Angewandte Chemie
Eine Zeitschrift der Gesellschaft Deutscher Chemiker

Supporting Information

69451 Weinheim, Germany

Highly Stereoselective Intermolecular Formal [3+3] Cycloaddition Reaction of Cyclic Enamines and Enones

Mohammad Movassaghi* and Bin Chen

Massachusetts Institute of Technology, Department of Chemistry, Massachusetts 02139

General Procedures. All reactions were performed in oven-dried or flame-dried round bottomed flasks or modified Schlenk (Kjeldahl shape) flasks. The flasks were fitted with rubber septa and reactions were conducted under a positive pressure of argon. Stainless steel syringes or cannulae were used to transfer air- and moisture-sensitive liquids. Flash column chromatography was performed as described by Still et al. using silica gel (60-Å pore size, 32–63 μm, standard grade, Sorbent Technologies) or non-activated alumina gel (80–325 mesh, chromatographic grade, EM Science).[1] Analytical thin–layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel or neutral alumina gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light and/or by exposure to an ethanolic phosphomolybdic acid (PMA), an acidic solution of *p*-anisaldehyde (anis), an aqueous solution of ceric ammonium molybdate (CAM), an aqueous solution of potassium permanganate ($KMnO_4$) or an ethanolic solution of ninhydrin followed by heating (<1 min) on a hot plate (~250 °C). Organic solutions were concentrated on Büchi R-200 rotary evaporators at ~20 Torr (house vacuum) at 25–35 °C, then at ~1 Torr (vacuum pump) unless otherwise indicated.

Materials. Commercial reagents and solvents were used as received with the following exceptions: Dichloromethane, diethyl ether, tetrahydrofuran, acetonitrile, and toluene were purchased from J.T.

[1] Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923–2925.

Baker (Cycletainer™) and were purified by the method of Grubbs et al. under positive argon pressure,[2] CuBr•Me_2S was purchased from Aldrich Chemicals and stored dry in a glove box. The molarity of *n*-butyllithium solutions was determined by titration using diphenylacetic acid as an indicator (average of three determinations).[3] Commercially available benzenethiol, cyclohex-2-enone, cyclopent-2-enone, 2-methylcyclopent-2-enone, 3-methylcyclopent-2-enone, 5-methyl-3,4-dihydro-2*H*-pyrrole, 2-methylpiperidine, and L-proline were purchased and used as received.

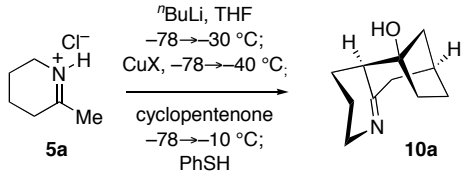
Instrumentation. Proton nuclear magnetic resonance (^{1}H NMR) spectra were recorded with a Varian inverse probe 500 INOVA spectrometer. Chemical shifts are recorded in parts per million from internal tetramethylsilane on the δ scale and are referenced from the residual protium in the NMR solvent ($CHCl_3$: δ 7.27, C_6H_6: δ7.16). Data is reported as follows: chemical shift [multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant(s) in Hertz, integration, assignment]. Carbon-13 nuclear magnetic resonance spectra were recorded with a Varian 500 INOVA spectrometer and are recorded in parts per million from internal tetramethylsilane on the δ scale and are referenced from the carbon resonances of the solvent ($CDCl_3$: δ 77.2, benzene-d_6: δ 128.0). Data is reported as follows: chemical shift [multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant(s) in Hertz, assignment]. Infrared data were obtained with a Perkin-Elmer 2000 FTIR and are reported as follows: [frequency of absorption (cm^{-1}), intensity of absorption (s = strong, m = medium, w = weak, br = broad), assignment]. Gas chromatograpy was performed on an Agilent Technologies 6890N Network GC System with a HP-5 5% Phenyl Methyl Siloxane column. Enantiomeric excess was determined by chiral HPLC analysis on Agilent Technologies 1100 Series HPLC system. We are grateful for the assistance of Dr. Peter Mueller (X-ray Crystallographic Laboratory, Department of Chemistry, Massachusetts Institute of Technology), Dr. Richard J. Staples (X-ray Crystallographic laboratory, Harvard University), and Mr. Michael A. Schmidt for assistance with crystal structures described here. We are grateful to Dr. Li Li for obtaining mass spectrometric data at the Department of Chemistry's Instrumentation Facility, Massachusetts Institute of Technology. Optical Rotation was recorded on a Jasco P-1010 Polarimeter.

[2] Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518–1520.
[3] Kofron, W. G.; Baclawski, L. M. *J. Org. Chem.* **1976**, *41*, 1879–1880.

Supplementary notes:

- The optimal organocuprate was prepared from CuBr•SMe_2 complex (Table S1).

Table S1. Copper Promoted Formal [3+3] Cycloaddition.



entry	Cu	CuX (equiv)	**5a** (equiv)	^{n}BuLi (equiv)	isolated yield (%)
1	CuBr•SMe_2	1.0	2.0	4.0	30
2	***CuBr·SMe₂***	***1.5***	***3.0***	***6.0***	***82***
3	CuCN	1.5	3.0	6.0	<5
4	CuI	1.5	3.0	6.0	20
5	Lipshutz reagents	1.5	1.5	3.0	30

Table S2. Rate of Deuterium Incorporation.[a]

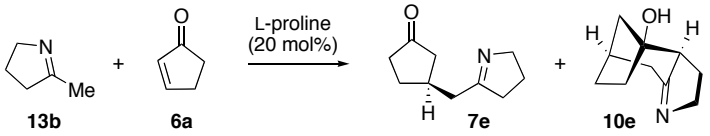
entry	imine	time (h)	position	*d*-incorporation (%)
1	**13a**	0.17	C3	95
2	**13a**	0.17	C2-Me	20
3	**13a**	9	C3	>99
4	**13a**	9	C2-Me	90
5	**13b**	15	C3	<1
6	**13b**	15	C2-Me	20
7	**13b**	96	C3	<25
8	**13b**	96	C2-Me	90

[a] Conditions: DMSO-d_6–D_2O (3:2), 23 °C.

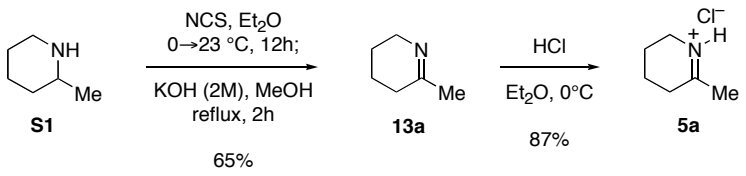
- Comparison of the rate of deuterium incorporation with imines **13a** and **13b** (Table S2).

- A series of potential catalysts (for example: 5-benzyl-2,2,3-trimethyl-4-oxo-imidazolidin-1-ium chloride, 5-pyrrolidin-2-yl-1*H*-tetrazole, and 2-[diphenyl-(trimethyl-silanyloxy)-methyl]-pyrrolidine) and reaction conditions were examined and the use of proline as catalyst in chloroform was found to be most effective. Under identical conditions (TFE, 80 °C), the use of pyrrolidine, acetic acid, or pyrrolidinium acetate as catalyst provided less than 5% yield of **7e** (Table S3).

Table S3. Catalytic Asymmetric Conjugate Addition of **13b** to **6a**.



entry	solvent	temp (°C)	time (h)	yield (%)	%ee of **10e**
1	MeCN, H_2O	50	20	**7e**, 31; **10e**, 23	<5
2	DMSO, H_2O	80	20	**10e**, 35	15
3	TFE, H_2O	80	20	**7e**, 40; **10e**, 21	<5
4	DMF, H_2O	80	20	**7e**, 25; **10e**, 33	17
5	Acetone	50	20	**7e**, 25;	20
6	***CH_3Cl***	***45***	***48***	***7e, 50;***	***52***
7	CH_3Cl	23	48	**7e**, 18;	35
8	CH_3Cl	45	48	**7e**, 37;	41

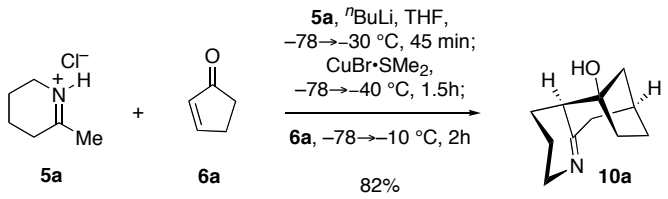


6-Methyl-2,3,4,5-tetrahydro-pyridine (13a) and 6-Methyl-2,3,4,5-tetrahydro-pyridinium chloride (5a):

2-Methylpiperidine (**S1**, 30.0 mL, 252 mmol, 1 equiv) was drop-wise added to a solution of *N*-chlorosuccinimide (NCS, 35.0 g, 258 mmol, 1.03 equiv) in Et_2O (300 mL) at 0 ºC. After 12 h, the reaction mixture was filtered, and the resulting filtrate was concentrated under reduced pressure at 23 °C. The residue was suspended in diethylether (100 mL), the mixture filtered, and the filtrate concentrated. A cold solution of potassium hydroxide in methanol (2M, 252 mL, 504 mmol, 2.0 equiv, 0 °C) was slowly added to the crude *N*-chloropiperidine sample. The reaction mixture was heated to reflux. After 2 h, the reaction mixture was allowed to cool to 23 °C, filtered, and concentrated to a minimum volume under reduced pressure. The viscous orange oil was dissolved in aqueous sodium hydroxide solution (1M, 50 mL) and extracted with ethyl acetate (3 × 150 mL). The combined organic layers were dried over anhydrous sodium sulfate, and concentrated under reduced pressure. The orange viscous liquid was distilled to afforded 6-methyl-2,3,4,5-tetrahydropyridine (**13a**, 16.0 g, 65%).[4] A solution of hydrogen chloride in Et_2O (2M, 99 mL, 198 mmol, 1.20 equiv) was added drop-wise to a solution of 6-methyl-2,3,4,5-tetrahydropyridine (16.0 g, 165 mmol, 1.00 equiv) in Et_2O (200 mL) at 0 ºC. After 10 min, the mixture separated into two distinct layers (a clear and colorless ethereal top layer and a pink bottom layer). The clear ethereal layer was decanted, and the pink bottom layer was concentrated under reduced pressure to afford a semi-solid. This residue was dried under reduced pressure (0.5 Torr) for 12 h at 50 ºC to remove the excess hydrogen chloride. The resulting dry pink-brown solid was dissolved in CH_2Cl_2 (20 mL) and this solution was added drop-wise to a vigorously stirred volume of Et_2O (150 mL) at 0 ºC. The ethereal layer was decanted and the resulting solid iminium chloride **5a** was dried under reduced pressure. Iminium chloride 5a was dried by concentration from benzene (3 × 100 mL) and was further dried under vacuum (0.5 Torr) for 24 h to afford a grayish white solid (19.0 g, 87%).

^{1}H NMR (500 MHz, $CDCl_3$, 20ºC):	3.68 (bs, 2H, $CH_2C\mathbf{H}_2N(H)=C$), 2.70 (bs, 2H, $CH_2C\mathbf{H}_2C=N(H)$), 2.60 (s, 3H, $C\mathbf{H}_3$), 1.80-1.96 (m, 4H, $C\mathbf{H}_2CH_2N(H)=C$, $C\mathbf{H}_2CH_2C=N(H)$).
^{13}C NMR (500 MHz, $CDCl_3$, 20ºC):	188.2, 44.8, 32.1, 25.1, 20.0, 17.8.
FTIR (neat):	3399 (br, N–H), 2960 (s, C–H), 1698 (s, C=N), 1639, 1452, 1387.
HRMS–ESI (*m*/*z*):	calcd for $C_6H_{12}N$ $[M\text{-}Cl]^+$: 98.0970, found: 98.0966
TLC (50% hexanes in acetone), *Rf*:	0.3 (ninhydrin)

[4] For prior syntheses of imine **13a**, see: T. N. Van, N. De Kimpe, *Tetrahedron*. **2000**, *56*, 7969 and B. G. Davis, M. A. T. Maughan, T. M. Chapman, R. Villard, S. Courtney, *Org. Lett*. **2002**, *4*, 103.

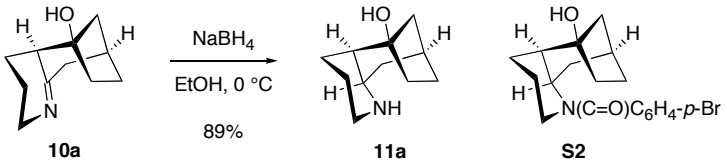


6-Aza-tricyclo[7.2.1.$0^{0,0}$]dodec-6-en-1-ol (10a, Scheme 2):

A solution of *n*-butyl lithium in hexanes (2.49 M, 2.40 mL, 6.00 mmol, 6.00 equiv) was added drop wise via gas–tight syringe to a well-stirred suspension of the iminium chloride salt **5a** (400 mg, 3.00 mmol, 3.00 equiv) in THF (3.0 mL) at –78 °C under an argon atmosphere. The resulting reaction mixture was allowed to gradually warm to –30 °C over 45 min. After which the reaction mixture turned into a clear yellow solution indicating the complete dissolution of the iminium chloride salt. The reaction mixture was then cooled back to –78 °C and the clear yellow solution of the lithioenamine was transferred via cannula under positive argon pressure to a stirring suspension of copper bromide–dimethyl sulfide (308 mg, 1.50 mmol, 1.50 equiv) in THF (2.0 mL) at –78 °C. The resulting red-orange reaction mixture was allowed to gradually warm to –40 °C over 1.5 h. The resulting clear and homogeneous red-orange solution was cooled to –78 °C and a solution of cyclopentenone (**6a**, 85 μL, 1.0 mmol, 1 equiv) in THF (2.0 mL) introduced. The slightly darker solution was allowed to warm to –10 °C over 2 h. A solution of thiophenol (0.16 mL, 1.5 mmol, 1.5 equiv) in absolute ethanol (200 proof, 1.0 mL) was added to the reaction mixture followed by dilution of the resulting mixture with an aqueous ammonium hydroxide–saturated aqueous ammonium chloride solution (1:9, 5 mL) and warming of the mixture to 0 °C. After 1 h of vigorous stirring at 0 °C, the mixture was extracted with dichloromethane (3 × 20 mL) and the combined organic layers were then dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure on a rotary evaporator. Purification of the residue by flash column chromatography (silica gel; 5% MeOH 2% Et_3N in CH_2Cl_2) afforded the desired product **10a** (147 mg, 82%) as an oil. The relative stereochemistry of imino alcohol **10a** was secured by X-ray crystallographic analysis of the corresponding amino alcohol derivative **S2** (see page S6).

^{1}H NMR (500 MHz, $CDCl_3$, 20ºC):	3.68-3.76 (m, 1H, CH_2CH_2N=C), 3.37-3.47 (m, 1H, CH_2CH_2N=C), 2.42-2.49 (m, 1H, CH_2CHC(OH)), 2.32-2.38 (m, 2H, CHCH_2C(OH), CHCH_2C=N), 2.18-2.25 (m, 1H, CHCH_2C=N), 1.92-2.08 (m, 3H, CH_2CH_2N=C, CH_2CHC(OH), CHCH_2C(OH)), 1.76-1.88 (m, 3H, CHCH_2C(OH), CH_2CH_2C(OH), CH_2CH_2C(OH)), 1.38-1.60 (m, 4H, CH_2CH_2C(OH), CH_2CH_2C(OH), CH_2CH_2N=C , CH_2CHC(OH)).
^{13}C NMR (500 MHz, $CDCl_3$, 20ºC):	170.5, 81.3, 50.9, 49.7, 47.2, 45.6, 33.3, 32.0, 28.3, 21.9, 20.8.
FTIR (neat):	3416 (br, O–H), 2941 (C–H), 1653 (s, C=N), 1446, 1320.
HRMS–ESI (*m*/*z*):	calcd for $C_{11}H_{18}NO$ $[M+H]^+$: 180.1388, found: 180.1378

TLC (5% MeOH and 2% Et_3N in CH_2Cl_2): 0.3 (Ninhydrin)



10a **11a** **S2**

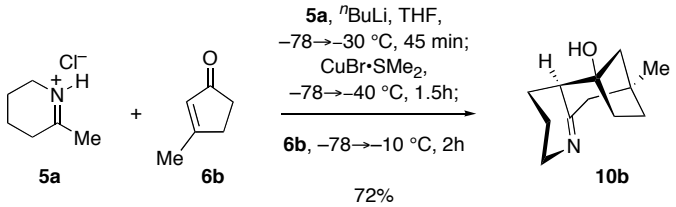
6-Aza-tricyclo[7.2.1.0^{0,0}]dodecan-1-ol (11a, Scheme 2):

Sodium borohydride (14.5 mg, 0.380 mmol, 1.50 equiv) was added to a solution of imino alcohol **10a** (45 mg, 0.25 mmol, 1 equiv) in absolute ethanol (200 proof, 2.5 mL, 0.1 M) at 0 ºC. After 20 min, the excess hydride was quenched by addition of aqueous saturated ammonium chloride solution (3 mL) and the resulting mixture was concentrated under reduced pressure. The residue was diluted with aqueous sodium hydroxide solution (1N, 3 mL) and extracted with dichloromethane (3 × 20 mL). The combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel; 10% MeOH and 2% Et_3N in CH_2Cl_2) afforded the desired product **11a**[5] (40 mg, 89%) as a viscous oil. The relative stereochemistry of amino alcohol **11a** was secured by X-ray crystallographic analysis of the corresponding *N*-4-bromobenzoyl derivative **S2** (see page S31).

^{1}H NMR (500 MHz, C_6D_6, 20ºC):	2.80-2.88 (m, 1H, CH_2CH_2(NH)), 2.80 (app-t, 1H, *J*= 5.3 Hz, CH_2CH(NH)), 2.66-2.74 (m, 1H, CH_2CH_2C(OH)), 2.41-2.49 (m, 1H, CH_2CH_2(NH)), 2.28-2.36 (m, 1H, CH_2CH_2(NH)), 2.03-2.09 (m, 1H, $CHCH_2C$(OH)), 1.91-1.99 (m, 1H, CH_2CH_2C(OH)), 1.76-1.88 (m, 2H, CH_2CH_2C(OH), CH_2CHC(OH)), 1.65-1.70 (m, 1H, $CHCH_2C$(OH)), 1.60-1.65 (m, 1H, CH_2CHC(OH)), 1.51-1.59 (m, 1H, CH_2CH(NH)), 1.40-1.48 (m, 1H, CH_2CH_2C(OH)), 1.16-1.28 (m, 3H, CH_2CH_2(NH), CH_2CHC(OH), $CHCH_2C$(OH)), 1.02 (app-d, 1H, *J*=14.0 Hz, CH_2CH(NH)).
^{13}C NMR (500 MHz, C_6D_6, 20ºC):	80.4, 56.5, 50.0, 48.6, 46.3, 41.1, 36.0, 35.9, 29.5, 24.4, 23.8.
FTIR (neat):	3346 (br, O–H), 2935 (w, C–H), 1452, 1318, 1101.
HRMS–ESI (*m*/*z*):	calcd for $C_{11}H_{20}NO$ $[M + H]^+$: 182.1545, found: 182.1537

TLC (50% MeOH and 2% Et_3N in CH_2Cl_2), *Rf*: 0.2 (Ninhydrin)

[5] We thank Mr. Robert W. Sindelar for assistance with initial spectroscopic analysis of **11a**.

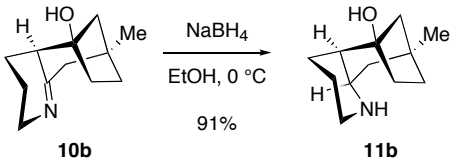


9-Methyl-6-aza-tricyclo[7.2.1.0^{0,0}]dodec-6-en-1-ol (10b, Table 1, Entry 1):

A solution of *n*-butyl lithium in hexanes (2.49 M, 2.40 mL, 6.00 mmol, 6.00 equiv) was added drop wise via gas–tight syringe to a well-stirred suspension of the iminium chloride salt **5a** (400 mg, 3.00 mmol, 3.00 equiv) in THF (3.0 mL) at –78 °C under an argon atmosphere. The resulting reaction mixture was allowed to gradually warm to –30 °C over 45 min. After which the reaction mixture turned into a clear yellow solution indicating the complete dissolution of the iminium chloride salt. The reaction mixture was then cooled back to –78 °C and the clear yellow solution of the lithioenamine was transferred via cannula under positive argon pressure to a stirring suspension of copper bromide–dimethyl sulfide (308 mg, 1.50 mmol, 1.50 equiv) in THF (2.0 mL) at –78 °C. The resulting red-orange reaction mixture was allowed to gradually warm to –40 °C over 1.5 h. The resulting clear and homogeneous red-orange solution was cooled to –78 °C and a solution of 3-methylcyclopentenone (**6b**, 101 μL, 1.00 mmol, 1 equiv) in THF (2.0 mL) introduced. The slightly darker solution was allowed to warm to –10 °C over 2 h. A solution of thiophenol (0.16 mL, 1.50 mmol, 1.50 equiv) in absolute ethanol (200 proof, 1.0 mL) was added to the reaction mixture followed by dilution of the resulting mixture with an aqueous ammonium hydroxide–saturated aqueous ammonium chloride solution (1:9, 5 mL) and warming of the mixture to 0 °C. After 1 h of vigorous stirring at 0 °C, the mixture was extracted with dichloromethane (3 × 20 mL) and the combined organic layers were then dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure on a rotary evaporator. Purification of the residue by flash column chromatography (silica gel; 5% MeOH and 2% Et_3N in CH_2Cl_2) afforded the desired product **10b** (140 mg, 72%) as a viscous oil.

^{1}H NMR (500 MHz, $CDCl_3$, 20°C):	3.66-3.74 (m, 1H, CH_2CH_2N=C), 3.35-3.45 (m, 1H, CH_2CH_2N=C), 2.37-2.43 (m, 1H, CH_2CHC=N), 2.19 (dq, *J* = 14.5, 3.2 Hz, 1H, C(CH_3)CH_2C=N), 2.09 (dd, *J* = 14.5, 2.7 Hz, 1H, C(CH_3)CH_2C=N), 1.94-2.01 (m, 1H, CH_2CHC=N), 1.91 (dd, *J* = 10.9, 2.7 Hz, 1H, C(OH)CH_2C(CH_3)), 1.74-1.85 (m, 2H, CH_2CH_2N=C, CH_2CH_2C(OH)), 1.67 (dt, *J* = 10.9, 2.2 Hz, 1H, C(OH)CH_2C(CH_3)), 1.38-1.64 (m, 5H, CH_2CH_2C(OH), CH_2CH_2C(OH), CH_2CH_2C(OH), CH_2CH_2N=C, CH_2CHC=N), 1.08 (s, 3H, C(CH_3)CH_2C=N).
^{13}C NMR (125.7 MHz, $CDCl_3$, 20°C):	170.3, 82.0, 54.5, 53.1, 50.3, 50.2, 39.9, 36.0, 33.8, 27.4, 22.3, 21.1.
FTIR (neat):	3376 (br, O–H), 2944 (s, C–H), 1655 (s, C=N), 1452, 1276.
HRMS–ESI (*m*/*z*):	calcd for $C_{12}H_{19}NO$ $[M]^+$: 193.1467, found: 193.1464

TLC (5% MeOH and 2% Et_3N in CH_2Cl_2), *Rf*: 0.3 (Ninhydrin)



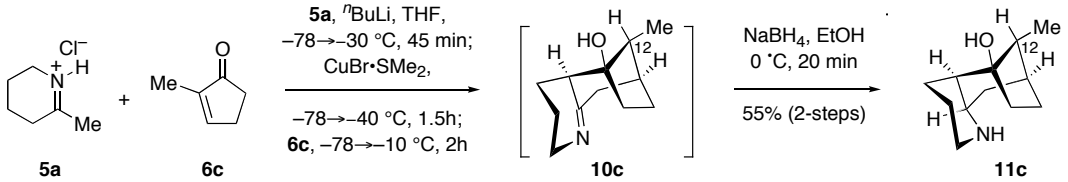
10b **11b**

9-Methyl-6-aza-tricyclo[7.2.1.$0^{0,0}$]dodecan-1-ol (11b, Table 1, Entry 1):

Sodium borohydride (16 mg, 0.43 mmol, 1.5 equiv) was added to a solution of imino alcohol **10b** (55 mg, 0.28 mmol, 1 equiv) in absolute ethanol (200 proof, 2.8 mL, 0.1 M) at 0 ºC. After 20 min, the excess hydride was quenched by addition of aqueous saturated ammonium chloride solution (3 mL) and the resulting mixture was concentrated under reduced pressure. The residue was diluted with aqueous sodium hydroxide solution (1N, 3 mL) and extracted with dichloromethane (3 × 20 mL). The combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel; 10% MeOH and 2% Et_3N in CH_2Cl_2) afforded the desired product **11b** (50 mg, 91%) as a very viscous oil.

^{1}H NMR (500 MHz, C_6D_6, 20°C):	2.80-2.86 (m, 1H, $CH_2C\mathbf{H}_2(NH)$), 2.79 (appt, 1H, *J*= 5.4 Hz, $CH_2C\mathbf{H}(NH)$), 2.66-2.72 (m, 1H, $CH_2C\mathbf{H}_2C(OH)$), 2.37-2.45 (m, 1H, $C\mathbf{H}_2CH_2(NH)$), 2.29-2.36 (m, 1H, $CH_2C\mathbf{H}_2(NH)$), 1.90-1.97 (m, 1H, $C\mathbf{H}_2CH_2C(OH)$), 1.71-1.83 (m, 1H, $C\mathbf{H}_2CHC(OH)$), 1.53-1.58 (m, 1H, $CH_2C\mathbf{H}C(OH)$), 1.40-1.52 (m, 3H, $C(CH_3)C\mathbf{H}_2C(OH)$, $CH_2C\mathbf{H}_2C(OH)$, $C\mathbf{H}_2CH_2C(OH)$), 1.35-1.40 (m, 1H, $C\mathbf{H}_2CH(NH)$), 1.16-1.26 (m, 2H, $C\mathbf{H}_2CH_2(NH)$, $C\mathbf{H}_2CHC(OH)$), 1.01-1.06 (m, 1H, $C(CH_3)C\mathbf{H}_2C(OH)$), 0.92 (s, 3H, $C(C\mathbf{H}_3)CH_2C(OH)$), 0.90 (dd, *J*= 13.9, 2.5 Hz, 1H, $C\mathbf{H}_2CH(NH)$).
^{13}C NMR (125.7 MHz, C_6D_6, 20°C):	80.8, 56.7, 56.3, 48.5, 48.2, 45.4, 40.6, 37.2, 36.4, 28.8, 24.2, 23.7.
FTIR (neat):	3394 (br, O–H), 2942 (w, C–H), 1646, 1452, 1310.
HRMS–ESI (*m*/*z*):	calcd for $C_{12}H_{22}NO$ $[M+H]^+$: 196.1701, found: 196.1691

TLC (50% MeOH and 2% Et_3N in CH_2Cl_2), *Rf*: 0.2 (Ninhydrin)



12-Methyl-6-aza-tricyclo[7.2.1.0^{0,0}]dodecan-1-ol (11c, Table 1, Entry 2):

A solution of *n*-butyl lithium in hexanes (2.49 M, 2.40 mL, 6.00 mmol, 6.00 equiv) was added drop wise via gas–tight syringe to a well-stirred suspension of the iminium chloride salt **5a** (400 mg, 3.00 mmol, 3.00 equiv) in THF (3.0 mL) at –78 °C under an argon atmosphere. The resulting reaction mixture was allowed to gradually warm to –30 °C over 45 min. After which the reaction mixture turned into a clear yellow solution indicating the complete dissolution of the iminium chloride salt. The reaction mixture was then cooled back to –78 °C and the clear yellow solution of the lithioenamine was transferred via cannula under positive argon pressure to a stirring suspension of copper bromide–dimethyl sulfide (308 mg, 1.50 mmol, 1.50 equiv) in THF (2.0 mL) at –78 °C. The resulting red-orange reaction mixture was allowed to gradually warm to –40 °C over 1.5 h. The resulting clear and homogeneous red-orange solution was cooled to –78 °C and a solution of 2-methylcyclopentenone (**6c**, 100 μL, 1.0 mmol, 1 equiv) in THF (2.0 mL) introduced. The slightly darker solution was allowed to warm to –10 °C over 2 h. A solution of thiophenol (0.16 mL, 1.5 mmol, 1.5 equiv) in absolute ethanol (200 proof, 1.0 mL) was added to the reaction mixture followed by dilution of the resulting mixture with an aqueous ammonium hydroxide–saturated aqueous ammonium chloride solution (1:9, 5 mL) and warming of the mixture to 0 °C. After 1 h of vigorous stirring at 0 °C, the mixture was extracted with dichloromethane (3 × 20 mL) and the combined organic layers were then dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure on a rotary evaporator. The resulting reaction crude was then passed through a plug of silica (silica gel; 5% methanol and 2% Et_3N in dichloromethane) to remove the nonpolar impurities and afforded the desired imino alcohol **10c** as a viscous oil.

Sodium borohydride (45 mg, 1.2 mmol, 1.2 equiv) was added to a solution of crude imino alcohol **10c** in absolute ethanol (200 proof, 10.0 mL, 0.1 M) at 0 ºC. After 20 min, the excess hydride was quenched by addition of aqueous saturated ammonium chloride solution (5 mL) and the resulting mixture was concentrated under reduced pressure. The residue was diluted slowly with aqueous sodium hydroxide solution (1N, 5 mL) and extracted with dichloromethane (3 × 20 mL). The combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel; 10% MeOH and 2% Et_3N in CH_2Cl_2) afforded the desired amino alcohol **11c** (108 mg, 55%, 8:1 diastereomeric mixture; major isomer shown) as a viscous oil. The relative stereochemistry was secured by a series of 2D-NMR experiments.

^{1}H NMR (500 MHz, C_6D_6, 20°C, 8:1 mixture of C12-diastereomers, minor diastereomer resonances not obscured noted by *): 2.78-2.86 (m, 2H, CH_2CH_2(NH), CH_2CH(NH)), 2.58-2.66 (m, 1H, CH_2CH_2C(OH)), 2.39-2.46 (m, 1H, CH_2CH_2(NH)), 2.26-2.33 (m, 1H, CH_2CH_2(NH)), 1.68-1.92 (m, 4H, $CHCH(CH_3)C$(OH), CH_2CH_2C(OH)), CH_2CH_2C(OH), CH_2CHC(OH)), 1.54-1.64 (m, 2H, $CHCH(CH_3)C$(OH), CH_2CHC(OH)), 1.42-1.54 (m, 1H, CH_2CH(NH)), 1.10-1.30 (m, 3H, CH_2CHC(OH), CH_2CH_2(NH),

$CH_2C\mathbf{H}_2C(OH)$), 1.07 (dd, *J*= 13.9, 3.2 Hz, 1H, $C\mathbf{H}_2CH(NH)$), 0.90 (d, *J*= 6.9 Hz, 3H, $C(C\mathbf{H}_3)$), 0.82 (d, *J*= 6.9 Hz, 3H, $C(C\mathbf{H}_3)$*).

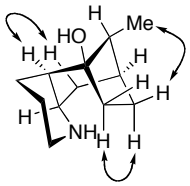
^{13}C NMR (125.7 MHz, C_6D_6, 20°C, 8:1 mixture of C12-diastereomers, resonances for the major diastereomer are listed): 81.9, 56.7, 52.2, 48.4, 47.7, 42.1, 42.0, 32.7, 26.7, 24.4, 23.9, 14.1.

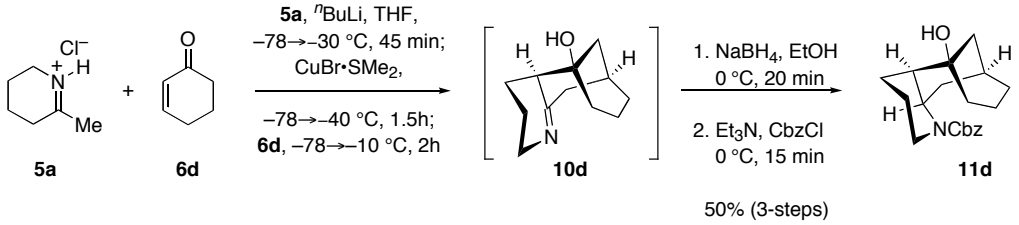
FTIR (neat): 3326 (br, O-H), 2929 (C–H), 1456, 1318, 1099.

HRMS–ESI (*m*/*z*): calcd for $C_{12}H_{22}NO$ $[M+H]^+$: 196.1701, found: 196.1701

TLC (50% MeOH and 2% Et_3N in CH_2Cl_2), *Rf*: 0.3 (Ninhydrin)

NOESY data (500 MHz, C_6D_6, 20°C):



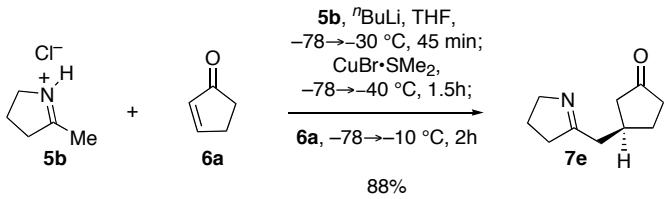


1-Hydroxy-6-aza-tricyclo[7.3.1.00,0]tridecane-6-carboxylic acid benzyl ester (11d, Table 1, Entry 3):

A solution of *n*-butyl lithium in hexanes (2.49 M, 2.40 mL, 6.00 mmol, 6.00 equiv) was added drop wise via gas–tight syringe to a well-stirred suspension of the iminium chloride salt **5a** (400 mg, 3.00 mmol, 3.00 equiv) in THF (3.0 mL) at –78 °C under an argon atmosphere. The resulting reaction mixture was allowed to gradually warm to –30 °C over 45 min. After which the reaction mixture turned into a clear yellow solution indicating the complete dissolution of the iminium chloride salt. The reaction mixture was then cooled back to –78 °C and the clear yellow solution of the lithioenamine was transferred via cannula under positive argon pressure to a stirring suspension of copper bromide–dimethyl sulfide (308 mg, 1.50 mmol, 1.50 equiv) in THF (2.0 mL) at –78 °C. The resulting red-orange reaction mixture was allowed to gradually warm to –40 °C over 1.5 h. The resulting clear and homogeneous red-orange solution was cooled to –78 °C and a solution of cyclohexenone (**6d**, 102 μL, 1.00 mmol, 1 equiv) in THF (2.0 mL) introduced. The slightly darker solution was allowed to warm to –10 °C over 2 h. A solution of thiophenol (0.16 mL, 1.50 mmol, 1.50 equiv) in absolute ethanol (200 proof, 1.0 mL) was added to the reaction mixture followed by dilution of the resulting mixture with an aqueous ammonium hydroxide–saturated aqueous ammonium chloride solution (1:9, 5 mL) and warming of the mixture to 0 °C. After 1 h of vigorous stirring at 0 °C, the mixture was extracted with dichloromethane (3 × 20 mL) and the combined organic layers were then dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure on a rotary evaporator. The resulting reaction crude was then passed through a plug of silica (silica gel; 5% methanol and 2% Et_3N in dichloromethane) to remove the nonpolar impurities and afforded the desired imino alcohol **10d** as a viscous oil.

Sodium borohydride (45 mg, 1.2 mmol, 1.2 equiv) was added to a solution of crude imino alcohol **10d** in absolute ethanol (200 proof, 10.0 mL, 0.1 M) at 0 ºC. After 20 min, the excess hydride was quenched by addition of aqueous saturated ammonium chloride solution (5 mL) and the resulting mixture was concentrated under reduced pressure. The residue was diluted with aqueous sodium hydroxide solution (1N, 5 mL) and extracted with dichloromethane (3 × 20 mL). The combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. Triethylamine (0.28 mL, 2.0 mmol, 2.0 equiv) was added to a solution of the corresponding crude amino alcohol in dichloromethane (10.0 mL, 0.1 M) at 0 ºC. Chlorobenzyl formate (0.22 mL, 1.5 mmol, 1.5 equiv) was then added dropwise to the solution with stirring. After 15 min, the reaction was quenched by addition of aqueous saturated ammonium chloride solution (5 mL) and extracted with dichloromethane (3 × 20 mL). The combined organic layers were concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel; 50% hexanes in ethyl acetate) afforded the desired carbamate **11d** (165 mg, 50%) as a viscous oil. Significant broadening of the ^1H and ^{13}C NMR resonances is due to atropisomerism of the carbamate. Relative stereochemistry is based on correlation with the detailed structure assignment of cyclohexenone derived product **10g** and related enclosed X-ray structures.

^{1}H NMR (500 MHz, $CDCl_3$, 20°C):	7.25-7.40 (m, 5H, Ar**H**), 5.20 (s, 2H, ArC**H**$_2$O(C=O)), 4.40-4.70 (m, 1H, CH_2C**H**$_2$N(C=O)), 4.00-4.20 (m, 1H, CH_2C**H**$_2$N(C=O)), 2.60-2.80 (m, 1H, CH_2C**H**N(C=O)), 1.05-2.40 (m, 16H).
^{13}C NMR (125.7 MHz, $CDCl_3$, 20°C):	156.0, 137.8, 128.5, 128.5, 128.0, 71.2, 66.9, 48.4, 45.6, 39.7, 38.6, 36.4, 31.1, 27.3, 25.5, 24.1, 20.9, 19.5.
FTIR (neat):	3429 (br, O–H), 2932 (w, C–H), 1694 (s, C=O), 1428, 1267.
HRMS–ESI (*m*/*z*):	calcd for $C_{20}H_{27}NNaO_3$ $[M+Na]^+$: 352.1889, found: 352.1896
TLC (50% hexanes in ethyl acetate), *Rf*:	0.3 ($KMnO_4$)



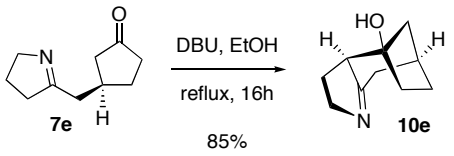
3-(4,5-Dihydro-3*H*-pyrrol-2-ylmethyl)-cyclopentanone (7e, Table 1, Entry 4):

A solution of *n*-butyl lithium in hexanes (2.49 M, 2.40 mL, 6.00 mmol, 6.00 equiv) was added drop wise via gas–tight syringe to a well-stirred suspension of the iminium chloride salt **5b**[6] (359 mg, 3.00 mmol, 3.00 equiv) in THF (3.0 mL) at –78 °C under an argon atmosphere. The resulting reaction mixture was allowed to gradually warm to –30 °C over 45 min. After which the reaction mixture turned into a clear yellow solution indicating the complete dissolution of the iminium chloride salt. The reaction mixture was then cooled back to –78 °C and the clear yellow solution of the lithioenamine was transferred via cannula under positive argon pressure to a stirring suspension of copper bromide–dimethyl sulfide (308 mg, 1.50 mmol, 1.50 equiv) in THF (2.0 mL) at –78 °C. The resulting red-orange reaction mixture was allowed to gradually warm to –40 °C over 1.5 h. The resulting clear and homogeneous red-orange solution was cooled to –78 °C and a solution of cyclopentenone (**6a**, 85 μL, 1.0 mmol, 1 equiv) in THF (2.0 mL) introduced. The slightly darker solution was allowed to warm to –10 °C over 2 h. A solution of thiophenol (0.16 mL, 1.5 mmol, 1.5 equiv) in absolute ethanol (200 proof, 1.0 mL) was added to the reaction mixture followed by dilution of the resulting mixture with an aqueous ammonium hydroxide–saturated aqueous ammonium chloride solution (1:9, 5 mL) and warming of the mixture to –10 °C. After 20 min of vigorous stirring, the mixture was extracted with dichloromethane (3 × 20 mL) and the combined organic layers were then dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure on a rotary evaporator. Purification of the residue by flash column chromatography (silica gel; 2% Et_3N in acetone-hexanes (3:2)) afforded the desired product **7e** (145 mg, 88%) as an oil.

^{1}H NMR (500 MHz, C_6D_6, 20°C):	3.70 (app t, J = 7.3 Hz, 2H, C**H**$_2$N=C), 2.15-2.26 (m, 2H, CH_2C**H**$_2$C=O), 1.93-1.97 (m, 1H, CHC**H**$_2$C=O), 1.90-1.94 (m, 2H, CHC**H**$_2$C=N), 1.83-1.90 (m, 2H, CH_2C**H**$_2$C=N), 1.74 (dd, J = 18.2, 8.7 Hz, 1H, CHC**H**$_2$C=O), 1.64-1.71 (m, 1H, C**H**$_2$$CH_2$C=O), 1.49-1.57 (m, 1H, C**H**$_2$$CH_2$C=O), 1.40-1.46 (m, 2H, C**H**$_2$$CH_2$N=C), 0.98-1.08 (m, 1H, C**H**CH_2C=N).
^{13}C NMR (125.7 MHz, C_6D_6, 20°C):	216.6, 175.0, 61.6, 45.3, 39.4, 38.4, 38.1, 34.6, 29.8, 23.1.
FTIR (neat):	2956 (w, C–H), 1738 (s, C=O), 1643 (s, C=N), 1404, 1158.
HRMS–ESI (*m*/*z*):	calcd for $C_{10}H_{15}NO$ $[M]^+$: 165.1154, found: 165.1153

TLC (2% Et_3N in acetone–hexanes (1:1)), *Rf*: 0.3 (Ninhydrin)

[6] 5-Methyl-3,4-dihydro-2H-pyrrole (**13b**) is commercially available as is readily converted to the HCl salt **5b**. Both **13b** and **5b** may be used for this chemistry. For consistency the described procedure utilizes the iminium chloride **5b**.

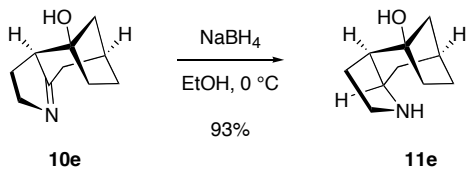


5-Aza-tricyclo[6.2.1.$0^{0,0}$]undec-5-en-1-ol (10e, Table 1, Entry 4):

1.8-Diazabicyclo[5.4.0]undec-7-ene (DBU, 70 μL, 0.45 mmol, 3.0 equiv) was added to a stirring solution of the imino ketone **7e** (25 mg, 0.15 mmol, 1 equiv) in absolute ethanol (200 proof, 1.5 mL, 0.1M) and the solution was heated to reflux under an argon atmosphere. After 16h, the reaction mixture was cooled to ambient temperature and concentrated under reduced pressure on a rotary evaporator. Purification of the residue by flash column chromatography (silica gel; 5% MeOH and 2% Et_3N in CH_2Cl_2) afforded the desired product **10e** (21 mg, 85%) as a viscous oil. The relative stereochemistry of imino alcohol **10e** was secured by X-ray crystallographic analysis of the corresponding amino alcohol **11e** (see page S41).

^{1}H NMR (500 MHz, C_6D_6, 20°C):	3.78-3.92 (m, 1H, $CH_2C\mathbf{H}_2N$=C), 3.48-3.60 (m, 1H, $CH_2C\mathbf{H}_2$N=C), 2.50-2.60 (m, 1H, $CH_2C\mathbf{H}$C=N), 2.40 (app d, *J* = 14.0 Hz, 1H, CHC$\mathbf{H}_2$C=N), 1.80-1.95 (m, 2H, C$\mathbf{H}CH_2$C=N, CHC$\mathbf{H}_2$C=N), 1.63-1.73 (m, 2H, C$\mathbf{H}_2CH_2$N=C, CHC$\mathbf{H}_2$C(OH)), 1.44-1.61 (m, 3H, C$\mathbf{H}_2CH_2$C(OH), $CH_2C\mathbf{H}_2$C(OH), C$\mathbf{H}_2CH_2$N=C), 1.30 (app-d, *J* = 11.3 Hz, 1H, CHC$\mathbf{H}_2$C(OH)), 1.05-1.17 (m, 2H, $CH_2C\mathbf{H}_2$C(OH), C$\mathbf{H}_2CH_2$C(OH)).
^{13}C NMR (125.7 MHz, C_6D_6, 20°C):	176.2, 81.1, 60.3, 46.5, 39.0, 34.1, 31.8, 30.4, 28.7, 24.3.
FTIR (neat):	3271 (s, O–H), 2946 (s, C–H), 1651 (s, C=N), 1451, 1319.
HRMS–ESI (*m*/*z*):	calcd for $C_{10}H_{16}NO$ $[M+H]^+$: 166.1232, found: 166.1226

TLC (5% MeOH and 2% Et_3N in CH_2Cl_2), *Rf*: 0.3 (Ninhydrin)



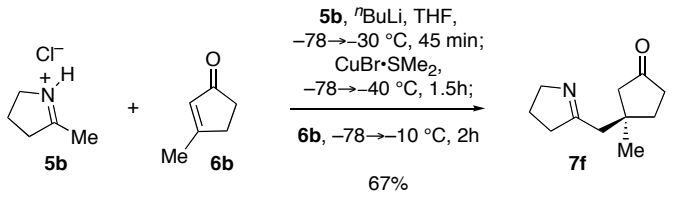
10e **11e**

5-Aza-tricyclo[6.2.1.0^{0,0}]undecan-1-ol (11e, Table 1, Entry 4):

Sodium borohydride (7.5 mg, 0.20 mmol, 1.5 equiv) was added to a solution of imino alcohol **10e** (21 mg, 0.13 mmol, 1 equiv) in absolute ethanol (200 proof, 1.5 mL, 0.1 M) at 0 ºC. After 20 min, the excess hydride was quenched by addition of aqueous saturated ammonium chloride solution (3 mL) and the resulting mixture was concentrated under reduced pressure. The residue was diluted slowly with aqueous sodium hydroxide solution (1N, 2 mL) and extracted with dichloromethane (3 × 20 mL). The combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel; 10% MeOH and 2% Et_3N in CH_2Cl_2) afforded the desired product **11e** (20 mg, 93%) as a viscous oil. The relative stereochemistry of amino alcohol **11e** was secured by X-ray crystallographic analysis of the corresponding tartaric acid salt (see page S41).

^{1}H NMR (500 MHz, C_6D_6, 20°C):	2.88 (td, 1H, *J* = 9.1, 2.6 Hz, CH_2CH_2(NH)), 2.82 (app-t, 1H, *J* = 6.7 Hz, CH_2CH(NH)), 2.42 (app q, *J* = 9.2 Hz, 1H, CH_2CH_2(NH)), 1.95-2.10 (m, 3H, $CHCH_2C$(OH), CH_2CH_2C(OH), CH_2CH_2(NH)), 1.61-1.92 (m, 5H, CH_2CH_2C(OH), CH_2CHC(OH), CH_2CH_2C(OH), $CHCH_2C$(OH), CH_2CH(NH)), 1.42-1.55 (m, 2H, $CHCH_2C$(OH), CH_2CH_2(NH)), 1.23-1.32 (m, 1H, CH_2CH_2C(OH)), 1.18 (app d, 1H, *J* = 10.3 Hz, CH_2CH(NH)).
^{13}C NMR (125.7 MHz, C_6D_6, 20°C):	80.7, 58.4, 50.2, 46.0, 36.5, 35.2, 33.1, 30.5, 29.0, 26.7.
FTIR (neat):	3370 (br, O–H), 2925 (w, C–H), 1647, 1532, 1410.
HRMS–ESI (*m/z*):	calcd for $C_{10}H_{17}NO$ $[M]^+$: 167.1310, found: 167.1307

TLC (50% MeOH and 2% Et_3N in CH_2Cl_2), *Rf*: 0.2 (Ninhydrin)

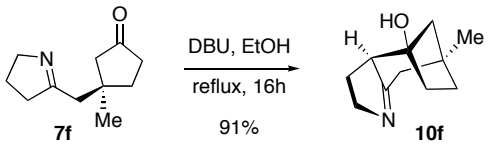


3-(4,5-Dihydro-3*H*-pyrrol-2-ylmethyl)-3-methyl-cyclopentanone (7f, Table 1, Entry 5):

A solution of *n*-butyl lithium in hexanes (2.49 M, 2.40 mL, 6.00 mmol, 6.00 equiv) was added drop wise via gas–tight syringe to a well-stirred suspension of the iminium chloride salt **5b** (359 mg, 3.00 mmol, 3.00 equiv) in THF (3.0 mL) at –78 °C under an argon atmosphere. The resulting reaction mixture was allowed to gradually warm to –30 °C over 45 min. After which the reaction mixture turned into a clear yellow solution indicating the complete dissolution of the iminium chloride salt. The reaction mixture was then cooled back to –78 °C and the clear yellow solution of the lithioenamine was transferred via cannula under positive argon pressure to a stirring suspension of copper bromide–dimethyl sulfide (308 mg, 1.50 mmol, 1.50 equiv) in THF (2.0 mL) at –78 °C. The resulting red-orange reaction mixture was allowed to gradually warm to –40 °C over 1.5 h. The resulting clear and homogeneous red-orange solution was cooled to –78 °C and a solution of 3-methylcyclopentenone (**6b**, 101 μL, 1.00 mmol, 1 equiv) in THF (2.0 mL) introduced. The slightly darker solution was allowed to warm to –10 °C over 2 h. A solution of thiophenol (0.16 mL, 1.5 mmol, 1.5 equiv) in absolute ethanol (200 proof, 1.0 mL) was added to the reaction mixture followed by dilution of the resulting mixture with an aqueous ammonium hydroxide–saturated aqueous ammonium chloride solution (1:9, 5 mL) and warming of the mixture to –10 °C. After 20 min of vigorous stirring, the mixture was extracted with dichloromethane (3 × 20 mL) and the combined organic layers were then dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure on a rotary evaporator. Purification of the residue by flash column chromatography (silica gel; 2% Et_3N in acetone-hexanes (1:3)) afforded the desired product **7f** (120 mg, 67%) as an oil.

^{1}H NMR (500 MHz, C_6D_6, 20°C):	3.66 (app t, J = 7.5 Hz, 2H, C**H**$_2$N=C), 2.12 (d, J = 17.7 Hz, 1H, CC**H**$_2$C=O), 1.83-2.01 (m, 7H, CC**H**$_2$C=O, CH$_2$C**H**$_2$C=O, CC**H**$_2$C=N, CH$_2$C**H**$_2$C=N), 1.52-1.60 (m, 1H, C**H**$_2$CH$_2$C=O), 1.29-1.39 (m, 3H, C**H**$_2$CH$_2$N=C, C**H**$_2$CH$_2$C=O), 0.82 (s, 3H, C**H**$_3$CCH$_2$C=N).
^{13}C NMR (125.7 MHz, C_6D_6, 20°C):	216.9, 174.5, 61.8, 52.3, 44.5, 40.0, 39.3, 36.9, 35.8, 26.0, 23.0.
FTIR (neat):	2954 (w, C–H), 1739 (s, C=O), 1636 (s, C=N), 1405, 1167.
HRMS–ESI (*m*/*z*):	calcd for $C_{11}H_{17}NO$ $[M]^+$: 179.1310, found: 179.1305

TLC (2% Et_3N in acetone–hexanes (1:3)), *Rf*: 0.3 (Ninhydrin)



8-Methyl-5-aza-tricyclo[6.2.1.$0^{0,0}$]undec-5-en-1-ol (10f, Table 1, Entry 5):

1.8-Diazabicyclo[5.4.0]undec-7-ene (DBU, 115 μL, 0.750 mmol, 3.00 equiv) was added to a stirring solution of the imino ketone **7f** (45 mg, 0.25 mmol, 1 equiv) in absolute ethanol (200 proof, 2.5 mL, 0.1M) and the solution was heated to reflux under an argon atmosphere. After 16h, the reaction mixture was cooled to ambient temperature and concentrated under reduced pressure on a rotary evaporator. Purification of the residue by flash column chromatography (silica gel; 5% MeOH and 2% Et_3N in CH_2Cl_2) afforded the desired product **10f** (41 mg, 91%) as a viscous oil.

^{1}H NMR (500 MHz, C_6D_6, 20°C): 3.84-3.94 (m, 1H, CH_2CH_2N=C), 3.52-3.64 (m, 1H, CH_2CH_2N=C), 2.62 (app t, *J* = 8.2 Hz, 1H, CH_2CHC=N), 2.35 (dd, *J* = 14.0, 2.1 Hz, 1H, $C(CH_3)CH_2C$=N), 1.82 (app d, *J* = 14.0 Hz, 1H, $C(CH_3)CH_2C$=N), 1.69-1.78 (m, 1H, CH_2CH_2N=C), 1.61 (dd, *J* = 11.1, 2.3 Hz, 1H, $C(CH_3)CH_2C(OH)$), 1.50-1.58 (m, 2H, $CH_2CH_2C(OH)$, $CH_2CH_2C(OH)$), 1.33 (d, *J* = 11.0 Hz, 1H, $C(CH_3)CH_2C(OH)$), 1.17-1.30 (m, 3H, CH_2CH_2N=C, $CH_2CH_2C(OH)$, $CH_2CH_2C(OH)$), 0.84 (s, 3H, $C(CH_3)CH_2C$=N).

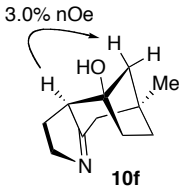
^{13}C NMR (125.7 MHz, C_6D_6, 20°C): 176.5, 81.0, 60.7, 60.6, 53.7, 45.7, 40.2, 35.9, 33.2, 27.3, 24.1.

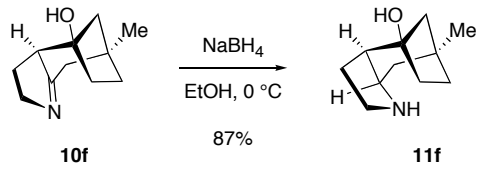
FTIR (neat): 3372 (br, O-H), 2952 (s, C–H), 1651 (s, C=N), 1452, 1305.

HRMS–ESI (*m*/*z*): calcd for $C_{11}H_{18}NO$ $[M+H]^+$: 180.1388, found: 180.1386

TLC (5% MeOH and 2% Et_3N in CH_2Cl_2), *Rf*: 0.3 (Ninhydrin)

nOe data (500 MHz, C_6D_6, 20°C):





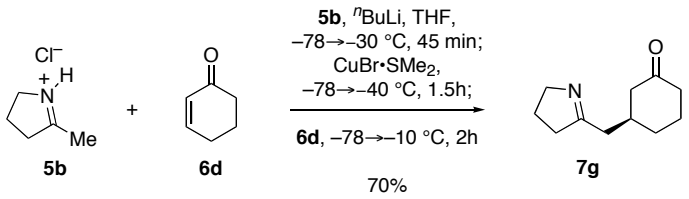
10f **11f**

8-Methyl-5-aza-tricyclo[6.2.1.0 ^{0,0}]undecan-1-ol (11f, Table 1, Entry 5):

Sodium borohydride (14 mg, 0.37 mmol, 1.5 equiv) was added to a solution of imino alcohol **10f** (45 mg, 0.25 mmol, 1 equiv) in absolute ethanol (200 proof, 2.5 mL, 0.1 M) at 0 ºC. After 20 min, the excess hydride was quenched by addition of aqueous saturated ammonium chloride solution (3 mL) and the resulting mixture was concentrated under reduced pressure. The residue was diluted with aqueous sodium hydroxide solution (1N, 3 mL) and extracted with dichloromethane (3 × 20 mL). The combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel; 10% MeOH and 2% Et_3N in CH_2Cl_2) afforded the desired product **11f** (39 mg, 87%) as a viscous oil.

^{1}H NMR (500 MHz, C_6D_6, 20°C):	2.84-2.94 (m, 2H, $CH_2C\mathbf{H}_2$(NH), $CH_2C\mathbf{H}$(NH)), 2.47 (app q, 1H, J = 9.1 Hz, $CH_2C\mathbf{H}_2$(NH)), 2.01-2.10 (m, 1H, $CH_2C\mathbf{H}_2C$(OH)), 1.93-2.02 (m, 1H, $CH_2C\mathbf{H}C$(OH)), 1.83-1.94 (m, 1H, $C\mathbf{H}_2CH_2$(NH)), 1.67-1.81 (m, 2H, $C\mathbf{H}_2CH_2C$(OH), $C\mathbf{H}_2CH_2$(NH)), 1.51 (dd, 1H, J = 10.7, 1.8 Hz, $C(CH_3)C\mathbf{H}_2C$(OH)), 1.30-1.48 (m, 4H, $CH_2C\mathbf{H}_2C$(OH), $C\mathbf{H}_2CH_2C$(OH), $C\mathbf{H}_2CH$(NH), $C\mathbf{H}_2CH$(NH)), 1.10 (app-d, 1H, J = 10.7 Hz, $C(CH_3)C\mathbf{H}_2C$(OH)), 0.98 (s, 3H, $C(C\mathbf{H}_3)$).
^{13}C NMR (125.7 MHz, C_6D_6, 20°C):	81.2, 58.9, 53.0, 49.5, 46.1, 43.9, 40.1, 36.0, 34.4, 28.8, 26.5.
FTIR (neat):	3408 (br, O–H), 2954 (s, C–H), 1643, 1419, 1355.
HRMS–ESI (*m*/*z*):	calcd for $C_{11}H_{19}NO$ $[M]^+$: 181.1467, found: 181.1456

TLC (50% MeOH and 2% Et_3N in CH_2Cl_2,), *Rf*: 0.2 (Ninhydrin)

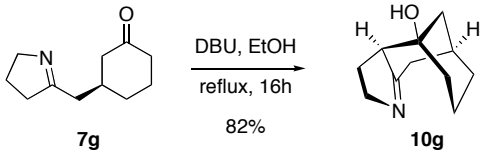


3-(4,5-Dihydro-3*H*-pyrrol-2-ylmethyl)-cyclohexanone (7g, Table 1, Entry 6):

A solution of *n*-butyl lithium in hexanes (2.49 M, 2.40 mL, 6.00 mmol, 6.00 equiv) was added drop wise via gas–tight syringe to a well-stirred suspension of the iminium chloride salt **5b** (359 mg, 3.00 mmol, 3.00 equiv) in THF (3.0 mL) at –78 °C under an argon atmosphere. The resulting reaction mixture was allowed to gradually warm to –30 °C over 45 min. After which the reaction mixture turned into a clear yellow solution indicating the complete dissolution of the iminium chloride salt. The reaction mixture was then cooled back to –78 °C and the clear yellow solution of the lithioenamine was transferred via cannula under positive argon pressure to a stirring suspension of copper bromide–dimethyl sulfide (308 mg, 1.50 mmol, 1.50 equiv) in THF (2.0 mL) at –78 °C. The resulting red-orange reaction mixture was allowed to gradually warm to –40 °C over 1.5 h. The resulting clear and homogeneous red-orange solution was cooled to –78 °C and a solution of cyclohexenone (**6d**, 102 μL, 1.0 mmol, 1 equiv) in THF (2.0 mL) introduced. The slightly darker solution was allowed to warm to –10 °C over 2 h. A solution of thiophenol (0.16 mL, 1.5 mmol, 1.5 equiv) in absolute ethanol (200 proof, 1.0 mL) was added to the reaction mixture followed by dilution of the resulting mixture with an aqueous ammonium hydroxide–saturated aqueous ammonium chloride solution (1:9, 5 mL) and warming of the mixture to –10 °C. After 20 min of vigorous stirring, the mixture was extracted with dichloromethane (3 × 20 mL) and the combined organic layers were then dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure on a rotary evaporator. Purification of the residue by flash column chromatography (silica gel; 2% Et_3N in acetone-hexanes (2:1)) afforded the desired product **7g** (125 mg, 70%) as an oil.

^{1}H NMR (500 MHz, C_6D_6, 20°C):	3.70 (app t, J = 7.3 Hz, 2H, C**H**$_2$N=C), 2.35-2.40 (m, 1H, CH_2C**H**$_2$C=O), 2.13-2.19 (m, 1H, CH_2C**H**$_2$C=O), 1.84-2.04 (m, 5H, CHC**H**$_2$C=O, CHC**H**$_2$C=N, CH_2C**H**$_2$C=N), 1.76-1.84 (m, 1H, CHC**H**$_2$C=O), 1.70-1.76 (m, 1H, C**H**$_2$$CH_2$C=O), 1.49-1.56 (m, 2H, C**H**$_2$$CH_2$C=O, C**H**$_2CHCH_2$C=O), 1.37-1.44 (m, 2H, C**H**$_2$$CH_2$N=C), 1.20-1.30 (m, 1H, C**H**$_2$CHCH_2C=O), 0.86-0.96 (m, 1H, CH_2C**H**CH_2C=O).
^{13}C NMR (125.7 MHz, C_6D_6, 20°C):	208.8, 174.6, 61.6, 48.3, 41.6, 40.6, 38.0, 36.7, 31.6, 25.4, 23.1.
FTIR (neat):	2937 (w, C–H), 1709 (s, C=O), 1642 (s, C=N), 1429, 1309, 1226.
HRMS–ESI (*m*/*z*):	calcd for $C_{11}H_{18}NO$ $[M+H]^+$: 180.1388, found: 180.1381

TLC (2% Et_3N in acetone–hexanes (2:1)), *Rf*: 0.2 (Ninhydrin)



5-Aza-tricyclo[6.3.1.0^{0,0}]dodec-5-en-1-ol (10g, Table 1, Entry 6):

1.8-Diazabicyclo[5.4.0]undec-7-ene (DBU, 128 μL, 0.840 mmol, 3.00 equiv) was added to a stirring solution of the imino ketone **7g** (50 mg, 0.28 mmol, 1 equiv) in absolute ethanol (200 proof, 3.0 mL, 0.1M) and the solution was heated to reflux under an argon atmosphere. After 16h, the reaction mixture was cooled to ambient temperature and concentrated under reduced pressure on a rotary evaporator. Purification of the residue by flash column chromatography (silica gel; 5% MeOH and 2% Et_3N in CH_2Cl_2) afforded the desired product **10g** (41 mg, 82%) as a yellow viscous oil. The relative stereochemistry was secured by a series of NMR experiments.

^{1}H NMR (500 MHz, $CDCl_3$, 20°C): 3.88-3.98 (m, 1H, CH_2CH_2N=C), 3.52-3.64 (m, 1H, CH_2CH_2N=C), 2.78 (app-t, J = 9.8 Hz, 1H, CH_2CHC(OH)), 2.58 (app-d, J = 14.3 Hz, 1H, CHCH_2C=N), 2.40-2.48 (m, 2H, CHCH_2C=N, CHCH_2C=N), 2.05-2.14 (m, 1H, CH_2CH_2N=C), 1.94-2.02 (m, 1H, CH_2CH_2C(OH)), 1.80-1.88 (m, 2H, CH_2CH_2C(OH)), 1.68-1.80 (m, 1H, CH_2CH_2N=C), 1.58-1.66 (m, 1H, CH_2CH_2CH), 1.44-1.56 (m, 2H, CH_2CH_2CH, CH_2CH_2C(OH)), 1.30-1.44 (m, 2H, CH_2CH_2C(OH), CH_2CH_2C(OH)).

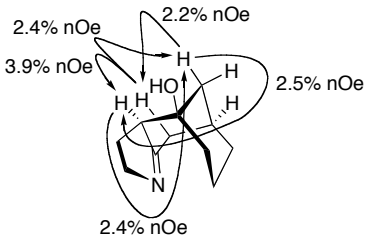
^{13}C NMR (125.7 MHz, C_6D_6, 20°C): 180.5, 71.0, 60.5, 58.9, 44.5, 36.6, 36.1, 32.0, 31.9, 24.3, 20.9.

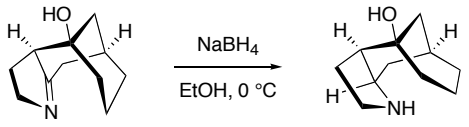
FTIR (neat): 3416 (br, O–H), 2930 (s, C–H), 1645 (s, C=N), 1460, 1088 (m).

HRMS–ESI (*m*/*z*): calcd for $C_{11}H_{18}NO$ $[M+H]^+$: 180.1388, found: 180.1377

TLC (5% MeOH and 2% Et_3N in CH_2Cl_2), *Rf*: 0.3 (Ninhydrin)

nOe Data (500 MHz, C_6D_6, 20°C):





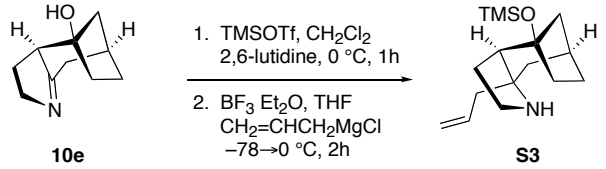
10g **11g**

5-Aza-tricyclo[6.3.1.$0^{0,0}$]dodecan-1-ol (11g, Table 1, Entry 6):

Sodium borohydride (12.6 mg, 0.33 mmol, 1.5 equiv) was added to a solution of imino alcohol **10g** (40 mg, 0.22 mmol, 1 equiv) in absolute ethanol (200 proof, 2.2 mL, 0.1 M) at 0 ºC. After 1 h, the excess hydride was quenched by addition of aqueous saturated ammonium chloride solution (3 mL) and the resulting mixture was concentrated under reduced pressure. The residue was diluted with aqueous sodium hydroxide solution (1N, 3 mL) and extracted with dichloromethane (3 × 20 mL). The combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel; 10% MeOH and 2% Et_3N in CH_2Cl_2) afforded the desired product **11g** (36 mg, 88%) as a white solid.

^{1}H NMR (500 MHz, C_6D_6, 20°C):	2.95 (td, 1H, *J* = 8.8, 2.1 Hz, $CH_2C\mathbf{H}_2(NH)$), 2.79-2.85 (m, 1H, $CH_2C\mathbf{H}(NH)$), 2.52-2.64 (m, 1H, $CH_2C\mathbf{H}_2C(OH)$), 2.41 (app-q, *J* = 8.8 Hz, 1H, $CH_2C\mathbf{H}_2(NH)$), 2.02-2.09 (m, 2H, $C\mathbf{H}CH_2C(OH)$, $C\mathbf{H}_2CH_2(NH)$), 1.68-1.87 (m, 4H, $CH_2C\mathbf{H}C(OH)$, $CHC\mathbf{H}_2C(OH)$, $C\mathbf{H}_2CH_2C(OH)$, $C\mathbf{H}_2CHCH_2$), 1.12-1.50 (m, 7H, $C\mathbf{H}_2CH(NH)$, $C\mathbf{H}_2CH(NH)$, $C\mathbf{H}_2CH_2(NH)$, $CHC\mathbf{H}_2C(OH)$, $CH_2C\mathbf{H}_2C(OH)$, $C\mathbf{H}_2CH_2C(OH)$, $C\mathbf{H}_2CHCH_2$).
^{13}C NMR (125.7 MHz, $CDCl_3$, 20°C):	70.9, 56.6, 49.6, 45.9, 43.9, 36.4, 34.5, 32.6, 32.2, 26.5, 20.0.
FTIR (neat):	3356 (br, O–H), 2926 (w, C–H), 1643, 1532, 1415.
HRMS–ESI (*m*/*z*):	calcd for $C_{11}H_{20}NO$ $[M+H]^+$: 182.1545, found: 182.1533
mp:	120°C

TLC (50% MeOH and 2% Et_3N in CH_2Cl_2,), *Rf*: 0.3 (Ninhydrin)



6-Allyl-1-(trimethyl-silanyloxy)-5-aza-tricyclo[6.2.1.$0^{0,0}$]undecane (S3):

Trimethylsilyl trifluoromethanesulfonate (28 μL, 0.16 mmol, 1.2 equiv) was added drop wise via gas–tight syringe to a well-stirred solution of imino alcohol **10e** (21 mg, 0.13 mmol, 1 equiv) and 2,6-lutidine (35 μL, 0.32 mmol, 2.4 equiv) in CH_2Cl_2 (2.0 mL) at –78 °C under an argon atmosphere. The resulting reaction mixture was allowed to gradually warm to 0 °C over 30 min. After which saturated aqueous ammonium chloride solution (5 mL) was added to the reaction mixture. The layers were separated and the aqueous layer was extracted with dichloromethane (2 x 10 mL). The combined organic layers were then dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure on a rotary evaporator. The crude reaction mixture was then passed through a plug of silica gel (5% MeOH and 2% Et_3N in CH_2Cl_2) to afford the corresponding *O*-TMS protected imino alcohol as a yellow viscous oil.

Boron trifluoride-diethyl etherate (48 μL, 0.39 mmol, 3.0 equiv) was added drop wise via gas–tight syringe to a well-stirred solution of the *O*-TMS protected imino alcohol in THF (1.0 mL) at –78 °C under an argon atmosphere. The resulting reaction mixture was allowed to gradually warm to -30 °C over 30 min. The reaction mixture was then cooled back to –78 °C and allylmagnesium chloride (2M in THF, 0.39 mL, 0.78 mmol, 6.0 equiv) was added drop wise via gas–tight syringe to the reaction mixture. The resulting light yellow reaction mixture was allowed to gradually warm to 0 °C over 2.0 h. After which saturated aqueous sodium bicarbonate solution (5 mL) was added to the reaction mixture. The layers were separated and the aqueous layer was extracted with dichloromethane (3 x 10 mL). The combined organic layers were then dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure on a rotary evaporator. Purification of the residue by flash column chromatography (silica gel: diam. 3.0 cm, ht, 15 cm; 2% Et_3N and 15% EtOAc in hexanes) afforded the desired amine **S3** (26 mg, 72%) as a yellow viscous oil. The relative stereochemistry was secured by a series of NMR experiments.

^{1}H NMR (500 MHz, C_6D_6, 20ºC): 5.56-5.66 (m, 1H, C**H**=CH_2), 5.01 (dd, *J* = 10.1, 2.4 Hz, 1H, CH=C**H**$_2$), 4.84-4.90 (m, 1H, CH=C**H**$_2$), 2.82-2.88 (m, 1H, CH_2C**H**$_2$(NH)), 2.68-2.75 (m, 1H, CH_2C**H**$_2$(NH)), 2.10-2.17 (m, 1H, C**H**$_2$$CH_2$(NH)), 1.90-2.10 (m, 4H, C**H**CH_2C(OTMS), CH_2C**H**C(OTMS), C**H**$_2$CH=CH_2, CH_2C**H**$_2$C(OTMS)), 1.70-1.88 (m, 5H, C**H**$_2$$CH_2$(NH), C**H**$_2$$CH_2$C(OTMS), C**H**$_2$$CH_2$C(OTMS), C**H**$_2$CH=$CH_2$, CHC**H**$_2$C(OTMS)), 1.56-1.64 (m, 1H, CH_2C**H**$_2$C(OTMS)), 1.32-1.43 (m, 2H, C**H**$_2$C(NH)CH_2CH=CH_2), 1.25-1.30 (m, 1H, CHC**H**$_2$C(OTMS)), 0.20 (s, 9H, Si(C**H**$_3$)$_3$).

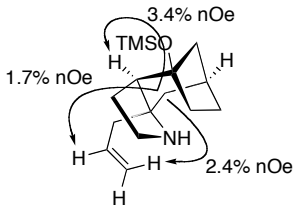
^{13}C NMR (125 MHz, C_6D_6, 20ºC): 135.5, 118.2, 84.1, 64.2, 56.3, 46.1, 45.3, 43.3, 41.1, 35.5, 33.8, 28.6, 26.1, 2.98.

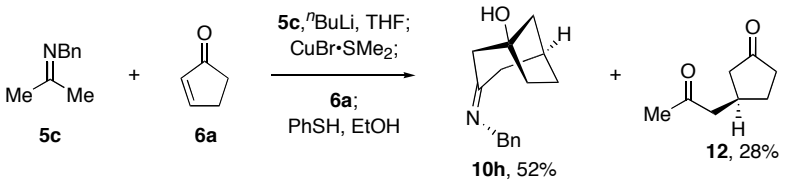
FTIR (neat): 3430 (N–H), 2943 (C–H), 1638 (s, C=C), 1249 (m), 1126 (m), 838 (s).

HRMS–ESI (*m*/*z*): calcd for $C_{16}H_{30}NOSi$ $[M+H]^+$: 280.2097, found: 280.2093

TLC (2% Et_3N and 15% EtOAc in hexanes) 0.3 ($KMnO_4$)

nOe Data (500 MHz, C_6D_6, 20°C):





3-Benzylimino-bicyclo[3.2.1]octan-1-ol (10h, equation 1):

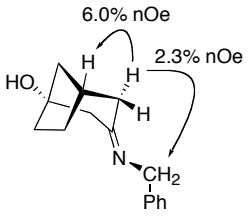
A solution of *n*-butyl lithium in hexanes (2.49 M, 2.40 mL, 6.00 mmol, 3.00 equiv) was added drop wise via gas–tight syringe to a well-stirred solution of imine **5c** (883 mg, 6.00 mmol, 3.00 equiv) in THF (6.0 mL) at –78 °C under an argon atmosphere. The resulting reaction mixture was allowed to gradually warm to –30 °C over 45 min. After which the reaction mixture turned into a clear yellow solution. The reaction mixture was then cooled back to –78 °C and the clear yellow solution of the lithioenamine was transferred via cannula under positive argon pressure to a stirring suspension of copper bromide–dimethyl sulfide (616 mg, 3.0 mmol, 1.50 equiv) in THF (4.0 mL) at –78 °C. The resulting red-orange reaction mixture was allowed to gradually warm to –40 °C over 1.5 h. The resulting clear and homogeneous red-orange solution was cooled to –78 °C and a solution of cyclopentenone (**6a**, 170 μL, 2.00 mmol, 1 equiv) in THF (2.0 mL) introduced. The slightly darker solution was allowed to warm to –10 °C over 2 h. A solution of thiophenol (0.31 mL, 3.0 mmol, 1.5 equiv) in absolute ethanol (200 proof, 1.0 mL) was added to the reaction mixture followed by dilution of the resulting mixture with an aqueous ammonium hydroxide–saturated aqueous ammonium chloride solution (1:9, 5 mL) and warming of the mixture to 0 °C. After 1 h of vigorous stirring at 0 °C, the mixture was extracted with dichloromethane (3 × 30 mL) and the combined organic layers were then dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure on a rotary evaporator. Purification of the residue by flash column chromatography (silica gel; 10% MeOH in CH_2Cl_2) afforded the desired product **10h** (240 mg, 52%) as a yellow viscous oil along with the diketone **12** (130 mg, 28%) as a slightly yellow oil. The relative stereochemistry was secured by a series of NMR experiments.

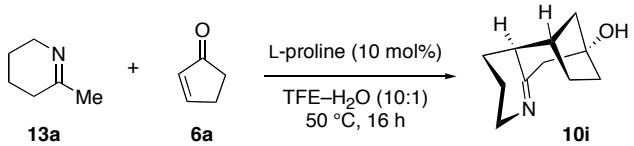
^{1}H NMR (500 MHz, $CDCl_3$, 20ºC):	7.24-7.38 (m, 5H, Ar**H**), 3.76-3.86 (m, 2H, PhC**H**$_2$N=C), 2.69 (app-d, *J* = 14.1 Hz, 1H, CHC**H**$_2$C=N), 2.56 (app-d, *J* = 13.8 Hz, 1H, CHC**H**$_2$C=N), 2.52-2.56 (m, 1H, C**H**CH$_2$C(OH)), 2.31-2.45 (m, 2H, C(OH)C**H**$_2$C=N), 1.94-2.06 (m, 1H, CH$_2$C**H**$_2$C(OH)), 1.92-1.96 (m, 2H, CHC**H**$_2$C(OH)), 1.72-1.80 (m, 2H, C**H**$_2$CH$_2$C(OH)), 1.49-1.58 (m, 1H, CH$_2$C**H**$_2$C(OH)), 1.42 (br-s, 1H, O**H**).
^{13}C NMR (125.7 MHz, $CDCl_3$, 20ºC):	211.7, 141.3, 129.2, 128.8, 127.7, 64.4, 55.8, 49.9, 49.0, 43.8, 35.9, 34.3, 29.7.
FTIR (neat):	3312 (br, O–H), 2945 (C–H), 1710 (s, C=N), 1452, 1332.

HRMS–ESI (*m*/*z*): calcd for $C_{15}H_{19}NNaO$ $[M+Na]^+$: 252.1364, found: 252.1368

TLC (10% MeOH in CH_2Cl_2) 0.3 ($KMnO_4$)

nOe data (500 MHz, C_6D_6, 20°C):





6-Aza-tricyclo[7.2.1.0^{0,0}]dodec-6-en-9-ol (10i, Table 2, Entry 1):

Cyclopentenone (**6a**, 170 μL, 2.00 mmol, 1 equiv) and imine **13a** (389 mg, 4.00 mmol, 2.00 equiv) were added sequentially to a solution of L-Proline (23 mg, 0.20 mmol, 0.10 equiv) in a mixture of 2,2,2-trifluoroethanol–water (10:1, 6.6 mL, 0.33 M) at 23 °C. The resulting solution was heated to 50 ºC under an argon atmosphere. After 16h, the reaction mixture was allowed to cooled to ambient temperature and concentrated under reduced pressure on a rotary evaporator. Purification of the residue by flash column chromatography (silica gel; 5% MeOH and 2% Et_3N in CH_2Cl_2) afforded the desired product **10i** (326 mg, 91%) as a light yellow solid. The relative stereochemistry of the imino alcohol **10i** was secured by X-ray crystallographic analysis of the corresponding amino alcohol **S4** (see page S37).

^{1}H NMR (500 MHz, $CDCl_3$, 20°C): 3.62-3.70 (m, 1H, $CH_2C\mathbf{H}_2N$=C), 3.32-3.42 (m, 1H, $CH_2C\mathbf{H}_2N$=C), 2.56 (dd, *J* = 2.3, 14.0 Hz, 1H, C(OH)C$\mathbf{H}_2$C=N), 2.49 (dd, *J* = 2.7, 13.8 Hz, 1H, C(OH)C$\mathbf{H}_2$C=N), 2.12-2.18 (m, 1H, CHC**H**C=N), 2.00-2.10 (m, 2H, CHC$\mathbf{H}_2$C(OH), C$\mathbf{H}_2CH_2N$=C), 1.59-1.82 (m, 6H, C$\mathbf{H}_2CH_2N$=C, C$\mathbf{H}_2$CHC=N, CHC$\mathbf{H}_2$C(OH), C$\mathbf{H}_2CH_2$C(OH), $CH_2C\mathbf{H}_2$C(OH), C**H**CH_2C(OH)), 1.30-1.56 (m, 3H, C$\mathbf{H}_2$CHC=N, C$\mathbf{H}_2CH_2$C(OH), $CH_2C\mathbf{H}_2$C(OH)).

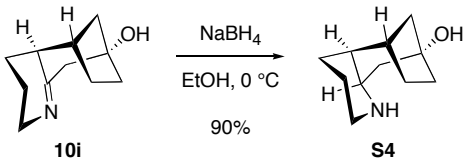
^{13}C NMR (125.7 MHz, $CDCl_3$, 20°C): 171.9, 78.5, 53.8, 49.5, 46.7, 44.8, 39.4, 36.1, 24.8, 24.7, 22.5.

FTIR (neat): 3291 (br, O–H), 2938 (s, C–H), 1656 (s, C=N), 1452, 1327.

HRMS–ESI (*m*/*z*): calcd for $C_{11}H_{18}NO$ $[M+H]^+$: 180.1388, found: 180.1377.

mp: 115 ºC

TLC (5% MeOH and 2% Et_3N in CH_2Cl_2,), *Rf*: 0.2 (Ninhydrin)



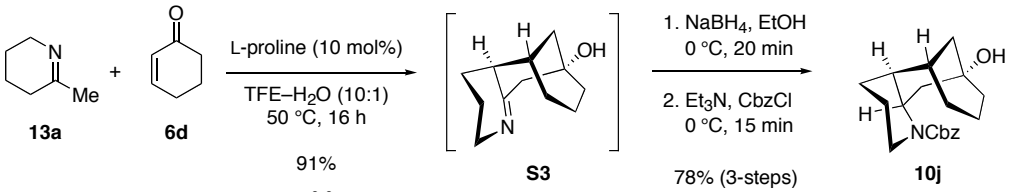
10i S4

6-Aza-tricyclo[7.2.1.$0^{0,0}$]dodecan-9-ol (S2, Table 2, Entry 1):

Sodium borohydride (14.2 mg, 0.380 mmol, 1.50 equiv) was added to a solution of imino alcohol **10h** (45 mg, 0.25 mmol, 1 equiv) in absolute ethanol (200 proof, 2.5 mL, 0.1 M) at 0 ºC. After 20 min, the excess hydride was quenched by addition of aqueous saturated ammonium chloride solution (3 mL) and the resulting mixture was concentrated under reduced pressure. The residue was diluted with aqueous sodium hydroxide solution (1N, 3 mL) and extracted with dichloromethane (3 × 20 mL). The combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel; 10% MeOH and 2% Et_3N in CH_2Cl_2) afforded the desired product **S4** (41 mg, 90%) as a white solid. The relative stereochemistry of the amino alcohol **S4** was secured by X-ray crystallographic analysis (see page S37).

^{1}H NMR (500 MHz, C_6D_6, 20°C):	2.75-2.82 (m, 1H, $CH_2C\mathbf{H}_2$(NH)), 2.68 (app-t, 1H, *J*= 5.3 Hz, $CH_2C\mathbf{H}$(NH)), 2.38-2.46 (m, 1H, $C\mathbf{H}_2CH$(NH)), 2.20-2.28 (m, 1H, $CH_2C\mathbf{H}_2$(NH)), 2.12-2.19 (m, 1H, $C\mathbf{H}_2CH$(NH)), 1.83-1.87 (m, 1H, $C\mathbf{H}CH_2C$(OH)), 1.66-1.83 (m, 4H, $CH_2C\mathbf{H}_2C$(OH), $C\mathbf{H}_2CH_2C$(OH), $C\mathbf{H}_2CH_2$(NH), $CHC\mathbf{H}_2C$(OH)), 1.25-1.56 (m, 5H, $CH_2C\mathbf{H}CH$, $CH_2C\mathbf{H}_2C$(OH), $CHC\mathbf{H}_2C$(OH), $C\mathbf{H}_2CHCH$, $C\mathbf{H}_2CH_2$(NH)), 1.13-1.23 (m, 2H, $C\mathbf{H}_2CHCH$), $C\mathbf{H}_2CH_2C$(OH)).
^{13}C NMR (125.7 MHz, C_6D_6, 20°C):	78.4, 55.3, 49.3, 48.3, 48.2, 43.5, 40.0, 37.3, 30.9, 28.2, 23.7.
FTIR (neat):	3330 (br, O–H), 2936 (s, C–H), 1453, 1338, 1102.
HRMS–ESI (*m*/*z*):	calcd for $C_{11}H_{20}NO$ $[M+H]^+$: 182.1539, found: 182.1533.
mp:	95°C

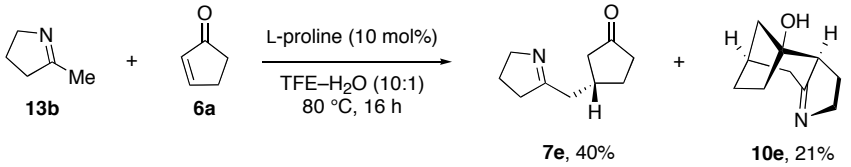
TLC (50% MeOH and 2% Et_3N in CH_2Cl_2,), *Rf*: 0.2 (Ninhydrin)



9-Hydroxy-6-aza-tricyclo[7.3.1.$0^{0,0}$]tridecane-6-carboxylic acid benzyl ester (10j, Table 2, Entry 2):

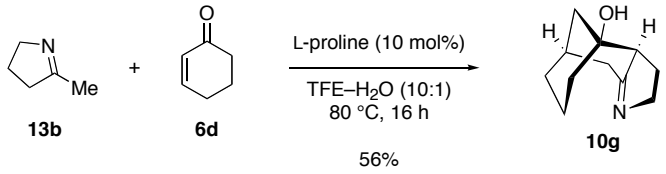
Cyclopentenone ((**6d**, 204 μL, 2.00 mmol, 1 equiv) and imine **13a** (389 mg, 4.00 mmol, 2.00 equiv) were added sequentially to a solution of L-proline (23 mg, 0.20 mmol, 0.10 equiv) in a mixture of 2,2,2-trifluoroethanol–water (10:1, 6.6 mL, 0.3 M) at 23 °C. The resulting solution was heated to 50 ºC under an argon atmosphere. After 16h, the reaction mixture was cooled to ambient temperature and concentrated under reduced pressure on a rotary evaporator. The crude imino alcohol was passed through a plug of silica gel (5% MeOH and 2% Et_3N in dichloromethane) to afford a yellow viscous oil. Sodium borohydride (90 mg, 2.4 mmol, 1.2 equiv) was added to a solution of crude imino alcohol **S3** in absolute ethanol (200 proof, 20.0 mL, 0.1 M) at 0 ºC. After 20 min, the excess hydride was quenched by addition of aqueous saturated ammonium chloride solution (5 mL) and the resulting mixture was concentrated under reduced pressure. The residue was diluted with aqueous sodium hydroxide solution (1N, 5 mL) and extracted with dichloromethane (3 × 30 mL). The combined organic layers were dried over anhydrous sodium sulfate, were filtered, and were concentrated under reduced pressure to give the corresponding amino alcohol. Triethylamine (0.56 mL, 4.0 mmol, 2.0 equiv) was added to a solution of crude amino alcohol in dichloromethane (20 mL, 0.1 M) at 0 ºC. Benzyl chloroformate (0.45 mL, 3.0 mmol, 1.5 equiv) was then added drop-wise to the solution. After 15 min, the reaction mixture was quenched by addition of aqueous saturated ammonium chloride solution (5 mL) and extracted with dichloromethane (3 × 20 mL). The combined organic layers were concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel; 50% hexanes in ethyl acetate) afforded the desired carbamate **10j** (514 mg, 78%) as a clear viscous oil. Significant broadening of the ^1H and ^{13}C NMR resonances is due to atropisomerism of the benzyl carbamate. Relative stereochemistry is based on correlation with the detailed structure assignment of cyclohexenone derived product **10g** and related enclosed X-ray structures.

^1H NMR (500 MHz, $CDCl_3$, 20°C):	7.25-7.45 (m, 5H, Ar**H**), 5.15 (s, 2H, ArC$\mathbf{H}_2$O(C=O)), 4.40-4.60 (m, 1H, $CH_2C\mathbf{H}_2$N(C=O)), 4.00-4.20 (m, 1H, $CH_2C\mathbf{H}_2$N(C=O)), 2.60-2.70 (m, 1H, $CH_2C\mathbf{H}$N(C=O)), 1.10-2.40 (m, 16H).
^{13}C NMR (125.7 MHz, $CDCl_3$, 20°C):	156.0, 137.7, 129.2, 128.6, 128.4, 70.5, 67.6, 48.1, 42.0, 40.5, 38.6, 37.0, 34.5, 32.2, 26.9, 26.1, 23.4, 23.3.
FTIR (neat):	3413 (br, O–H), 2933 (w, C–H), 1695 (s, C=O), 1428, 1269.
HRMS–ESI (*m/z*):	calcd for $C_{20}H_{27}NNaO_3$ $[M+Na]^+$: 352.1889, found: 352.1892
TLC (50% hexanes in ethyl acetate), *Rf*:	0.3 ($KMnO_4$)



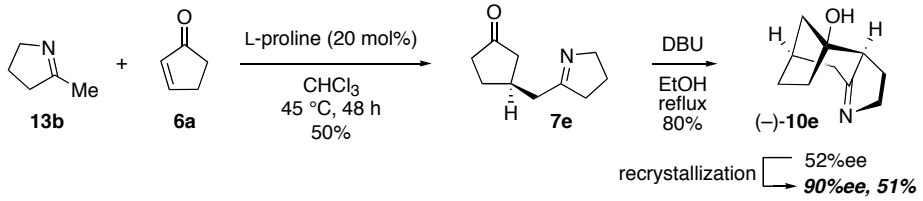
3-(4,5-Dihydro-3*H*-pyrrol-2-ylmethyl)-cyclopentanone (7e) and 5-Aza-tricyclo[6.2.1.$0^{0,0}$]undec-5-en-1-ol (10e, Table 2, Entry 3):

Cyclopentenone (**6a**, 170 μL, 2.00 mmol, 1 equiv) and imine **13b** (195 μL, 2.00 mmol, 2.00 equiv) were added sequentially to a solution of L-Proline (12 mg, 0.10 mmol, 0.10 equiv) in a mixture of 2,2,2-trifluoroethanol–water (10:1, 3.3 mL, 0.3 M) at 23 °C. The resulting solution was heated to 80 ºC under an argon atmosphere. After 16h, the reaction mixture was cooled to ambient temperature and concentrated under reduced pressure on a rotary evaporator. Purification of the residue by flash column chromatography (silica gel; 5% MeOH and 2% Et_3N in CH_2Cl_2) afforded the imino ketone **7e** (65 mg, 40%) and the imino alcohol **10e** (35 mg, 21%). Please see page S14 for complete characterization data.



5-Aza-tricyclo[6.3.1.$0^{0,0}$]dodec-5-en-1-ol (10g, Table 2, Entry 4):

Cyclohexenone (**6d**, 102 μL, 1.00 mmol, 1 equiv) and imine **13b** (195 μL, 2.00 mmol, 2.00 equiv) were added sequentially to a solution of L-Proline (12 mg, 0.10 mmol, 0.10 equiv) in a mixture of 2,2,2-trifluoroethanol–water (10:1, 3.3 mL, 0.3 M) at 23 °C. The resulting solution was heated to 80 ºC under an argon atmosphere. After 16h, the reaction mixture was cooled to ambient temperature and concentrated under reduced pressure on a rotary evaporator. Purification of the residue by flash column chromatography (silica gel; 5% MeOH and 2% Et_3N in CH_2Cl_2) afforded the desired imino ketone **10g** (100 mg, 56%) as an oil. Please see page S20 for complete characterization data.



Cyclopentenone (**6a**, 425 μL, 5.00 mmol, 1 equiv) and imine **13b** (975 μL, 10.0 mmol, 2.00 equiv) were added sequentially to a solution of L-Proline (115 mg, 1.00 mmol, 0.20 equiv) in chloroform (10 mL) at 23 °C. The resulting solution was heated to 45 ºC under an argon atmosphere. After 48h, the reaction mixture was cooled to ambient temperature and concentrated under reduced pressure on a rotary evaporator. Purification of the residue by flash column chromatography (silica gel: diam. 3.0 cm, ht, 15 cm; 5% MeOH and 2% Et_3N in CH_2Cl_2) afforded the desired imino ketone **7e** (415 mg, 50%) as an oil. 1,8-Diazabicyclo[5.4.0]undec-7-ene (DBU, 1.17 mL, 0.45 mmol, 3.0 equiv) was added to a stirring solution of the imino ketone **7e** (415 mg, 2.5 mmol, 1 equiv) in absolute ethanol (200 proof, 25 mL, 0.1M) and the solution was heated to reflux under an argon atmosphere. After 16 h, the reaction mixture was cooled to ambient temperature and concentrated under reduced pressure on a rotary evaporator. Purification of the residue by flash column chromatography (silica gel; 5% MeOH and 2% Et_3N in CH_2Cl_2) afforded the desired iminoalcohol **10e** (332 mg, 80%, 52% ee) as a solid. A 100-mg portion of the iminoalcohol **10e** was dissolved in diethylether-^{n}pentane (1:1, 2 mL) at ambient temperature and cooled to 5 °C. After 24h, the mother liquor containing the optically active imino alcohol **10e** was removed, leaving behind the nearly racemic solid imino alcohol **10e**. Concentration of the mother liquor gave the desired optically active imino alcohol **10e** (51 mg, 51%, 90% ee, $[\alpha]^{22}_D = -25$ (*c* 0.25, CH_2Cl_2)). The optical activity of imine **10e** was determined by chiral HPLC analysis of a derivative[7]: [Chiralpak AD-H; 3.0mL/min; 10% ^{i}PrOH in hexanes; t_R(major) = 4.62 min, t_R(minor) = 6.20 min]. Please see page S14 for full characterization data for product **10e**.

Absolute stereochemistry: The imino alcohol **10e** (90% ee) was reduced to the corresponding amino alcohol **11e** (see above). The resulting amino alcohol **11e** was co-crystallized with L-(+)-tartaric acid (2 equiv) in MeCN (0.2 M); for X-ray data see page S41.

[7] Derivatization by reduction and benzoylation ($NaBH_4$, EtOH; BzCl, pyridine, CH_2Cl_2, 90% 2-steps).

X-Ray Structure of the *N*-(4-Bromobenzoyl) Amino Alcohol Derivative S2:

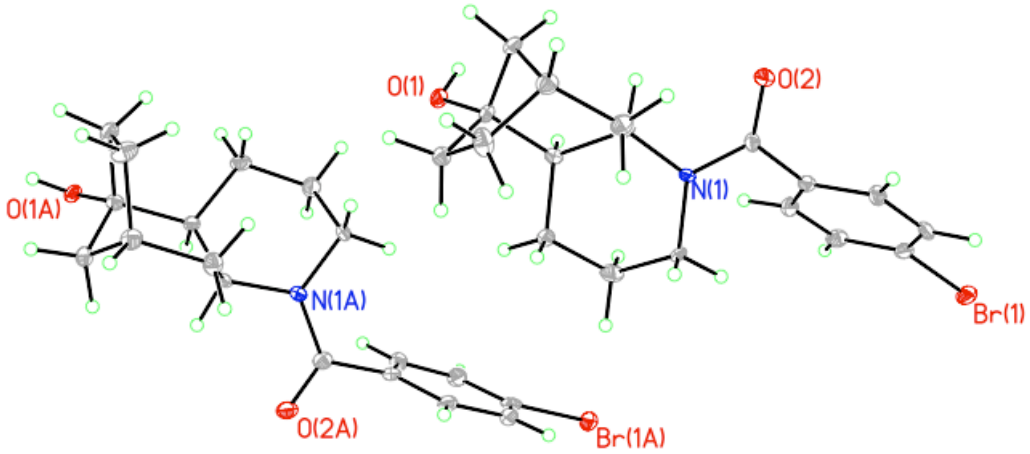


Table S4. Crystal data and structure refinement for S2.

Identification code	05209	
Empirical formula	C18 H22 Br N O2	
Formula weight	364.28	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 6.7557(9) Å	α= 92.158(4)°.
	b = 13.2982(17) Å	β= 92.622(4)°.
	c = 17.691(2) Å	γ = 96.339(4)°.
Volume	1576.5(3) $Å^3$	
Z	4	
Density (calculated)	1.535 Mg/m^3	
Absorption coefficient	2.615 mm^{-1}	
F(000)	752	
Crystal size	0.20 x 0.20 x 0.20 mm^3	
Theta range for data collection	1.15 to 29.57°.	
Index ranges	-9<=h<=9, -18<=k<=18, -24<=l<=24	
Reflections collected	33544	
Independent reflections	8817 [R(int) = 0.0504]	
Completeness to theta = 29.57°	99.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.6229 and 0.6229	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8817 / 415 / 399	
Goodness-of-fit on F^2	1.228	
Final R indices [I>2sigma(I)]	R1 = 0.0941, wR2 = 0.2265	
R indices (all data)	R1 = 0.1010, wR2 = 0.2299	
Largest diff. peak and hole	7.293 and -1.214 $e.Å^{-3}$	

Highest residual electron density resides 1.12 Å away from the bromine atom due to incomplete absorption correction.

Table S5. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for S2. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1A)	-6130(1)	4087(1)	8906(1)	18(1)
Br(1)	-5894(1)	-742(1)	6104(1)	18(1)
N(1)	704(7)	3638(3)	6220(2)	11(1)
O(1A)	925(6)	12168(3)	8640(2)	12(1)
O(1)	1271(6)	7331(3)	6342(2)	13(1)
N(1A)	325(7)	8483(3)	8789(2)	12(1)
C(1)	432(9)	3419(4)	7017(3)	12(1)
C(1A)	-197(9)	8182(4)	7993(3)	13(1)
O(2A)	939(6)	8032(3)	9998(2)	15(1)
O(2)	979(6)	3030(3)	5013(2)	15(1)
C(2A)	-1269(9)	8975(4)	7612(3)	16(1)
C(2)	-538(9)	4261(5)	7403(3)	18(1)
C(3A)	79(8)	9973(4)	7668(3)	14(1)
C(3)	768(9)	5262(4)	7332(3)	15(1)
C(4)	1085(8)	5502(4)	6508(3)	10(1)
C(4A)	655(8)	10317(4)	8490(3)	10(1)
C(5)	1822(8)	4607(4)	6048(3)	11(1)
C(5A)	1514(8)	9478(4)	8951(3)	11(1)
C(6)	4086(8)	4592(4)	6140(3)	14(1)
C(6A)	3740(8)	9427(4)	8855(3)	15(1)
C(7)	5194(8)	5660(4)	6062(3)	15(1)
C(7A)	4899(8)	10500(4)	8927(3)	14(1)
C(8)	3787(8)	6369(4)	5723(3)	12(1)
C(8A)	3599(8)	11268(4)	9256(3)	12(1)
C(9A)	2082(8)	11331(4)	8587(3)	11(1)
C(9)	2461(8)	6513(4)	6392(3)	11(1)
C(10)	4067(8)	6801(4)	7037(3)	13(1)
C(10A)	3509(8)	11527(4)	7939(3)	13(1)
C(11)	5799(9)	6190(5)	6844(3)	18(1)
C(11A)	5296(8)	10940(5)	8128(3)	17(1)
C(12A)	77(8)	7843(4)	9367(3)	12(1)
C(12)	294(8)	2925(4)	5647(3)	11(1)
C(13)	-1149(8)	2025(4)	5785(3)	11(1)
C(13A)	-1395(8)	6929(4)	9231(3)	13(1)
C(14)	-639(8)	1042(4)	5692(3)	14(1)
C(14A)	-826(8)	5954(4)	9315(3)	12(1)
C(15A)	-2233(9)	5110(4)	9207(3)	14(1)
C(15)	-2053(8)	217(4)	5793(3)	13(1)
C(16)	-3969(9)	377(4)	5971(3)	14(1)
C(16A)	-4215(9)	5227(4)	9049(3)	15(1)
C(17)	-4528(8)	1353(4)	6053(3)	14(1)
C(17A)	-4813(8)	6191(4)	8982(3)	14(1)
C(18)	-3079(8)	2160(4)	5974(3)	14(1)
C(18A)	-3391(8)	7034(4)	9064(3)	14(1)

Table S6. Bond lengths [Å] and angles [°] for S2.

Br(1A)-C(16A)	1.882(6)
Br(1)-C(16)	1.894(5)
N(1)-C(12)	1.359(6)
N(1)-C(1)	1.466(6)
N(1)-C(5)	1.470(7)
O(1A)-C(9A)	1.432(6)
O(1)-C(9)	1.426(6)
N(1A)-C(12A)	1.359(7)
N(1A)-C(1A)	1.467(6)
N(1A)-C(5A)	1.481(7)
C(1)-C(2)	1.514(8)
C(1A)-C(2A)	1.506(8)
O(2A)-C(12A)	1.239(7)
O(2)-C(12)	1.240(6)
C(2A)-C(3A)	1.522(8)
C(2)-C(3)	1.526(8)
C(3A)-C(4A)	1.527(7)
C(3)-C(4)	1.524(7)
C(4)-C(5)	1.557(7)
C(4)-C(9)	1.574(7)
C(4A)-C(5A)	1.555(7)
C(4A)-C(9A)	1.568(7)
C(5)-C(6)	1.533(7)
C(5A)-C(6A)	1.530(7)
C(6)-C(7)	1.545(8)
C(6A)-C(7A)	1.548(8)
C(7)-C(8)	1.529(8)
C(7)-C(11)	1.547(8)
C(7A)-C(8A)	1.534(8)
C(7A)-C(11A)	1.571(8)
C(8)-C(9)	1.534(7)
C(8A)-C(9A)	1.541(7)
C(9A)-C(10A)	1.544(7)
C(9)-C(10)	1.545(7)
C(10)-C(11)	1.539(8)
C(10A)-C(11A)	1.540(8)
C(12A)-C(13A)	1.488(8)
C(12)-C(13)	1.493(7)
C(13)-C(18)	1.390(8)
C(13)-C(14)	1.393(7)
C(13A)-C(18A)	1.391(8)
C(13A)-C(14A)	1.404(7)
C(14)-C(15)	1.395(8)
C(14A)-C(15A)	1.390(8)
C(15A)-C(16A)	1.382(8)
C(15)-C(16)	1.383(8)
C(16)-C(17)	1.395(8)
C(16A)-C(17A)	1.393(8)
C(17)-C(18)	1.386(8)
C(17A)-C(18A)	1.392(8)
C(12)-N(1)-C(1)	122.9(4)
C(12)-N(1)-C(5)	117.8(4)
C(1)-N(1)-C(5)	118.1(4)
C(12A)-N(1A)-C(1A)	123.7(5)
C(12A)-N(1A)-C(5A)	118.0(4)
C(1A)-N(1A)-C(5A)	117.4(4)
N(1)-C(1)-C(2)	109.9(4)
N(1A)-C(1A)-C(2A)	110.7(4)
C(1A)-C(2A)-C(3A)	108.7(5)
C(1)-C(2)-C(3)	109.1(5)
C(2A)-C(3A)-C(4A)	111.9(4)
C(4)-C(3)-C(2)	111.9(5)
C(3)-C(4)-C(5)	112.6(4)
C(3)-C(4)-C(9)	114.8(4)
C(5)-C(4)-C(9)	110.3(4)
C(3A)-C(4A)-C(5A)	112.7(4)
C(3A)-C(4A)-C(9A)	114.5(4)
C(5A)-C(4A)-C(9A)	110.3(4)
N(1)-C(5)-C(6)	112.7(4)
N(1)-C(5)-C(4)	111.0(4)
C(6)-C(5)-C(4)	112.5(4)
N(1A)-C(5A)-C(6A)	112.1(4)
N(1A)-C(5A)-C(4A)	111.1(4)
C(6A)-C(5A)-C(4A)	113.3(4)
C(5)-C(6)-C(7)	111.1(4)
C(5A)-C(6A)-C(7A)	110.7(4)
C(8)-C(7)-C(6)	110.7(4)
C(8)-C(7)-C(11)	101.8(4)
C(6)-C(7)-C(11)	111.5(5)
C(8A)-C(7A)-C(6A)	110.7(4)
C(8A)-C(7A)-C(11A)	101.0(4)
C(6A)-C(7A)-C(11A)	111.6(5)
C(7)-C(8)-C(9)	100.5(4)
C(7A)-C(8A)-C(9A)	100.8(4)
O(1A)-C(9A)-C(8A)	114.9(4)
O(1A)-C(9A)-C(10A)	107.4(4)
C(8A)-C(9A)-C(10A)	100.3(4)
O(1A)-C(9A)-C(4A)	109.6(4)
C(8A)-C(9A)-C(4A)	109.7(4)
C(10A)-C(9A)-C(4A)	114.7(4)
O(1)-C(9)-C(8)	115.5(4)
O(1)-C(9)-C(10)	107.2(4)
C(8)-C(9)-C(10)	100.4(4)
O(1)-C(9)-C(4)	109.5(4)
C(8)-C(9)-C(4)	110.0(4)
C(10)-C(9)-C(4)	114.1(4)
C(11)-C(10)-C(9)	104.9(4)
C(11A)-C(10A)-C(9A)	105.5(4)
C(10)-C(11)-C(7)	105.5(4)
C(10A)-C(11A)-C(7A)	105.2(4)
O(2A)-C(12A)-N(1A)	122.1(5)
O(2A)-C(12A)-C(13A)	120.6(5)
N(1A)-C(12A)-C(13A)	117.2(5)
O(2)-C(12)-N(1)	122.4(5)
O(2)-C(12)-C(13)	119.9(5)
N(1)-C(12)-C(13)	117.6(5)
C(18)-C(13)-C(14)	118.7(5)
C(18)-C(13)-C(12)	119.7(5)
C(14)-C(13)-C(12)	121.5(5)
C(18A)-C(13A)-C(14A)	118.9(5)
C(18A)-C(13A)-C(12A)	120.1(5)
C(14A)-C(13A)-C(12A)	120.9(5)
C(13)-C(14)-C(15)	120.0(5)
C(15A)-C(14A)-C(13A)	120.1(5)
C(16A)-C(15A)-C(14A)	120.3(5)
C(16)-C(15)-C(14)	119.9(5)
C(15)-C(16)-C(17)	121.3(5)
C(15)-C(16)-Br(1)	120.0(4)
C(17)-C(16)-Br(1)	118.8(4)

Table S7. Bond lengths [Å] and angles [°] for S2 (Continued).

C(15A)-C(16A)-C(17A)	120.3(5)
C(15A)-C(16A)-Br(1A)	120.4(4)
C(17A)-C(16A)-Br(1A)	119.3(4)
C(18)-C(17)-C(16)	117.7(5)
C(18A)-C(17A)-C(16A)	119.4(5)
C(17)-C(18)-C(13)	122.3(5)
C(13A)-C(18A)-C(17A)	120.9(5)

Symmetry transformations used to generate equivalent atoms:

Table S8. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for S2. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + \ldots + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1A)	25(1)	13(1)	15(1)	1(1)	-2(1)	-5(1)
Br(1)	23(1)	14(1)	15(1)	3(1)	1(1)	-4(1)
N(1)	18(2)	10(2)	5(2)	0(1)	0(2)	-2(2)
O(1A)	17(2)	8(2)	13(2)	2(1)	3(1)	2(1)
O(1)	17(2)	8(2)	13(2)	2(1)	-2(1)	4(1)
N(1A)	17(2)	11(2)	7(2)	0(2)	0(2)	1(2)
C(1)	22(3)	7(2)	9(2)	4(2)	2(2)	3(2)
C(1A)	21(3)	9(2)	8(2)	-2(2)	0(2)	0(2)
O(2A)	17(2)	16(2)	12(2)	4(1)	0(1)	0(2)
O(2)	19(2)	14(2)	11(2)	0(1)	4(1)	-1(2)
C(2A)	21(3)	13(2)	15(2)	2(2)	-6(2)	1(2)
C(2)	22(3)	20(3)	12(2)	1(2)	8(2)	-1(2)
C(3A)	16(2)	16(2)	10(2)	5(2)	-2(2)	1(2)
C(3)	24(3)	10(2)	11(2)	1(2)	4(2)	3(2)
C(4)	10(2)	12(2)	8(2)	1(2)	1(2)	2(2)
C(4A)	12(2)	10(2)	9(2)	1(2)	1(2)	2(2)
C(5)	15(2)	9(2)	10(2)	4(2)	2(2)	1(2)
C(5A)	12(2)	13(2)	6(2)	0(2)	0(2)	0(2)
C(6)	13(2)	14(2)	15(2)	2(2)	1(2)	1(2)
C(6A)	15(2)	12(2)	17(2)	0(2)	-1(2)	4(2)
C(7)	13(2)	16(2)	15(2)	1(2)	2(2)	0(2)
C(7A)	12(2)	16(2)	15(2)	2(2)	1(2)	1(2)
C(8)	15(2)	9(2)	13(2)	4(2)	2(2)	1(2)
C(8A)	11(2)	11(2)	13(2)	0(2)	0(2)	0(2)
C(9A)	11(2)	12(2)	9(2)	1(2)	0(2)	1(2)
C(9)	14(2)	7(2)	12(2)	2(2)	0(2)	1(2)
C(10)	17(2)	11(2)	12(2)	1(2)	-2(2)	0(2)
C(10A)	16(2)	10(2)	15(2)	2(2)	3(2)	1(2)
C(11)	17(3)	16(3)	22(3)	-4(2)	-5(2)	5(2)
C(11A)	10(2)	22(3)	20(3)	7(2)	5(2)	1(2)
C(12A)	11(2)	13(2)	12(2)	2(2)	4(2)	2(2)
C(12)	14(2)	9(2)	12(2)	-1(2)	0(2)	2(2)
C(13)	14(2)	11(2)	9(2)	1(2)	0(2)	0(2)
C(13A)	18(2)	12(2)	7(2)	3(2)	1(2)	1(2)
C(14)	17(2)	12(2)	13(2)	-1(2)	1(2)	5(2)
C(14A)	15(2)	13(2)	10(2)	3(2)	1(2)	3(2)
C(15A)	19(2)	11(2)	12(2)	1(2)	2(2)	3(2)
C(15)	17(2)	12(2)	9(2)	-3(2)	0(2)	4(2)
C(16)	19(2)	13(2)	9(2)	2(2)	0(2)	-4(2)
C(16A)	20(3)	19(2)	6(2)	3(2)	5(2)	2(2)
C(17)	12(2)	17(2)	14(2)	3(2)	4(2)	3(2)
C(17A)	13(2)	15(2)	14(2)	2(2)	-2(2)	-1(2)
C(18)	17(2)	12(2)	14(2)	1(2)	3(2)	4(2)
C(18A)	17(2)	12(2)	13(2)	0(2)	-1(2)	3(2)

Table S9. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for S2.

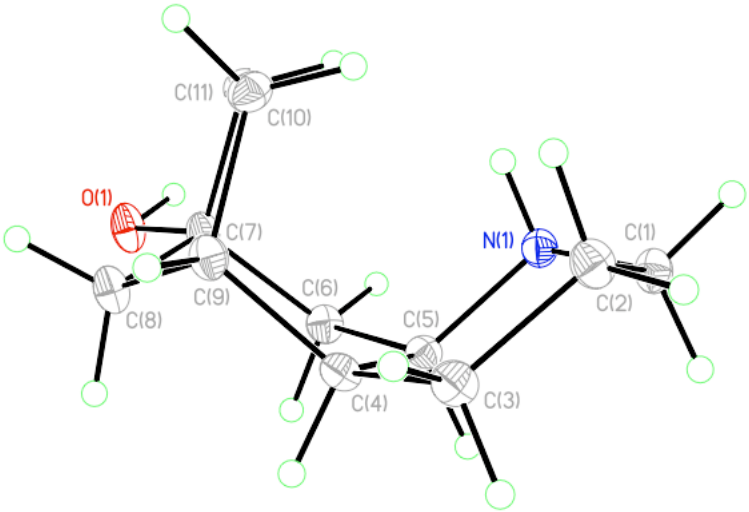
	x	y	z	U(eq)
H(1A)	1687	12713	8654	19
H(1)	543	7256	5941	19
H(1B)	1742	3358	7276	15
H(1C)	-417	2768	7051	15
H(1A1)	-1062	7529	7962	15
H(1A2)	1031	8087	7726	15
H(2A1)	-2527	9054	7862	20
H(2A2)	-1599	8768	7074	20
H(2A)	-693	4121	7944	22
H(2B)	-1878	4300	7162	22
H(3A1)	-615	10500	7420	17
H(3A2)	1303	9895	7395	17
H(3A)	131	5815	7580	18
H(3B)	2077	5229	7599	18
H(4)	-258	5596	6278	12
H(4A)	-611	10450	8727	12
H(5)	1510	4717	5501	13
H(5A)	1363	9659	9498	13
H(6A)	4521	4122	5750	17
H(6B)	4432	4342	6644	17
H(6A1)	3935	9111	8352	18
H(6A2)	4266	9000	9247	18
H(7)	6384	5629	5749	18
H(7A)	6173	10502	9238	17
H(8A)	3006	6051	5273	15
H(8B)	4518	7018	5587	15
H(8A1)	4383	11931	9383	15
H(8A2)	2939	11017	9712	15
H(10A)	4507	7537	7054	16
H(10B)	3545	6616	7533	16
H(10C)	2845	11279	7446	16
H(10D)	3949	12260	7914	16
H(11A)	5985	5685	7230	22
H(11B)	7056	6645	6822	22
H(11C)	6560	11396	8147	20
H(11D)	5374	10386	7744	20
H(14)	670	934	5560	16
H(14A)	525	5871	9445	15
H(15A)	-1831	4450	9243	17
H(15)	-1700	-453	5739	15
H(17)	-5856	1460	6158	17
H(17A)	-6178	6271	8882	17
H(18)	-3418	2830	6052	17
H(18A)	-3791	7691	9005	17

Table S10. Hydrogen bonds for S2 [Å and °].

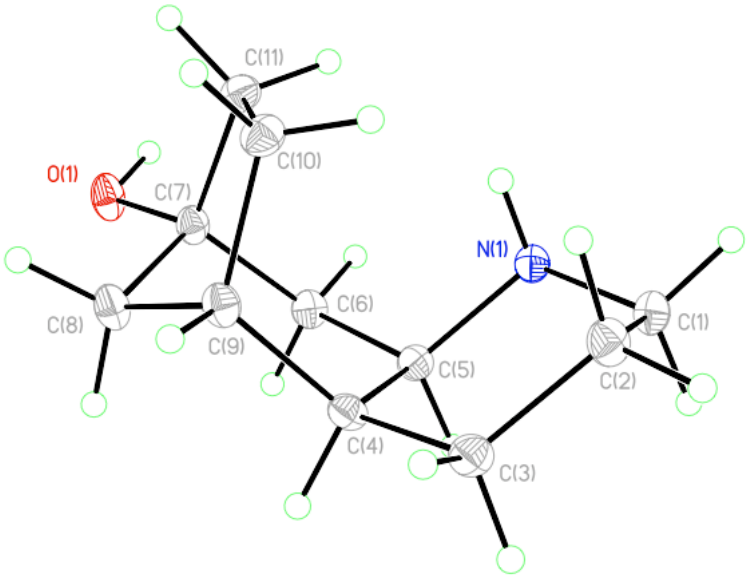
D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1)...O(2)#1	0.84	1.94	2.775(6)	174.1
O(1A)-H(1A)...Br(1A)#2	0.84	2.24	3.065(4)	169.5

Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,-z+1 #2 x+1,y+1,z

X-Ray Structure of Amino Alcohol S4:



View A:



View B:

Table S11. Crystal data and structure refinement for S4.

Identification code	05177	
Empirical formula	C11 H19 N O	
Formula weight	181.27	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 10.6609(3) Å	α= 90°.
	b = 7.5019(2) Å	β= 96.6050(10)°.
	c = 12.1799(3) Å	γ = 90°.
Volume	967.65(4) $Å^3$	
Z	4	
Density (calculated)	1.244 Mg/m^3	
Absorption coefficient	0.079 mm^{-1}	
F(000)	400	
Crystal size	0.17 x 0.15 x 0.05 mm^3	
Theta range for data collection	1.92 to 29.57°.	
Index ranges	-14<=h<=14, -10<=k<=10, -16<=l<=16	
Reflections collected	20630	
Independent reflections	2714 [R(int) = 0.0262]	
Completeness to theta = 29.57°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9961 and 0.8815	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2714 / 2 / 124	
Goodness-of-fit on F^2	1.050	
Final R indices [I>2sigma(I)]	R1 = 0.0429, wR2 = 0.1174	
R indices (all data)	R1 = 0.0473, wR2 = 0.1216	
Largest diff. peak and hole	0.490 and -0.193 $e.Å^{-3}$	

Table S12. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for S4. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	3540(1)	1531(1)	3760(1)	17(1)
N(1)	3893(1)	5715(1)	1068(1)	13(1)
C(4)	1767(1)	5760(1)	1837(1)	13(1)
C(3)	1268(1)	6956(1)	856(1)	15(1)
C(2)	2019(1)	6860(1)	-138(1)	16(1)
C(1)	3428(1)	7042(1)	227(1)	15(1)
C(5)	3224(1)	5837(1)	2067(1)	12(1)
C(6)	3740(1)	4442(1)	2933(1)	14(1)
C(8)	1640(1)	2977(1)	2928(1)	15(1)
C(9)	1252(1)	3834(1)	1798(1)	14(1)
C(10)	1843(1)	2527(1)	1021(1)	15(1)
C(11)	3024(1)	1754(1)	1728(1)	15(1)
C(7)	3045(1)	2646(1)	2870(1)	13(1)

Table S13. Bond lengths [Å] and angles [°] for S4.

O(1)-C(7)	1.4218(11)
N(1)-C(1)	1.4721(12)
N(1)-C(5)	1.4819(12)
C(4)-C(3)	1.5398(12)
C(4)-C(9)	1.5444(13)
C(4)-C(5)	1.5475(12)
C(3)-C(2)	1.5273(13)
C(2)-C(1)	1.5229(13)
C(5)-C(6)	1.5415(12)
C(6)-C(7)	1.5358(13)
C(8)-C(7)	1.5277(12)
C(8)-C(9)	1.5321(12)
C(9)-C(10)	1.5467(13)
C(10)-C(11)	1.5540(13)
C(11)-C(7)	1.5406(13)
C(1)-N(1)-C(5)	111.97(7)
C(3)-C(4)-C(9)	115.57(7)
C(3)-C(4)-C(5)	111.62(7)
C(9)-C(4)-C(5)	112.74(7)
C(2)-C(3)-C(4)	114.95(7)
C(1)-C(2)-C(3)	110.72(8)
N(1)-C(1)-C(2)	112.59(7)
N(1)-C(5)-C(6)	110.66(7)
N(1)-C(5)-C(4)	114.63(7)
C(6)-C(5)-C(4)	111.71(7)
C(7)-C(6)-C(5)	115.42(7)
C(7)-C(8)-C(9)	101.04(7)
C(8)-C(9)-C(4)	107.86(7)
C(8)-C(9)-C(10)	101.33(7)
C(4)-C(9)-C(10)	116.34(7)
C(9)-C(10)-C(11)	104.96(7)
C(7)-C(11)-C(10)	105.37(7)
O(1)-C(7)-C(8)	109.90(7)
O(1)-C(7)-C(6)	110.08(7)
C(8)-C(7)-C(6)	108.98(8)
O(1)-C(7)-C(11)	113.44(8)
C(8)-C(7)-C(11)	101.52(7)
C(6)-C(7)-C(11)	112.53(7)

Symmetry transformations used to generate equivalent atoms:

Table S14. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for S4. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + \ldots + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	14(1)	22(1)	17(1)	9(1)	3(1)	4(1)
N(1)	13(1)	13(1)	13(1)	1(1)	2(1)	-1(1)
C(4)	13(1)	14(1)	11(1)	0(1)	2(1)	3(1)
C(3)	15(1)	16(1)	14(1)	2(1)	1(1)	4(1)
C(2)	16(1)	18(1)	12(1)	3(1)	0(1)	2(1)
C(1)	16(1)	15(1)	13(1)	3(1)	1(1)	-1(1)
C(5)	14(1)	13(1)	11(1)	0(1)	0(1)	0(1)
C(6)	13(1)	16(1)	12(1)	1(1)	-1(1)	0(1)
C(8)	12(1)	19(1)	15(1)	4(1)	3(1)	1(1)
C(9)	11(1)	17(1)	14(1)	2(1)	1(1)	0(1)
C(10)	17(1)	14(1)	15(1)	-1(1)	-1(1)	-2(1)
C(11)	17(1)	13(1)	16(1)	0(1)	2(1)	1(1)
C(7)	12(1)	14(1)	12(1)	3(1)	2(1)	1(1)

Table S15. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2 x\ 10^{\ 3}$) for S4.

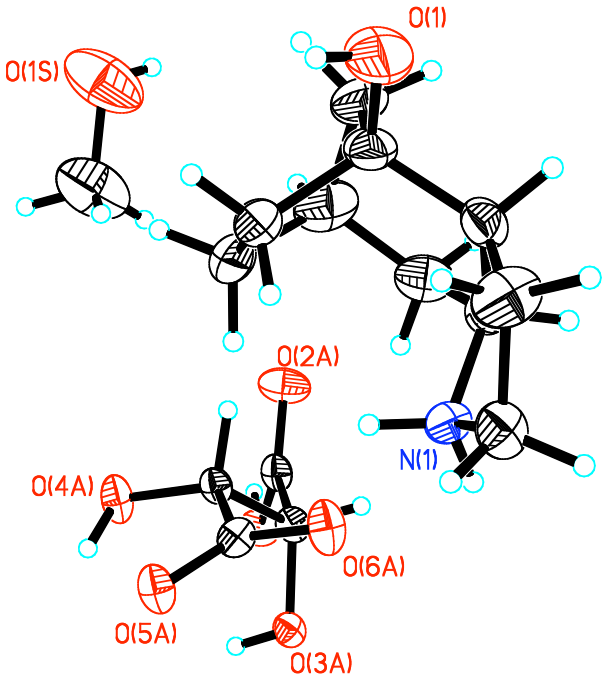
	x	y	z	U(eq)
H(1O)	4320(12)	1282(19)	3700(12)	21
H(1N)	3789(12)	4623(16)	787(11)	16
H(4)	1450	6316	2498	15
H(3A)	382	6620	610	18
H(3B)	1267	8207	1115	18
H(2A)	1852	5707	-522	19
H(2B)	1743	7827	-664	19
H(1A)	3895	6903	-425	18
H(1B)	3602	8252	531	18
H(5)	3437	7032	2402	15
H(6A)	4639	4222	2849	16
H(6B)	3703	4953	3678	16
H(8A)	1501	3795	3540	18
H(8B)	1179	1849	3013	18
H(9)	311	3840	1630	16
H(10A)	2088	3158	364	19
H(10B)	1240	1565	771	19
H(11A)	2956	443	1793	18
H(11B)	3801	2041	1392	18

Table S16. Hydrogen bonds for S4 [Å and °].

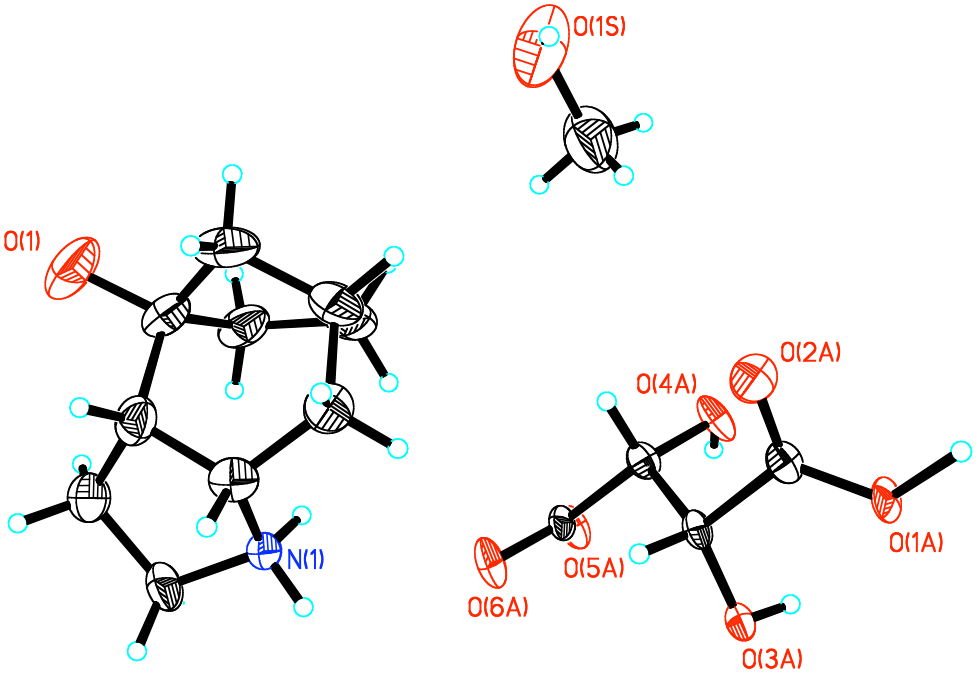
D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1O)...N(1)#1	0.863(12)	1.942(13)	2.7892(10)	166.8(14)
N(1)-H(1N)...O(1)#2	0.889(11)	2.601(12)	3.2618(11)	131.8(11)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,y-1/2,-z+1/2 #2 x,-y+1/2,z-1/2

X-Ray Structure of Amino Alcohol 11e:



View A:



View B:

Table S17. Crystal data and structure refinement for 11e.

Identification code	mm01	
Empirical formula	C15 H27 N O8	
Formula weight	349.38	
Temperature	193(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2	
Unit cell dimensions	a = 29.454(8) Å	α= 90°.
	b = 7.4447(19) Å	β= 90°.
	c = 7.662(2) Å	γ = 90°.
Volume	1680.1(8) $Å^3$	
Z	4	
Density (calculated)	1.381 Mg/m^3	
Absorption coefficient	0.112 mm^{-1}	
F(000)	752	
Crystal size	0.20 x 0.14 x 0.04 mm^3	
Theta range for data collection	1.38 to 25.00°.	
Index ranges	-35<=h<=34, -7<=k<=8, -9<=l<=9	
Reflections collected	11074	
Independent reflections	2964 [R(int) = 0.0595]	
Completeness to theta = 25.00°	100.0 %	
Absorption correction	Empirical	
Max. and min. transmission	0.9955 and 0.9780	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2964 / 10 / 226	
Goodness-of-fit on F^2	1.157	
Final R indices [I>2sigma(I)]	R1 = 0.0586, wR2 = 0.1243	
R indices (all data)	R1 = 0.0670, wR2 = 0.1277	
Absolute structure parameter	0.2(17)	
Largest diff. peak and hole	0.216 and -0.199 $e.Å^{-3}$	

Table S18. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) For 11e. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
N(1)	1688(1)	2323(4)	480(3)	27(1)
O(1)	225(1)	1534(4)	-792(5)	63(1)
C(1)	1692(1)	1435(5)	-1258(4)	35(1)
C(2)	1207(1)	758(6)	-1517(5)	49(1)
C(3)	971(1)	847(5)	254(5)	36(1)
C(4)	569(1)	2172(5)	355(5)	36(1)
C(5)	702(1)	4120(5)	-31(5)	41(1)
C(6)	851(1)	4938(6)	1729(6)	47(1)
C(7)	792(1)	3398(6)	3053(5)	49(1)
C(8)	1208(1)	2235(6)	3245(5)	40(1)
C(9)	1352(1)	1286(5)	1556(5)	33(1)
C(10)	411(1)	2278(6)	2222(6)	52(1)
O(1A)	2336(1)	7228(3)	9276(2)	24(1)
O(2A)	1683(1)	5827(3)	8697(3)	34(1)
O(3A)	2662(1)	6734(3)	6076(3)	20(1)
O(4A)	1826(1)	8836(3)	5481(3)	27(1)
O(5A)	2093(1)	7958(3)	2263(2)	26(1)
O(6A)	2078(1)	5010(3)	2807(3)	29(1)
C(1A)	2053(1)	6416(4)	8272(4)	21(1)
C(2A)	2215(1)	6126(4)	6383(3)	20(1)
C(3A)	1881(1)	6995(4)	5117(4)	20(1)
C(4A)	2032(1)	6592(4)	3248(4)	20(1)
O(1S)	42(1)	6830(5)	6266(4)	80(1)
C(1S)	476(2)	7571(7)	5989(6)	73(2)

Table S19. Bond lengths [Å] and angles [°] for 11e.

N(1)-C(1)	1.487(4)	N(1)-C(9)	1.502(4)
N(1)-H(1A)	0.9200	N(1)-H(1B)	0.9200
O(1)-C(4)	1.424(4)	O(1)-H(1G)	0.8400
C(1)-C(2)	1.526(5)	C(1)-H(1C)	0.9900
C(1)-H(1D)	0.9900	C(2)-C(3)	1.526(5)
C(2)-H(2B)	0.9900	C(2)-H(2C)	0.9900
C(3)-C(9)	1.536(5)	C(3)-C(4)	1.543(5)
C(3)-H(3)	1.0000	C(4)-C(10)	1.507(5)
C(4)-C(5)	1.531(5)	C(5)-C(6)	1.543(6)
C(5)-H(5A)	0.9900	C(5)-H(5B)	0.9900
C(6)-C(7)	1.541(6)	C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900	C(7)-C(8)	1.509(6)
C(7)-C(10)	1.535(6)	C(7)-H(7A)	1.0000
C(8)-C(9)	1.534(5)	C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900	C(9)-H(9)	1.0000
C(10)-H(10A)	0.9900	C(10)-H(10B)	0.9900
O(1A)-C(1A)	1.285(4)	O(1A)-H(1AA)	1.19(5)
O(2A)-C(1A)	1.221(4)	O(3A)-C(2A)	1.413(4)
O(3A)-H(3A)	0.8400	O(4A)-C(3A)	1.408(4)
O(4A)-H(4A)	0.8400	O(5A)-C(4A)	1.279(4)
O(6A)-C(4A)	1.232(4)	C(1A)-C(2A)	1.539(4)
C(2A)-C(3A)	1.525(4)	C(2A)-H(2A)	1.0000
C(3A)-C(4A)	1.529(4)	C(3A)-H(3A1)	1.0000
O(1S)-C(1S)	1.410(6)	O(1S)-H(1S)	0.8400
C(1S)-H(1S1)	0.9800	C(1S)-H(1S2)	0.9800
C(1S)-H(1S3)	0.9800		

C(1)-N(1)-C(9)	105.5(3)	C(1)-N(1)-H(1A)	110.6
C(9)-N(1)-H(1A)	110.6	C(1)-N(1)-H(1B)	110.6
C(9)-N(1)-H(1B)	110.6	H(1A)-N(1)-H(1B)	108.8
C(4)-O(1)-H(1G)	109.5	N(1)-C(1)-C(2)	104.9(3)
N(1)-C(1)-H(1C)	110.8	C(2)-C(1)-H(1C)	110.8
N(1)-C(1)-H(1D)	110.8	C(2)-C(1)-H(1D)	110.8
H(1C)-C(1)-H(1D)	108.8	C(1)-C(2)-C(3)	107.2(3)
C(1)-C(2)-H(2B)	110.3	C(3)-C(2)-H(2B)	110.3
C(1)-C(2)-H(2C)	110.3	C(3)-C(2)-H(2C)	110.3
H(2B)-C(2)-H(2C)	108.5	C(2)-C(3)-C(9)	104.7(3)
C(2)-C(3)-C(4)	114.9(3)	C(9)-C(3)-C(4)	113.1(3)
C(2)-C(3)-H(3)	107.9	C(9)-C(3)-H(3)	107.9
C(4)-C(3)-H(3)	107.9	O(1)-C(4)-C(10)	112.5(3)
O(1)-C(4)-C(5)	112.2(3)	C(10)-C(4)-C(5)	102.3(3)
O(1)-C(4)-C(3)	107.7(3)	C(10)-C(4)-C(3)	108.6(3)
C(5)-C(4)-C(3)	113.6(3)	C(4)-C(5)-C(6)	106.1(3)
C(4)-C(5)-H(5A)	110.5	C(6)-C(5)-H(5A)	110.5
C(4)-C(5)-H(5B)	110.5	C(6)-C(5)-H(5B)	110.5
H(5A)-C(5)-H(5B)	108.7	C(7)-C(6)-C(5)	104.4(3)
C(7)-C(6)-H(6A)	110.9	C(5)-C(6)-H(6A)	110.9
C(7)-C(6)-H(6B)	110.9	C(5)-C(6)-H(6B)	110.9
H(6A)-C(6)-H(6B)	108.9	C(8)-C(7)-C(10)	108.8(4)
C(8)-C(7)-C(6)	113.5(3)	C(10)-C(7)-C(6)	102.3(3)
C(8)-C(7)-H(7A)	110.6	C(10)-C(7)-H(7A)	110.6
C(6)-C(7)-H(7A)	110.6	C(7)-C(8)-C(9)	114.0(3)
C(7)-C(8)-H(8A)	108.8	C(9)-C(8)-H(8A)	108.8
C(7)-C(8)-H(8B)	108.8	C(9)-C(8)-H(8B)	108.8
H(8A)-C(8)-H(8B)	107.7	N(1)-C(9)-C(8)	114.1(3)
N(1)-C(9)-C(3)	103.6(3)	C(8)-C(9)-C(3)	116.4(3)
N(1)-C(9)-H(9)	107.4	C(8)-C(9)-H(9)	107.4
C(3)-C(9)-H(9)	107.4	C(4)-C(10)-C(7)	101.3(3)
C(4)-C(10)-H(10A)	111.5	C(7)-C(10)-H(10A)	111.5
C(4)-C(10)-H(10B)	111.5	C(7)-C(10)-H(10B)	111.5
H(10A)-C(10)-H(10B)	109.3	C(1A)-O(1A)-H(1AA)	117(2)
C(2A)-O(3A)-H(3A)	109.5	C(3A)-O(4A)-H(4A)	109.5
O(2A)-C(1A)-O(1A)	126.1(3)	O(2A)-C(1A)-C(2A)	118.5(3)
O(1A)-C(1A)-C(2A)	115.4(3)	O(3A)-C(2A)-C(3A)	111.0(2)
O(3A)-C(2A)-C(1A)	113.5(2)	C(3A)-C(2A)-C(1A)	109.9(2)
O(3A)-C(2A)-H(2A)	107.4	C(3A)-C(2A)-H(2A)	107.4
C(1A)-C(2A)-H(2A)	107.4	O(4A)-C(3A)-C(2A)	111.2(2)
O(4A)-C(3A)-C(4A)	114.2(2)	C(2A)-C(3A)-C(4A)	108.9(2)
O(4A)-C(3A)-H(3A1)	107.4	C(2A)-C(3A)-H(3A1)	107.4
C(4A)-C(3A)-H(3A1)	107.4	O(6A)-C(4A)-O(5A)	125.7(3)
O(6A)-C(4A)-C(3A)	118.4(3)	O(5A)-C(4A)-C(3A)	115.9(3)
C(1S)-O(1S)-H(1S)	109.5	O(1S)-C(1S)-H(1S1)	109.5
O(1S)-C(1S)-H(1S2)	109.5	H(1S1)-C(1S)-H(1S2)	109.5
O(1S)-C(1S)-H(1S3)	109.5	H(1S1)-C(1S)-H(1S3)	109.5
H(1S2)-C(1S)-H(1S3)	109.5		

Symmetry transformations used to generate equivalent atoms:

Table S20. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 11e. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
N(1)	24(1)	31(2)	25(1)	-7(1)	-1(1)	-1(1)
O(1)	42(2)	57(2)	91(3)	4(2)	-31(2)	-11(2)
C(1)	46(2)	34(2)	26(2)	-10(2)	3(2)	-2(2)
C(2)	41(2)	66(3)	41(2)	-24(2)	-7(2)	0(2)
C(3)	37(2)	27(2)	46(2)	-1(2)	-6(2)	-5(2)
C(4)	25(2)	39(2)	45(2)	7(2)	-7(2)	-5(2)
C(5)	28(2)	35(2)	61(3)	9(2)	-8(2)	2(2)
C(6)	34(2)	34(2)	73(3)	-8(2)	9(2)	7(2)
C(7)	41(2)	67(3)	39(2)	-12(2)	15(2)	2(2)
C(8)	39(2)	53(2)	27(2)	-1(2)	6(2)	-8(2)
C(9)	33(2)	32(2)	35(2)	6(2)	1(2)	2(2)
C(10)	32(2)	53(3)	71(3)	10(2)	17(2)	3(2)
O(1A)	32(1)	35(1)	6(1)	-3(1)	0(1)	-4(1)
O(2A)	32(1)	52(2)	18(1)	5(1)	5(1)	-11(1)
O(3A)	25(1)	18(1)	18(1)	-5(1)	2(1)	-2(1)
O(4A)	48(2)	18(1)	15(1)	0(1)	9(1)	6(1)
O(5A)	44(2)	21(1)	12(1)	1(1)	4(1)	2(1)
O(6A)	50(2)	22(1)	16(1)	-6(1)	-3(1)	3(1)
C(1A)	30(2)	21(2)	12(1)	4(1)	1(1)	2(2)
C(2A)	27(2)	21(2)	11(1)	-2(1)	2(1)	-1(1)
C(3A)	26(2)	19(2)	14(1)	2(1)	3(1)	0(1)
C(4A)	23(2)	25(2)	12(1)	-6(1)	-4(1)	2(1)
O(1S)	101(3)	88(3)	53(2)	8(2)	-15(2)	-47(2)
C(1S)	89(4)	73(3)	57(3)	-17(3)	22(3)	-28(3)

Table S21. Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2 x\ 10^3$) for 11e.

	x	y	z	U(eq)
H(1A)	1601	3505	380	32
H(1B)	1972	2280	981	32
H(1G)	153	2353	-1494	95
H(1C)	1910	424	-1275	42
H(1D)	1776	2300	-2184	42
H(2B)	1210	-492	-1956	59
H(2C)	1046	1521	-2375	59
H(3)	854	-380	533	44
H(5A)	440	4789	-516	50
H(5B)	954	4164	-884	50
H(6A)	1171	5337	1679	56
H(6B)	657	5976	2042	56
H(7A)	694	3877	4213	59
H(8A)	1149	1316	4151	48
H(8B)	1463	2991	3657	48
H(9)	1499	126	1893	40
H(10A)	389	1072	2759	63
H(10B)	113	2890	2315	63
H(3A)	2682	7822	6358	30
H(4A)	1966	9446	4731	41
H(2A)	2210	4803	6155	23
H(3A1)	1580	6404	5293	24
H(1S)	55	6070	7075	121
H(1S1)	466	8870	6192	110
H(1S2)	572	7338	4785	110
H(1S3)	693	7020	6797	110
H(1AA)	2233(16)	7370(70)	10770(70)	89(16)

Table S22. Torsion angles [°] for 11e.

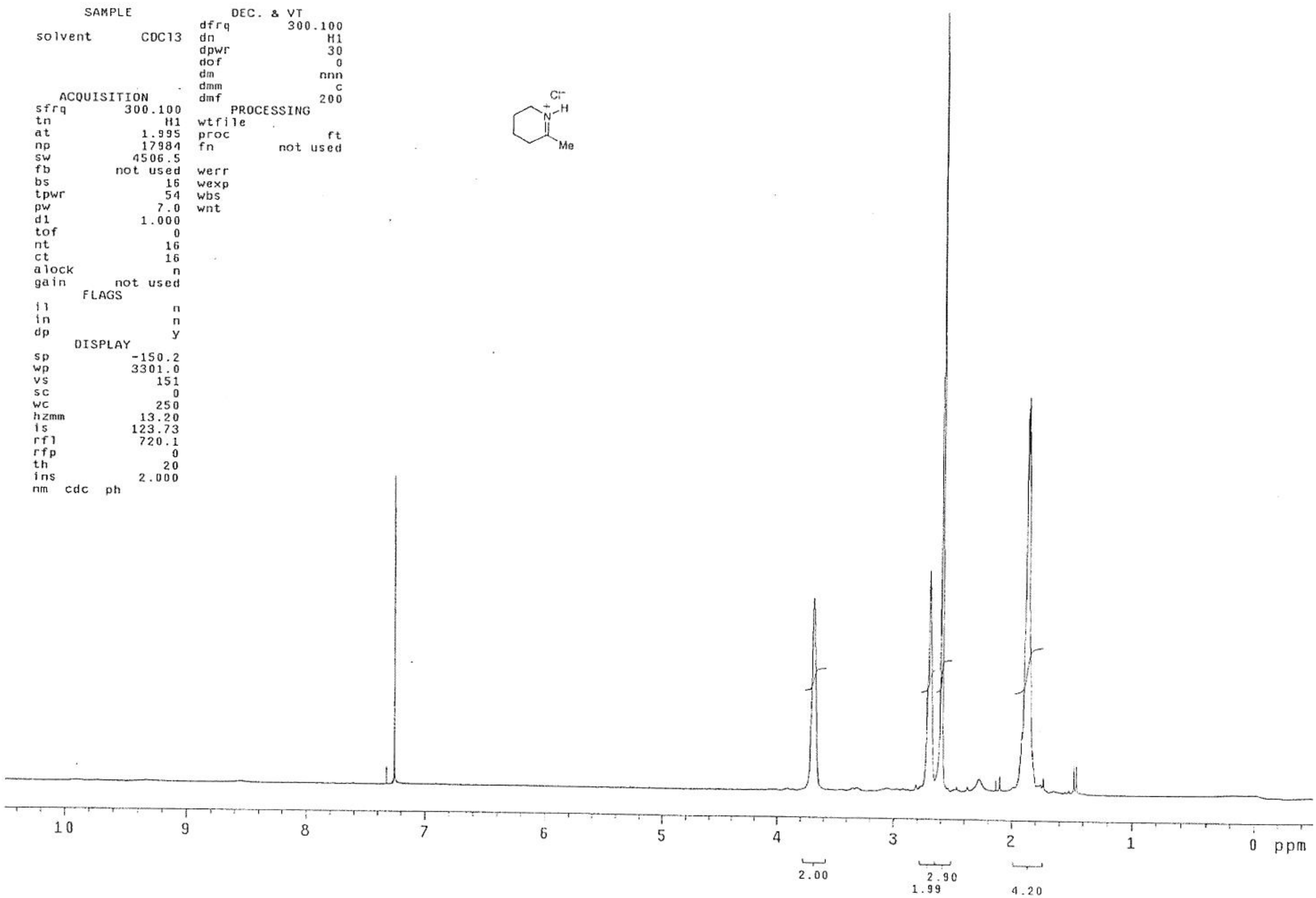
C(9)-N(1)-C(1)-C(2)	32.7(4)
N(1)-C(1)-C(2)-C(3)	-14.3(4)
C(1)-C(2)-C(3)-C(9)	-9.0(4)
C(1)-C(2)-C(3)-C(4)	115.7(3)
C(2)-C(3)-C(4)-O(1)	64.9(4)
C(9)-C(3)-C(4)-O(1)	-174.9(3)
C(2)-C(3)-C(4)-C(10)	-173.0(3)
C(9)-C(3)-C(4)-C(10)	-52.9(4)
C(2)-C(3)-C(4)-C(5)	-60.0(4)
C(9)-C(3)-C(4)-C(5)	60.2(4)
O(1)-C(4)-C(5)-C(6)	149.8(3)
C(10)-C(4)-C(5)-C(6)	29.0(4)
C(3)-C(4)-C(5)-C(6)	-87.8(4)
C(4)-C(5)-C(6)-C(7)	-0.4(4)
C(5)-C(6)-C(7)-C(8)	89.4(4)
C(5)-C(6)-C(7)-C(10)	-27.7(4)
C(10)-C(7)-C(8)-C(9)	52.2(5)
C(6)-C(7)-C(8)-C(9)	-61.0(5)
C(1)-N(1)-C(9)-C(8)	-165.9(3)
C(1)-N(1)-C(9)-C(3)	-38.4(3)
C(7)-C(8)-C(9)-N(1)	91.5(4)
C(7)-C(8)-C(9)-C(3)	-29.0(5)
C(2)-C(3)-C(9)-N(1)	28.6(4)
C(4)-C(3)-C(9)-N(1)	-97.3(3)
C(2)-C(3)-C(9)-C(8)	154.6(3)
C(4)-C(3)-C(9)-C(8)	28.8(4)
O(1)-C(4)-C(10)-C(7)	-166.9(3)
C(5)-C(4)-C(10)-C(7)	-46.3(4)
C(3)-C(4)-C(10)-C(7)	74.1(4)
C(8)-C(7)-C(10)-C(4)	-74.2(4)
C(6)-C(7)-C(10)-C(4)	46.2(4)
O(2A)-C(1A)-C(2A)-O(3A)	-175.2(3)
O(1A)-C(1A)-C(2A)-O(3A)	2.7(4)
O(2A)-C(1A)-C(2A)-C(3A)	59.8(4)
O(1A)-C(1A)-C(2A)-C(3A)	-122.2(3)
O(3A)-C(2A)-C(3A)-O(4A)	-69.8(3)
C(1A)-C(2A)-C(3A)-O(4A)	56.7(3)
O(3A)-C(2A)-C(3A)-C(4A)	57.0(3)
C(1A)-C(2A)-C(3A)-C(4A)	-176.6(2)
O(4A)-C(3A)-C(4A)-O(6A)	-178.2(3)
C(2A)-C(3A)-C(4A)-O(6A)	56.7(4)
O(4A)-C(3A)-C(4A)-O(5A)	0.6(4)
C(2A)-C(3A)-C(4A)-O(5A)	-124.4(3)

Symmetry transformations used to generate equivalent atoms:

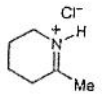
Table S23. Hydrogen bonds for 11e [Å and °].

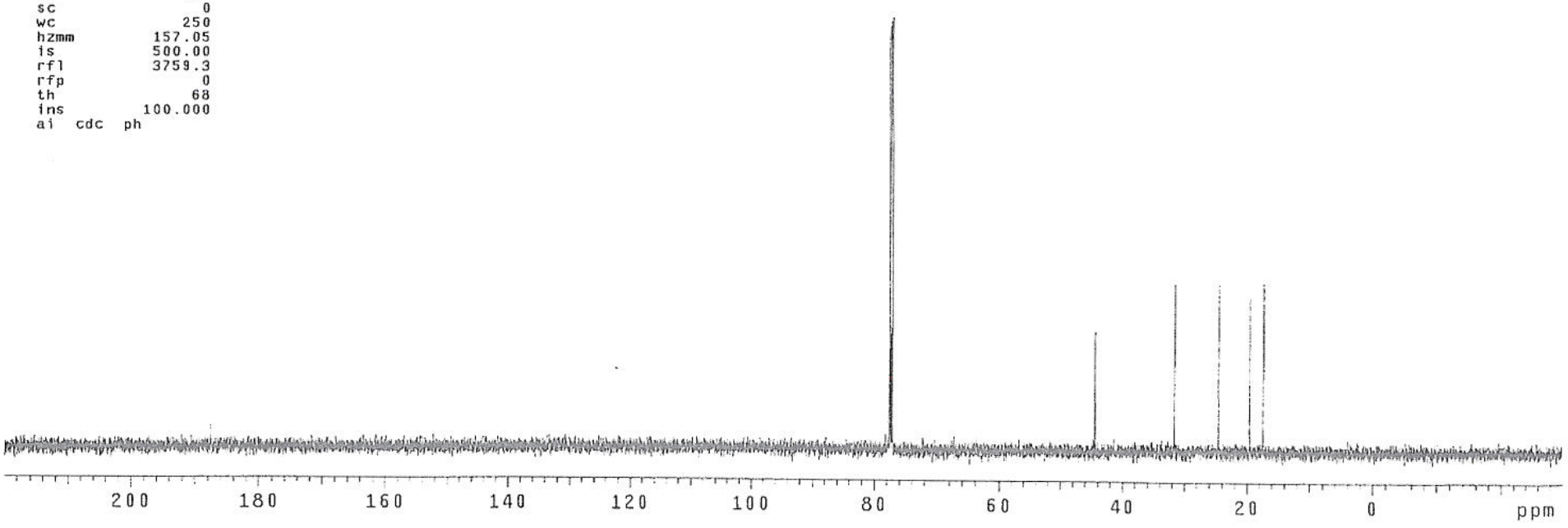
D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1S)-H(1S)...O(1S)#1	0.84	2.26	2.735(7)	115.8
O(4A)-H(4A)...O(3A)#2	0.84	2.12	2.891(3)	152.6
O(4A)-H(4A)...O(5A)	0.84	2.22	2.669(3)	113.3
O(3A)-H(3A)...O(6A)#2	0.84	1.89	2.696(3)	161.1
N(1)-H(1A)...O(2A)#3	0.92	2.17	2.945(4)	141.3
N(1)-H(1B)...O(1A)#4	0.92	2.05	2.880(4)	149.8
N(1)-H(1B)...O(3A)#4	0.92	2.53	3.289(3)	139.8
O(1)-H(1G)...O(1S)#5	0.84	1.91	2.679(5)	152.0
O(1A)-H(1AA)...O(5A)#6	1.19(5)	1.29(5)	2.459(3)	165(5)
O(1A)-H(1AA)...O(6A)#6	1.19(5)	2.40(5)	3.259(3)	128(3)

Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,z #2 -x+1/2,y+1/2,-z+1 #3 x,y,z-1
#4 -x+1/2,y-1/2,-z+1 #5 -x,-y+1,z-1 #6 x,y,z+1


SAMPLE
solvent CDCl3
ACQUISITION
sfrq 300.100
tn H1
at 1.995
np 17984
sw 4506.5
fb not used
bs 16
tpwr 54
pw 7.0
d1 1.000
tof 0
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -150.2
wp 3301.0
vs 151
sc 0
wc 250
hzmm 13.20
is 123.73
rfl 720.1
rfp 0
th 20
ins 2.000
nm cdc ph
DEC. & VT
dfrq 300.100
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
Cl⁻
N
H
Me
10
9
8
7
6
5
4
3
2
1
0 ppm
2.00
2.90
1.99
4.20

SAMPLE		DEC. & VT	
solvent	CDCl3	dfrq	499.747
		dn	H1
		dpwr	34
		dof	0
		dm	yyy
		dmm	w
C13080606.fid		dmf	10000
ACQUISITION		dseq	
sfrq	125.673	dres	1.0
tn	C13	homo	n
at	2.001	PROCESSING	
np	125638	lb	1.00
sw	31397.2	wtfile	
fb	not used	proc	ft
bs	8	fn	131072
tpwr	58	math	f
pw	6.7		
d1	3.000	werr	
tof	0	wexp	
nt	220	wbs	
ct	220	wnt	
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-3758.8		
wp	31396.7		
vs	655		
sc	0		
wc	250		
hzmm	157.05		
is	500.00		
rfl	3759.3		
rfp	0		
th	68		
ins	100.000		
ai cdc ph			





SAMPLE	
solvent	CDCl3

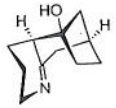
ACQUISITION	
sfrq	499.758
tn	H1
at	3.277
np	65536
sw	9998.8
fb	not used
bs	16
tpwr	56
pw	8.2
d1	0
tof	1498.1
nt	16
ct	16
alock	n
gain	not used

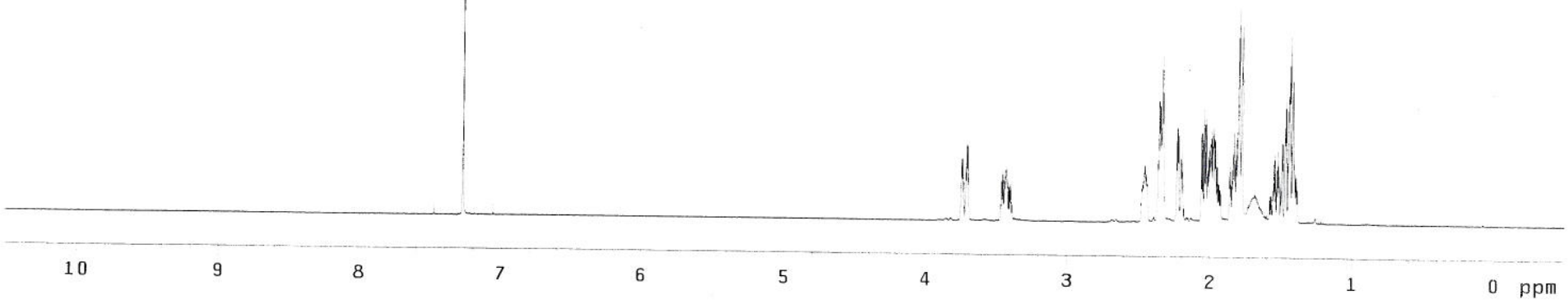
FLAGS	
il	n
in	n
dp	y
hs	nn

DISPLAY	
sp	-250.1
wp	5497.1
vs	261
sc	0
wc	250
hzmm	21.99
is	33.57
rfl	1002.5
rfp	0
th	7
ins	1.000
nm	cdc ph

DEC. & VT	
dfrq	125.677
dn	C13
dpwr	34
dof	1498.1
dm	nnn
dmm	w
dmf	10000
dseq	
dres	1.0
homo	n

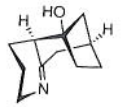
PROCESSING	
wtfile	
proc	ft
fn	65536
math	f
werr	
wexp	
wbs	
wnt	

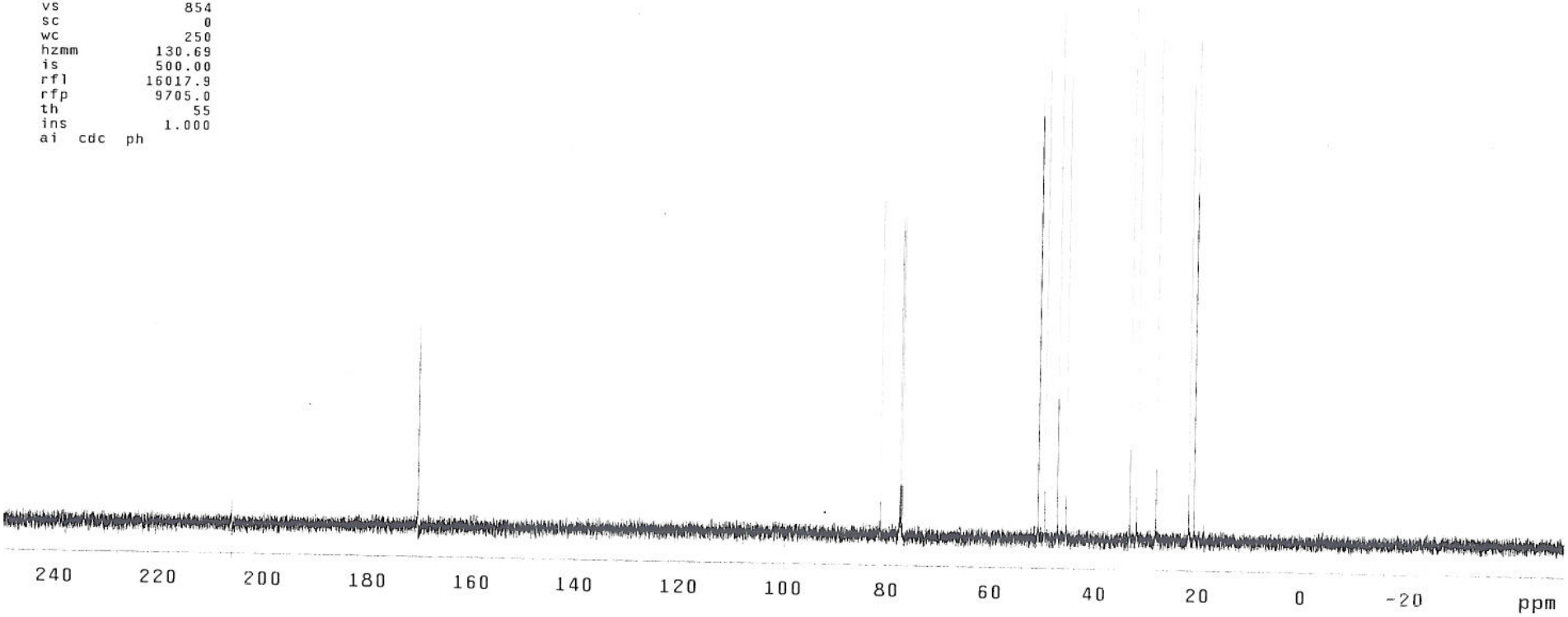




SAMPLE	
solvent	CDCl3
file /	
	onc_C.fid
ACQUISITION	
sfrq	125.676
tn	C13
at	0.869
np	65536
sw	37718.1
fb	not used
bs	16
ss	1
tpwr	58
pw	7.5
d1	3.000
tof	615.5
nt	350
ct	350
alock	n
gain	not used
FLAGS	
il	n
in	n
dp	y
hs	nn
DISPLAY	
sp	-6312.3
wp	37717.5
vs	854
sc	0
wc	250
hzmm	130.69
is	500.00
rfl	16017.9
rfp	9705.0
th	55
ins	1.000
ai cdc ph	

DEC. & VT	
dfrq	499.756
dn	H1
dpwr	34
dof	0
dm	yyy
dmm	w
dmf	10000
dseq	
dres	1.0
homo	n
PROCESSING	
lb	1.00
wtfile	
proc	ft
fn	131072
math	f
werr	
wexp	
wbs	
wnt	





SAMPLE	
solvent	Benzene
file	

ACQUISITION	
sfrq	499.758
tn	H1
at	3.277
np	65536
sw	9998.8
fb	not used
bs	1
tpwr	56
pw	8.2
d1	0
tof	1498.1
nt	16
ct	16
alock	n
gain	not used

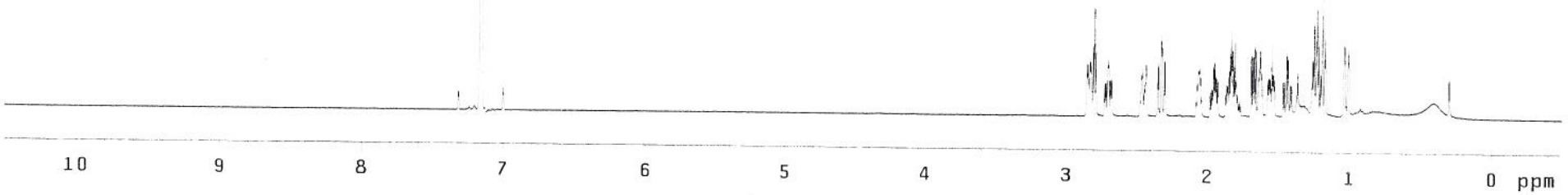
FLAGS	
il	n
in	n
dp	y
hs	nn

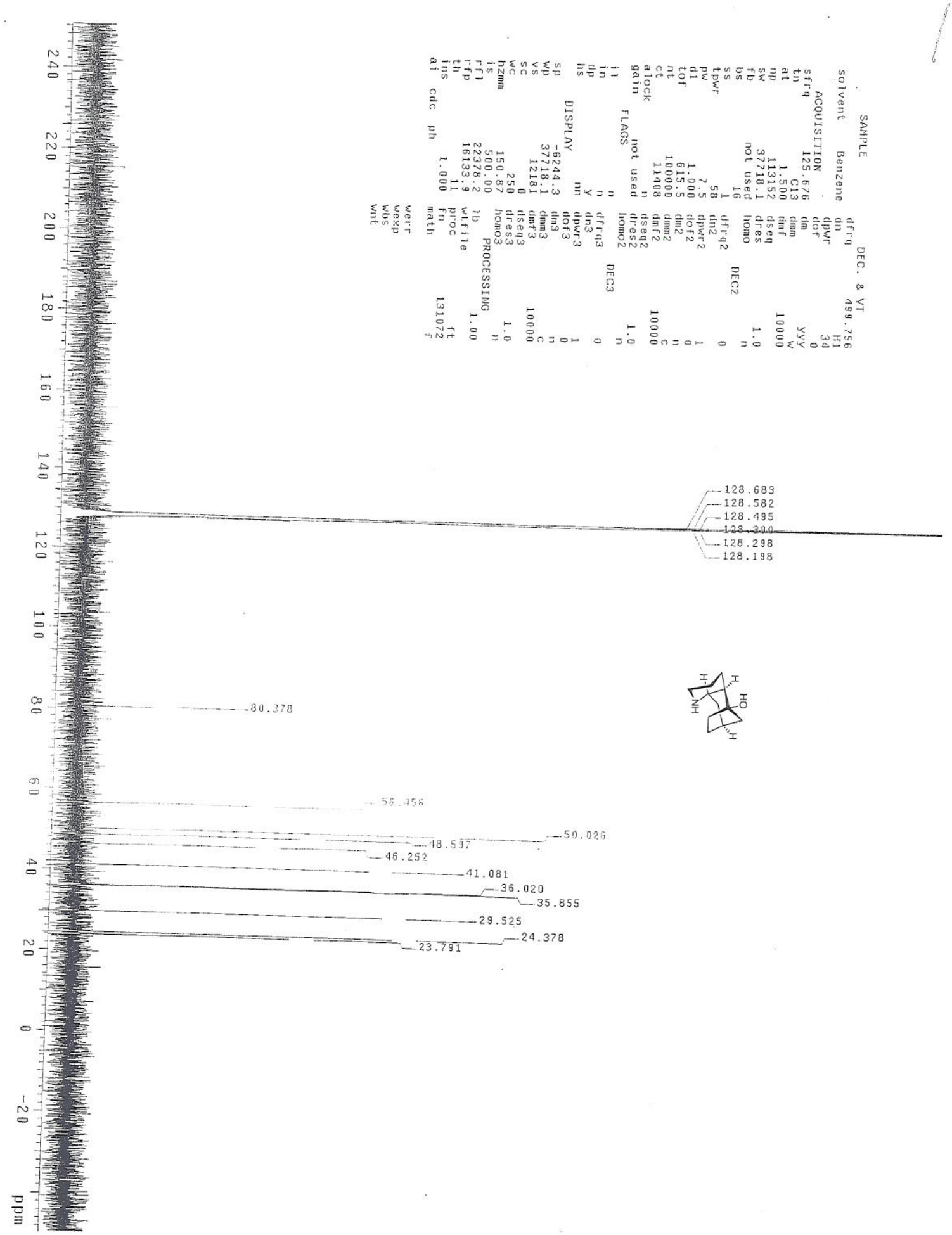
DISPLAY	
sp	-250.2
wp	5497.1
vs	536
sc	0
wc	250
hzmm	21.99
is	109.53
rfl	4563.5
rfp	3578.2
th	7
ins	1.000

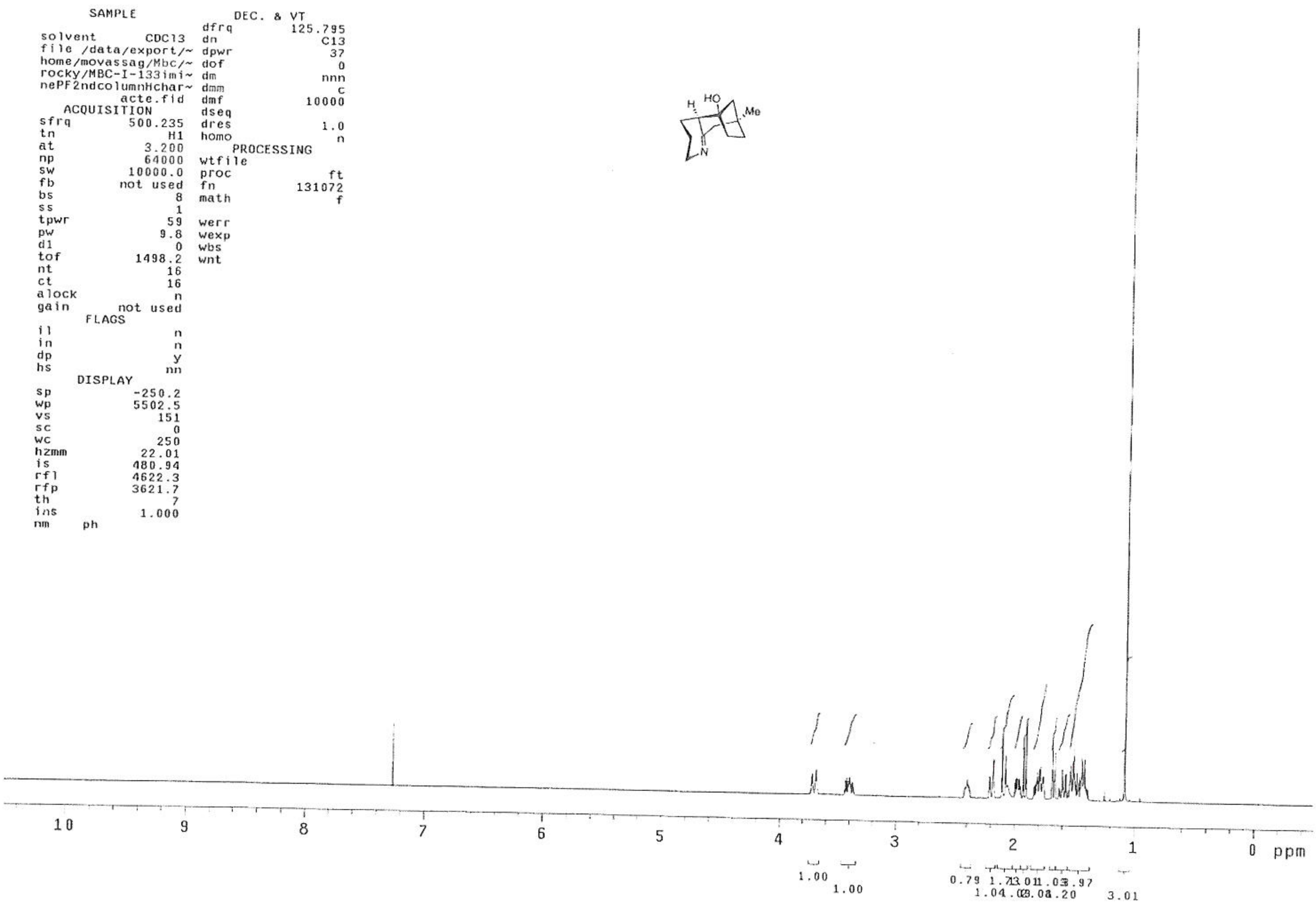
nm cdc ph

DEC. & VT	
dfrq	125.677
dn	C13
dpwr	34
dof	1498.1
dm	nnn
dmm	w
dmf	10000
dseq	
dres	1.0
homo	n

PROCESSING	
wtfile	
proc	ft
fn	65536
math	f
werr	
wexp	
wbs	
wnt	

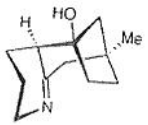



SAMPLE
solvent Benzene
ACQUISITION
sfrq 125.676
tn C13
at 1.500
np 113152
sw 37718.1
fb not used
bs 16
ss 1
tpwr 58
pw 7.5
pw1 1.000
d1 615.5
tof 100000
nt 11408
ct
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -6244.3
wp 37718.1
vs 12181
sc 0
wc 250
hzmm 150.87
is 500.00
rfl 22378.2
rfp 16133.9
th 11
ins 1.000
ai cdc ph
wnt
wbs
wexp
werr
DEC. & VT
dfrq
dn
dpwr
dof
dm
dmm
dmf
dseq
dres
homo
DEC2
dfrq2
dn2
dpwr2
dof2
dm2
dmm2
dmf2
dseq2
dres2
homo2
DEC3
dfrq3
dn3
dpwr3
dof3
dm3
dmm3
dmf3
dseq3
dres3
homo3
PROCESSING
lb
wtfile
proc
fn
math
499.756
H1
34
0
yyy
w
10000
1.0
n
0
1
0
n
c
10000
1.0
n
1.00
ft
131072
f
128.683
128.582
128.495
128.390
128.298
128.198
80.378
56.456
50.026
48.597
46.252
41.081
36.020
35.855
29.525
24.378
23.791
ppm

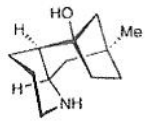

SAMPLE
solvent CDCl3
file /data/export/~
home/movassag/Mbc/~
rocky/MBC-I-133imi~
nePF2ndcolumnHchar~
acte.fid
ACQUISITION
sfrq 500.235
tn H1
at 3.200
np 64000
sw 10000.0
fb not used
bs 8
ss 1
tpwr 59
pw 9.8
d1 0
tof 1498.2
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 5502.5
vs 151
sc 0
wc 250
hzmm 22.01
is 480.94
rfl 4622.3
rfp 3621.7
th 7
ins 1.000
nm ph
DEC. & VT
dfrq 125.795
dn C13
dpwr 37
dof 0
dm nnn
dmm c
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
HO
H
Me
N
10
9
8
7
6
5
4
3
2
1
0 ppm
1.00
1.00
0.79
1.04
3.01

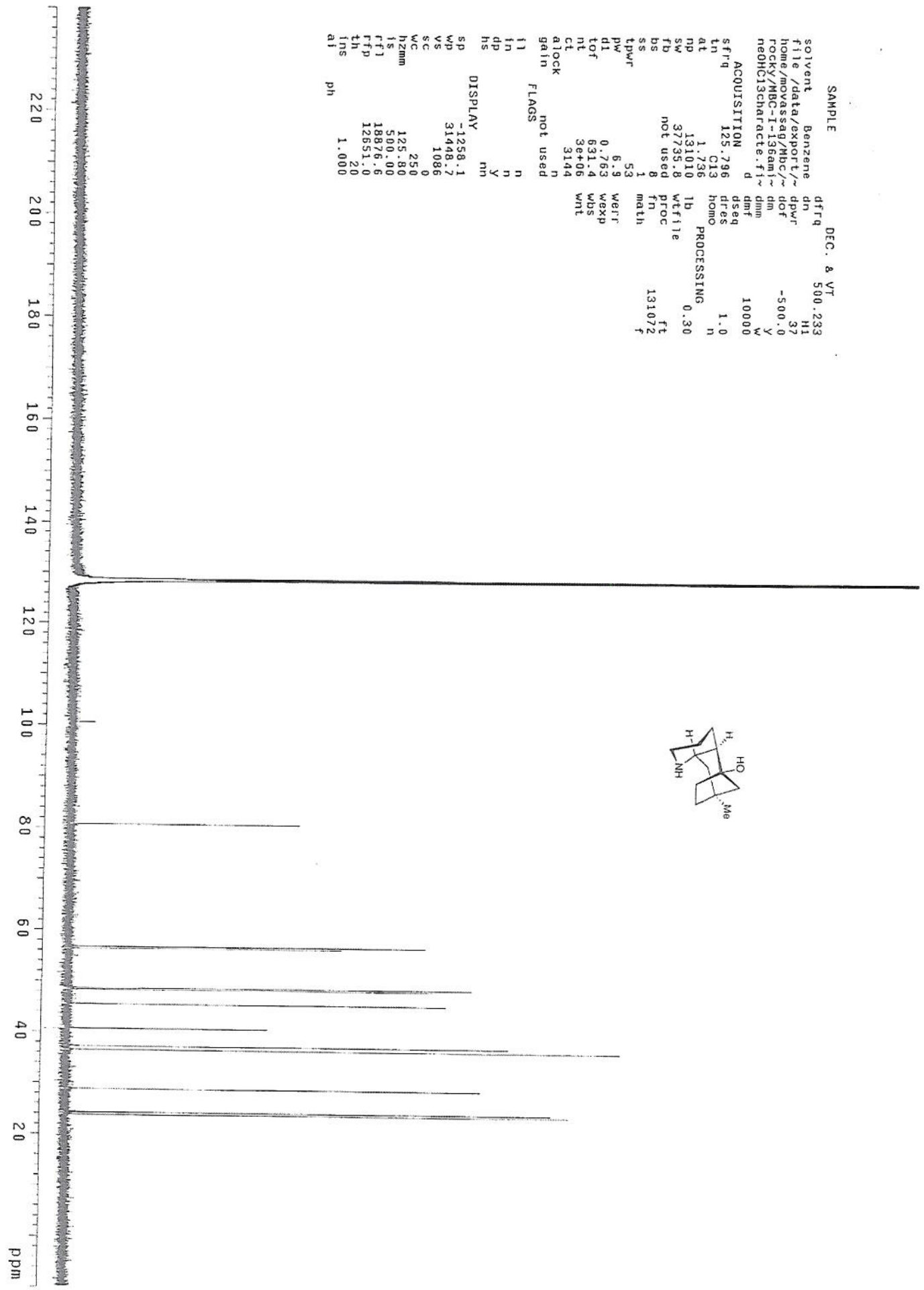
SAMPLE	
solvent	CDCl3
file	/data/export/~
home/movassag/Mbc/~	
rocky/MBC-I-133imi~	
neC13characteinCDC~	
	13.fid
ACQUISITION	
sfrq	125.796
tn	C13
at	1.736
np	131010
sw	37735.8
fb	not used
bs	8
ss	1
tpwr	53
pw	6.9
d1	0.763
tof	631.4
nt	200000
ct	808
alock	n
gain	not used
FLAGS	
il	n
in	n
dp	y
hs	nn
DISPLAY	
sp	-1258.3
wp	31448.1
vs	1346
sc	0
wc	250
hzmm	125.79
is	500.00
rfl	15911.8
rfp	9686.0
th	43
ins	1.000
ai	ph

DEC. & VT	
dfrq	500.233
dn	H1
dpwr	37
dof	-500.0
dm	y
dmm	w
dmf	10000
dseq	
dres	1.0
homo	n
PROCESSING	
lb	0.30
wtfile	
proc	ft
fn	131072
math	f
werr	
wexp	
wbs	
wnt	



SAMPLE		DEC. & VT	
solvent	Benzene	dfrq	125.795
file	/data/export/~	dn	C13
home/movassag/Mbc/~		dpwr	37
rocky/MBC-I-136ami~		dof	0
neOHHchara501.fid		dm	nnn
ACQUISITION		dmm	c
sfrq	500.235	dmf	10000
tn	H1	dseq	
at	3.200	dres	1.0
np	64000	homo	n
sw	10000.0	PROCESSING	
fb	not used	wtfile	
bs	8	proc	ft
ss	1	fn	131072
tpwr	59	math	f
pw	9.8	werr	
d1	0	wexp	
tof	1498.2	wbs	
nt	16	wnt	
ct	16		
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-250.2		
wp	5502.5		
vs	151		
sc	0		
wc	250		
hzmm	22.01		
is	575.40		
rfl	4577.3		
rfp	3576.7		
th	7		
ins	1.000		
nm	ph		




SAMPLE
solvent Benzene
file /data/export/~
home/movassag/Mbc/~
rocky/MBC-I-136am1~
ne0HC13characte.fi~
d
ACQUISITION
sfrq 125.796
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 3e+06
ct 3144
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1258.1
wp 31448.7
vs 1086
sc 0
wc 250
hzmm 125.80
is 500.00
rfl 18876.6
rfp 12651.0
th 20
ins 1.000
ai ph
dfrq DEC. & VT
dn H1
dpwr 37
dof -500.0
dm y
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
lb 0.30
wtfile
proc ft
fn 131072
math f
wexp
wbs
wnt
wer
HO
NH
Me
220
200
180
160
140
120
100
80
60
40
20
ppm

SAMPLE	
solvent	Benzene

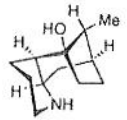
ACQUISITION	
sfrq	499.749
tn	H1
at	3.277
np	65536
sw	9998.8
fb	not used
bs	16
tpwr	56
pw	8.2
d1	0
tof	1498.1
nt	48
ct	48
alock	n
gain	not used

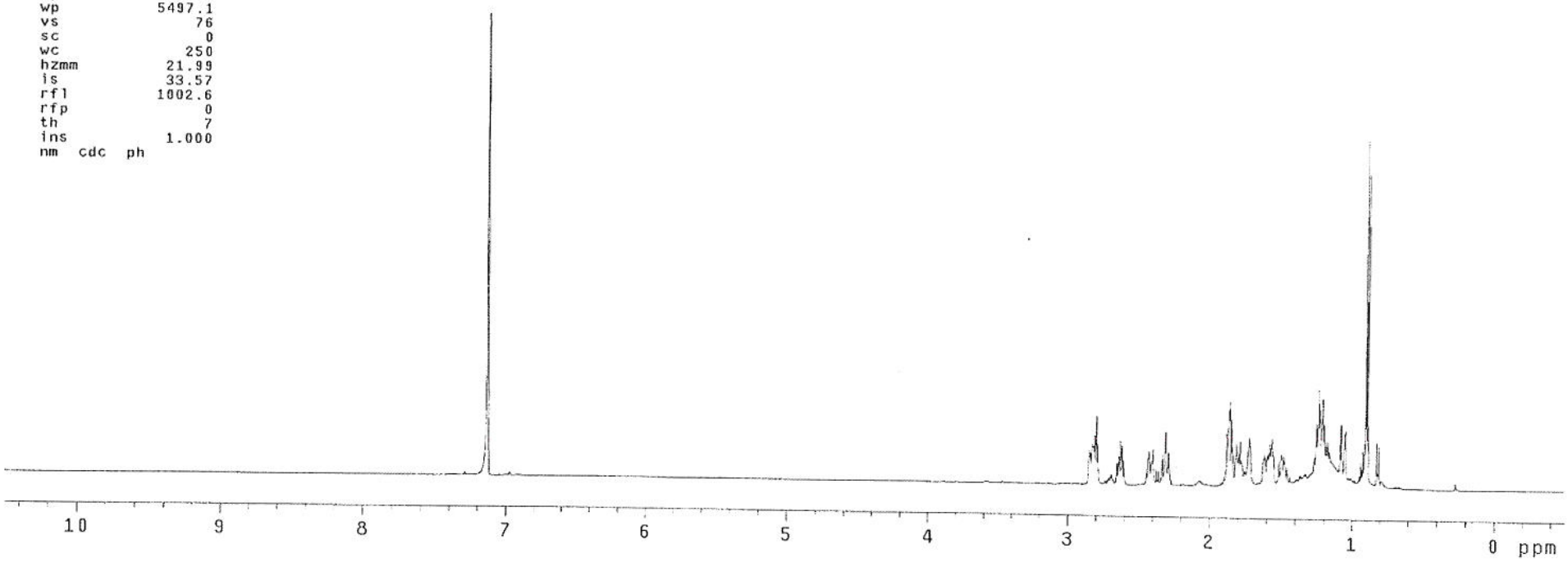
FLAGS	
il	n
in	n
dp	y
hs	nn

DISPLAY	
sp	-250.2
wp	5497.1
vs	76
sc	0
wc	250
hzmm	21.99
is	33.57
rfl	1002.6
rfp	0
th	7
ins	1.000
nm	cdc ph

DEC. & VT	
dfrq	125.674
dn	C13
dpwr	34
dof	1498.1
dm	nnn
dmm	w
dmf	10000
dseq	
dres	1.0
homo	n

PROCESSING	
wtfile	
proc	ft
fn	65536
math	f
werr	
wexp	
wbs	
wnt	





SAMPLE	
solvent	Benzene
file	/data/export/~
	home/movassag/Mbc/~
	rocky/MBC-II-54ami~
	noOHC13.fid

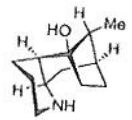
ACQUISITION	
sfrq	125.796
tn	C13
at	1.736
np	131010
sw	37735.8
fb	not used
bs	8
ss	1
tpwr	53
pw	6.9
d1	0.763
tof	631.4
nt	1e+09
ct	11552
alock	n
gain	not used

FLAGS	
il	n
in	n
dp	y
hs	nn

DISPLAY	
sp	-6225.0
wp	37735.3
vs	1606
sc	0
wc	250
hzmm	150.94
is	500.00
rfl	18876.6
rfp	12651.0
th	20
ins	1.000
ai	ph

DEC. & VT	
dfrq	500.233
dn	H1
dpwr	37
dof	-500.0
dm	y
dmm	w
dmf	10000
dseq	
dres	1.0
homo	n

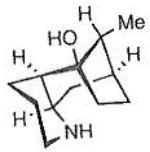
PROCESSING	
lb	0.30
wtfile	
proc	ft
fn	131072
math	f
werr	
wexp	
wbs	
wnt	

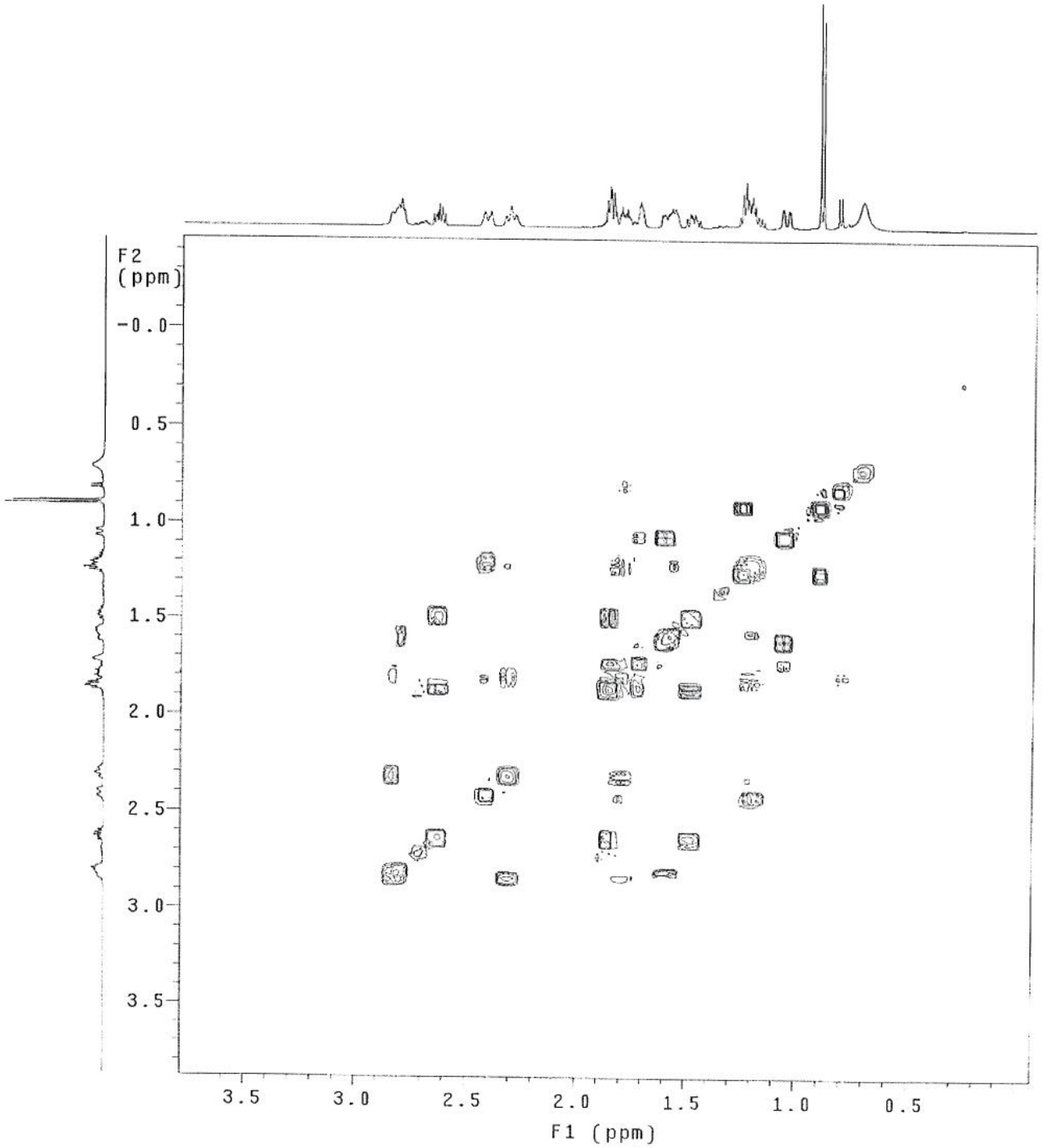


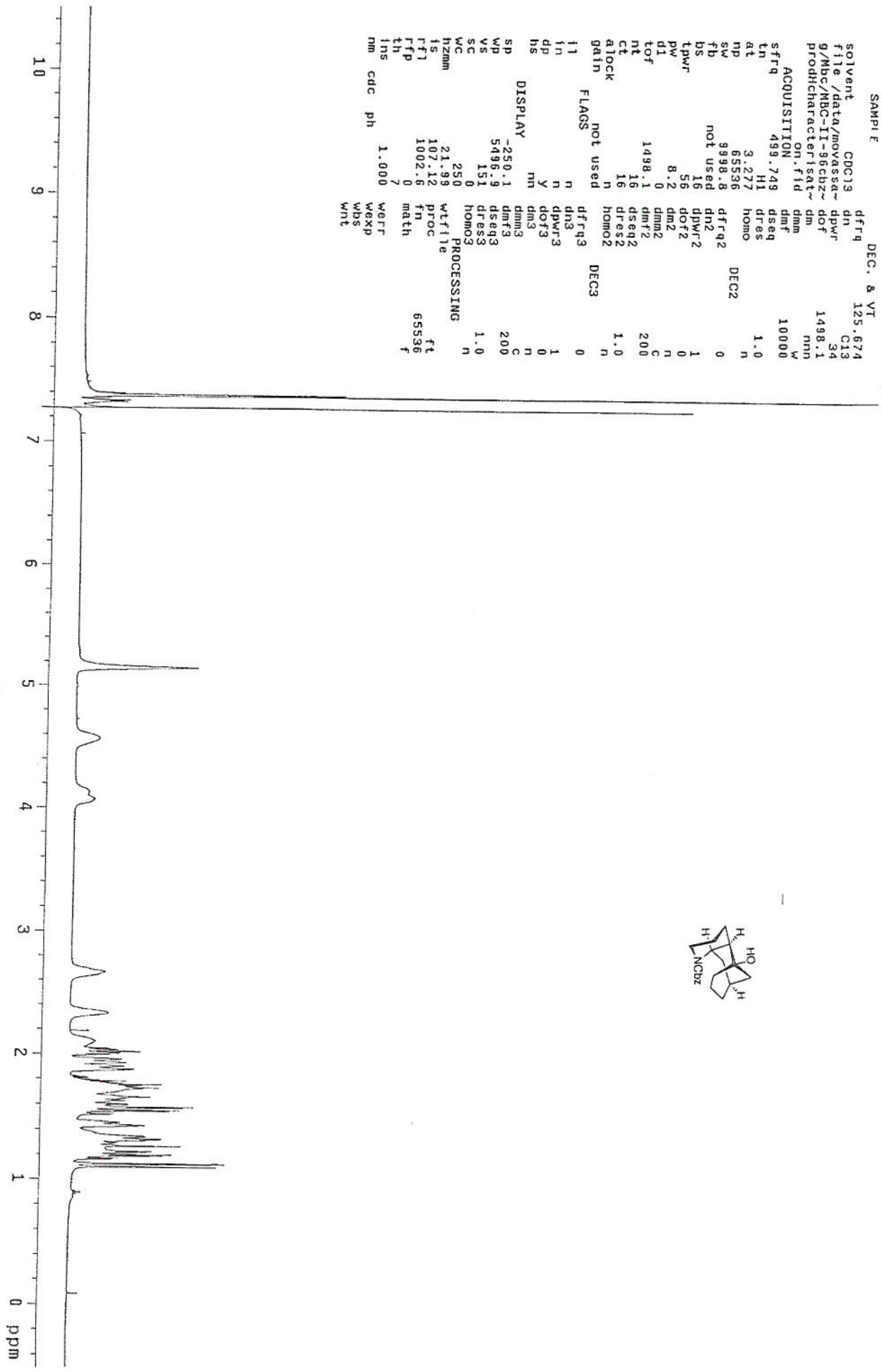
Pulse Sequence: gCOSY

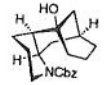
Solvent: Benzene
Temp. 22.0 C / 295.1 K
File: MBC-III-84aminoOHgCOSY
INOVA-500 "zippy"

PULSE SEQUENCE: gCOSY
Relax. delay 1.000 sec
Acq. time 0.195 sec
Width 5252.1 Hz
2D Width 5252.1 Hz
22 repetitions
128 increments
OBSERVE H1, 499.7446971 MHz
DATA PROCESSING
Sq. sine bell 0.097 sec
F1 DATA PROCESSING
Sq. sine bell 0.024 sec
FT size 2048 x 2048
Total time 0 min, -1 sec






SAMPLE
solvent CDCl3
file /data/movassa~
g/Mbc/MBC-II-96cbz~
prodHcharacterisat~
ACQUISITION
sfrq 499.749
tn H1
at 3.277
np 65536
sw 9998.8
fb not used
bs 16
tpwr 56
pw 8.2
d1 0
tof 1498.1
nt 16
ct 16
alock n
gain n
FLAGS
not used
DISPLAY
-250.1
5496.9
151
0
250
21.99
107.12
1002.6
0
7
1.000
cdc ph
dfrq
dn
dpwr
dof
dm
dmm
dmf
dseq
dres
homo
DEC2
dfrq2
dn2
dpwr2
dof2
dm2
dmm2
dmf2
dseq2
dres2
homo2
DEC3
dfrq3
dn3
dpwr3
dof3
dm3
dmm3
dmf3
dseq3
dres3
homo3
PROCESSING
wtfile
proc
fn
math
werr
wexp
wbs
wnt
DEC. & VT
125.674
C13
34
1498.1
nnn
w
10000
1.0
n
n
DEC2
0
1
0
200
n
DEC3
0
1
0
n
c
200
1.0
n
ft
65536
f
HO
H
H
NCbz
10
9
8
7
6
5
4
3
2
1
0 ppm



Current Data Parameters

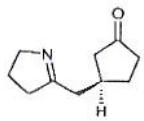
EXPNO	1
PROCNO	1

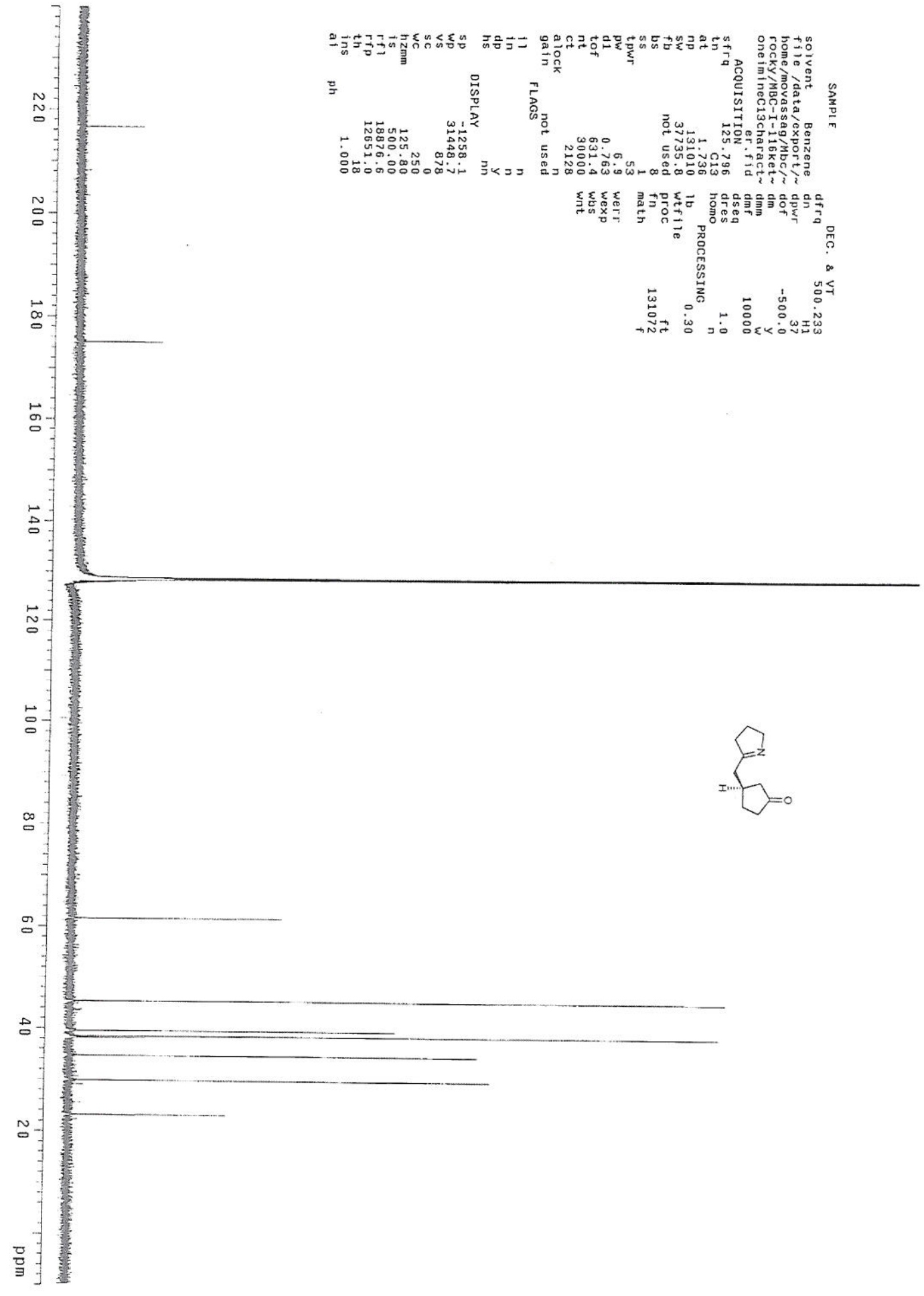
F2 - Acquisition Parameters

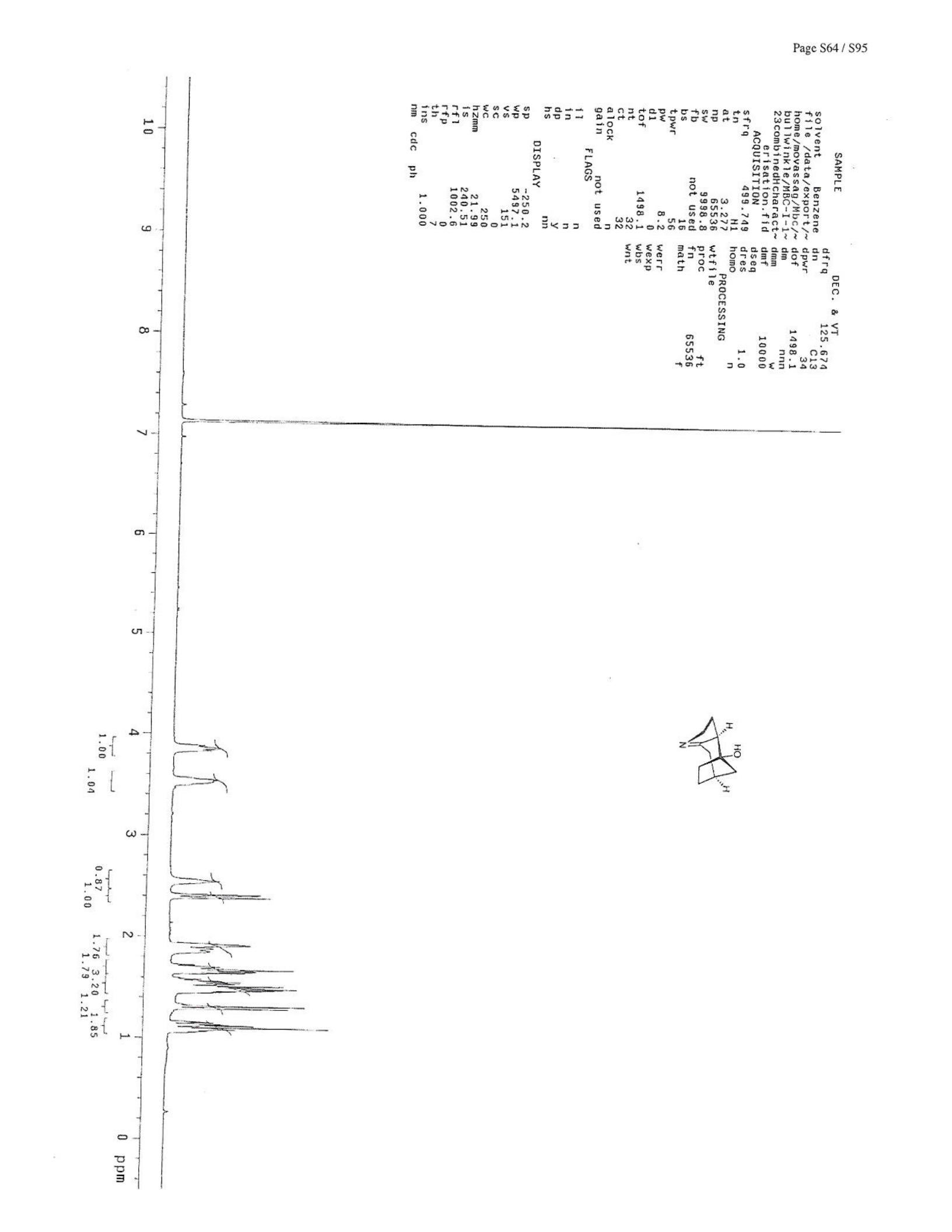
Time	20.07	
INSTRUM	spect	
PROBHD	5 mm QNP 1H/1	
PULPROG	zgpg30	
TD	65536	
SOLVENT	C6D6	
NS	13647	
DS	4	
SWH	23980.814	Hz
FIDRES	0.365918	Hz
AQ	1.3664756	sec
RG	1024	
DW	20.850	usec
DE	6.00	usec
TE	294.8	K
D1	2.00000000	sec
d11	0.03000000	sec
DELTA	1.89999998	sec
MCREST	0.00000000	sec
MCWRK	0.01500000	sec

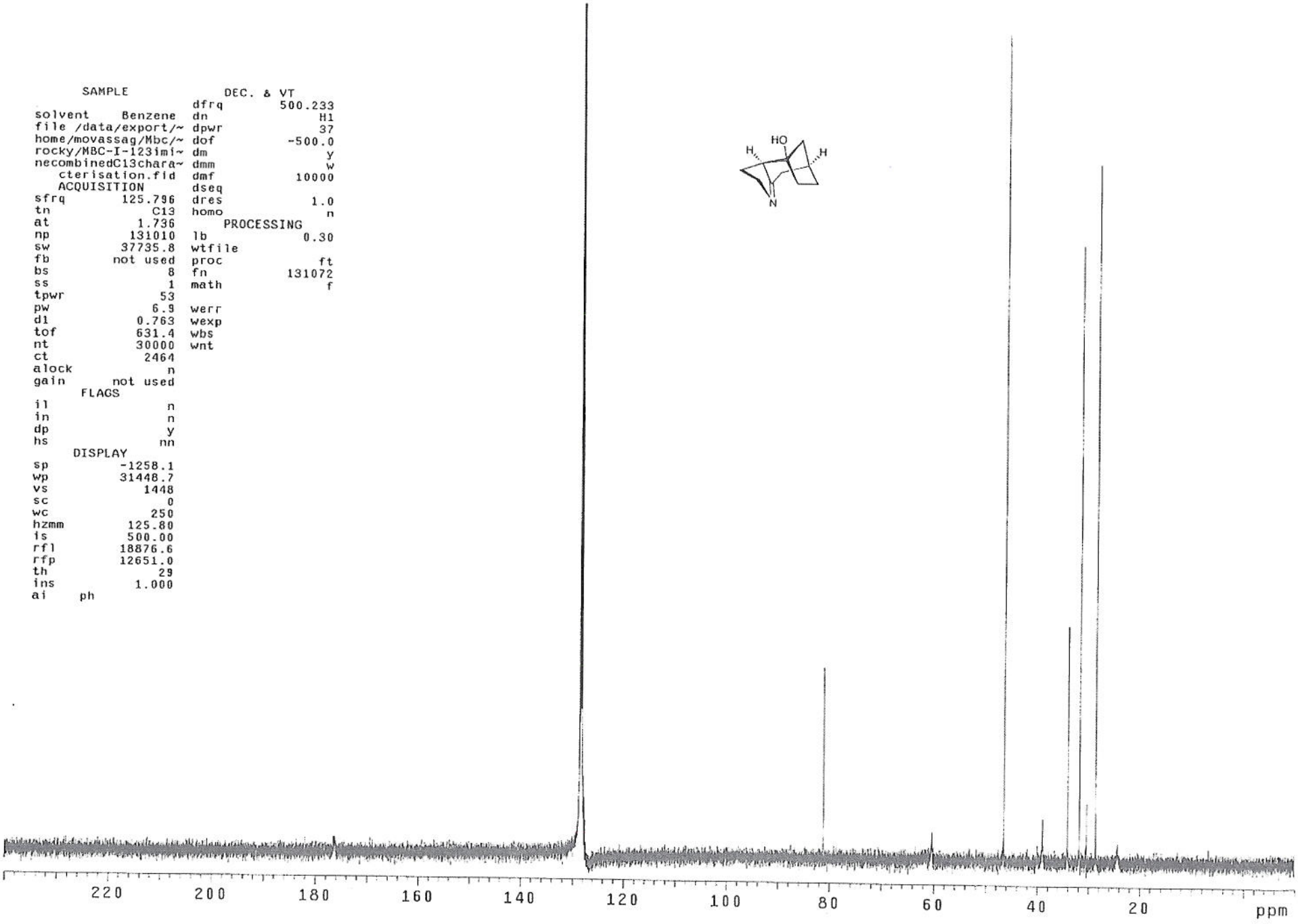
SAMPLE	
solvent	Benzene
file	/data/export/~
	home/movassag/Mbc/~
	bullwinkle/MBC-I-1~
	16PFinBenzene.fid
ACQUISITION	
sfrq	499.749
tn	H1
at	3.277
np	65536
sw	9998.8
fb	not used
bs	16
tpwr	56
pw	8.2
d1	0
tof	1498.1
nt	32
ct	32
alock	n
gain	not used
FLAGS	
il	n
in	n
dp	y
hs	nn
DISPLAY	
sp	-249.9
wp	5497.1
vs	151
sc	0
wc	250
hzmm	21.99
is	446.24
rfl	4566.8
rfp	3578.2
th	7
ins	2.000
nm cdc ph	

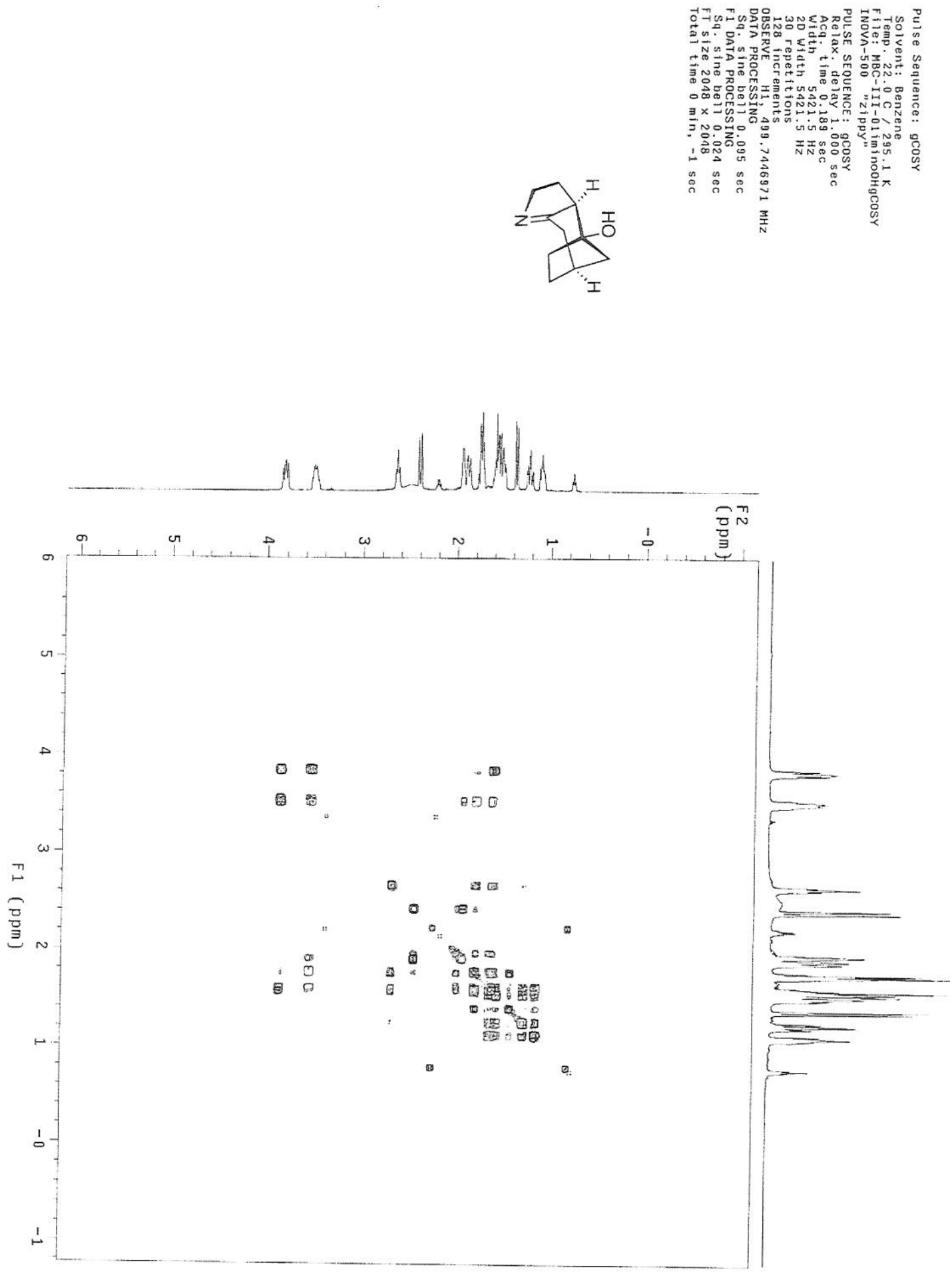
DEC. & VT	
dfrq	125.674
dn	C13
dpwr	34
dof	1498.1
dm	nnn
dmm	w
dmf	10000
dseq	
dres	1.0
homo	n
PROCESSING	
wtfile	
proc	ft
fn	65536
math	f
werr	
wexp	
wbs	
wnt	

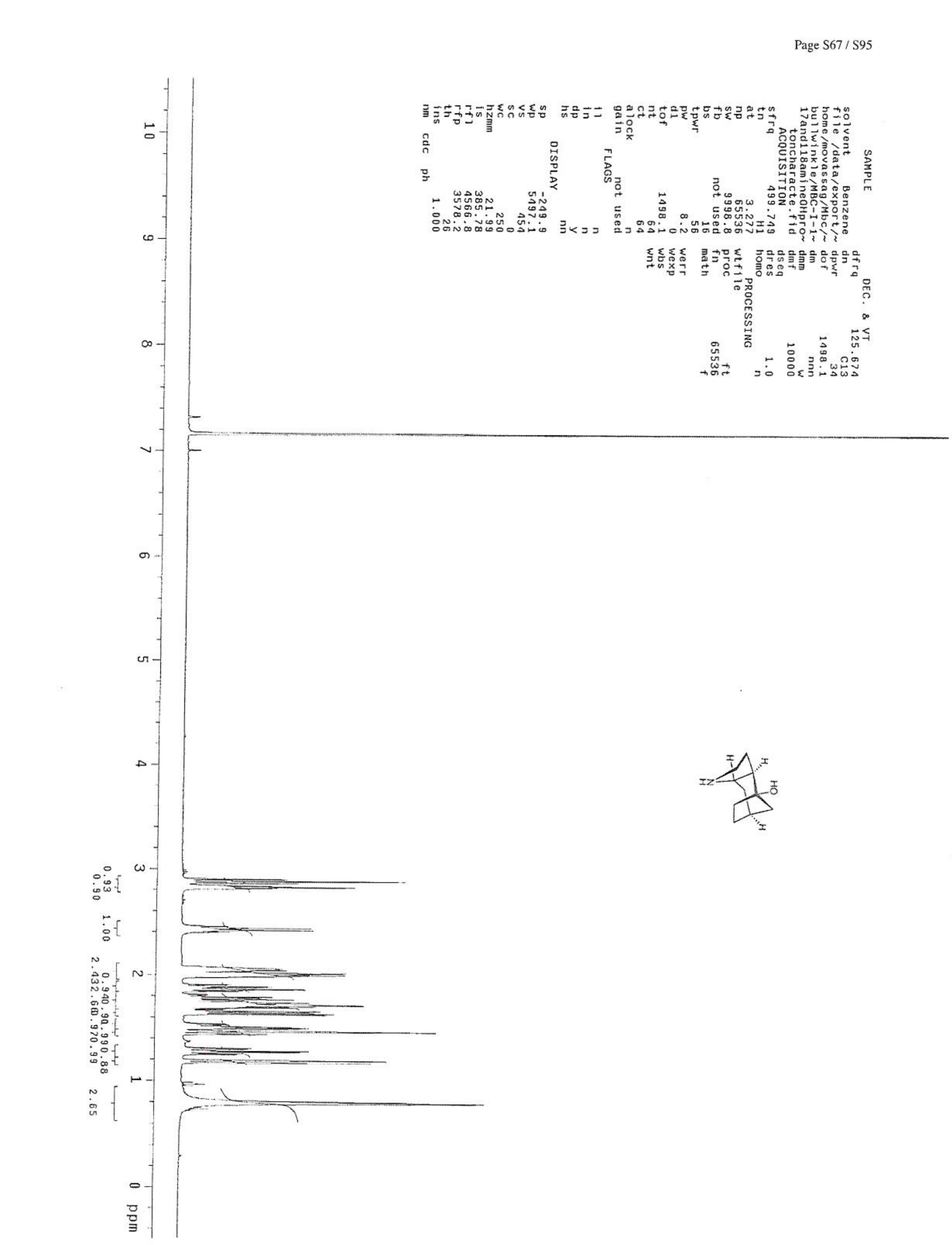


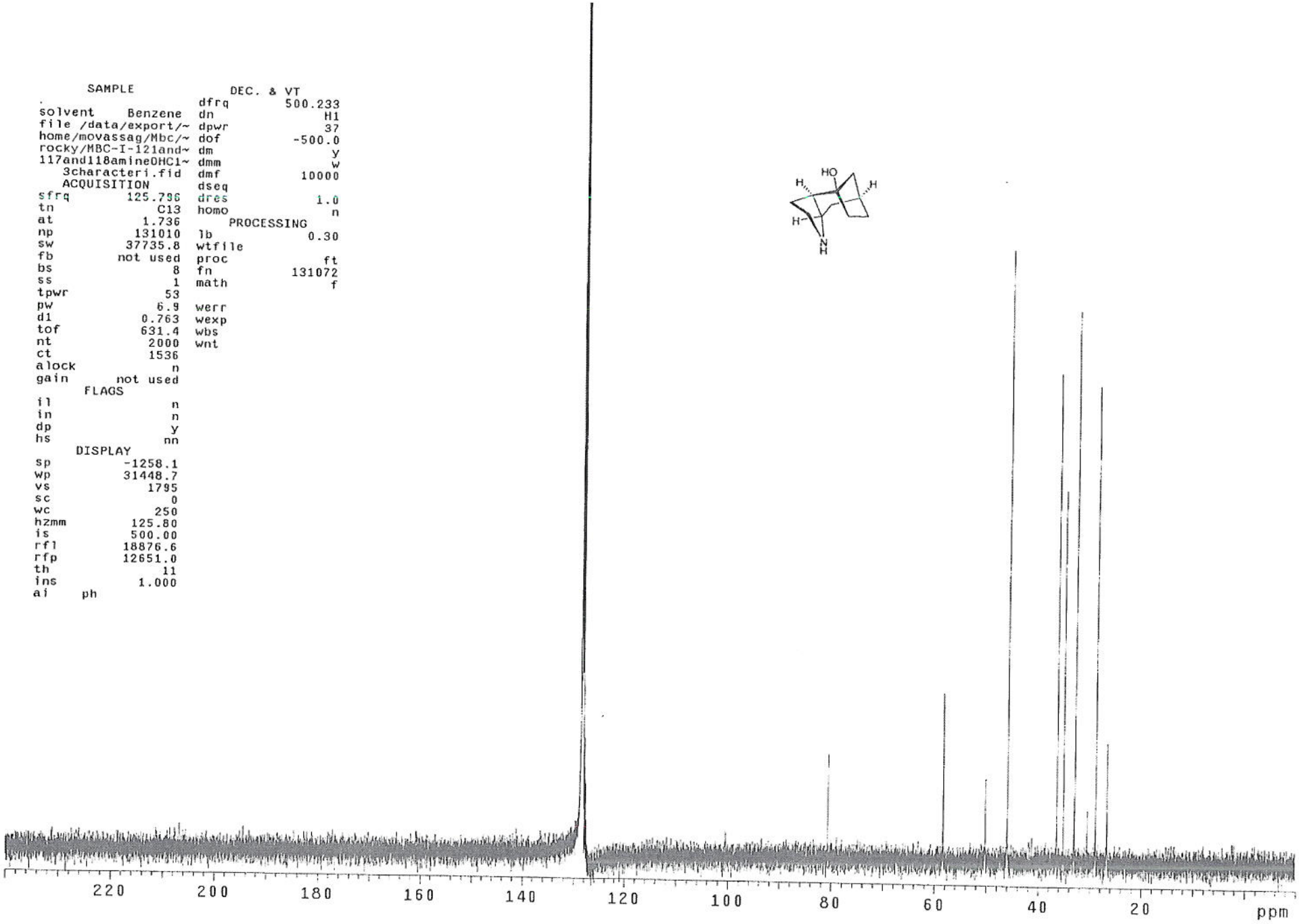

SAMPLE
solvent Benzene
file /data/export/~
home/movassag/Mbc/~
rocky/MBC-I-116ket~
oneImineC13charact~
ACQUISITION
sfrq 125.796
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 30000
ct 2128
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1258.1
wp 31448.7
vs 878
sc 0
wc 250
hzmm 125.80
is 500.00
rfl 18876.6
rfp 12651.0
th 18
ins 1.000
ai ph
dfrq DEC. & VT
dn 500.233
dpwr H1
dof 37
dm -500.0
dmm y
dmf w
dseq 10000
dres 1.0
homo n
PROCESSING
lb 0.30
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
N
H
O
220
200
180
160
140
120
100
80
60
40
20
ppm

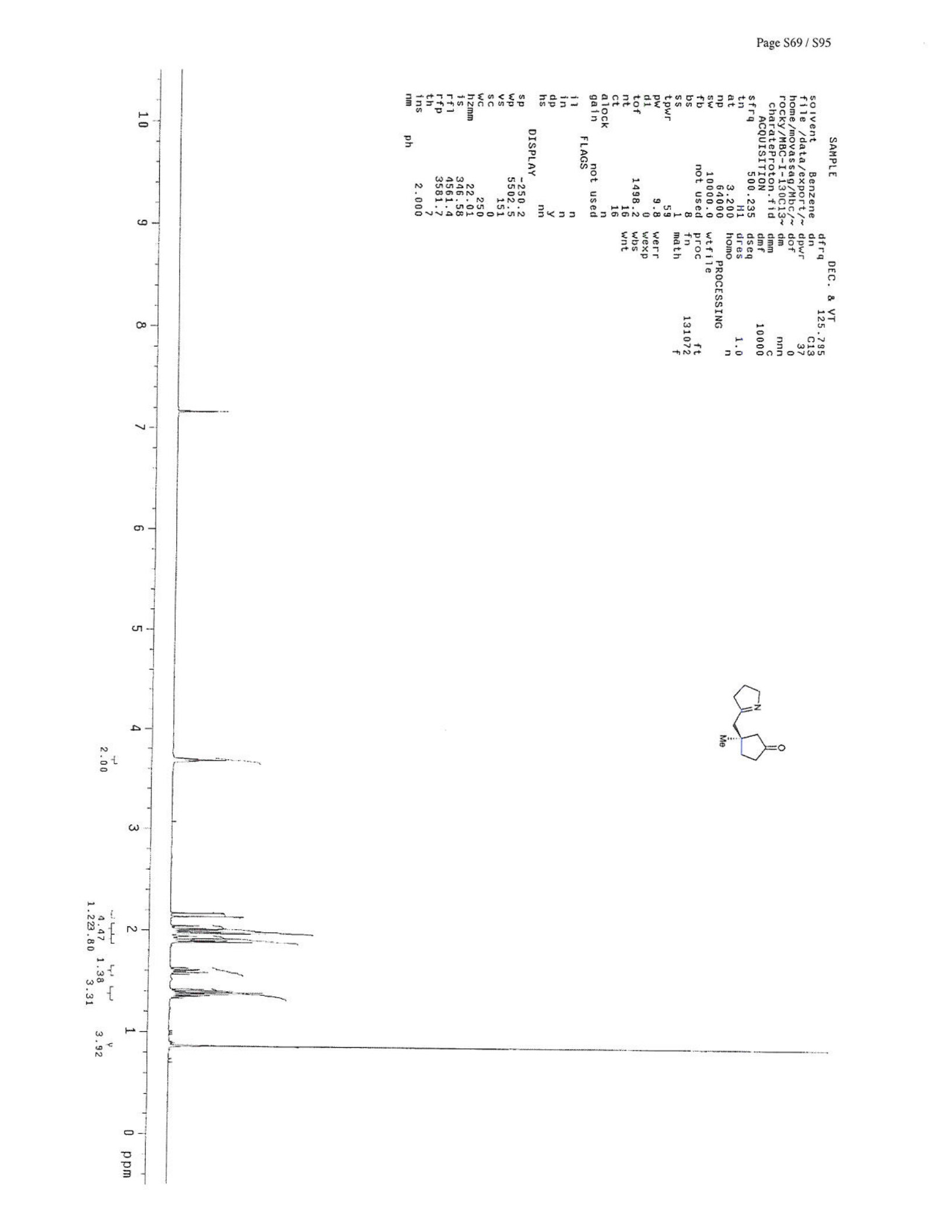

SAMPLE
solvent Benzene
file /data/export/~
home/movassag/Mbc/~
bullwinkle/MBC-I-1~
23combinedHcharact~
erlsation.fid
ACQUISITION
sfrq 499.749
tn H1
at 3.277
np 65536
sw 9998.8
fb not used
bs 16
tpwr 56
pw 8.2
d1 0
tof 1498.1
nt 32
ct 32
alock n
gain not used
FLAGS
fl n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 5497.1
vs 151
sc 0
wc 250
hzmm 21.99
is 240.51
rfl 1002.6
rfp 0
th 7
ins 1.000
nm cdc ph
dfrq DEC. & VT
dn C13
dpwr 34
dof 1498.1
dm nnn
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc
fn ft
math 65536 f
werr
wexp
wbs
wnt
H
HO
N
H
10
9
8
7
6
5
4
3
2
1
0 ppm
1.00
1.04
0.87
1.00
1.76
3.20
1.85
1.79
1.21


SAMPLE
solvent Benzene
file /data/export/~
home/movassag/Mbc/~
rocky/MBC-I-123imi~
necombinedC13chara~
cterisation.fid
ACQUISITION
sfrq 125.796
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 30000
ct 2464
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1258.1
wp 31448.7
vs 1448
sc 0
wc 250
hzmm 125.80
is 500.00
rfl 18876.6
rfp 12651.0
th 29
ins 1.000
ai ph
DEC. & VT
dfrq 500.233
dn H1
dpwr 37
dof -500.0
dm y
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
lb 0.30
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
HO
H
H
N
220
200
180
160
140
120
100
80
60
40
20
ppm


Pulse Sequence: gCOSY
Solvent: Benzene
Temp. 22.0 C / 295.1 K
File: MBC-III-01iminooHgCOSY
INOVA-500 "zippy"
PULSE SEQUENCE: gCOSY
Relax. delay 1.000 sec
Acq. time 0.189 sec
Width 5421.5 Hz
2D Width 5421.5 Hz
30 repetitions
128 increments
OBSERVE H1, 499.7446971 MHz
DATA PROCESSING
Sq. sine bell 0.095 sec
F1 DATA PROCESSING
Sq. sine bell 0.024 sec
FT size 2048 x 2048
Total time 0 min, -1 sec
H
HO
N
H
F2 (ppm)
F1 (ppm)

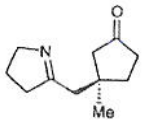

SAMPLE
solvent Benzene
file /data/export/~
home/movassag/Mbc/~
bullwinkle/MBC-I-1~
17and18amineOHpro~
toncharacte-fid
ACQUISITION
sfrq 499.749
tn H1
at 3.277
np 65536
sw 9998.8
fb not used
bs 16
tpwr 56
pw 8.2
d1 0
tof 1498.1
nt 64
ct 64
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 5497.1
vs 454
sc 0
wc 250
hzmm 21.99
is 385.78
rfl 4566.8
rfp 3578.2
th 26
ins 1.000
nm cdc ph
DEC. & VT
dfrq 125.674
dn C13
dpwr 34
dof 1498.1
dm nnn
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 65536
math f
wexp
wbs
wnt
HO
H
N
H
0 ppm
1 2 3 4 5 6 7 8 9 10
0.93
0.90
1.00
0.94 0.90 0.99 0.88
2.43 2.60 3.97 0.99
2.65

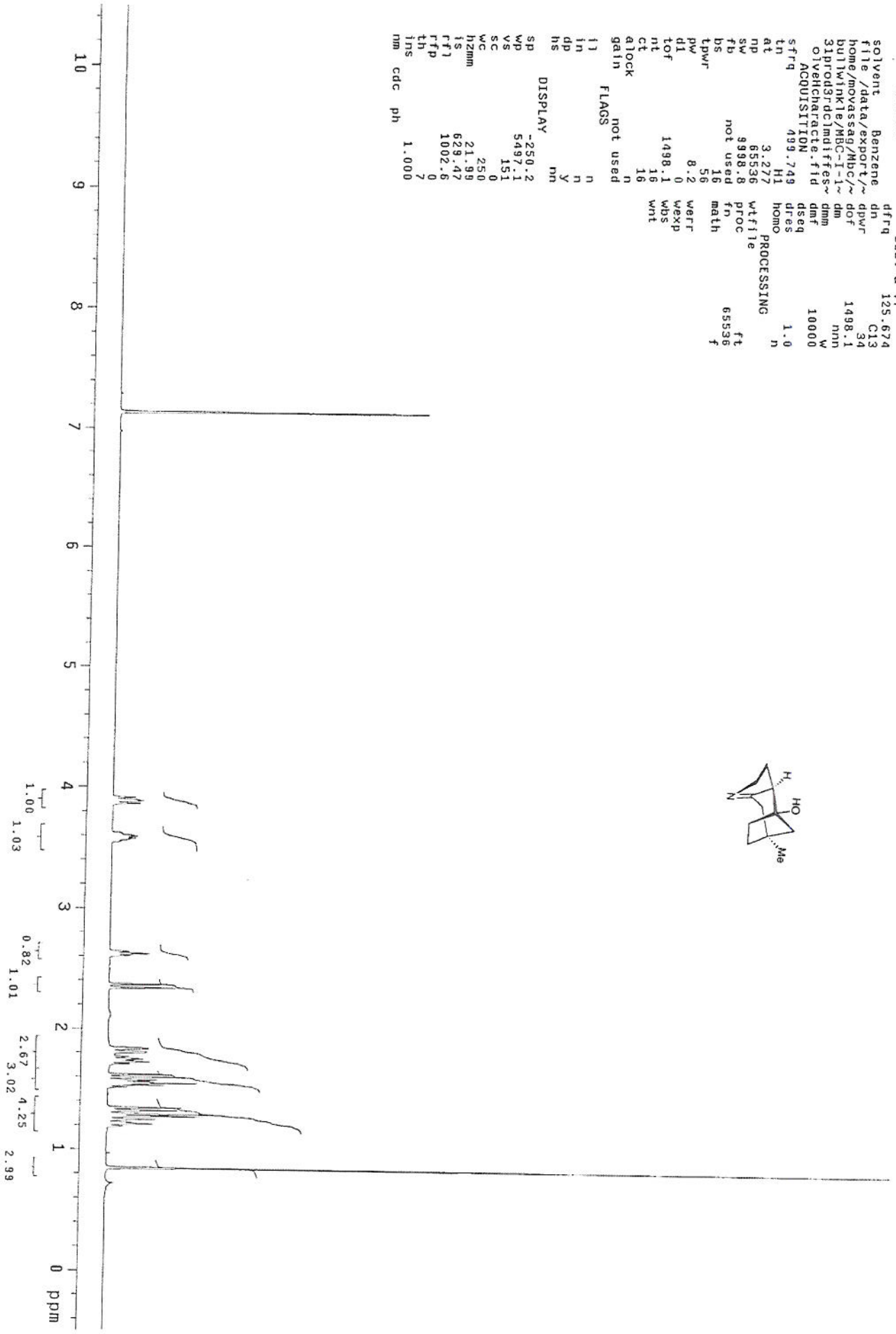

SAMPLE
solvent Benzene
file /data/export/~
home/movassag/Mbc/~
rocky/MBC-I-121and~
117and118amineOHCl~
3characteri.fid
ACQUISITION
sfrq 125.796
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 2000
ct 1536
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1258.1
wp 31448.7
vs 1795
sc 0
wc 250
hzmm 125.80
is 500.00
rfl 18876.6
rfp 12651.0
th 11
ins 1.000
ai ph
DEC. & VT
dfrq 500.233
dn H1
dpwr 37
dof -500.0
dm y
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
lb 0.30
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
HO
H
H
H
N
H
220
200
180
160
140
120
100
80
60
40
20
ppm

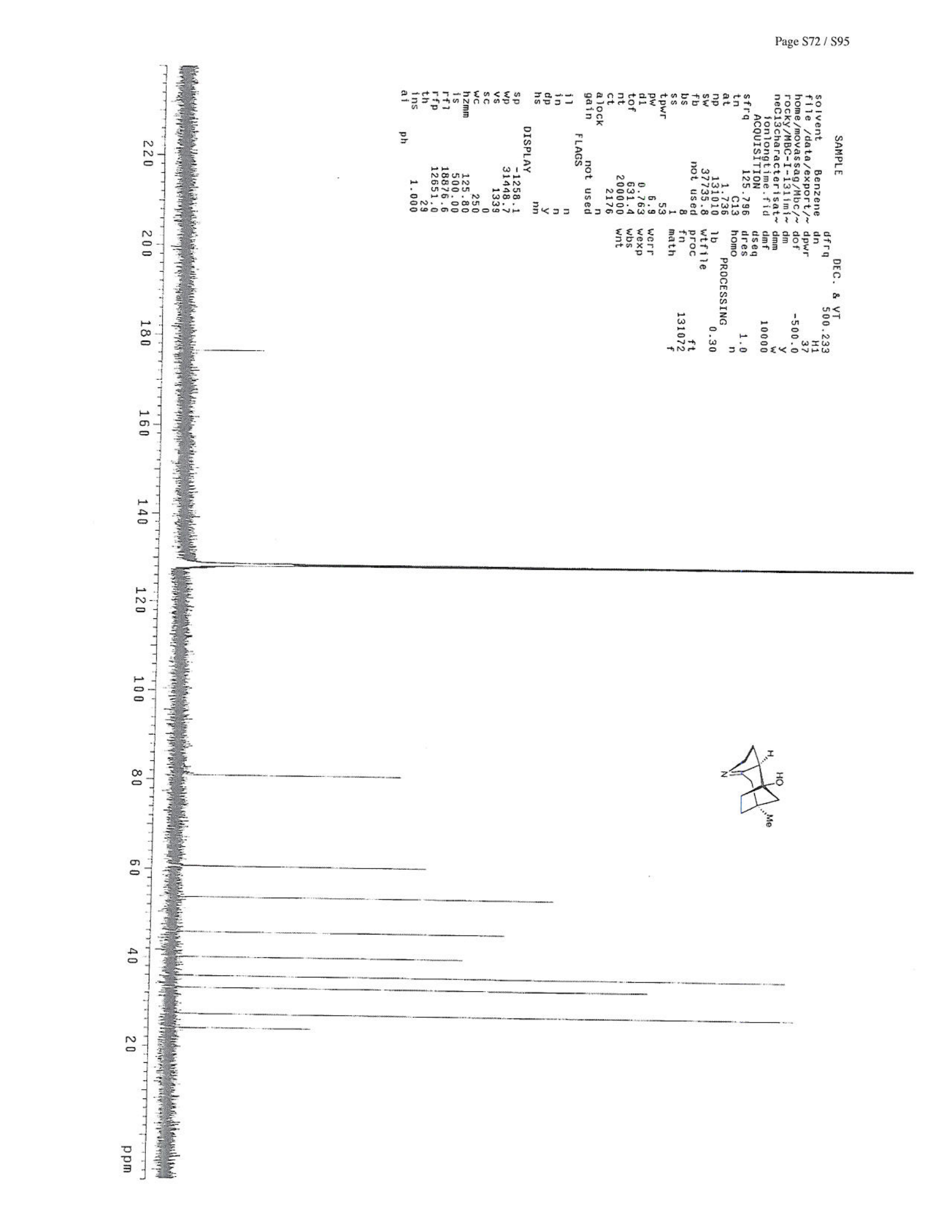

SAMPLE
solvent Benzene
file /data/export/~
home/movassag/Mbc/~
rocky/MBC-I-130C13~
charateProton.fid
ACQUISITION
sfrq 500.235
tn H1
at 3.200
np 64000
sw 10000.0
fb not used
bs 8
ss 1
tpwr 59
pw 9.8
d1 0
tof 1498.2
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 5502.5
vs 151
sc 0
wc 250
hzmm 22.01
is 346.58
rfl 4561.4
rfp 3581.7
th 7
ins 2.000
nm ph
dfrq
dn C13
dpwr 37
dof 0
dm nnn
dmm c
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
DEC. & VT
125.795
N
Me
O
10 9 8 7 6 5 4 3 2 1 0 ppm
2.00
4.47
1.22
3.80
1.38
3.31
3.92

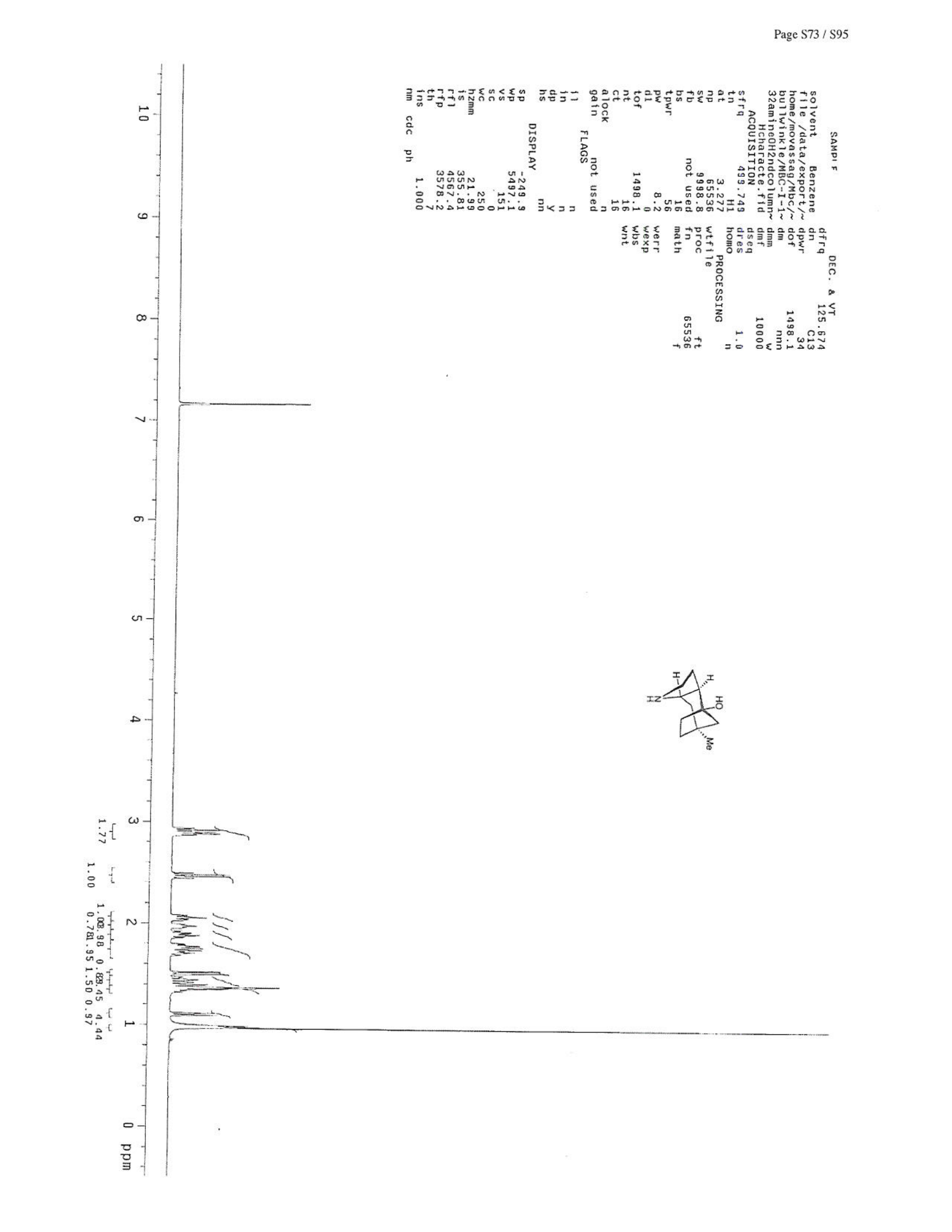
SAMPLE	
solvent	Benzene
file	/data/export/~
	home/movassag/Mbc/~
	rocky/MBC-I-130ket~
	oneimineC13charact~
	er.fid
ACQUISITION	
sfrq	125.796
tn	C13
at	1.736
np	131010
sw	37735.8
fb	not used
bs	8
ss	1
tpwr	53
pw	6.9
d1	0.763
tof	631.4
nt	200000
ct	3688
alock	n
gain	not used
FLAGS	
il	n
in	n
dp	y
hs	nn
DISPLAY	
sp	-1258.1
wp	31448.7
vs	473
sc	0
wc	250
hzmm	125.80
is	500.00
rfl	18876.6
rfp	12651.0
th	20
ins	1.000
ai	ph

DEC. & VT	
dfrq	500.233
dn	H1
dpwr	37
dof	-500.0
dm	y
dmm	w
dmf	10000
dseq	
dres	1.0
homo	n
PROCESSING	
lb	0.30
wtfile	
proc	ft
fn	131072
math	f
werr	
wexp	
wbs	
wnt	



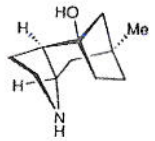

SAMPLE
solvent Benzene
file /data/export/~
home/movassag/Mbc/~
bullwinkle/MBC-1-1~
31prod3rdclmdiffes~
olveHcharacte.fid
ACQUISITION
sfrq 499.749
tn H1
at 3.277
np 65536
sw 9998.8
fb not used
bs 16
tpwr 56
pw 8.2
d1 0
tof 1498.1
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 5497.1
vs 151
sc 0
wc 250
hzmm 21.99
is 629.47
rfl 1002.6
rfp 0
th 7
ins 1.000
nm cdc ph
dfrq
dn
dpwr
dof
dm
dmm
dmf
dseq
dres
homo
PROCESSING
wtfile
proc
fn
math
werr
wexp
wbs
wnt
DEC. & VT
125.674
C13
34
1498.1
nnn
w
10000
1.0
n
n
ft
65536
f
H
HO
N
Me
0 ppm
1 2 3 4 5 6 7 8 9 10
1.00
1.03
0.82
1.01
2.67
3.02
4.25
2.99


SAMPLE
solvent Benzene
file /data/export/~
home/movassag/Mbc/~
rocky/MBC-I-131imi~
neC13characterisat~
ionlongtime.fid
ACQUISITION
sfrq 125.796
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 200000
ct 2176
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1258.1
wp 31448.7
vs 1339
sc 0
wc 250
hzmm 125.80
is 500.00
rfl 18876.6
rfp 12651.0
th 29
ins 1.000
ai ph
dfrq DEC. & VT
dn H1
dpwr 37
dof -500.0
dm y
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
lb 0.30
wtfile
proc ft
fn 131072
math f
wcrr
wexp
wbs
wnt
H
N
HO
Me
220
200
180
160
140
120
100
80
60
40
20
ppm


SAMPLE
solvent Benzene
file /data/export/~
home/movassag/Mbc/~
bullwinkle/MBC-1-1~
32amineOH2ndcolumn~
Hcharacte.fid
ACQUISITION
sfrq 499.749
tn H1
at 3.277
np 65536
sw 9998.8
fb not used
bs 16
tpwr 56
pw 8.2
d1 0
tof 1498.1
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -249.9
wp 5497.1
vs 151
sc 0
wc 250
hzmm 21.99
is 355.81
rfl 4567.4
rfp 3578.2
th 7
ins 1.000
nm cdc ph
dfrq DEC. & VT
dn C13
dpwr 34
dof 1498.1
dm nnn
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 65536
math f
wexp
wbs
wnt
H
HO
H
N
H
Me
10
9
8
7
6
5
4
3
2
1
0 ppm
1.77
1.00
1.00 0.98 0.62 0.45 0.44
0.78 1.95 1.50 0.97

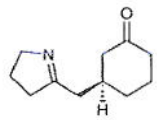
SAMPLE	
solvent	Benzene
file	/data/export/~
	home/movassag/Mbc/~
	rocky/MBC-I-132ami~
	noOHC13characteris~
	ation.fid
ACQUISITION	
sfrq	125.796
tn	C13
at	1.736
np	131010
sw	37735.8
fb	not used
bs	8
ss	1
tpwr	53
pw	6.9
d1	0.763
tof	631.4
nt	200000
ct	2016
alock	n
gain	not used
FLAGS	
il	n
in	n
dp	y
hs	nn
DISPLAY	
sp	-1258.1
wp	31448.7
vs	3480
sc	0
wc	250
hzmm	125.80
is	500.00
rfl	18876.6
rfp	12651.0
th	20
ins	1.000
ai	ph

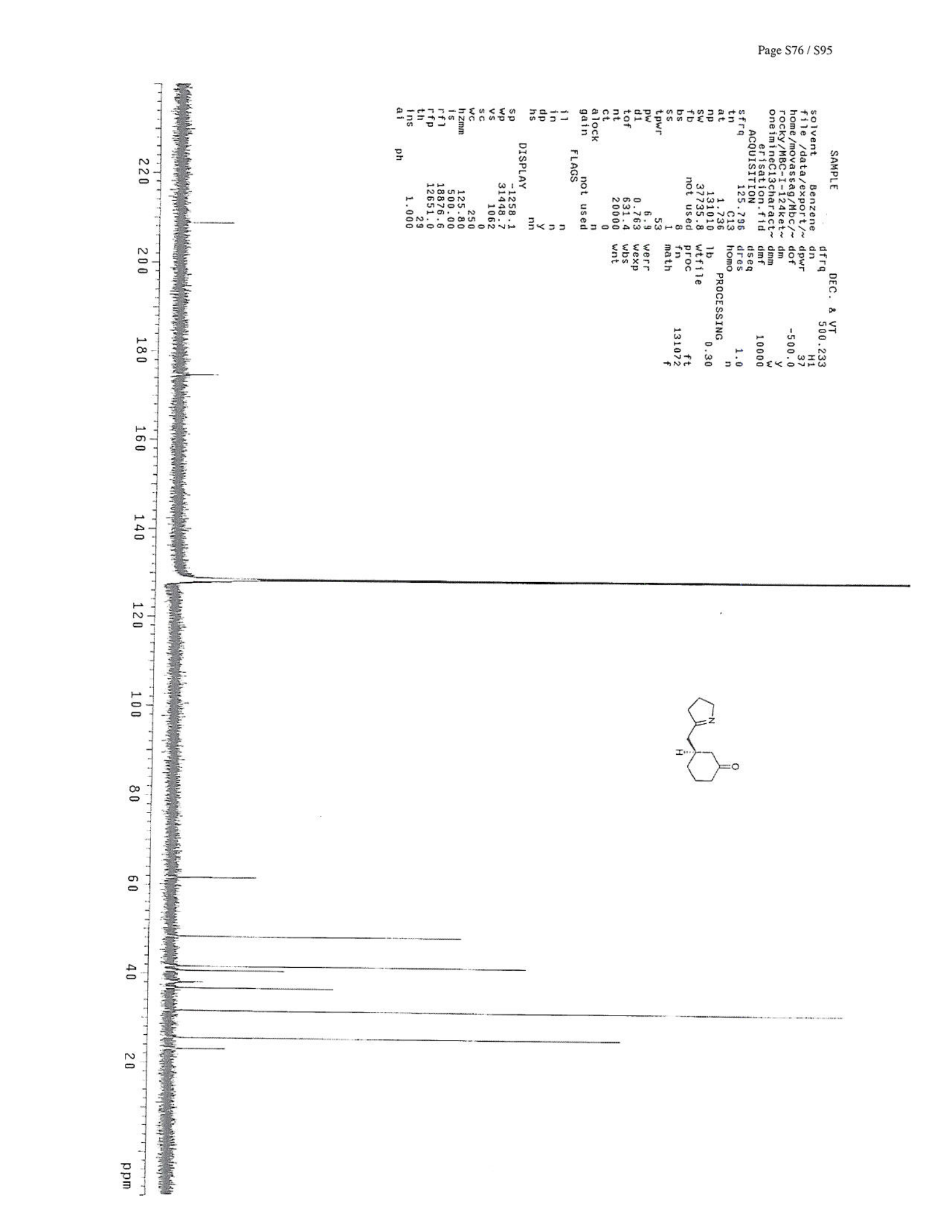
DEC. & VT	
dfrq	500.233
dn	H1
dpwr	37
dof	-500.0
dm	y
dmm	w
dmf	10000
dseq	
dres	1.0
homo	n
PROCESSING	
lb	0.30
wtfile	
proc	ft
fn	131072
math	f
werr	
wexp	
wbs	
wnt	

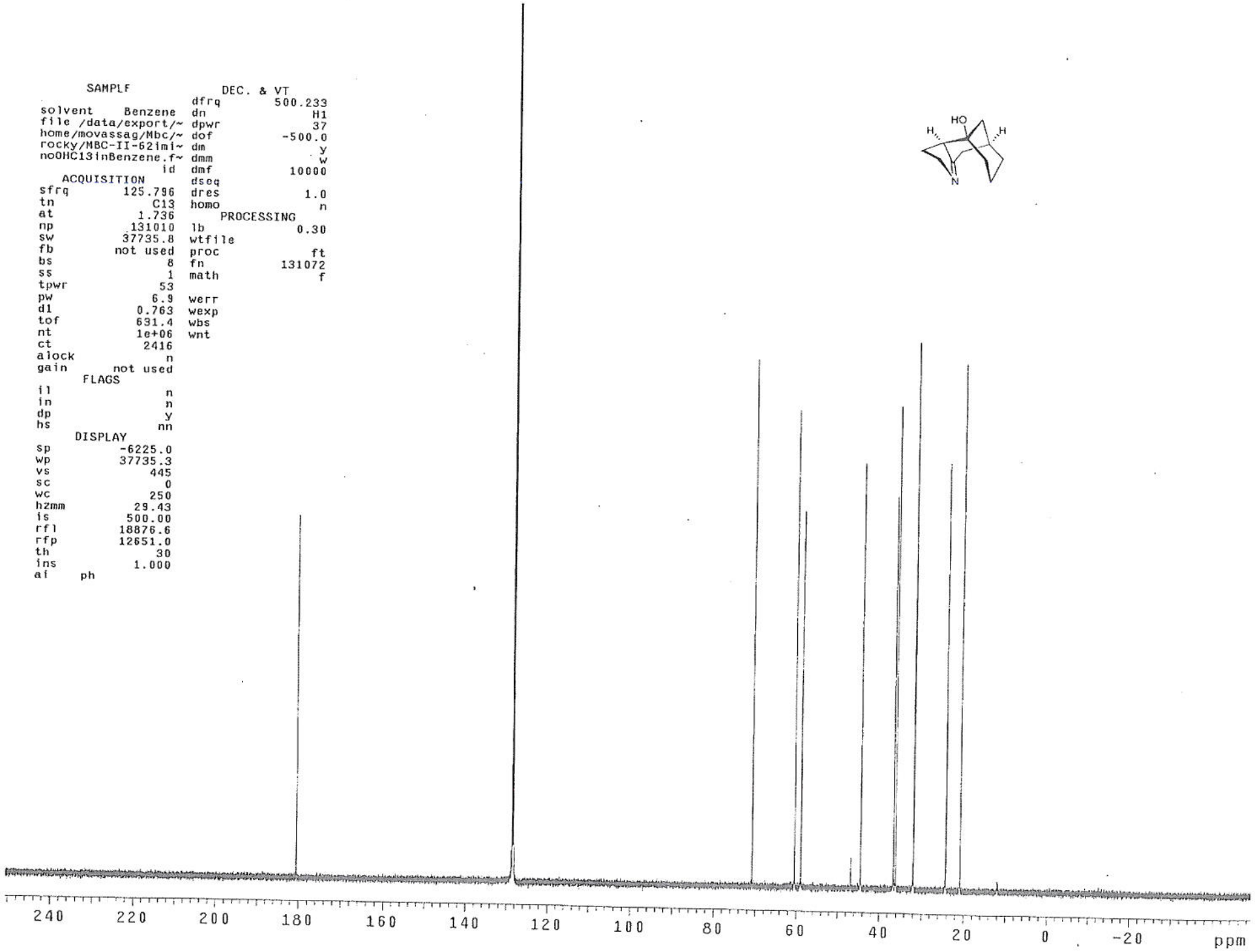


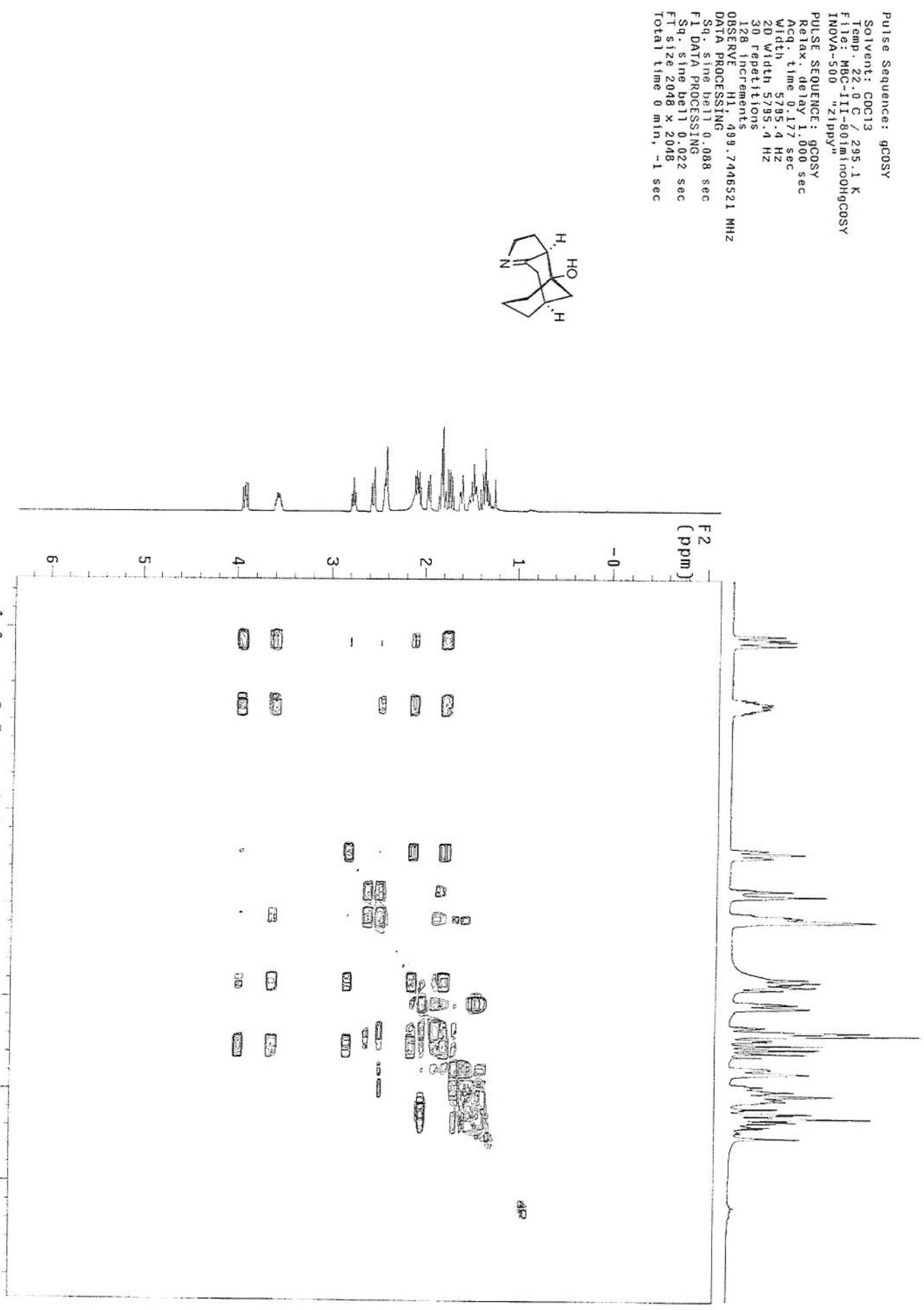
SAMPLE	
solvent	Benzene
file	/data/export/~
home/movassag/Mbc/~	
rocky/MBC-I-124pro~	
dHcharact501.fid	
ACQUISITION	
sfrq	500.235
tn	H1
at	3.200
np	64000
sw	10000.0
fb	not used
bs	8
ss	1
tpwr	59
pw	9.8
dl	0
tof	1498.2
nt	16
ct	16
alock	n
gain	not used
FLAGS	
il	n
in	n
dp	y
hs	nn
DISPLAY	
sp	-250.2
wp	5502.5
vs	151
sc	0
wc	250
hzmm	22.01
is	164.71
rfl	4561.5
rfp	3581.7
th	7
ins	2.000
nm cdc ph	

DEC. & VT	
dfrq	125.795
dn	C13
dpwr	37
dof	0
dm	nnn
dmm	c
dmf	10000
dseq	
dres	1.0
homo	n
PROCESSING	
wtfile	
proc	ft
fn	131072
math	f
werr	
wexp	
wbs	
wnt	



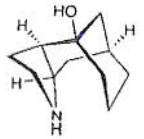

SAMPLE
solvent Benzene
file /data/export/~
home/movassag/Mbc/~
rocky/MBC-I-124ket~
oneiminеC13charact~
erisation.fid
ACQUISITION
sfrq 125.796
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 20000
ct 0
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1258.1
wp 31448.7
vs 1062
sc 0
wc 250
hzmm 125.80
is 500.00
rfl 18876.6
rfp 12651.0
th 29
ins 1.000
ai ph
DEC. & VT
dfrq 500.233
dn H1
dpwr 37
dof -500.0
dm y
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
lb 0.30
wtfile
proc
fn ft
math 131072 f
werr
wexp
wbs
wnt
N
H
O
220
200
180
160
140
120
100
80
60
40
20
ppm

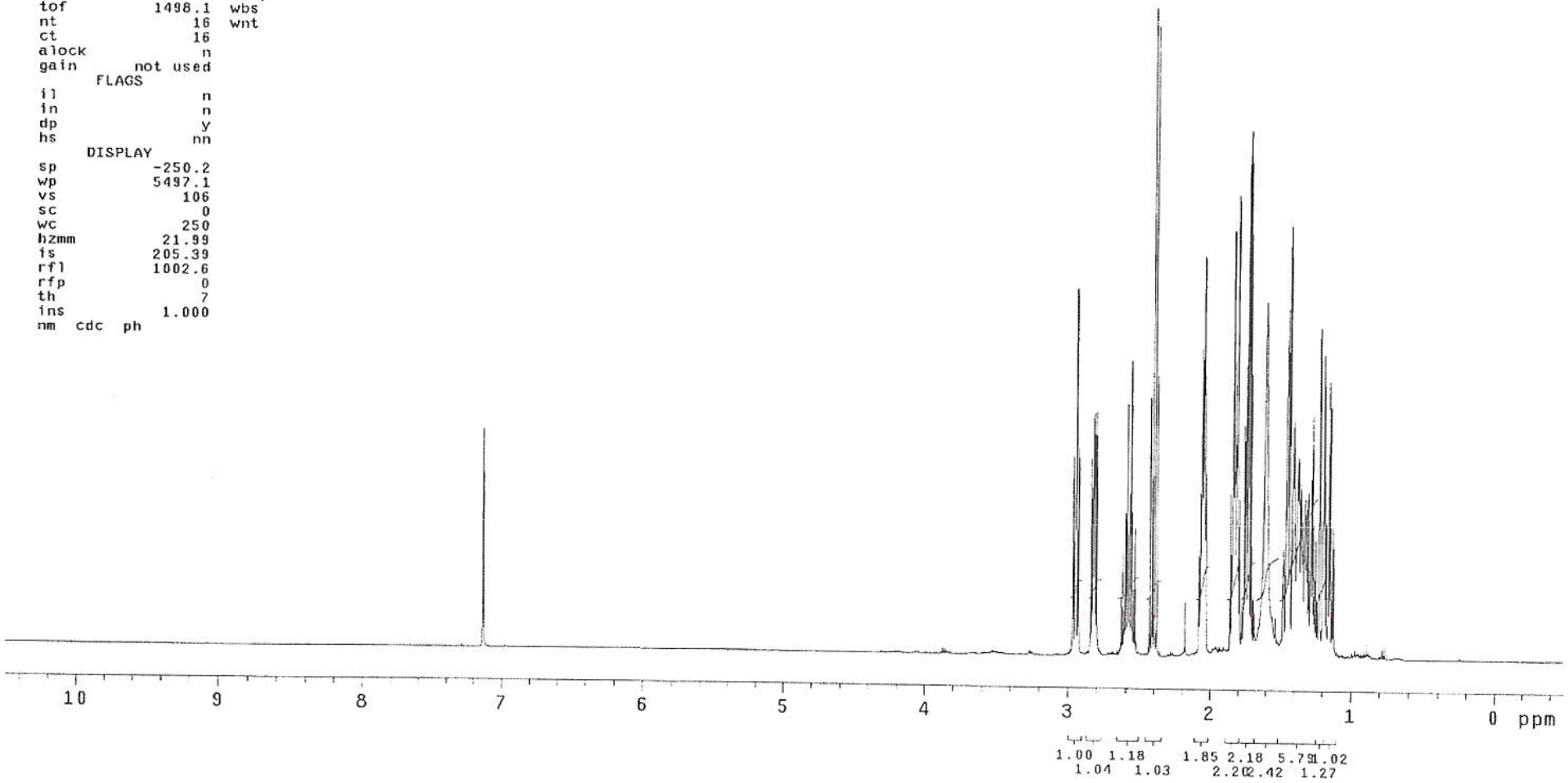

SAMPLE
solvent Benzene
file /data/export/~
home/movassag/Mbc/~
rocky/MBC-II-62imi~
noOHC13inBenzene.f~
id
ACQUISITION
sfrq 125.796
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 1e+06
ct 2416
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -6225.0
wp 37735.3
vs 445
sc 0
wc 250
hzmm 29.43
is 500.00
rfl 18876.6
rfp 12651.0
th 30
ins 1.000
ai ph
DEC. & VT
dfrq 500.233
dn H1
dpwr 37
dof -500.0
dm y
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
lb 0.30
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
HO
H
H
N
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm

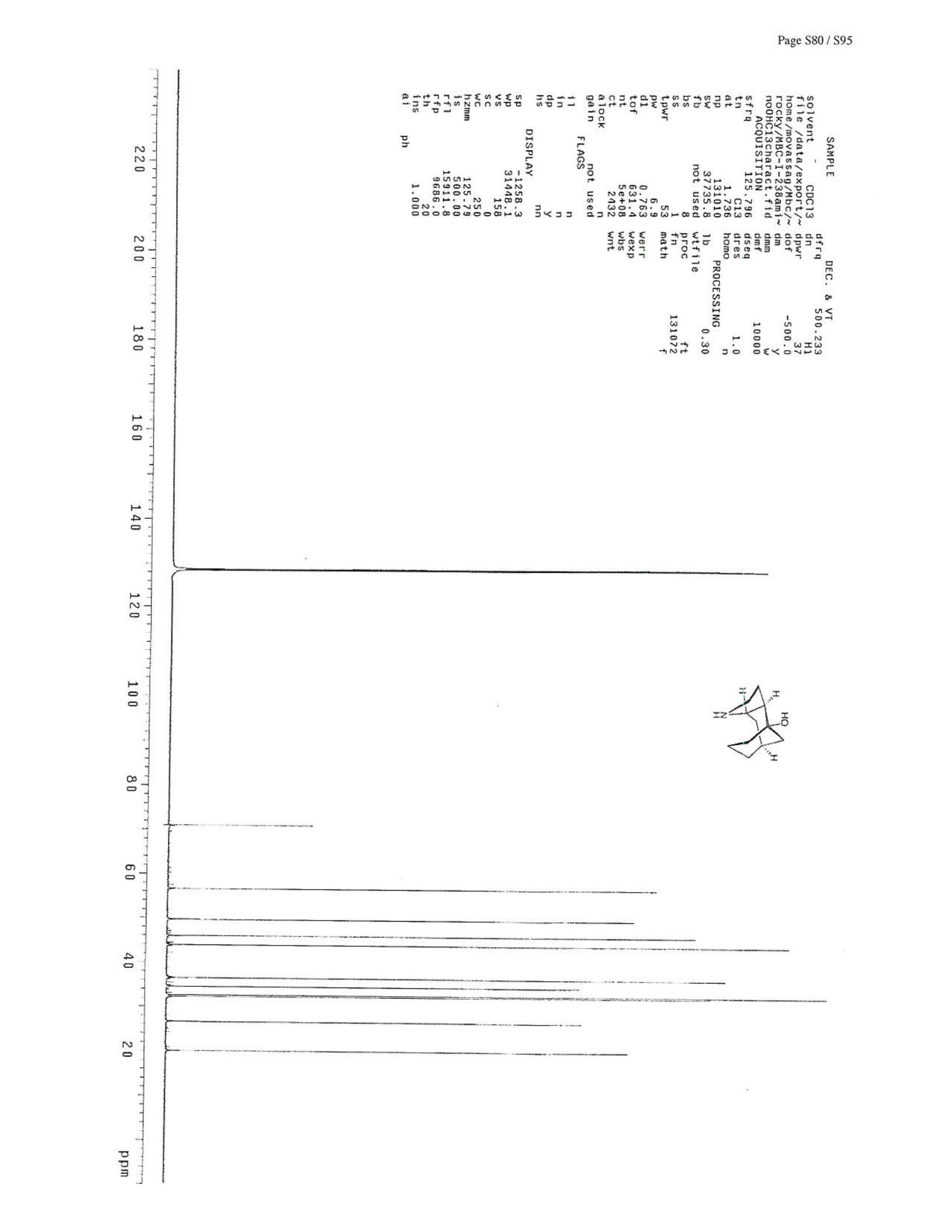

Pulse Sequence: gCOSY
Solvent: CDCl3
Temp. 22.0 C / 295.1 K
File: MBC-III-80iminooHgCOSY
INOVA-500 "zippy"
PULSE SEQUENCE: gCOSY
Relax. delay 1.000 sec
Acq. time 0.177 sec
Width 5795.4 Hz
2D Width 5795.4 Hz
30 repetitions
128 increments
OBSERVE H1, 499.7466521 MHz
DATA PROCESSING
Sq. sine bell 0.088 sec
F1 DATA PROCESSING
Sq. sine bell 0.022 sec
FT size 2048 x 2048
Total time 0 min, -1 sec
HO
H
N
H
F2 (ppm)
F1 (ppm)
4R

SAMPLE	
solvent	Benzene
file	/data/export/~
	home/movassag/Mbc/~
	bullwinkle/MBC-I-2~
	38aminoOHHcharacte~
	risation.fid
ACQUISITION	
sfrq	499.749
tn	H1
at	3.277
np	65536
sw	9998.8
fb	not used
bs	16
tpwr	56
pw	8.2
d1	0
tof	1498.1
nt	16
ct	16
alock	n
gain	not used
FLAGS	
il	n
in	n
dp	y
hs	nn
DISPLAY	
sp	-250.2
wp	5497.1
vs	106
sc	0
wc	250
hzmm	21.99
is	205.39
rfl	1002.6
rfp	0
th	7
ins	1.000
nm cdc ph	

DEC. & VT	
dfrq	125.674
dn	C13
dpwr	34
dof	1498.1
dm	nnn
dmm	w
dmf	10000
dseq	
dres	1.0
homo	n
PROCESSING	
wtfile	
proc	ft
fn	65536
math	f
werr	
wexp	
wbs	
wnt	






SAMPLE
solvent CDCl3
file /data/export/~
home/movassag/Mbc/~
rocky/MBC-I-238ami~
noOHC13charact.fid
ACQUISITION
sfrq 125.796
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 5e+08
ct 2432
alock wnt
gain n
FLAGS
not used
il n
in n
dp y
hs nn
DISPLAY
sp -1258.3
wp 31448.1
vs 158
sc 0
wc 250
hzmm 125.79
is 500.00
rfl 15911.8
rfp 9686.0
th 20
ins 1.000
ai ph
dfrq
dn
dpwr
dof
dm
dmm
dmf
dseq
dres
homo
PROCESSING
lb
wtfile
proc
fn
math
werr
wexp
wbs
wnt
DEC. & VT
500.233
H1
37
-500.0
y
w
10000
1.0
n
0.30
ft
131072
f
HO
H
N
H
H
H
220
200
180
160
140
120
100
80
60
40
20
ppm

expl s2pul

SAMPLE	
solvent	Benzene

ACQUISITION	
sfrq	499.749
tn	H1
at	3.001
np	63050
sw	10504.2
fb	not used
bs	8
tpwr	56
pw	8.9
d1	2.000
tof	1519.5
nt	24
ct	24
alock	n
gain	not used

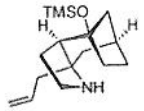
FLAGS	
il	n
in	n
dp	y
hs	nn

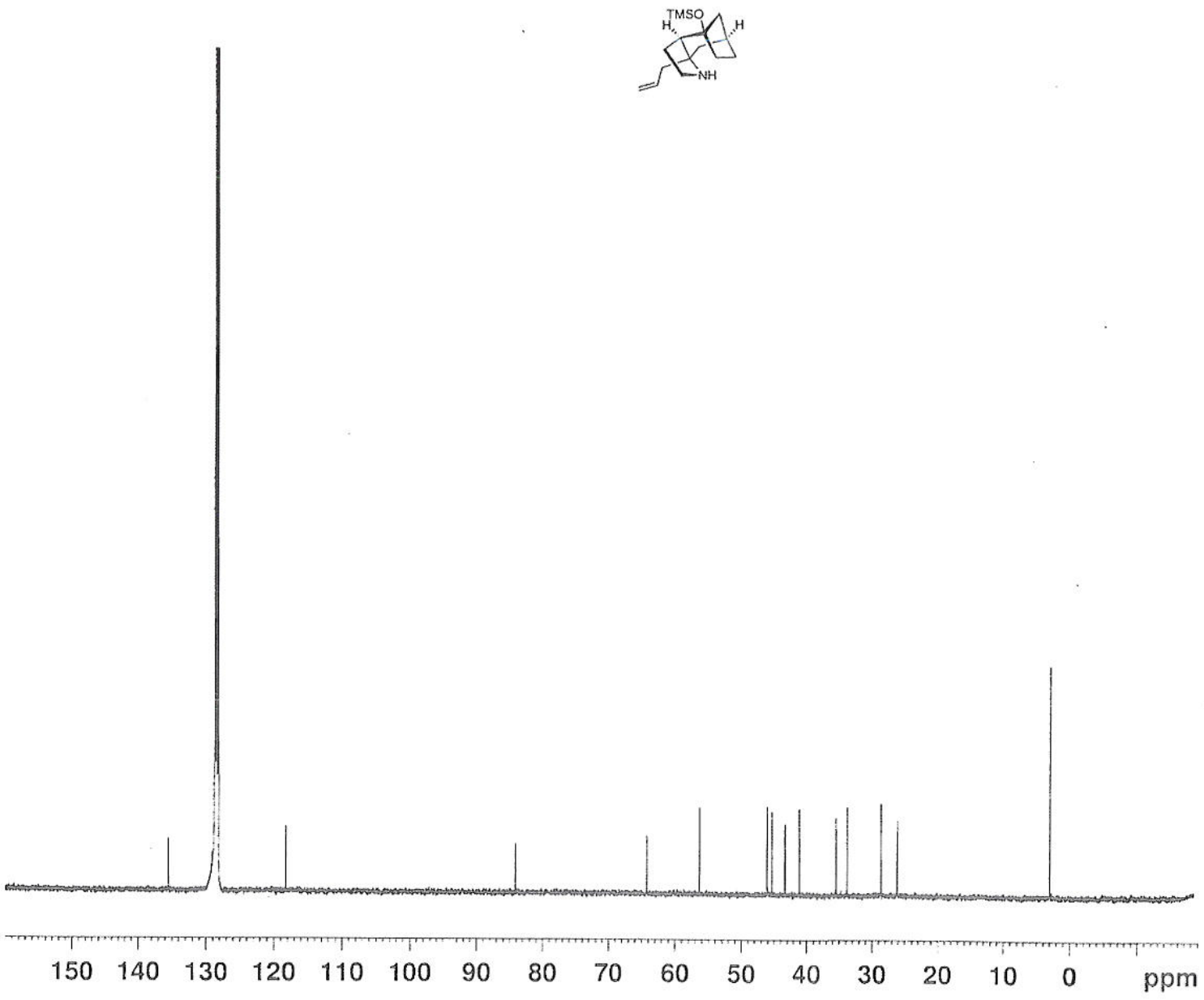
DISPLAY	
sp	-249.9
wp	5497.2
vs	151
sc	0
wc	250
hzmm	21.99
is	33.57
rfl	1233.8
rfp	0
th	7
ins	100.000

nm cdc ph

DEC. & VT	
dfrq	125.673
dn	C13
dpwr	30
dof	0
dm	nnn
dmm	w
dmf	10000
dseq	
dres	1.0
homo	n

PROCESSING	
wtfile	
proc	ft
fn	131072
math	f
werr	
wexp	
wbs	
wnt	wft





Current Data Parameters

EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Time	20.00
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	C6D6
NS	14565
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	2896.3
DW	20.850 usec
DE	6.00 usec
TE	294.8 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

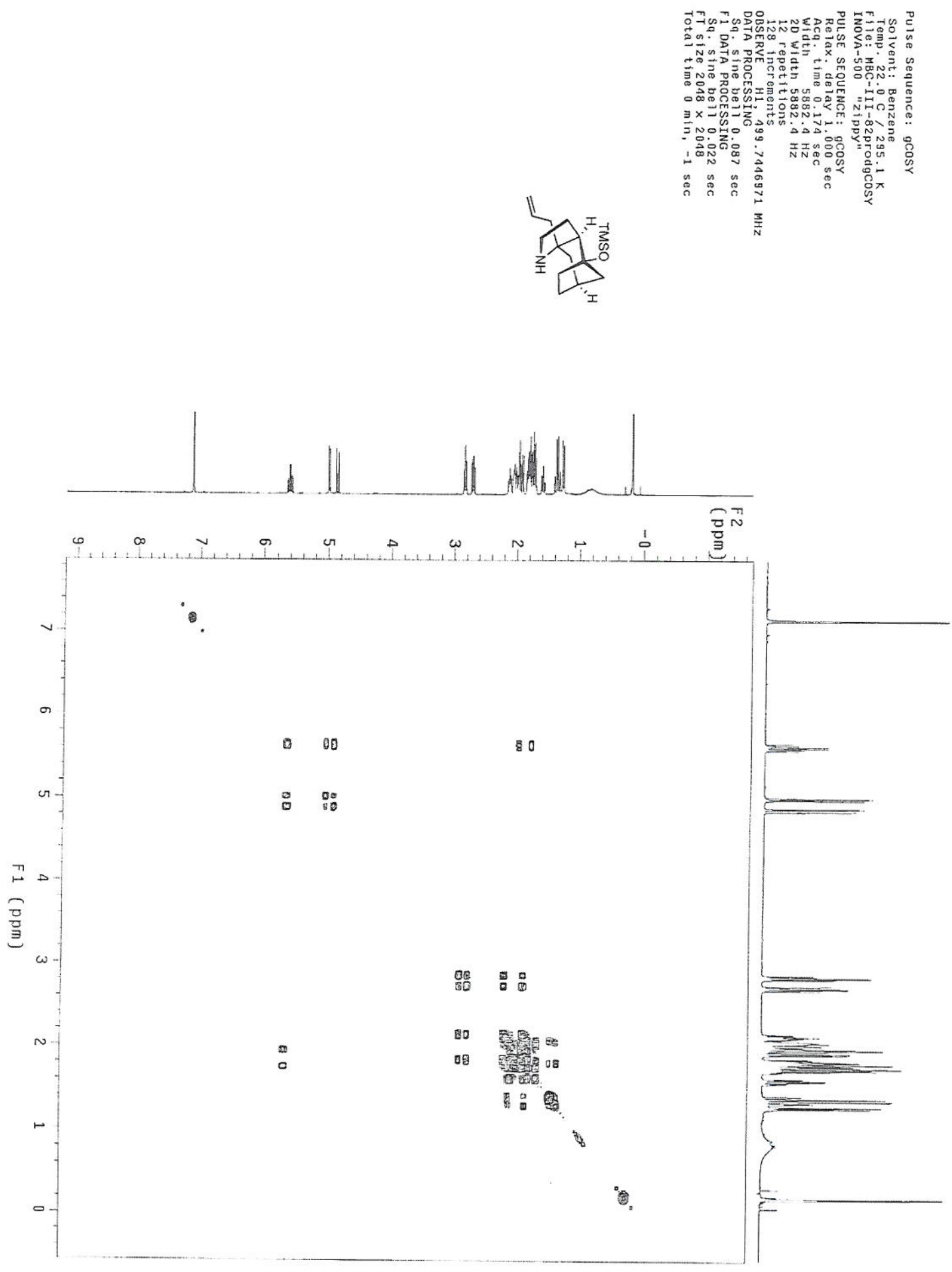
NUC1	13C
P1	8.50 usec
PL1	3.00 dB
SFO1	100.6228298 MHz

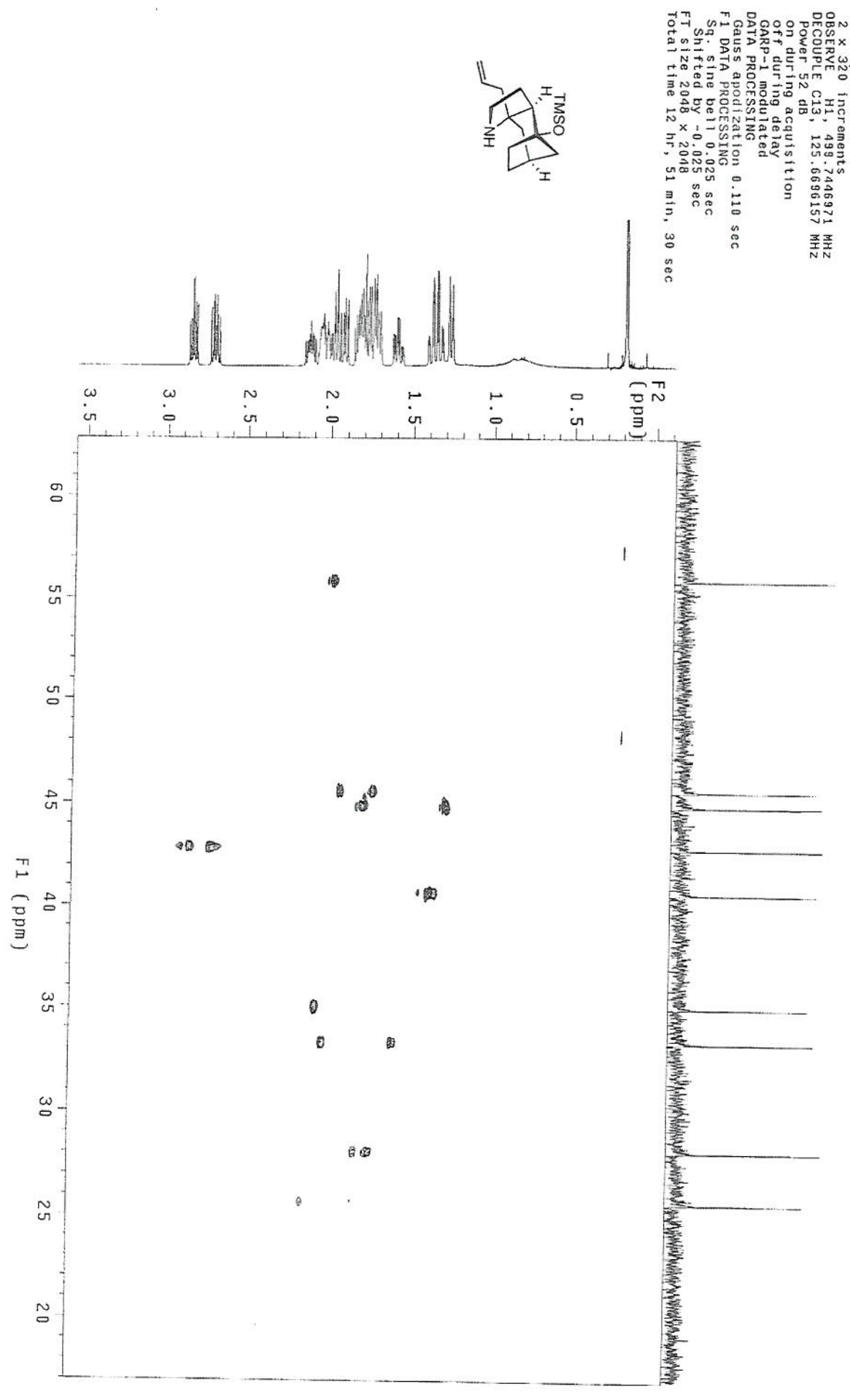
======== CHANNEL f2 ========

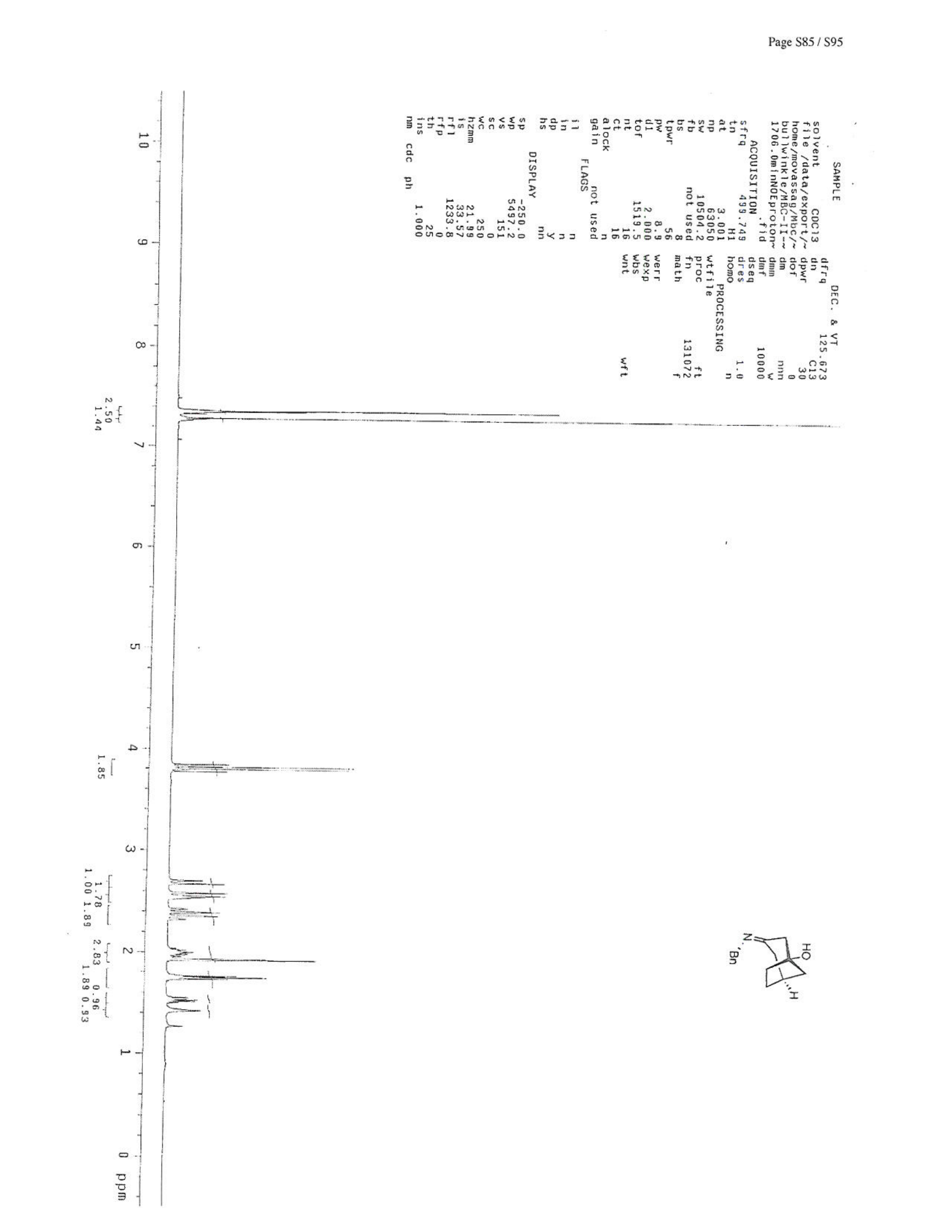
CPDPRG2	waltz16
NUC2	1H
PCPD2	88.01 usec
PL2	3.00 dB
PL12	22.00 dB
PL13	22.00 dB
SFO2	400.1316005 MHz

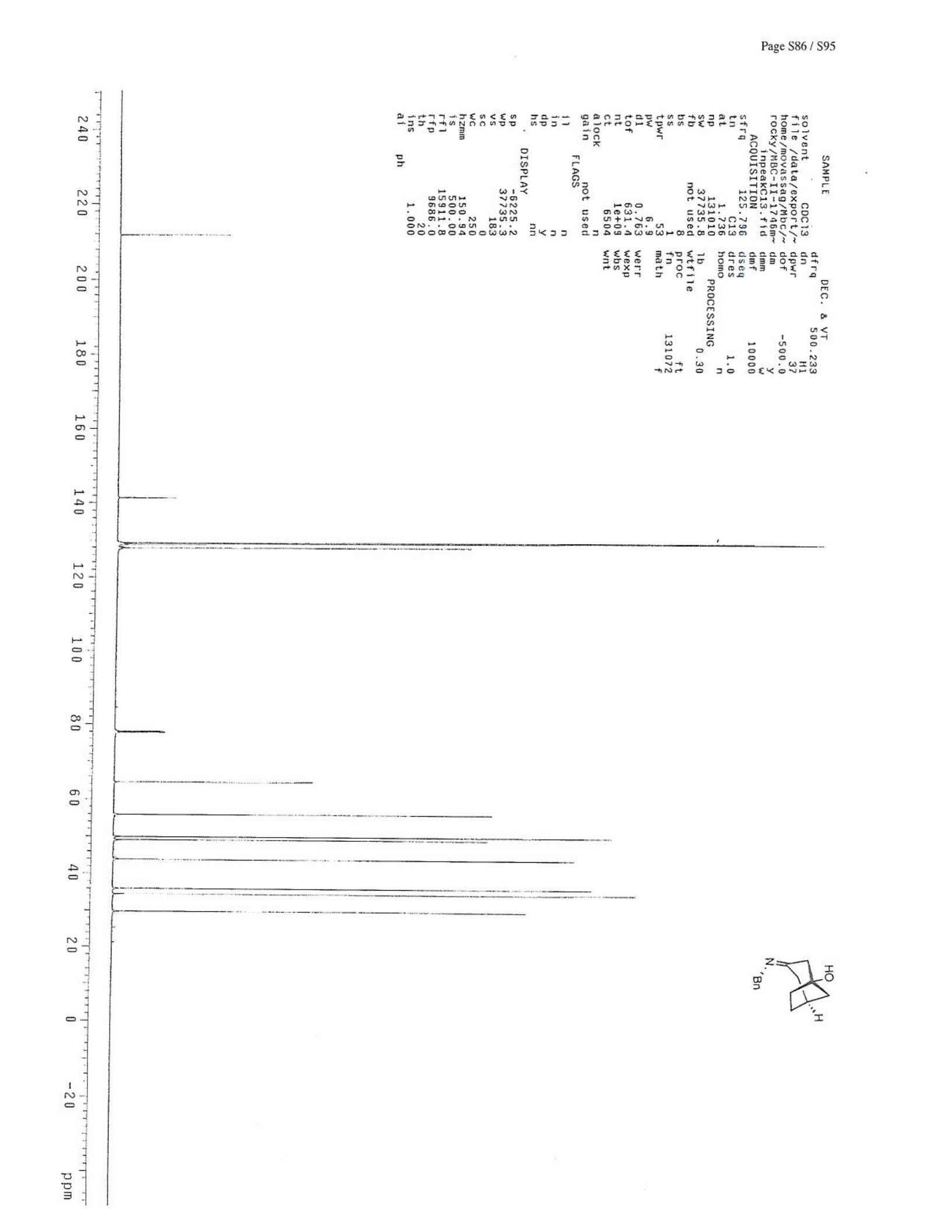
F2 - Processing parameters

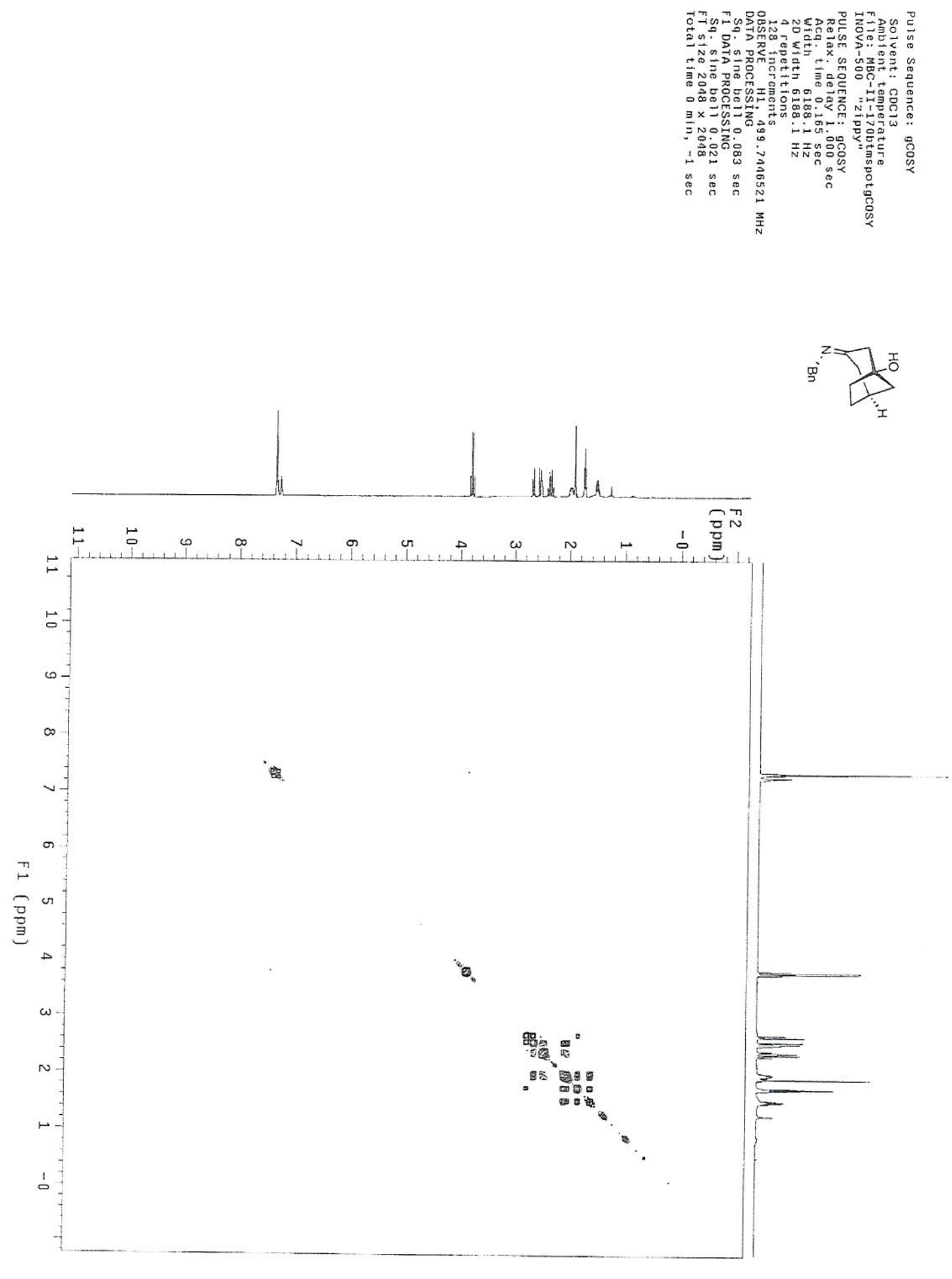
SI	32768
SF	100.6127505 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

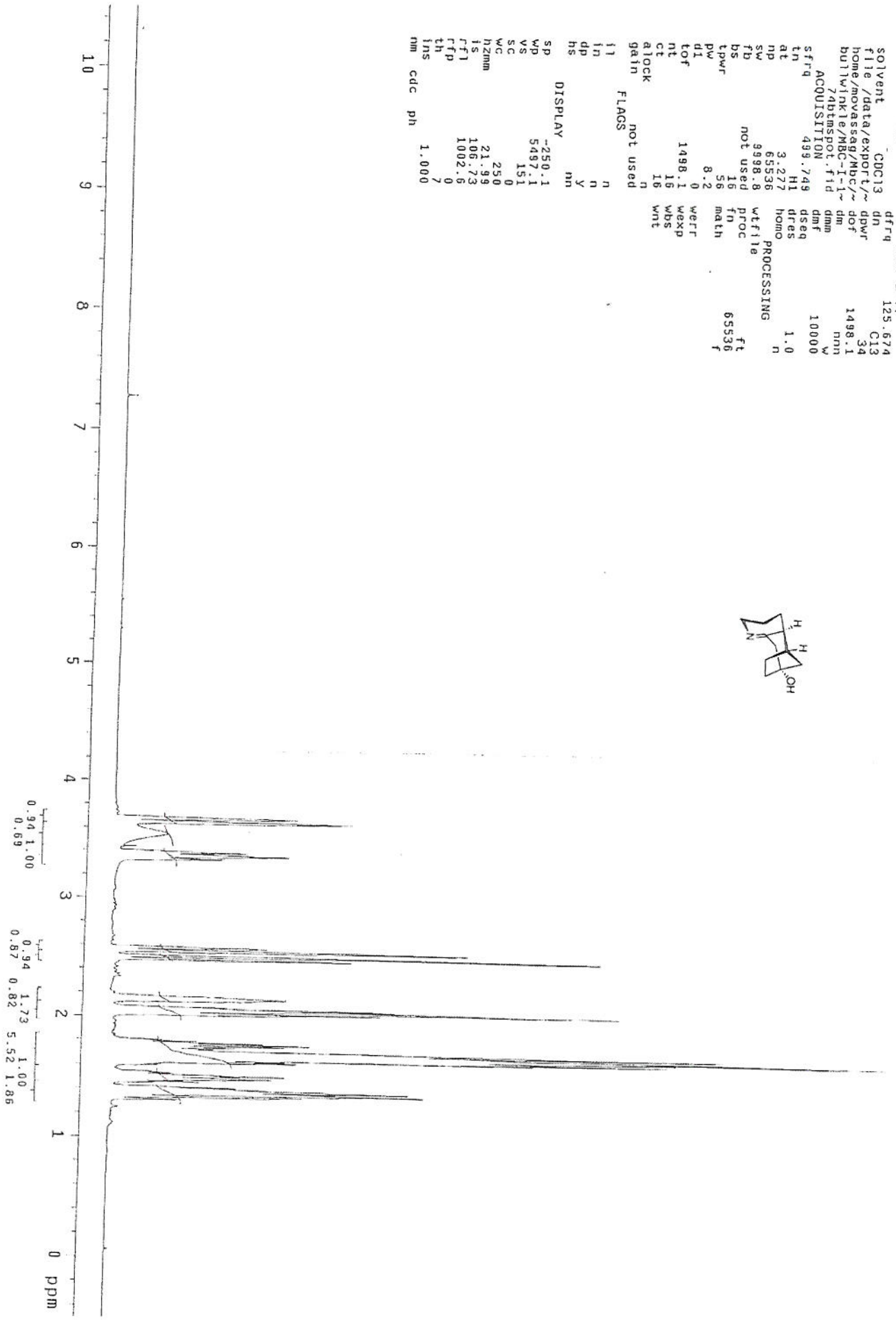

Pulse Sequence: gCOSY
Solvent: Benzene
Temp. 22.0 C / 295.1 K
File: MBC-III-82prodgCOSY
INOVA-500 "zippy"
PULSE SEQUENCE: gCOSY
Relax. delay 1.000 sec
Acq. time 0.174 sec
Width 5882.4 Hz
2D Width 5882.4 Hz
12 repetitions
128 increments
OBSERVE H1, 499.7446971 MHz
DATA PROCESSING
Sq. sine bell 0.087 sec
F1 DATA PROCESSING
Sq. sine bell 0.022 sec
FT size 2048 x 2048
Total time 0 min, -1 sec
TMSO
H
NH
H
F2 (ppm)
F1 (ppm)


STANDARD PROTON PARAMETERS
Pulse Sequence: HSQC
Solvent: Benzene
Ambient temperature
User: l-14-87
File: MBC-III-82prodHSQC
INOVA-500 "Zippy"
PULSE SEQUENCE: HSQC
Relax. delay 1.000 sec
Acq. time 0.238 sec
Width 4293.7 Hz
2D Width 20202.0 Hz
44 repetitions
2 x 320 increments
OBSERVE H1, 499.7446971 MHz
DECOUPLE C13, 125.6696157 MHz
Power 52 dB
on during acquisition
off during delay
GARP-1 modulated
DATA PROCESSING
Gauss apodization 0.110 sec
F1 DATA PROCESSING
Sq. sine bell 0.025 sec
Shifted by -0.025 sec
FT size 2048 x 2048
Total time 12 hr, 51 min, 30 sec
TMSO
H
NH
H
F2 (ppm)
F1 (ppm)

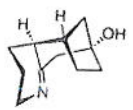

SAMPLE
solvent CDCl3
file /data/export/~
home/movassag/Mbc/~
bullwinkle/MBC-II~~
1706.0m!NOEproton~
.fid
ACQUISITION
sfrq 499.749
tn H1
at 3.001
np 63050
sw 10504.2
fb not used
bs 8
tpwr 56
pw 8.9
d1 2.000
tof 1519.5
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.0
wp 5497.2
vs 151
sc 0
wc 250
hzmm 21.99
is 33.57
rfl 1233.8
rfp 0
th 25
ins 1.000
nm cdc ph
dfrq DEC. & VT
dn 125.673
dpwr C13
dof 30
dm 0
nnn nnn
dmm w
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt wft
2.50
1.44
1.85
1.78
1.00 1.89
2.83 0.96
1.89 0.93
HO
N
Bn
H
10 9 8 7 6 5 4 3 2 1 0 ppm

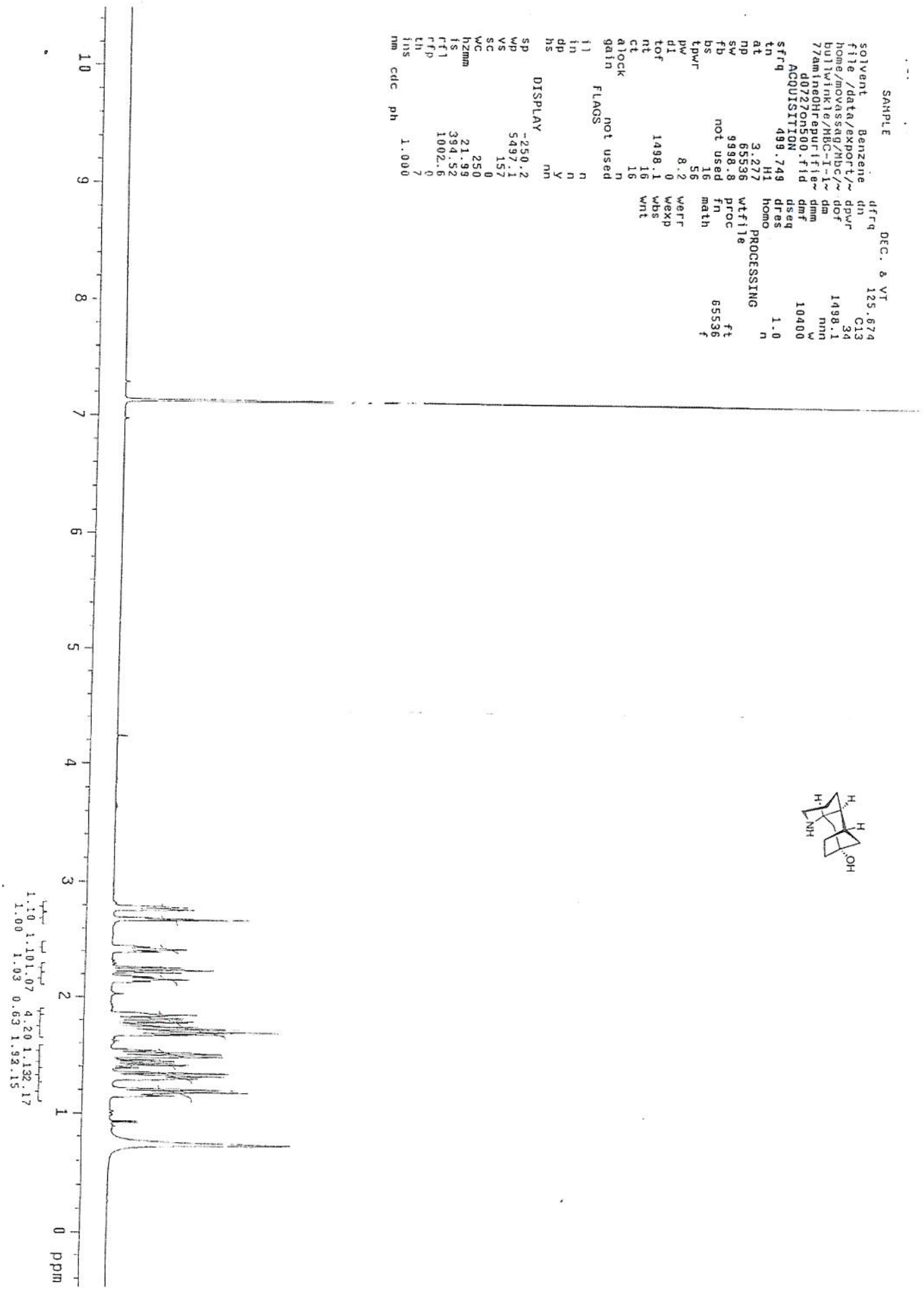

SAMPLE
solvent CDCl3
file /data/export/~
home/movassag/Mbc/~
rocky/MBC-II-1746m~
inpeakC13.fid
ACQUISITION
sfrq 125.796
tn C13
at 1.736
np 131010
sw 37735.8
fb not used
bs 8
ss 1
tpwr 53
pw 6.9
d1 0.763
tof 631.4
nt 1e+09
ct 6504
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -6225.2
wp 37735.3
vs 183
sc 0
wc 250
hzmm 150.94
is 500.00
rfl 15911.8
rfp 9686.0
th 20
ins 1.000
ai ph
dfrq
dn
dpwr
dof
dm
dmm
dmf
dseq
dres
homo
PROCESSING
lb
wtfile
proc
fn
math
werr
wexp
wbs
wnt
DEC. & VT
500.233
H1
37
-500.0
y
w
10000
1.0
n
0.30
ft
131072
f
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm
HO
N
Bn
H


Pulse Sequence: gCOSY
Solvent: CDCl3
Ambient temperature
File: MBC-II-170btmspotgCOSY
INOVA-500 "zippy"
PULSE SEQUENCE: gCOSY
Relax. delay 1.000 sec
Acq. time 0.165 sec
Width 6188.1 Hz
2D Width 6188.1 Hz
4 repetitions
128 increments
OBSERVE H1, 499.7446521 MHz
DATA PROCESSING
Sq. sine bell 0.083 sec
F1 DATA PROCESSING
Sq. sine bell 0.021 sec
FT size 2048 x 2048
Total time 0 min, -1 sec
HO
N
Bn
H
F2 (ppm)
F1 (ppm)


SAMPLE
solvent CDCl3
file /data/export/~
home/movassag/Mbc/~
bullwinkle/MBC-I-1~
7.4btmspot.fid
ACQUISITION
sfrq 499.749
tn H1
at 3.277
np 65536
sw 9998.8
fb not used
bs 16
tpwr 56
pw 8.2
d1 0
tof 1498.1
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.1
wp 5497.1
vs 151
sc 0
wc 250
hzmm 21.99
is 106.73
rfl 1002.6
rfp 0
th 7
ins 1.000
nm cdc ph
DEC. & VT
dfrq 125.674
dn C13
dpwr 34
dof 1498.1
dm nnn
dmm w
dmf 10000
dseq 1.0
dres n
homo
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
H1
H
OH
N
0.94 1.00
0.69
0.94 1.73
0.67 0.82 5.52 1.00 1.86
10 9 8 7 6 5 4 3 2 1 0 ppm

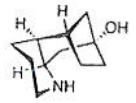
SAMPLE		DEC. & VT	
solvent	Benzene	dfrq	500.233
file	/data/export/~	dn	H1
	home/movassag/Mbc/~	dpwr	37
	rocky/MBC-I-199pro~	dof	-500.0
	dC13.fid	dm	y
ACQUISITION		dmm	w
sfrq	125.796	dmf	10000
tn	C13	dseq	
at	1.736	dres	1.0
np	131010	homo	n
sw	37735.8	PROCESSING	
fb	not used	lb	0.30
bs	8	wtfile	
ss	1	proc	ft
tpwr	53	fn	131072
pw	6.9	math	f
d1	0.763		
tof	631.4	werr	
nt	2e+07	wexp	
ct	1168	wbs	
alock	n	wnt	
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-6225.0		
wp	37735.3		
vs	2970		
sc	0		
wc	250		
hzmm	10.88		
is	500.00		
rfl	18876.6		
rfp	12651.0		
th	28		
ins	1.000		
ai	ph		

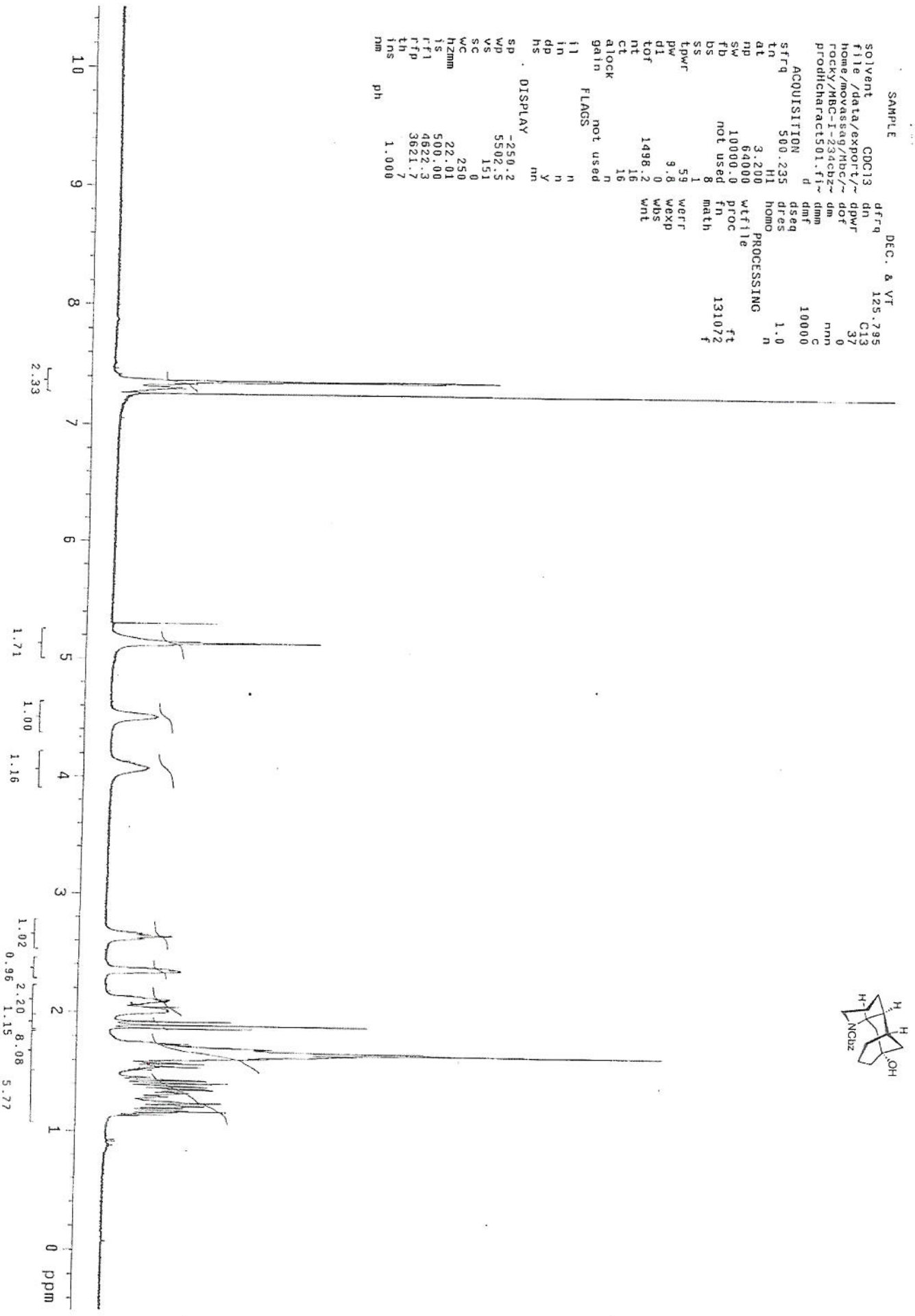



SAMPLE
solvent Benzene
file /data/export/~
home/movassag/Mbc/~
bullwinkle/MBC-I-1~
77amineOHrepurifie~
d0727n500.fid
ACQUISITION
sfrq 499.749
tn H1
at 3.277
np 65536
sw 9998.8
fb not used
bs 16
tpwr 56
pw 8.2
d1 0
tof 1498.1
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 5497.1
vs 157
sc 0
wc 250
hzmm 21.99
is 394.52
rfl 1002.6
rfp 0
th 7
ins 1.000
nm cdc ph
dfrq
dn
dpwr
dof
dm
dmm
dmf
dseq
dres
homo
PROCESSING
wtfile
proc
fn
math
werr
wexp
wbs
wnt
DEC. & VT
125.674
C13
34
1498.1
nnn
w
10400
1.0
n
ft
65536
f
H
H
H
NH
OH
0
1
2
3
4
5
6
7
8
9
10
ppm
1.10 1.10 1.07 4.20 1.13 2.17
1.00 1.03 0.63 1.93 1.15

SAMPLE	
solvent	Benzene
file	/data/export/~
	home/movassag/Mbc/~
	rocky/MBC-I-177ami~
	neOHrepurified0727~
	C13.fid
ACQUISITION	
sfrq	125.796
tn	C13
at	1.736
np	131010
sw	37735.8
fb	not used
bs	8
ss	1
tpwr	53
pw	6.9
d1	0.763
tof	631.4
nt	2e+07
ct	1328
alock	n
gain	not used
FLAGS	
il	n
in	n
dp	y
hs	nn
DISPLAY	
sp	-6225.0
wp	37735.3
vs	1491
sc	0
wc	250
hzmm	10.91
is	500.00
rfl	18876.6
rfp	12651.0
th	20
ins	1.000
at	pl

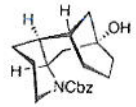
DEC. & VT	
dfrq	500.233
dn	H1
dpwr	37
dof	-500.0
dm	y
dmm	w
dmf	10000
dseq	
dres	1.0
homo	n
PROCESSING	
lb	0.30
wtfile	
proc	ft
fn	131072
math	f
werr	
wexp	
wbs	
wnt	

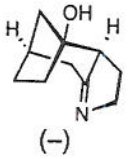



SAMPLE
solvent CDCl3
file /data/export/~
home/movassag/mbc/~
rocky/MBC-I-234cbz~
prodHcharact501.fi~
ACQUISITION
sfrq 500.235
tn H1
at 3.200
np 64000
sw 10000.0
fb not used
bs 8
ss 1
tpwr 59
pw 9.8
d1 0
tof 1498.2
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 5502.5
vs 151
sc 0
wc 250
hzmm 22.01
is 500.00
rfl 4622.3
rfp 3621.7
th 7
ins 1.000
nm ph
dfrq
dn
dpwr
dof
dm
dmm c
dmf 10000
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
DEC. & VT
125.795
C13
37
0
nnn
2.33
1.71
1.00
1.16
1.02
0.96
2.20
1.15
8.08
5.77
10
9
8
7
6
5
4
3
2
1
0
ppm
H
H
H
NCbz
OH

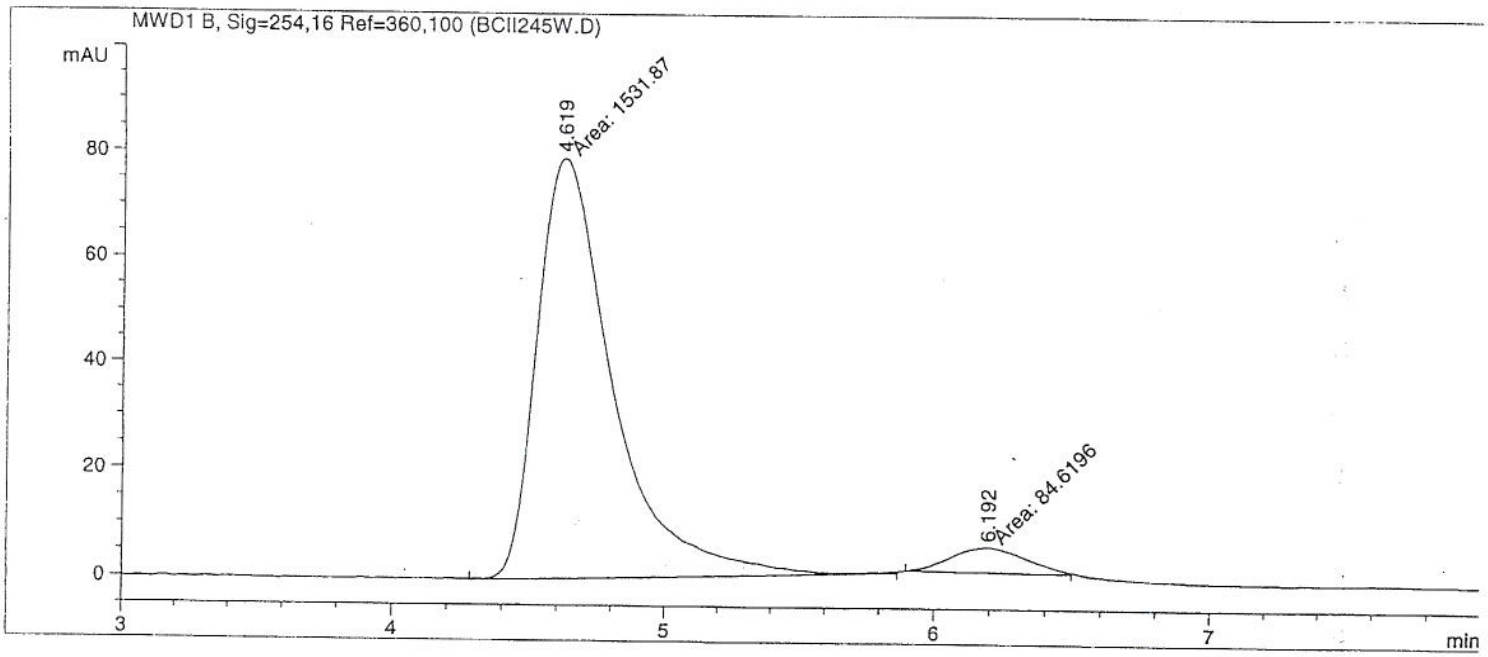
exp5 s2pul

SAMPLE		DEC. & VT	
solvent	CDCl3	dfrq	500.233
		dn	H1
		dpwr	37
		dof	-500.0
	.a	dm	y
ACQUISITION		dmm	w
sfrq	125.796	dmf	10000
tn	C13	dseq	
at	1.736	dres	1.0
np	131010	homo	n
sw	37735.8	PROCESSING	
fb	not used	lb	0.30
bs	8	wtfile	
ss	1	proc	ft
tpwr	53	fn	131072
pw	6.9	math	f
d1	0.763	werr	
tof	631.4	wexp	
nt	1e+08	wbs	
ct	4952	wnt	
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-6225.2		
wp	37735.3		
vs	1041		
sc	0		
wc	250		
hzmm	150.94		
is	500.00		
rfl	15911.8		
rfp	9686.0		
th	20		
ins	1.000		
ai ph			

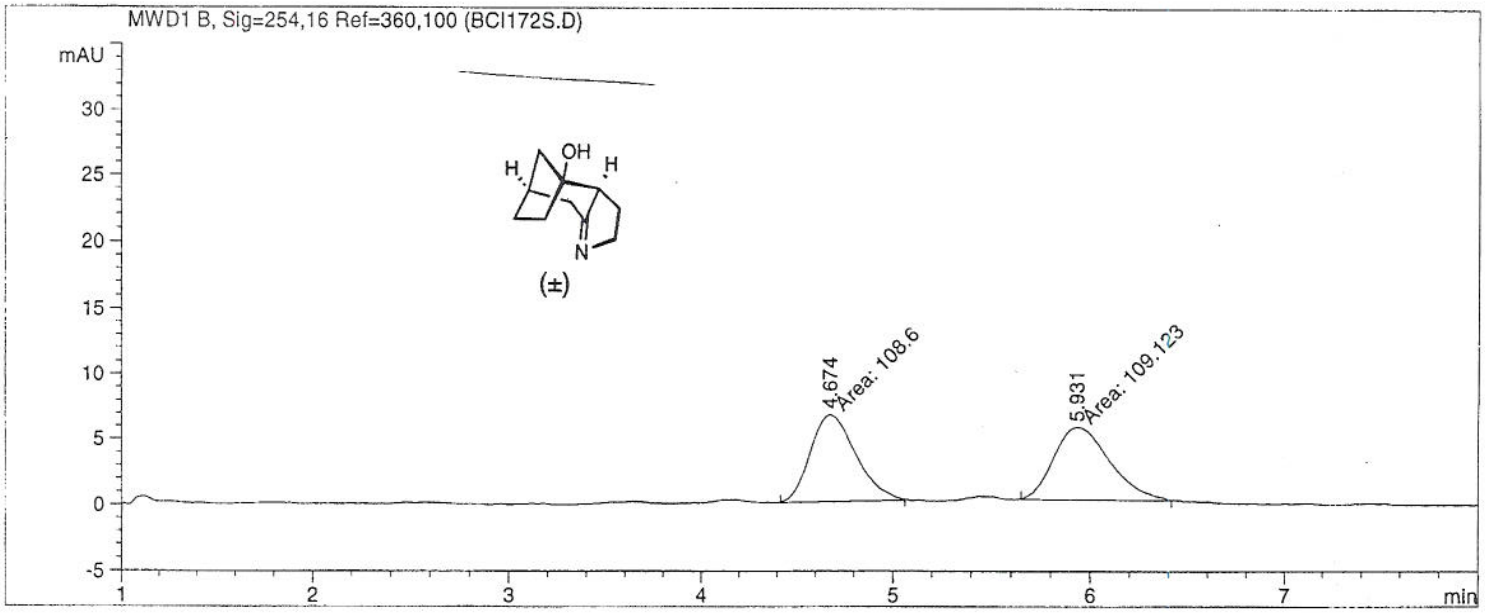




(–)




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   4.619 MM    0.3228 1531.86792   79.09542  94.7652
   2   6.192 MM    0.3153   84.61961    4.47352   5.2348

Totals :                  1616.48753   83.56894

 Results obtained with enhanced integrator!
=====================================================================
                        *** End of Report ***
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   4.674 MM    0.2731  108.60015    6.62695  49.8798
   2   5.931 MM    0.3316  109.12340    5.48517  50.1202

Totals :                   217.72355   12.11212

 Results obtained with enhanced integrator!
=====================================================================
                        *** End of Report ***
```