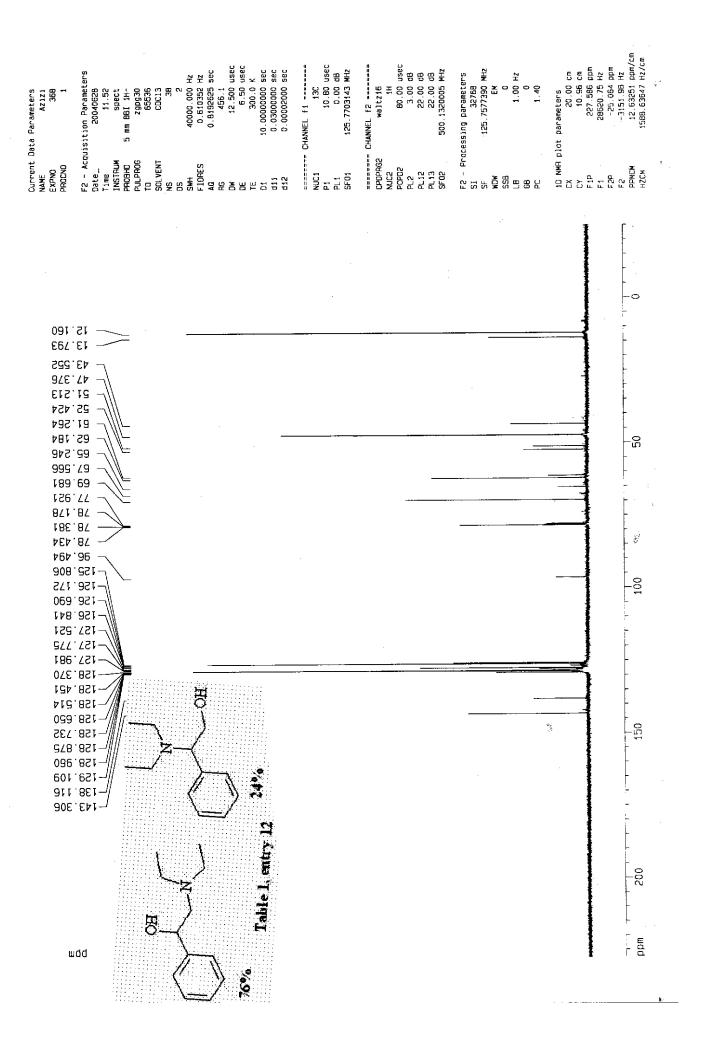
/

udd

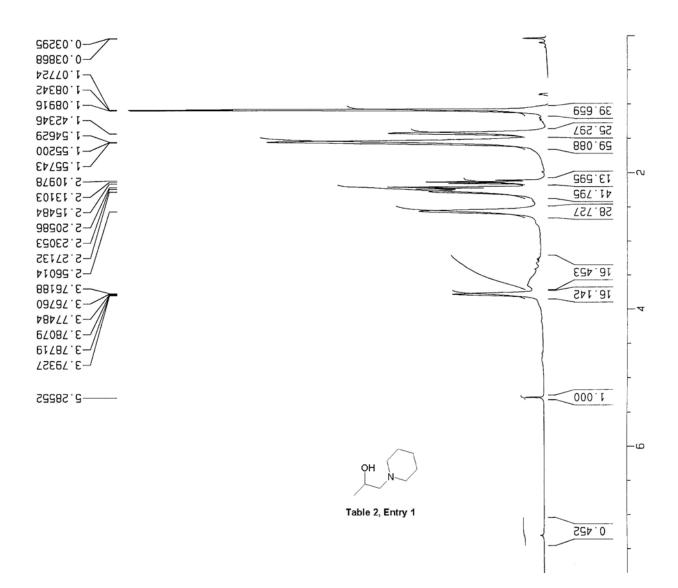
	.ers HZ Sec Usec K	sec usec MHz	ers MHZ Hz	cm cm ppr HZ HZ hz cm HZ/cm
Sula Purameters Poorhassan 20	Dn Param 2004061 01.5 10.5 10.5 10.5 10.5 276 276 275 275 275 0.31526 0.31526 0.31526 1 58008 1 580 1 5 1 5 1 5 1 5 1 5 1 5 1 5 1 5 1 5 1 5	5:00000000 = CHANNEL f1 ==== 1H 9.30 500.1330885	scessing paramete 32766 506.1300000 EM 0 0.30 0.30	Jiut parameters 10.00 11.00 5001.30 -0.500 -250.07 0.58333 291.74249
Current NAME EXPNO PROCNO	F2 - Aci Date Time INSTRUM FINSTRUM FINSTRUM FUCHING SOLVENT SOLVENT SOLVENT FIDRES FIDRES FIDRES FIDRES FIDRES FIDRES FIDRES FIDRES FIDERS FIDRES FI	DI NUCI P1 P1 SF01 SF01	RSS SS SS SS SS SS SS SS SS SS SS SS SS	10 NMR 1 CX F1 F1 F2 F2 PUNUV H2 CM



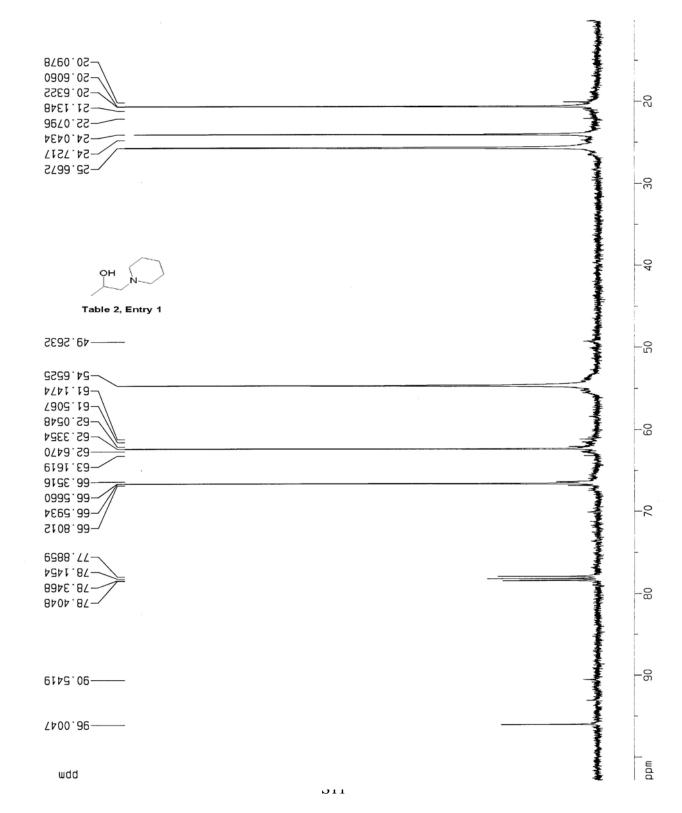




- Parameters Yosefi 134 1	tion Parameters 20040828 15.07 spect spect 2930 32768 CDC13 4 4 0 315264 Hz 0.315264 Hz 1.5860696 sec 5.0 Usec 5.50 Usec 5.50 Usec	CHANNEL f1 ======= 1 H 9.30 usec 3.00 dB 500.1330885 MHz essing parameters 32768 500.1300000 MHz EM 0 0 130 Hz 0 1.00	parameters 18.00 cm 11.00 cm 10.000 ppm 5001.30 Hz 0.000 ppm 0.000 Hz 0.55556 ppm/cm 277.85001 Hz/cm
Current Data NAME EXPNO PROCNO	F2 - Acquisi Date INSTRUM 5 PROBHD 5 PULPAOG 10 NNS SOLVENT NS SMH AG AG AG AG AG AG AG CE TE TE DM	====== CHANNEI NUC1 P1 PL1 500 F2 - Processing S1 500 MDW SSB LLB CG CG	10 NMR plot CX CY F1 F1 F2 PPMCM H2CM



10 NMR plot parameters CX 20.00 cm CY 21.64 cm F1P 102.862 ppm F1 12935.66 Hz F2P 10.091 ppm F2 1268.99 Hz PPMCM 4.63855 ppm/cm HZCM 583.33331 Hz/cm 40000.000 Hz 0.510352 Hz 0.8192625 sec 32000 12.500 usec 5.50 usec 300.0 K 5.0000000 sec 0.03000000 sec 13C 10.80 usec 0.00 dB 125.7703143 MHz F2 - Processing parameters SI 32768 SF 125.757390 MHz WDW EM SSB 1.00 Hz CB 1.40 PC 1.40 CHANNEL f1 ====== F2 - Acquisition Parameters Date____20040904 20040904 14.37 5 mm BB1 1H-290930 65536 C0513 69 Current Data Parameters NAME Azizi 407 CPDPRG2 CPDPRG2 NUC2 PCPD2 PL2 PL13 PL13 SF02 SF02 NUC1 NUC1 P1 PL1 SF01 INSTRUM EXPNO PROCNO Time



0.00 Hz 0.55556 ppm/cm 277.85001 Hz/cm F2 - Acquisition Parameters Date_____20040828 Time_____15.03 INSTRUM spect PROBHO 5 mm BBI 1H-PUL PROG 2930 TD 32768 SOLVENT COC13 35.9 48.400 usec 6.50 usec 295.0 K 10.0000000 sec 1H 9.30 Usec 3.00 dB 500.1330885 MHz
 1D NMR plot parameters

 CX
 18.00 cm

 CY
 10.96 cm

 CY
 10.96 cm

 F1P
 10.000 ppm

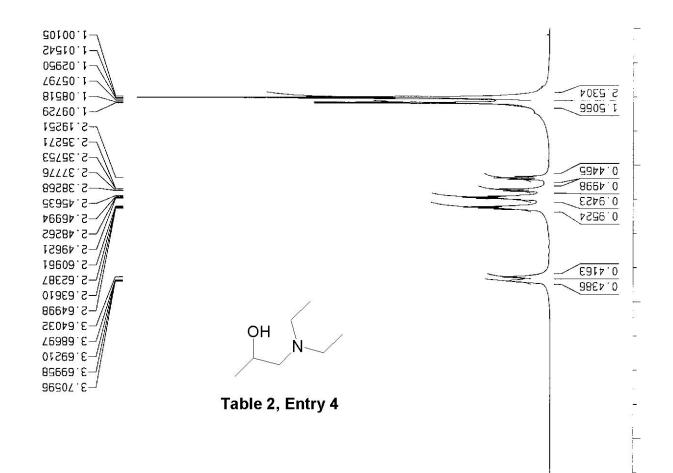
 F2P
 0.000 ppm

 F2
 0.000 Hz

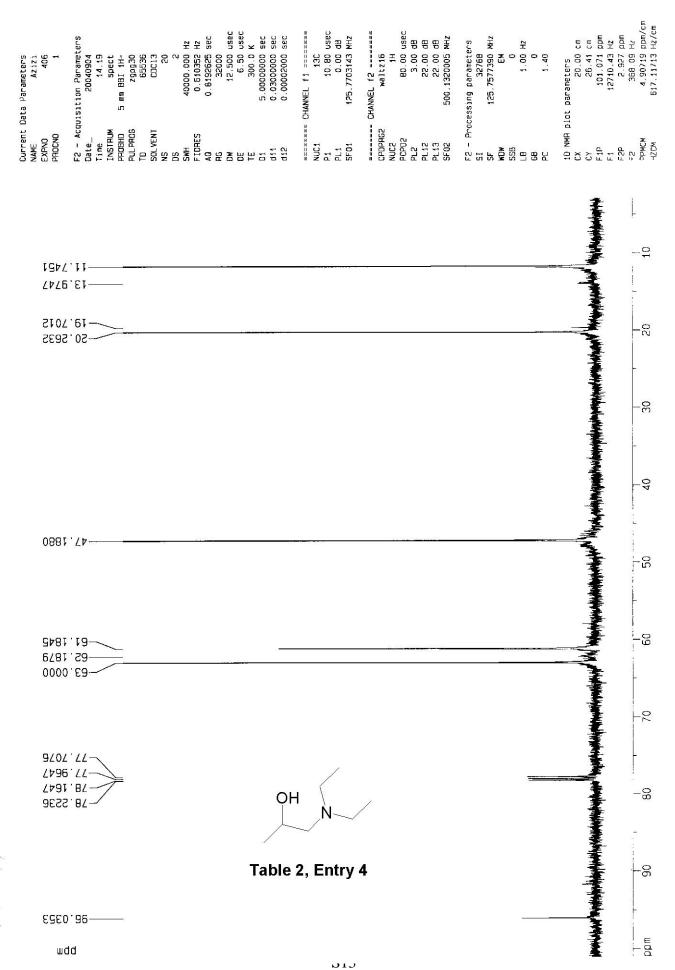
 F2
 0.000 Hz

 F2
 0.000 Hz

 F2
 0.000 Hz
 F2 - Processing parameters SI 32768 SF 500.1300000 MHZ WDW EM 0 SSB 0.30 HZ CB 0.30 HZ GB 0.30 HZ CB 7.00 10330.578 Hz 0.315264 Hz 1.5860696 sec 500.1300000 MHz EM 0 0.30 Hz 1.00 f1 ====== Current Data Parameters NAME Yosefi EXPND 133 PPOCNO 1 0 CHANNEL -----PROBHD PULPROG SOLVENT NS DS SWH FIDRES NUC1 P1 PL1 SF01 SHORE HO

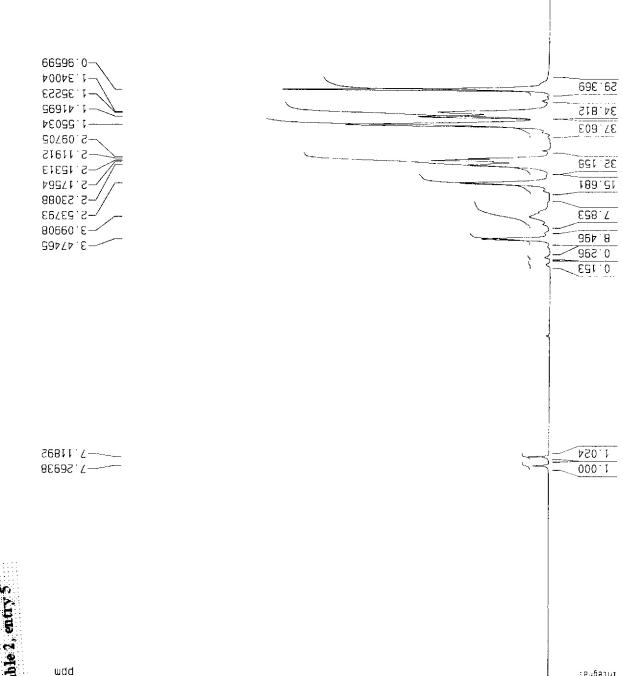


Y33 1HNMR in CDC13 at 298K 83/06/07

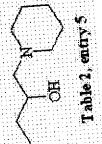


y-33 13CNMH in CDC13 at 298k 83/06/14

10 NWR plot parameters CX 18.00 cm CY 7.06 cm F1P 10.864 ppm F1 5433.28 H2 -0.551 ppm F2 -275.73 H2 PPMCM 0.53417 ppm/cm H7CM 317.16500 H2/cm 143.7 48.400 usec 6.50 usec 300.0 K 1H 9.30 usec 3.00 dB 500.1330885 MHz F2 - Pracessing parameters SI 32768 SF 500.1300000 MH2 WDW EM 0 SSB 0.30 H2 CB 0.30 H2 CB 0.30 H2 CB 0.30 H2 10330.578 Hz 0.315264 Hz 1.5860696 sec F2 - Acquisition Parameters Date____20040517 5.00000000 sec ====== CHANNEL f1 ===== 12.40 spect 5 mm 881 1H-2930 32768 32768 Azizi 330 Current Data Parameters œ n, Date. Time INSTRUM PUL PROG TD SOLVENT NS DS SWH DROBHD PROCNO FIDRES EXPNO NAME NUC1 P1 PL1 SF01 2 M D H D H D Å0



59 1HNMR in CDC13 at 298K 83/02/28



isrgetni

 1D NNR plot parameters

 CX
 20.00 cm

 CY
 21.59 cm

 F1P
 231.310 ppm

 F2
 25.089.05 Hz

 F2
 25.089.05 Hz

 F2
 30.598 pcm

 F2
 38.65.73 Hz

 F2
 38.65.73 Hz

 F2MCH
 13.04936 fcm

 H2CM
 156.65 Hz/cm

 CPOPAGE
 CHANNEL
 F2
 <thF2</th>
 F2
 F2
 F2 - Processing parameters SI 32769 MDW 125./5/290 MHz MDW 558 125./5/290 MHz MDW 569 11.00 Hz 69 11.00 Hz 69 11.40 ²2 - Acquisition Parameters Date 20040525 Time 10.53 INSTRUM soect PROBHI 5 mm GW H/ PULPROG 5 mm GW H/ PULPROG 5536 SOLVENT C606 NS / 40000 000 Hz SMH 40000 000 Hz FLDRES 0.510352 Hz 40000 000 Hz 0.610352 Hz 0.8192855 sec 3649.1 12.550 usec 6.50 usec 6.50 usec 0.0000000 sec 0.03000000 sec Current Data Parameters NAME AZIZI 330 EXPNO PROCNO NUC2 PCPD2 PL12 PL13 SFD2 SFD2 AQ 90 01 01 01 01 01 01 01 01 01 01 01 -÷ 105.01 ------ 54.836 - 56.468 E12.85 -678'99 ---59 13CNMH in C6D6 at 298K 83/03/05 906°99 — 190.89 1 I K. C. J. . ~ 127.807 000.851-S91.851-Table 2, entry 5 同 10 wdd

 ID NMH plot parameters

 CX
 18.00 cm

 CY
 11.24 cm

 F1P
 10.781 ppm

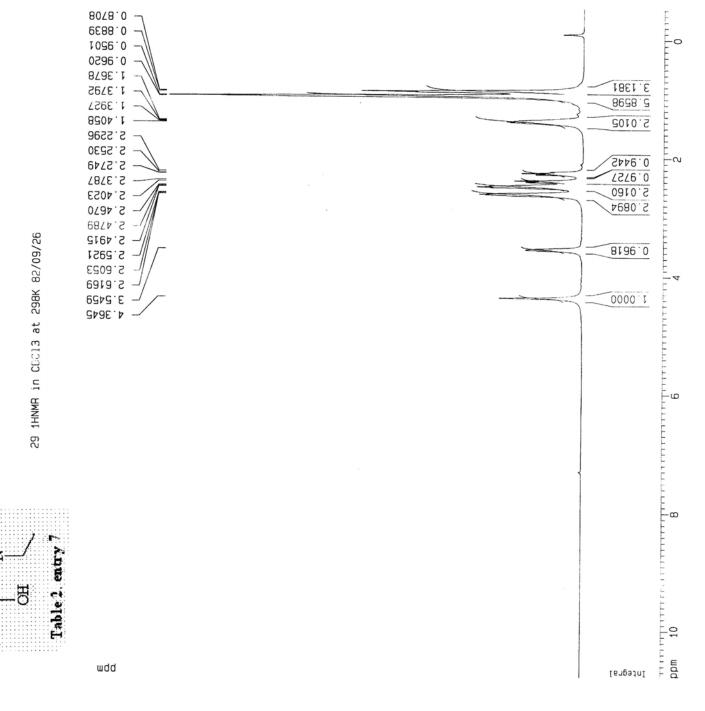
 F1
 5391.95 Hz

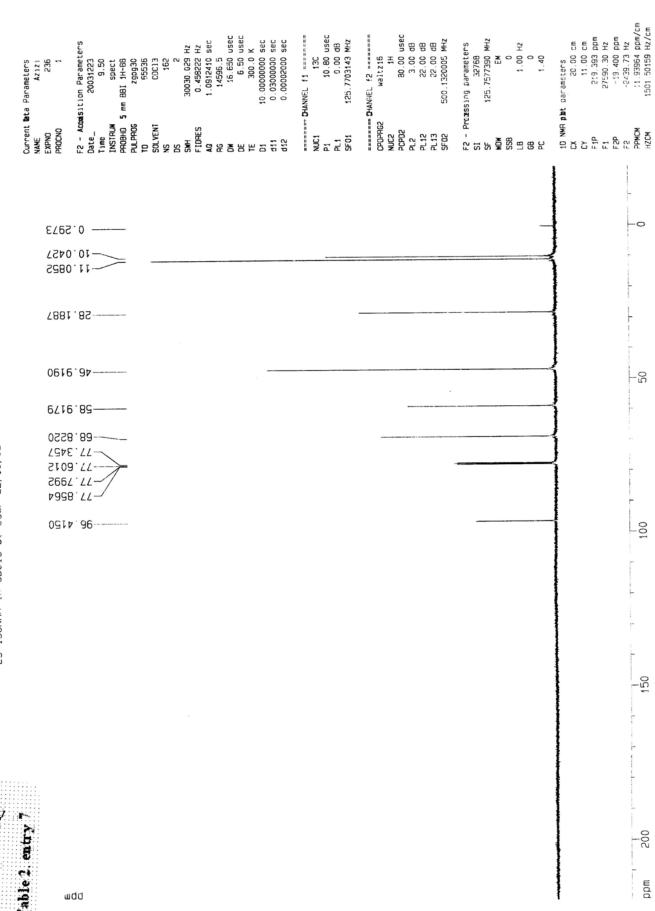
 F2P
 -0.524 ppm

 F2
 -261.95 Hz

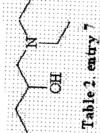
 PPMCM
 0.628005 ppm/cm

 HZCM
 314.10544 Hz/cm
 1H 9.30 usec 3.00 dB 500.1330885 MHz 6.50 usec 300.0 K F2 - Processing parameters SI 32760 SF 500.1300000 MHz WDW EM SSB 0 LB 0.30 Hz GB 0.30 Hz GB 0 10330.578 Hz 0.315264 Hz 1.5860696 sec 48.400 usec F2 - Acquisition Parameters Date____20031217 5.00000000 sec CHANNEL f1 ====== 15.38 spect 1H-BB zg30 32768 CDC13 25.4 Current Data Parameters ŝ 231 AZIZI 5 mm BBI 16 17 INSTRUM PROBHD TD SOL VENT EXPNO PULPROG F I DRES Time NAME NUC1 NS DS WH P1 PL1 SF01

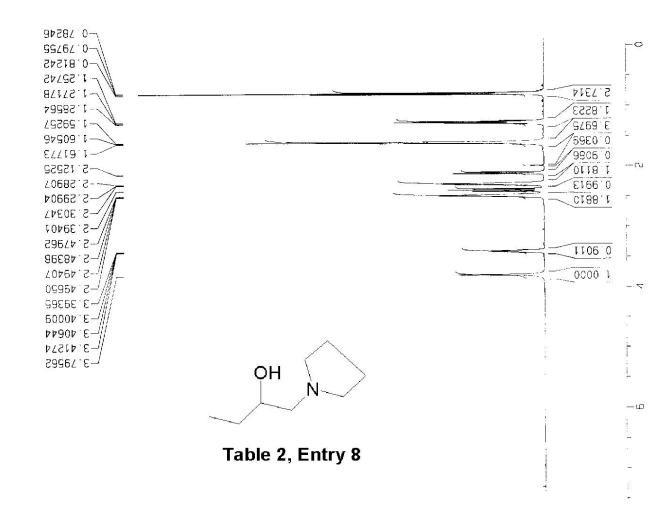




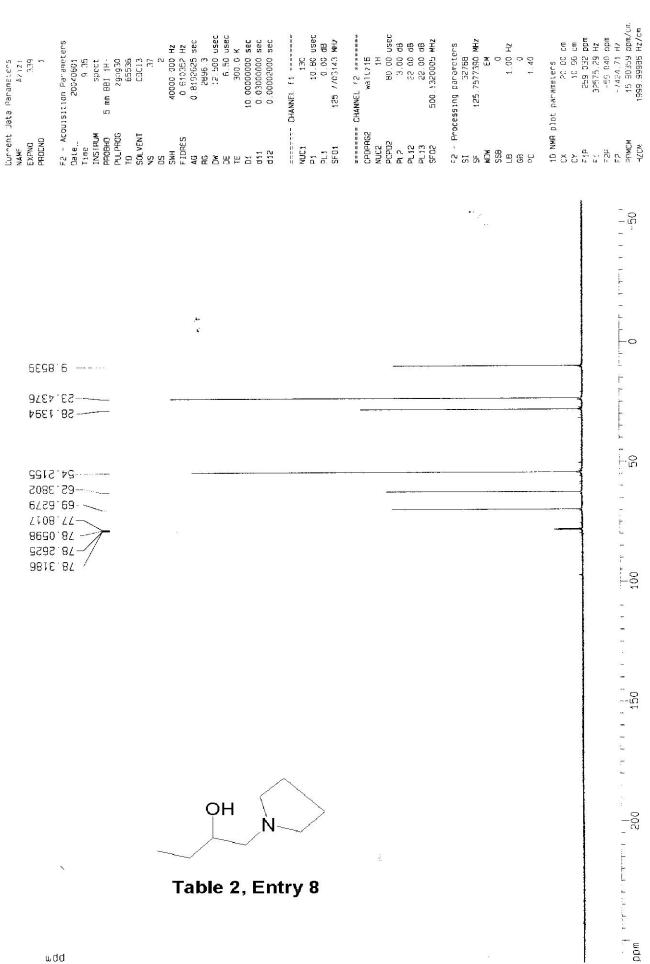
29 13CNMR in CDC13 at 298K 82/10/02



uneters 32768 500.1300000 MHz EM 0.0 0.30 Hz 0.30 Hz 0 352 ppm -181.26 Hz 0 61720 ppm/cm 308.67999 Hz/cm 1D NWH plot parameters CX 18.00 cm CY 11.00 cm F1P 10.747 ppm F1P 5374.98 Hz F2P 0 352 ppm F2P -181.26 Hz PPMCM 0 61720 ppm/cr 6.50 usec 300.0 K 5.0000000 sec 1H 10.50 Usec 3.00 dB 500.1330885 MHz F2 - Processing parameters SI 32768 SF 500.1300000 MHz WDW EM 0 SSB 0.30 H2 GB 0.30 H2 GB 7.00 10330.578 Hz 0.315264 Hz 1.5860696 sec 10.1 48.400 usec CHANNEL f1 -----F2 - Acquisition Parameters Current Data Parameters NAME A7171 EXPND 334 PAOCNO 1 10.40 spect 1.mm 0N¹1H/ 2930 22768 22768 22768 20040525 n PROBHD PULPROG TD SOLVENT NS SWH **INSTRUM** FIDRES Date NUC1 P1 PL1 SF01 Time AG DE DE DE DE DE

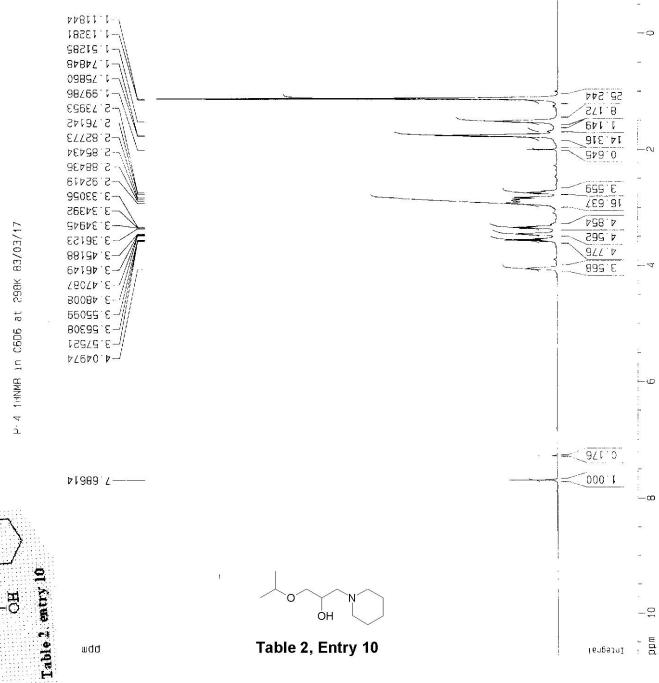


39-1 IHNWH in CDC13 at 298K 83/03/05



udd

500.1300000 MHz 500.1300000 MHz EM 0.30 Hz 0.30 Hz 0.62978 ppm/cm 314.97333 Hz/cm 48.400 usec 6.50 usec 300.0 K 5.0000000 sec 10 NMH plot parameters CX 18.00 cm EY 10.755 ppm F1 5363.69 H2 F2P -0.611 ppm F2P -0.611 ppm F2P -0.612 ppm/cn F2 PPMCM 314.97333 H2/cm 1H 9.30 Usec 3.00 dB 500.1330885 MHz F2 - Processing parameters SI 32768 MDW EM SSB 500.1300000 MHz EM SSB 0.30 Hz C 1.00 PC 1.00 10330.578 Hz 0.315264 Hz 1.5860696 sec CHANNEL f1 ====== F2 - Acquisition Parameters 10.08 spect 5 mm 881 1H-32768 C606 18 0E6z B 19 20040605 Current Oata Parameters Poorhassan 1-----INSTRUM PROBHD PULPROG EXPNO PROCNO Date Time P1 P1 PL1 SF01 NAME



-

10 NMR plot parameters CX 20.00 cm F1P 20.046 pm f1P 2003.19 H2 -25 482 ppm r2 -3204.52 H2 -3204.52 H2 -22 -3204.52 H2 -22 -3204.52 H2 -22 -3204.52 H2 13 2 40000.000 Hz 0.6192625 sec 812.7 12.500 usec 6.50 usec
 Emergence
 Mailt f2
 Emergence

 CPDPRG2
 waltz16
 1H

 NUC2
 B0.00
 usec

 PCPD2
 B0.00
 usec

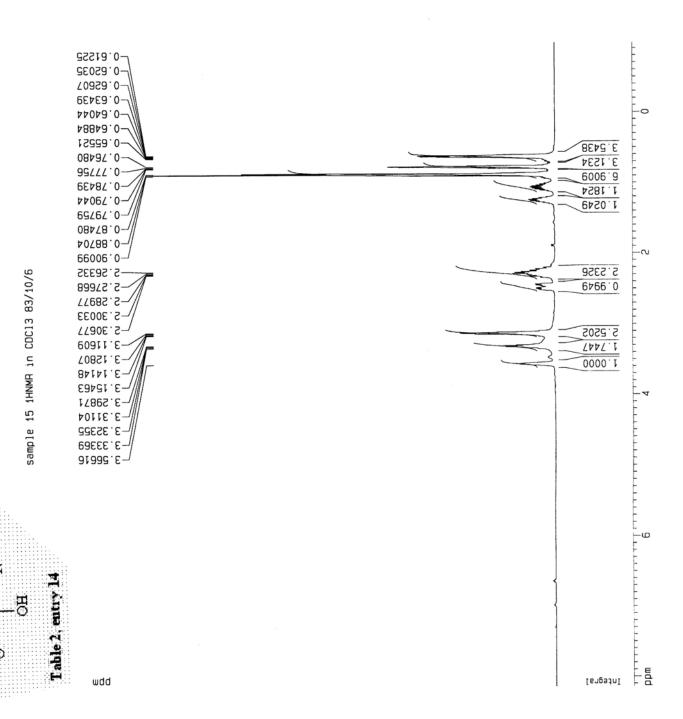
 PL12
 3.00
 dB

 PL12
 22.00
 dB

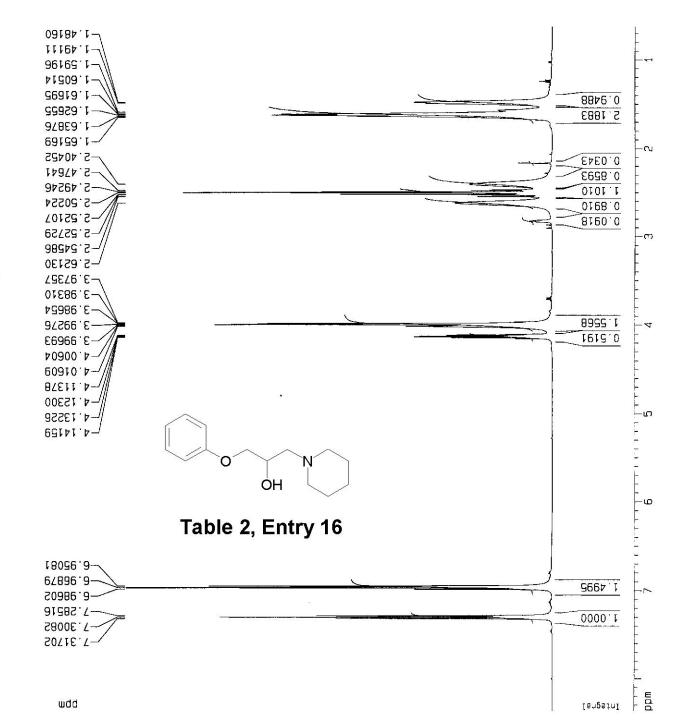
 PL13
 520.00
 dB
 13C 10.80 usec 0.00 dB 125.7703143 MHz 10.0000000 sec 0.03000000 sec 0.00002000 sec CHANNEL f1 -----F2 - Acquisition Parameters Current Data Parameters NAME A7171 EXPNO 358 PROCNO 1 11.02 spect mm BBI 1H-zgpg30 65536 C606 20040609 s Time INSTRUM PROBHD TD SOLVENT NS DS SWH FIDRES PUL PROG Date_ NUC1 P1 PL1 SF01 AQ DF DF d11 d12 0 ŀ - 55.546 -- 53'180 55.262 20 968.42 ----200 150 150 150 rie.5a ----085.88 ----E85.17 -- 72.015 IGL'6L -112.86 — 410.7S1-901.851--128.299 0 он Table 2, Entry 10

91 13CNMR in C6D6 at 298K 83/03/20

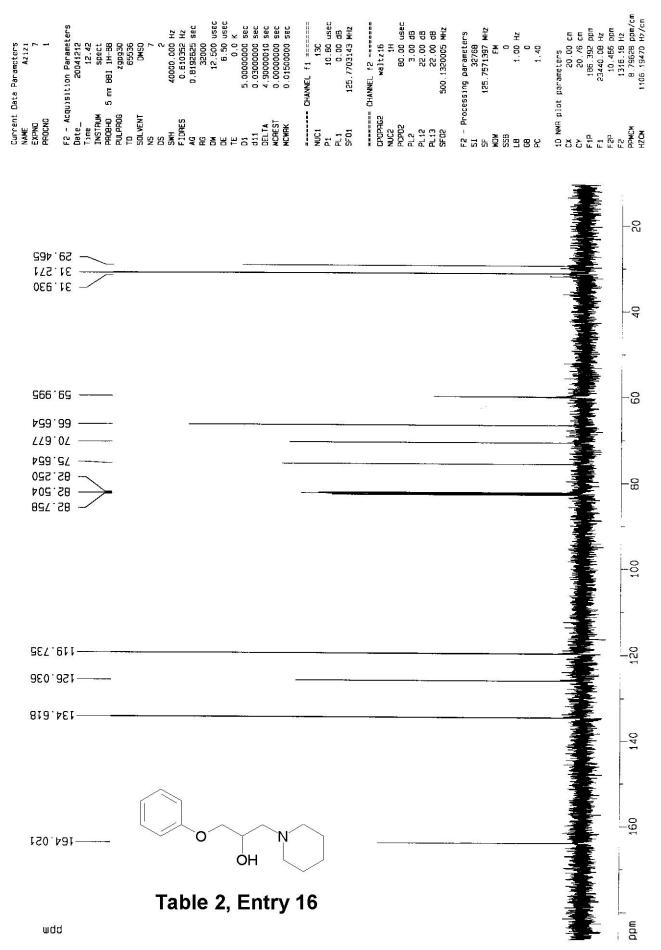
1D NWH plot parameters CX 11.43 cm F1P 00.71.43 cm F1 4071.28 H2 F2P -0.976 ppm F2P -0.976 ppm F2 0.50649 ppm/cm H2CM 253.31247 H2/cm 1H 9.30 USEC 3.00 dB 500.1330885 MHz 10330.578 Hz 0.315264 Hz 1.5860696 sec 48.400 usec 6.50 usec 5.0000000 sec 0.00000000 sec 0.01500000 sec F2 - Processing parameters SI 32768 SF 500.1300000 MHz MDW EM SSB 0.30 Hz CB 0.30 Hz CB 0.30 Hz CB 0.30 Hz ======= CHANNEL f1 ====== F2 - Acquisition Parameters 0.0 K 5 mm BBI 1H-BB 2930 32768 COC13 28.5 Azizi 26 14.57 Current Data Parameters 20041226 PROBHD PULPROG TD SOL VENT NS DS SWH INSTRUM MCREST MCWRK PROCNO FIDRES Date_ EXPNO P1 PL1 SF01 Time NAME NUC1 AC NO H 5



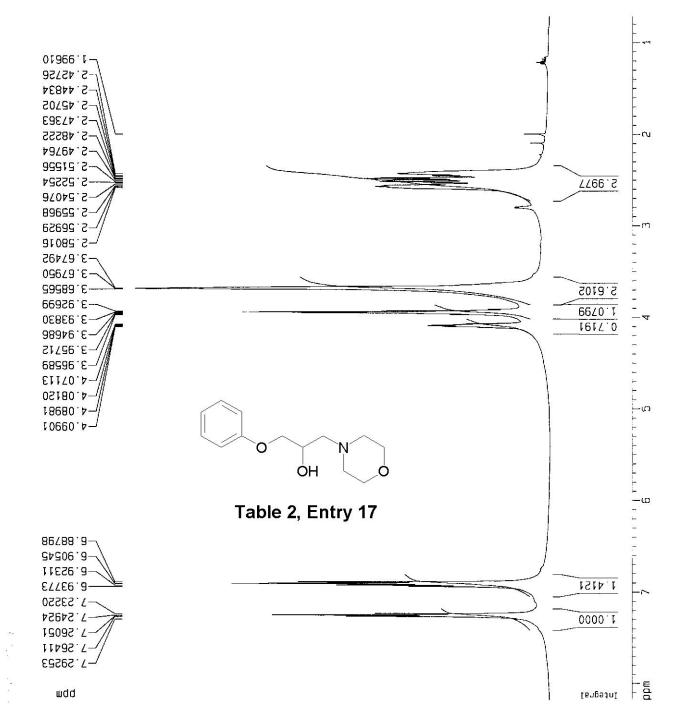
Data Parameters Azizi 6	isition Parameters 20041212 12.41 12.41 5 mm BBI 1H-BB 2g30 32768 11-B 2g30 32768 11-3 4 1.5860695 sec 11.3 48.400 usec 6.50 usec 0.0 K 5.00000000 sec 0.01500000 sec 0.01500000 sec	CHANNEL f1 ======= 1H 9.30 usec 3.00 dB 500.1330885 MHz essing parameters 32768 500.1300000 MHz EM 0 0.30 Hz 0 1.00	1t parameters 18.00 cm 11.32 cm 8.369 ppm 4185.56 Hz 0.623 ppm 311.59 Hz 0.43033 ppm/cn 215.22038 Hz/cm
Current D NAME EXPND PROCNO	F2 - Acqui: Date_ INSTRUM PROBHO 5 PULPROG TD PULPROG TD SOLVENT SOLVENT SOLVENT TD DM DM DM DM DM DM DM DM MCMRK MCMRK	NUCL NUCL SSI SSI CB CB CB CB CB CC CC CC CC CC CC CC CC	10 NMR p1 CX CY F1 F1 F2 PPMCM HZCM

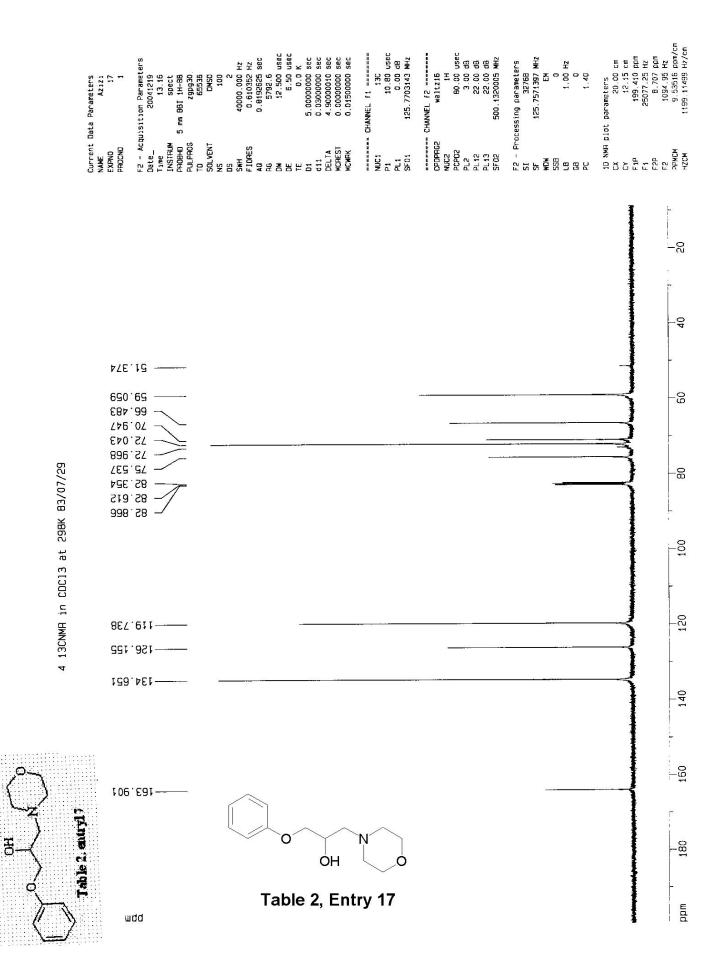


•

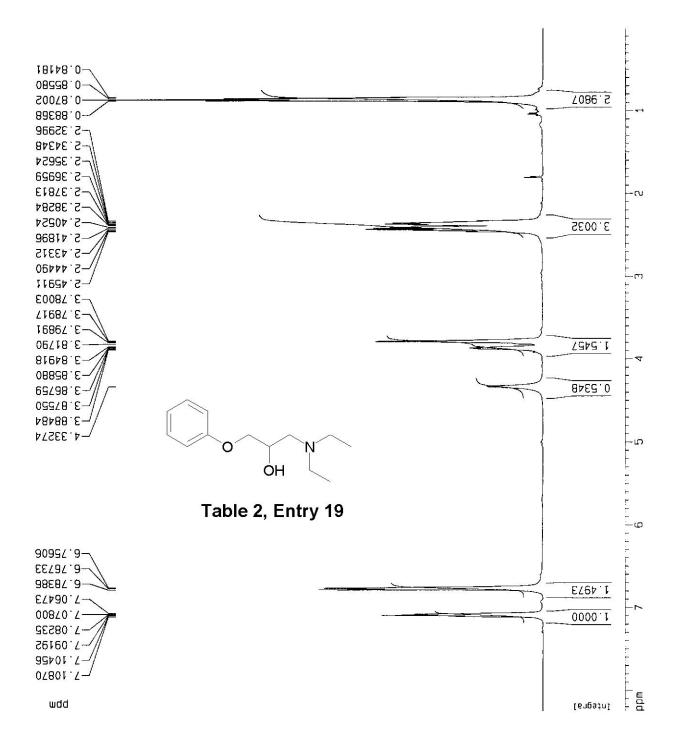


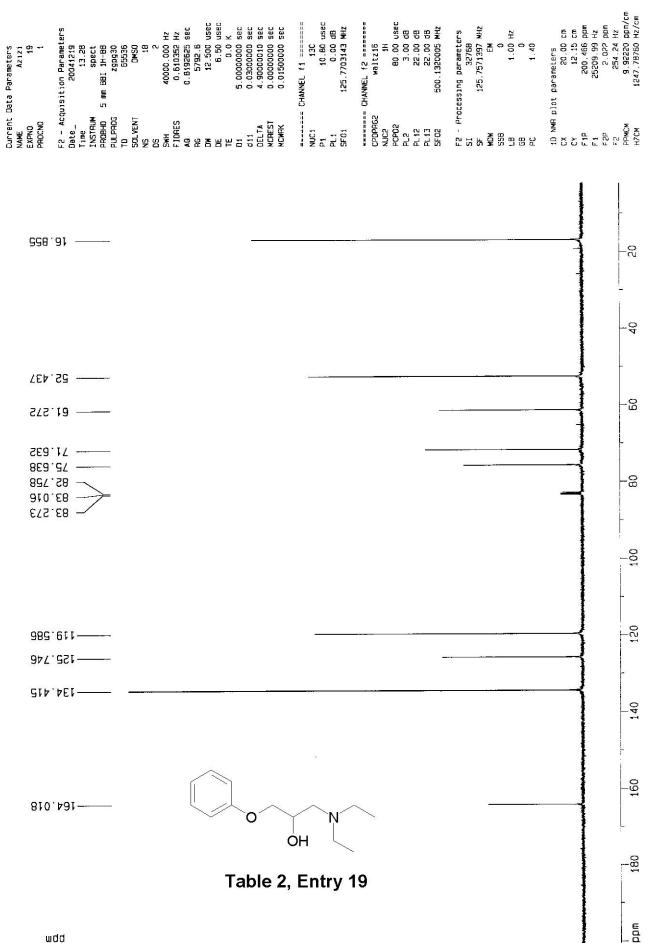
	Lers Hz sec	usec sec sec usec db	PIS MHZ HZ	cm cm Hz Ppm Hz Hz/cm Hz/cm
arameters Azizi 16	Param 13.0 13.0 5pec 5pec 2g3 3276 5276 5276 531526 586069 586069	48.400 48.400 6.50 0.0 0.00000000 0.01500000 0.1500000 0.1500000 0.11 11 11 12 13 0.30 9.30		rameters 18.00 11.00 8.186 4094.14 0.714 357.30 0.41510 0.41510 207.60197
t Data Par	a cquisiti. 6 G mail	5 CHANN(5 CHANN(5 CHANN(5 CHANN(5 CHANN(5 CHANN(5 CHANN(5 CHANN(5 CHANN(5 CHANN(5 CHAN)(5 CHANN(5 CHAN)(5 C	005 200	plot par
Curren NAME EXPNO PROCNO	H L C PRAH	HG DEM MCCHE MC MCCHE MCCHE MCCHE MCCHE MC	PC SSB SSB FC FB FC FB FC FB FC FB FC FB FC FB FC FF FC FC F	10 NMR CX CY F1P F2P F2P F2CM H2CM

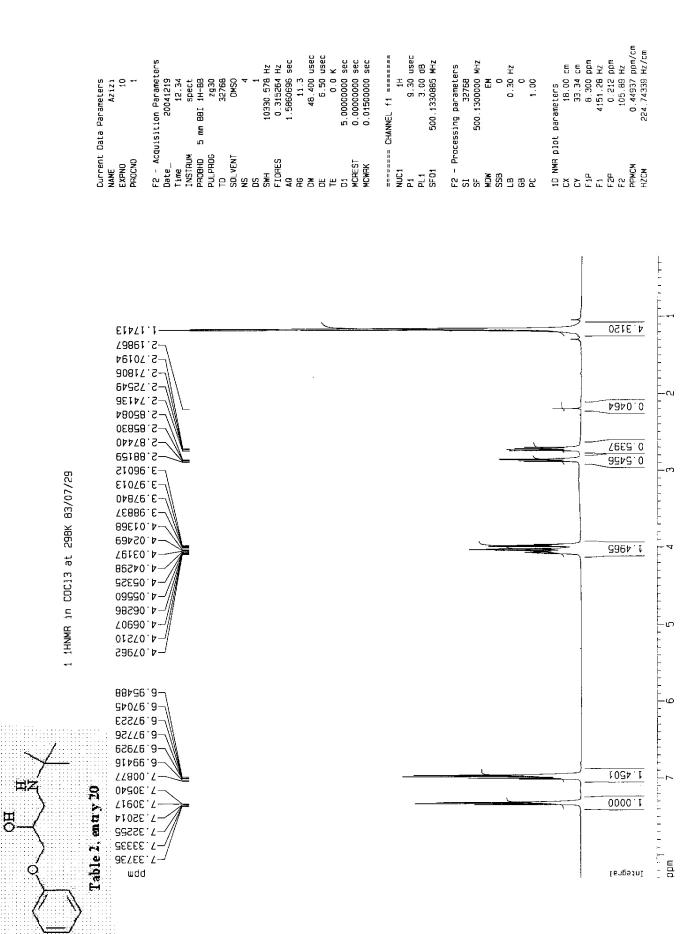


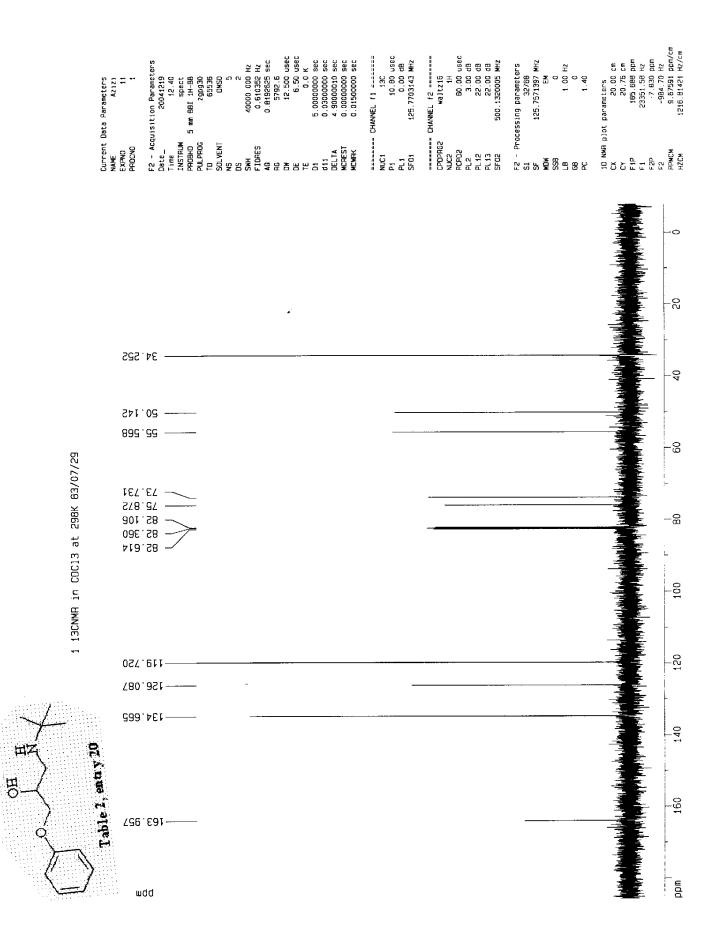


	s	Hz Hz Sec usec Sec Sec Sec	usec dB MHz	ers MHZ Hz	сл сл ррп Hz Hz Ppn/сл Hz/сп
Current Data Parameters NAME Azizi EXPNO 19 PPDCNO 1	F2 - Acquisition Parameti Date_ 20041219 Time 203041219 Time 2031219 Time 2031219 INSTRUM 5 pect PROBHD 5 mm BBI 1H-BB PULPROG 5 2930 TD 32766 SOLVENT CDC13 NS 4	H 10330.578 DRES 0.315264 1.5860696 8 48.400 6.50 0.0 0.0 HEST 0.01500000 HEST 0.01500000	===== CHANNEL f1 ==== 1H 9.30 1 3.00 1 500.1330885	F2 - Processing parameter SI 32768 SF 500.1300000 M NDW EN 0.1300000 M NDW EN 0.1300000 M EN 0.130000 M C 0 0 C 0 0 C 0 0 C 0 0 0 C 0 0 0 0 0 0	10 NMR plot parameters CX 18.00 CY 11.82 F1P 8.255 F1 4128.42 F2P 0.005 F2P 0.45826 F2P 0.45826 F2P 0.45826 F2P 0.45826 F2CM 2.45826

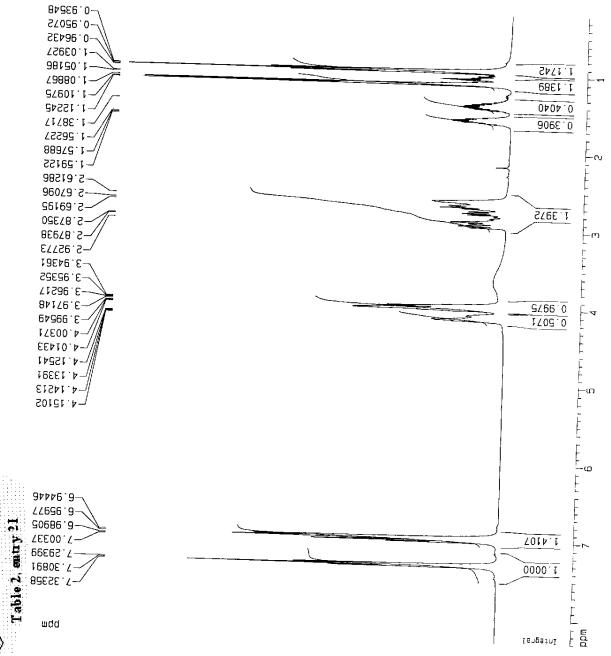








Data Parameters Azizi A	Acquisition Perameters 20041212 12.50 12.50 12.50 2030 2030 2030 2030 2030 1 10330.578 Hz 0.315264 Hz 1.5860695 sec 11.3 4 41.3 6.50 usec 0.0000000 sec 0.0000000 sec	CHANNEL f1 ***********************************	t parameters 18.00 cm 11.09 cm 8.300 ppm 4151.28 H2 0.212 ppm 105.89 H2 105.89 H2 224.74333 H2/cm
Current NAME EXPNO PROCND	F2 - Acc Date Time INSTRUM PROBHO PLLPROG TD PLLPROG TD SWH SWH FIDRES AG BK FIDRES AG DW DE DW DE DW DE DW DC DW DC DW DC DW DC DC DW DC DC DC DC DC DC DC DC DC DC DC DC DC		10 NMR pla CX CY F1P F1 F1 F2P F2P PPMCM AZCM

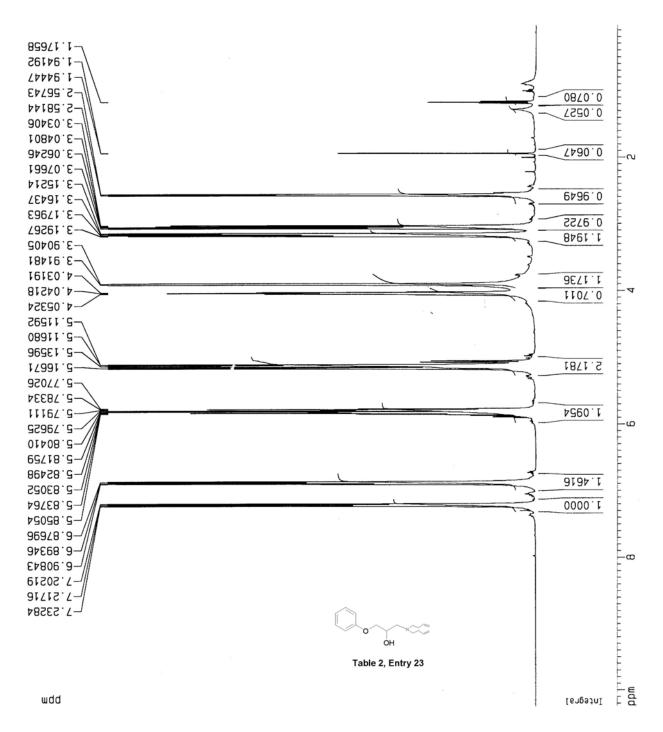


#2 1HNMA in CDC13 at 298K 83/07/22

μZ

HO

	s S	Hz Hz Sec Sec Sec Sec Sec	usec usec ers Hz	ст ст Н2 Ppm H2 H2 H2/ст H2/ст
Parameters Azizi 12	tion Parame 20041219 12.47 5pect m BBI 1H-BB 2930 32768 32768 32768	10330.578 0.315264 1.5860696 8 48.400 6.50 0.0000000 0.00000000 0.01500000	- f1 === 1H 9.30 3.00 3.00 32068 32768 paramet 1330000 0 0 0 0 0 1.000 1.000	parameters 18.00 33.34 10.243 5122.62 0.029 14.47 0.56742 283.78619
Current Data F NAME EXPNO PROCNO	F2 - Acquisit. Date INSTRUM PROBHD 5 mm PULPHOG TD NS NS NS DS DS DS DS DS DS	REST CRES	HILL CHANNEL NUC1 P1 PL1 500 SF01 500 SF01 500 SF2 - Processing SF SSB MDM MDM SSB SSB SSB SSB SSB SSB SSB SSB SSB SS	110 NMR plot p CY CY F1p F1 F2 F2 F2 F2 F2 F2 F2 F2 F2 F2 F2 F2 F2



 F2
 - Processing parameters

 SI
 32768

 SF
 125.757397 MHz

 MDN
 22769

 SSB
 125.7571397 MHz

 SSB
 120.00 Hz

 B
 10.00 Hz

 B
 0

 C
 1.00 Hz

 B
 0

 B
 0

 C
 1.40

 D
 1.40

 C
 20.00 cm

 CY
 20.00 cm

 CY
 20.00 cm

 F1
 21935.66 Hz

 F1
 21935.60 Hz

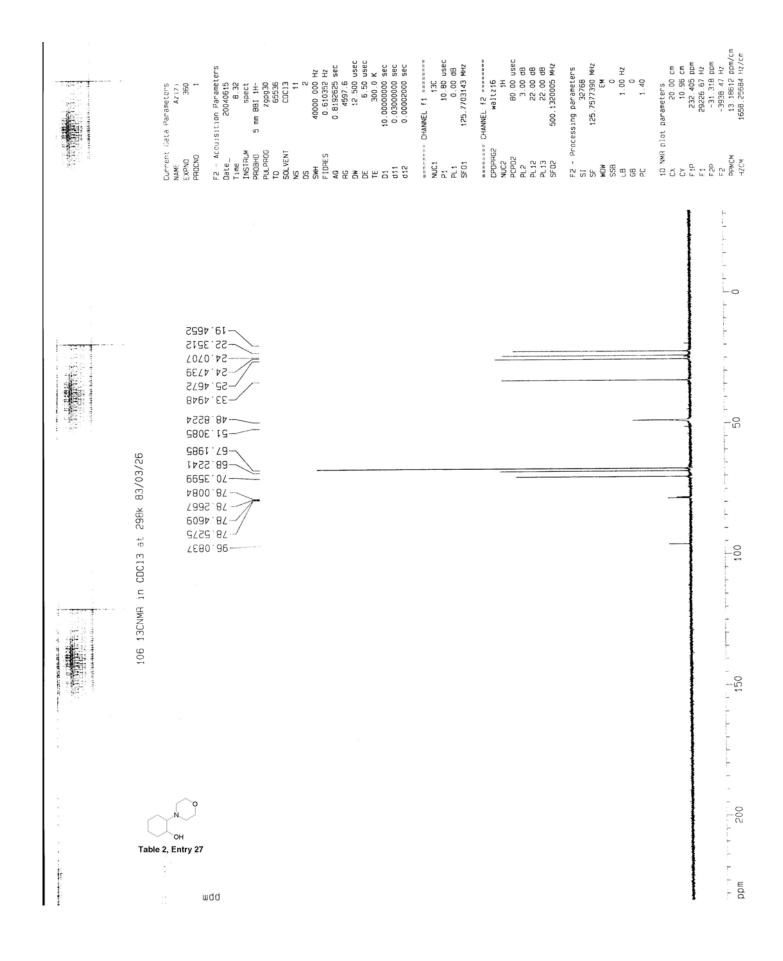
 F2
 4988.75 Hz

 PPMCM
 6.773795 ppm/cm

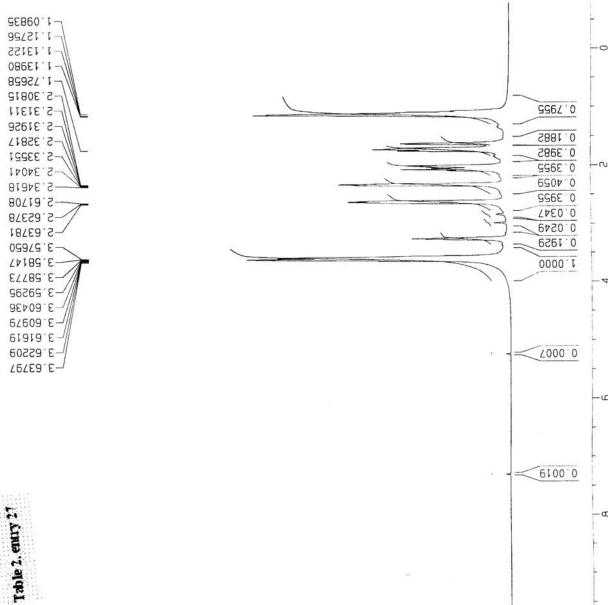
NNEL f2 ======== waltz16 14 B0.00 usec 3.00 dB 22.00 dB 22.00 dB 22.00 dB F2 - Acquisition Parameters Date______20041219 Time______20041219 INSTRUM______22.49 INSTRUM______22.49 PULPHOS_____209430 TD______55536 SOLVENT_____DMS0 13C 10.80 usec 0.00 dB 125.7703143 MHz ----- CHANNEL f1 -----Current Data Parameters NAME Azizi EXPNO 13 PROCNO 1 CHANNEL CPDPRC2 CPDPRC2 NUC2 PCPD2 PC2 PC13 PC13 SF02 SF02 d 11 DELTA MCREST MCMRK NUC1 P1 PL1 SF01 013.32 ------3 801.10 -- 95.406 £6£'99 — 808.17 -S19.27 ----08 - 82.602 198.58 711.58 -100 £02'611-120 -151'322 -153.158 155.948 134 264 -140.370 140 842.041-169.141-0 ÓН 160 Table 2, Entry 23 E10.431-----. mdd

N

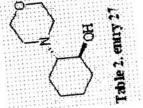
wdd



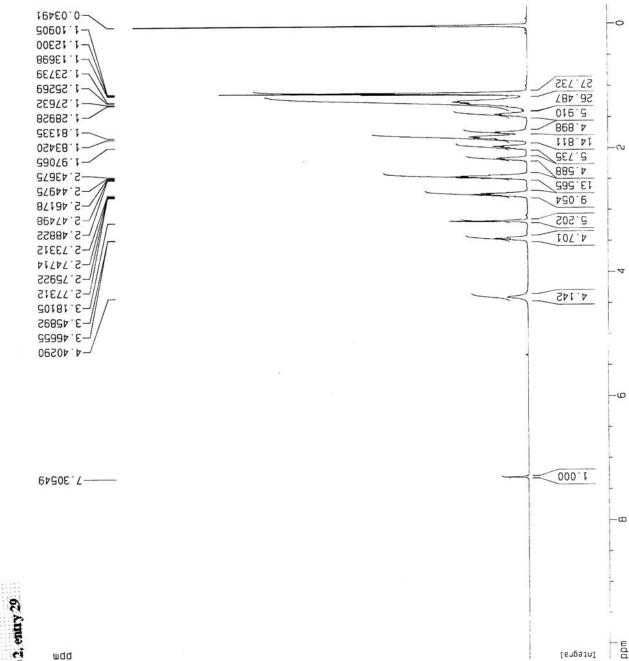
10 NWA plot parameters CY 7.06 cm F1P 10.768 ppm F1 5385.46 Hz F2P -0.794 ppm F2 -0.794 ppm F2 -0.64232 ppm/cm H2CM 321.24341 Hz/cm 1H 9.30 USEC 3.00 dB 500.1330885 MHZ 18 48.400 usec 6.50 usec 300.0 K F2 - Processing parameters SI 32768 SF 500.1300000 MHz WDW EM 0 SSB 0.30 H2 CB 0.30 H2 GB 0.30 H2 10330.578 Hz 0.315264 Hz 1.5860696 sec 5.00000000 sec F2 - Acquisition Parameters Current Data Parameters NAME Azizi EXPND 313 PAOCNO 1 11.10 spect 1H-BB 2930 32768 COC13 8 CHANNEL f1 ==== 20040501 mm 881 ŝ ------PULPROG TD SOLVENT NS DS SWH FIDRES **NURTRUM** рновно Date_ NUC1 P1 PL1 SF01 Time AG BC BC

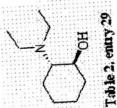


37 1HNMR in CDC13 at 298K 83/02/12



1D NNH plot parameters CX 10.79 cm F1P 10.72 ppm F1 5137.24 H2 F2P -0.304 ppm F2P -152.22 H2 PPMCM 293.85861 H2/Cm 64 48.400 usec 6.50 usec 300.0 K 5.0000000 sec 1H 9.30 Usec 3.00 dB 500.1330885 MHz 2 - Processing parameters 1 32768 F 500.1300000 MHz DW EM 10330.578 Hz 0.315264 Hz 1.5860696 sec 0 0.30 Hz 0 1.00 F2 - Acquisition Parameters 20031214 9.42 spect 5 mm BBI 1H-BB zg30 32768 CDC13 16 CHANNEL f1 ==== 2 AZIZI Current Data Parameters 217 : INSTRUM PROBHD TD SOL VENT NS DS SWH FIDRES PULPROG EXPNO Date_ ----Time PL1 PL1 SF01 NAME AG NO HIG





10 NMR plot parameters CX 11:50 cm 71:50 cm 71:50 cm 71:50 20 pm 71:50 20 pm 71:50 30 Hz 72:20 20 pm 72:20 20 pm 73 Hz 75:0159 Hz/cm CHANNEL F2
 waltz16
 H
 B0 00 UBEC
 3.00 dB
 22.00 dB
 22.00 dB
 520.01320005 MHz - CHANNEL F1 ======= 13C 10.80 usec 0.00 dB 125 //03143 MHz F2 - Processing parameters SI 32769 MHz MDW EM SSB 125.757790 MHz NDW EM SSB 100 Hz 68 100 Hz 68 100 Hz
 F2 - Acquisition Parameters

 Date
 20031215

 Time
 8.28

 INSTRUM
 5.28

 INSTRUM
 5.28

 PAGBHD
 5 mm BB1 14-BB

 PULPHOG
 0.45525

 PULPHOG
 0.45525

 PULPHOG
 0.45525

 PULPHOG
 0.45525

 PULPHOG
 0.45525

 PULPHOG
 0.45525

 PULPHOG
 1.0912.029

 PULPHOG
 0.45525

 PULPHOG
 0.45625

 PULPHOG
 1.0912.020

 PULPHOG
 0.0303000000

 PULPHOG
 0.000000000
 Current Data Parameters NAME Azizi EXPNO 221 PAGCNO 1 6 E M CP0PRG2 NUC2 PCP02 PL12 PL13 PL13 SF02 NUC1 P1 PL1 SF01 100 75 50 50 25 0 - 0.2982 501.11-14.1092 1029.01--23.3488 -54.3815 -24.5292 -25.7008 8667.56-1678.64--23.4233 P858.884 -70.1352 -77.3802 17563.77-S108.77-150 1250 1255 ppm 200 175 Table 2, entry 29 H wdd

Sample (20) 13C, NMR in CDC13 at 298K 82/09/240

References:

- 1. Chini, M.; Crotti, P.; Macchia, F.J. Org. Chem. 1991, 56, 5939.
- 2. Yadav, J. S.; Reddy, B. V. S.; Basak, A. K.; Narsaiah, A. V. Tetrahedron Lett. **2003**, *44*, 1047.
- 3. Chakraborti, A. K.; Rudrawar, S.; Kondaskar, A. Org. Biomol. Chem. 2004, 2, 1277.
- 4. Zhao, P-Q. Xu, L-W. Xia, C-G. Synlett 2004, 846.
- 5. Curini, M.; Epifano, F.; Marcotullio, M. C.; Rosati, O. Eur. J. Org. Chem. 2001, 4149.
- 6. Sagava, S.; Abe, H.; Hase, Y.; Inaba, T. J. Org. Chem. 1999, 64, 4962.
- 7. Mojtahedi, M. M.; Saidi, M. R.; Bolourtchian, M. J. Chem. Res. (S) 1999, 128.
- 8. Ollevier, T.; Lavie-Compin, G. Tetrahedron Lett. 2004, 45, 49.
- 9. Fan, R.-H.; Hou, X.-L. J. Org. Chem. 2003, 68, 726.
- 10. Azizi, N.; Saidi, M. R. Can. J. Chem. 2005, 83, 505.
- 11. Surendra, K.; Krishnaveni, N. S.; Rao, K. R.; Synlett, 2005, 506.