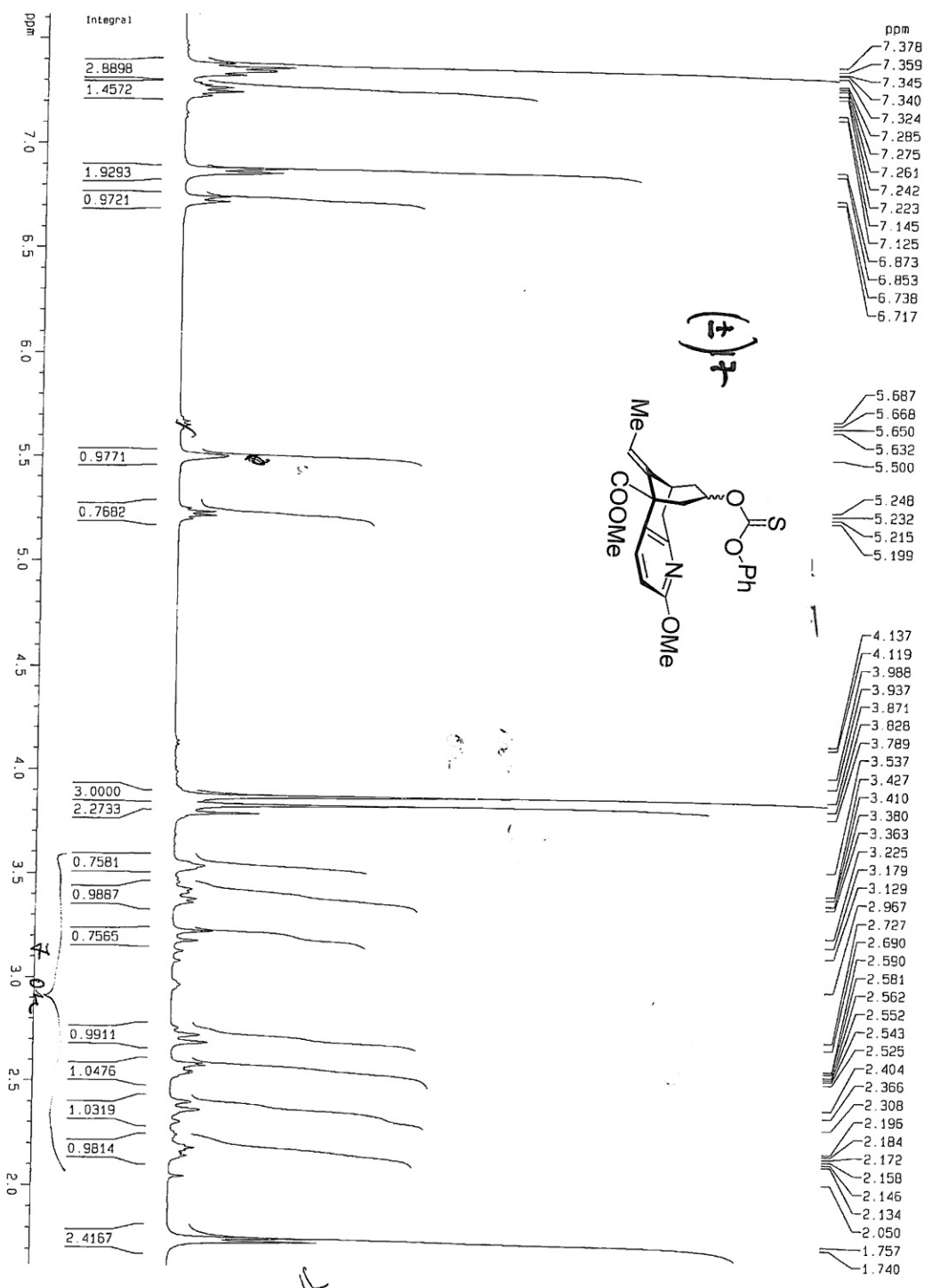
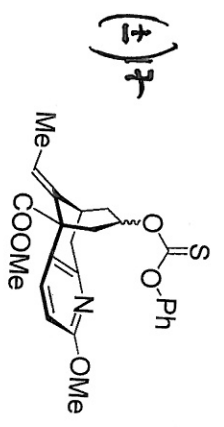


Sample: *Marcus* **(±)17**  
 Solvent: *CDCl3*  
 Standard 1-D Survey Proton NMR Experiment  
 Bruker DPX-400 NMR Spectrometer  
 October 14, 2004 / D. Lankin



Current Data Parameters  
 NAME: marcus  
 EXPNO: 76  
 PROCNO: 1

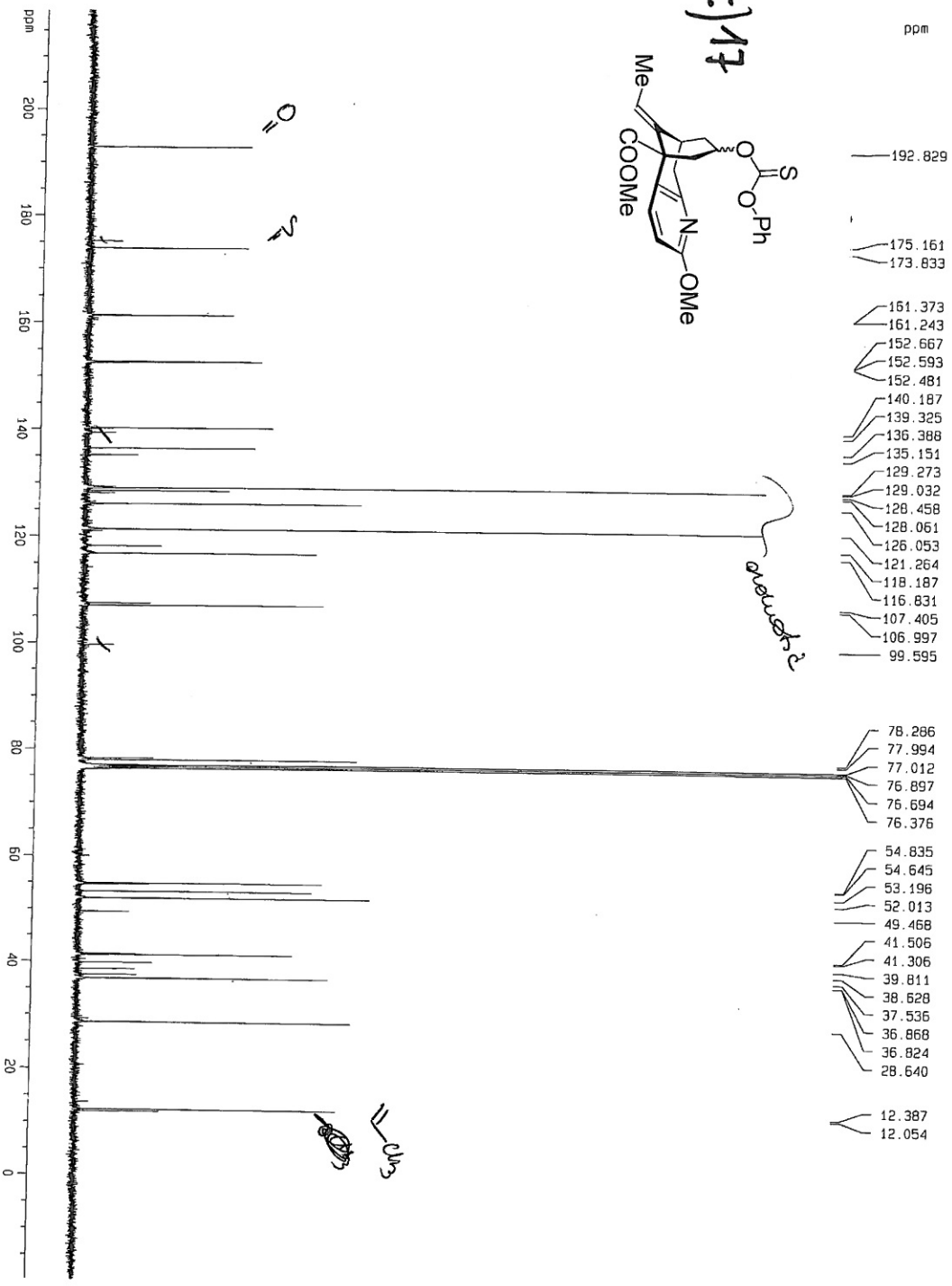
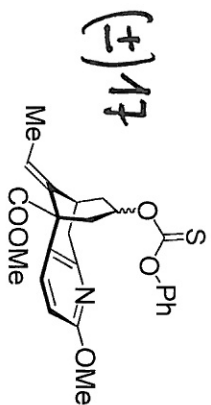
F2 - Acquisition Parameters  
 Date\_ Time: 20101005 15.22  
 INSTRUM: spect  
 PROBHD: 5 mm QNP 1H/1  
 PULPROG: zg30  
 TD: 65536  
 SOLVENT: CDCl3  
 NS: 16  
 DS: 2  
 SMH: 8278.146 Hz  
 FIDRES: 0.126314 Hz  
 AQ: 3.9584243 sec  
 RG: 64  
 DE: 60.400 usec  
 TE: 299.2 K  
 D1: 1.00000000 sec  
 MDCREST: 0.00000000 sec  
 MCMARK: 0.01500000 sec

===== CHANNEL f1 =====  
 NUC1: 1H  
 P1: 12.00 usec  
 PL1: 0.00 dB  
 SFO1: 400.1324710 MHz

F2 - Processing parameters  
 SI: 262144  
 SF: 400.1300037 MHz  
 MDW: EM  
 SSB: 0  
 LB: 0.30 Hz  
 GB: 0  
 PC: 1.00

1D NMR plot parameters  
 CX: 28.00 cm  
 CY: 8.00 cm  
 F1P: 7.617 ppm  
 F1: 3047.78 Hz  
 F2P: 1.625 ppm  
 F2: 650.15 Hz  
 PPMICH: 0.21400 ppm/cm  
 HZCM: 85.62978 Hz/cm

Sample: **(±)17**  
 Solvent: 1,1,1-Trichloroethane  
 1-D Carbon-13 NMR Spectrum  
 with H1Z2-16 (ppm) Decoupling  
 Bruker DPX-400 NMR Spectrometer  
 October 14, 2004 / D. Linkin



```

Current Data Parameters
NAME          marcus
EXPNO        77
PROCNO       1

F2 - Acquisition Parameters
Date_        20101005
Time         15.28
INSTRUM     spect
PROBHD      5 mm QNP 1H/1
PULPROG     zgpg30
TD           65536
SOLVENT     CDCl3
NS           1340
DS           4
SWH          23980.814 Hz
FIDRES      0.365918 Hz
AQ           1.3664756 sec
RG           574.7
DK           20.850 usec
DE           6.00 usec
TE           299.2 K
D1           1.00000000 sec
d11          0.03000000 sec
DELTA       0.89999998 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

***** CHANNEL f1 *****
NUC1         13C
P1           10.00 usec
PL1          -3.00 dB
SFO1        100.628303 MHz

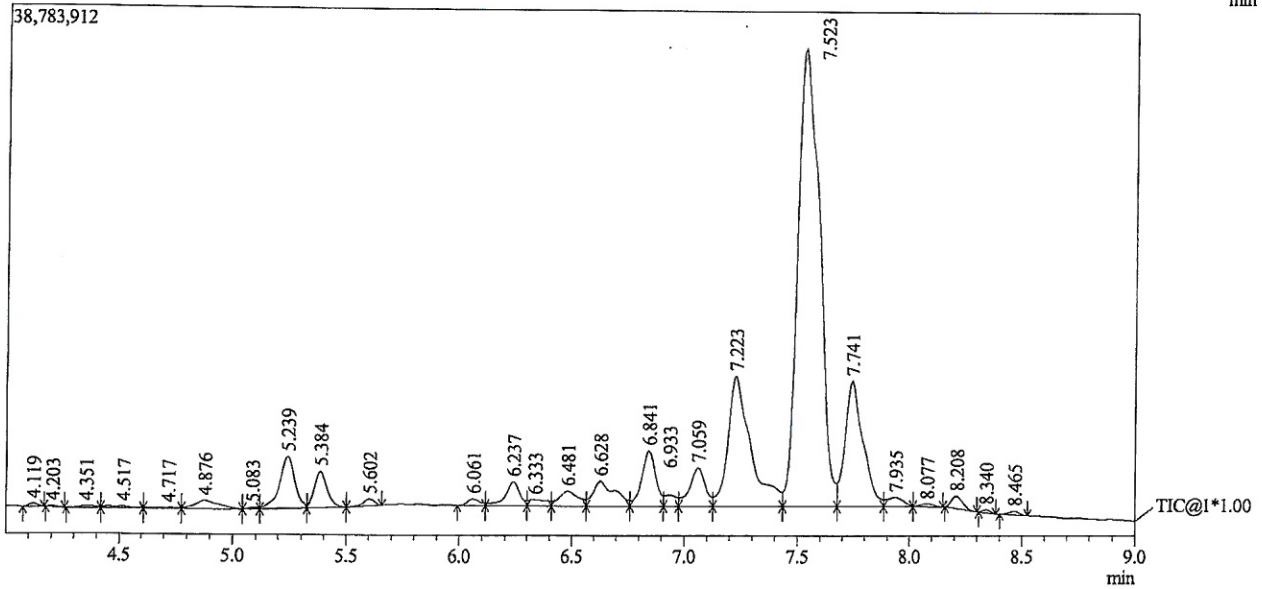
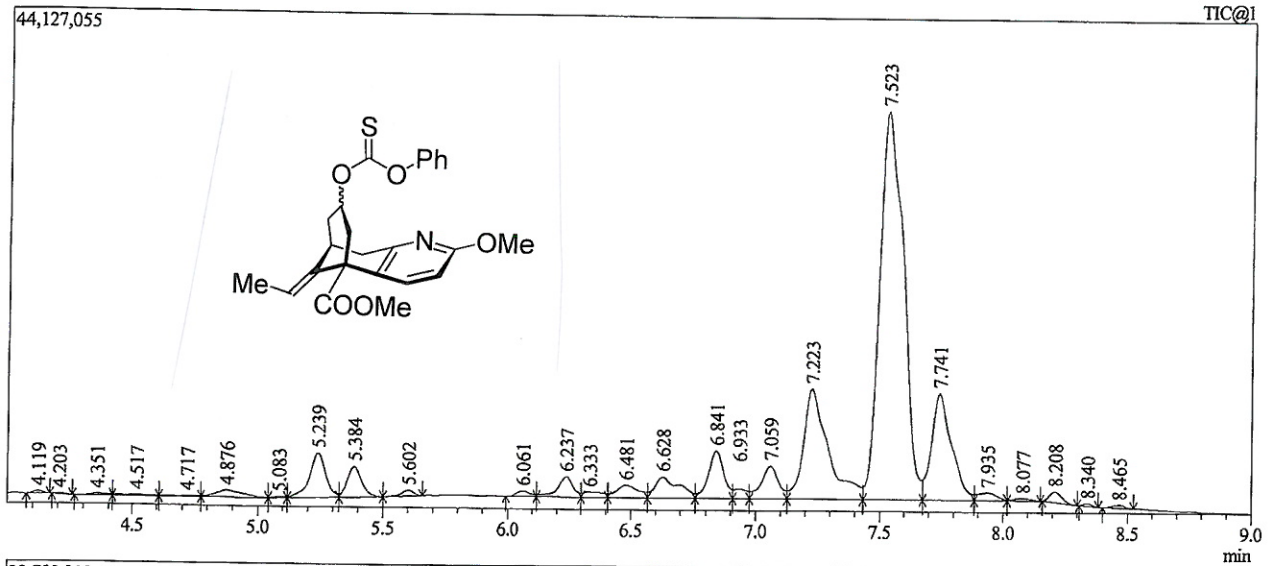
***** CHANNEL f2 *****
CPDPRG2     waltz16
NUC2         1H
PCPD2       80.00 usec
PL2         0.00 dB
PL12        17.00 dB
PL13        17.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI           32768
SF           100.6128056 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40

1D NMR plot parameters
CX           28.00 cm
CY           15.00 cm
F1P          218.770 ppm
F1           22014.04 Hz
F2P          -19.578 ppm
F2           -1969.77 Hz
PPOCKM      B 51241 DDM/cm
WZCH        856.45770 Hz/cm
  
```

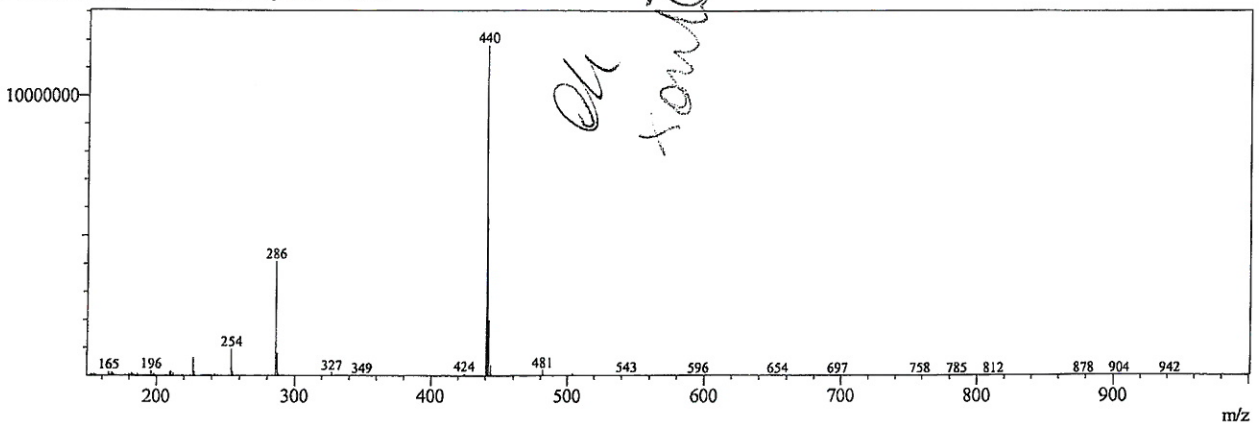
==== Shimadzu LCMSsolution Data Report ====

<Chromatogram>



<Spectrum>

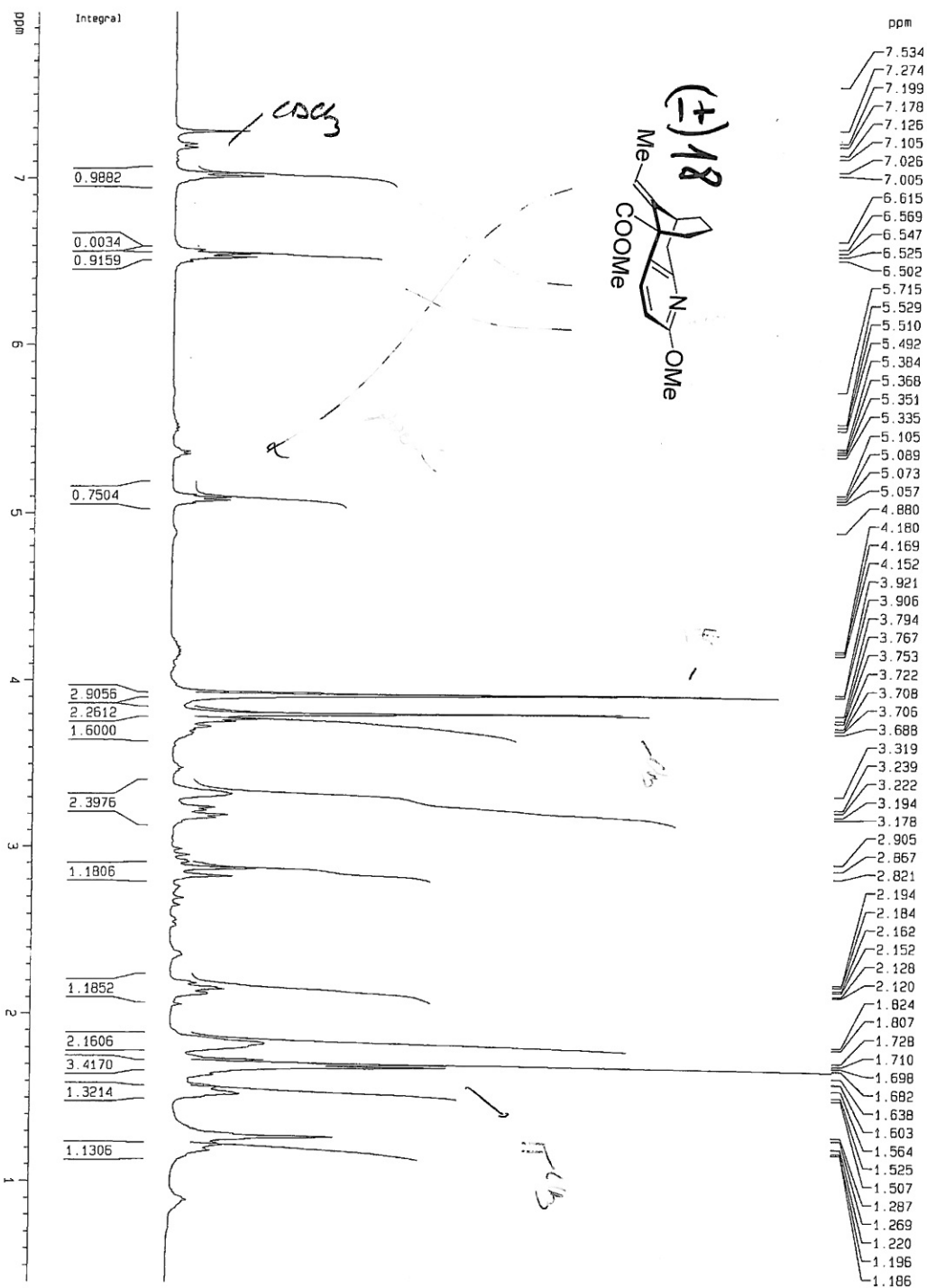
Retention Time: 7.517 (Scan#: 423)  
Max Peak: 850 Base Peak: 440.30 (11841900)  
Spectrum: Averaged 7.500-7.592 (421-432)  
Background: None Polarity: Pos Segment: 1 - Event 1



Standard 1-D Survey Proton NMR experiment  
 Bruker DPX-400 NMR Spectrometer  
 October 14, 2004 / D. Lankin

Sample: 1

(±) 18



- 7.534
- 7.274
- 7.199
- 7.178
- 7.126
- 7.105
- 7.026
- 7.005
- 6.615
- 6.569
- 6.547
- 6.525
- 6.502
- 5.715
- 5.529
- 5.510
- 5.492
- 5.384
- 5.368
- 5.351
- 5.335
- 5.105
- 5.089
- 5.073
- 5.057
- 4.880
- 4.180
- 4.169
- 4.152
- 3.921
- 3.906
- 3.794
- 3.767
- 3.753
- 3.722
- 3.708
- 3.706
- 3.688
- 3.319
- 3.239
- 3.222
- 3.194
- 3.178
- 2.905
- 2.867
- 2.821
- 2.194
- 2.184
- 2.162
- 2.152
- 2.128
- 2.120
- 1.824
- 1.807
- 1.728
- 1.710
- 1.698
- 1.682
- 1.638
- 1.603
- 1.564
- 1.525
- 1.507
- 1.287
- 1.269
- 1.220
- 1.196
- 1.186

Current Data Parameters  
 NAME: marcus  
 EXPNO: 103  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_: 2010118  
 Time: 20.16  
 INSTRUM: spect  
 PROBHD: 5 mm QNP 1H/1  
 PULPROG: zg30  
 TD: 65536  
 SOLVENT: CDCl3  
 NS: 16  
 DS: 2  
 SMH: 8278.146 Hz  
 FIDRES: 0.126314 Hz  
 AQ: 3.9584243 sec  
 RG: 128  
 DK: 60.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 D1: 1.00000000 sec  
 MPREST: 0.00000000 sec  
 MCMWK: 0.01500000 sec

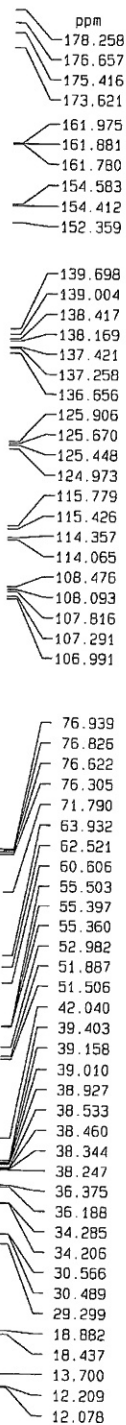
===== CHANNEL f1 =====  
 NUC1: 1H  
 P1: 12.00 usec  
 PL1: 0.00 dB  
 SF01: 400.1324710 MHz

F2 - Processing parameters  
 SI: 262144  
 SF: 400.130037 MHz  
 MDW: EM  
 SSB: 0  
 LB: 0.30 Hz  
 GB: 0  
 PC: 1.00

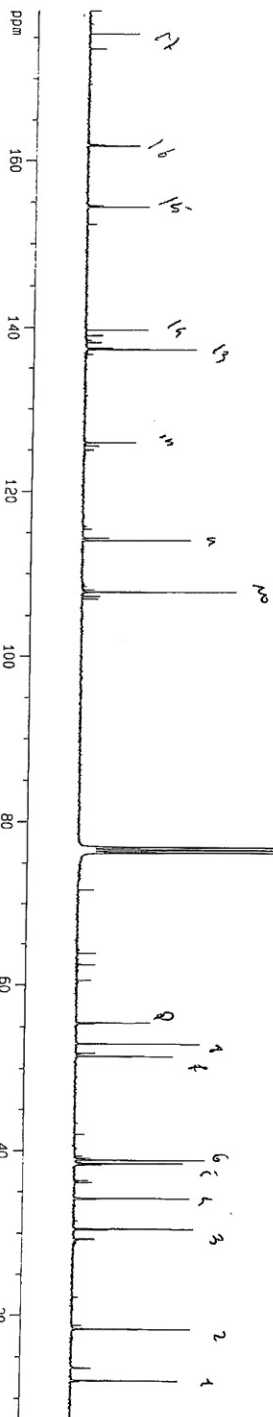
1D NMR plot parameters  
 CX: 28.00 cm  
 CY: 12.00 cm  
 F1P: 7.995 ppm  
 F1: 3199.21 Hz  
 F2P: 0.405 ppm  
 F2: 162.21 Hz  
 PPMCM: 0.27107 ppm/cm  
 HZCM: 108.46438 Hz/cm

Sample: 1  
 Solvent: 1-0 Carbon-13 NMR Spectrum  
 with Wa1z16 (CPD) Decoupling  
 Bruker DPX-400 NMR Spectrometer  
 October 14, 2004 / D. Linkin

(+) 18



*Chromatic f on  
 kubo 1 ok  
 Hphhc 2 on*



Current Data Parameters  
 NAME Marcuss  
 EXPNO 104  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20101118  
 Time 20.36  
 INSTRUM spect  
 PROBRD 5 mm QNP 1H/1  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 15488  
 DS 4  
 SWH 23980.814 Hz  
 FIDRES 0.365918 Hz  
 AQ 1.3664756 sec  
 RG 574.7  
 DW 20.850 usec  
 DE 6.00 usec  
 TE 298.2 K  
 D1 1.00000000 sec  
 d11 0.03000000 sec  
 DELTA 0.89999998 sec  
 MCREST 0.00000000 sec  
 KMCRK 0.01500000 sec

==== CHANNEL f1 =====  
 NUC1 13C  
 P1 10.00 usec  
 PL1 -3.00 dB  
 SFO1 100.6228303 MHz

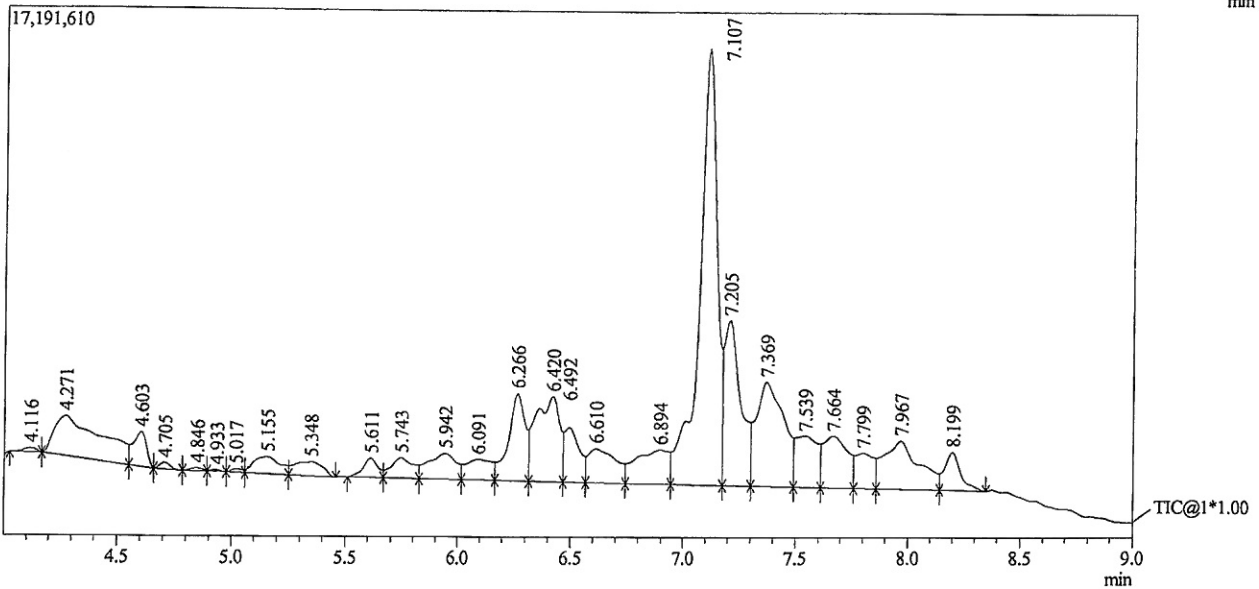
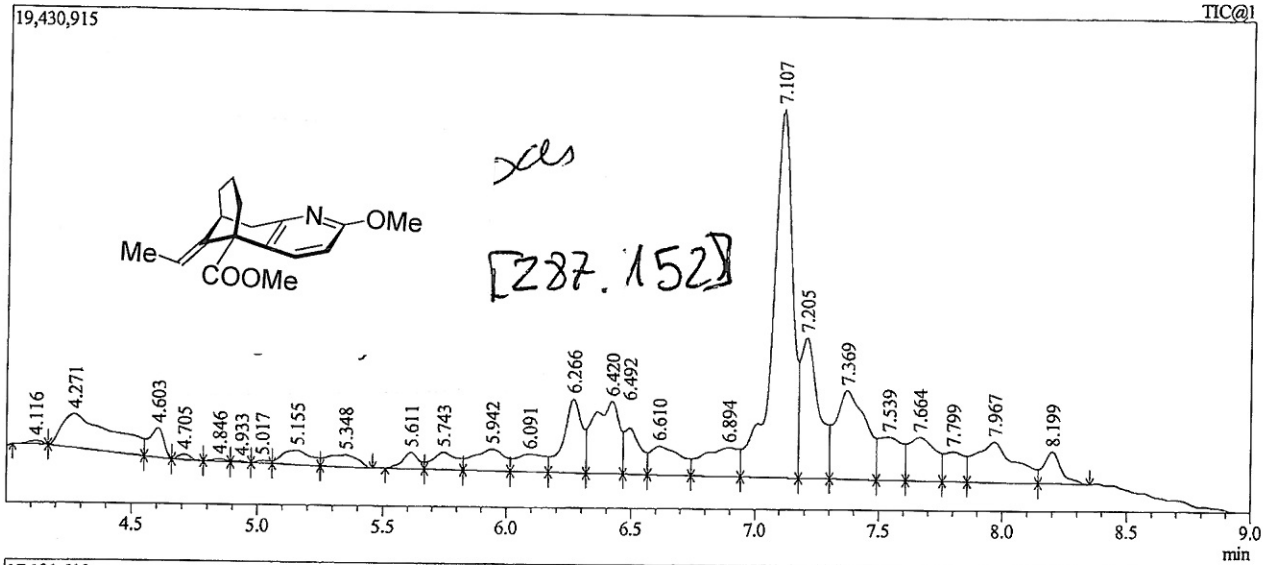
==== CHANNEL f2 =====  
 CPDPRG2 wa1z16  
 NUC2 1H  
 PCPD2 80.00 usec  
 PL2 0.00 dB  
 PL12 17.00 dB  
 PL13 17.00 dB  
 SFO2 400.1315005 MHz

F2 - Processing parameters  
 SI 32758  
 SF 100.6128095 MHz  
 MDN EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

1D NMR plot parameters  
 CX 28.00 cm  
 CY 3.00 cm  
 F1P 178.319 ppm  
 F1 17941.13 Hz  
 F2P 7.551 ppm  
 F2 759.75 Hz  
 PPKCH E.09863 ppm/cm  
 HZCM 613.62061 Hz/cm

==== Shimadzu LCMsolution Data Report =====

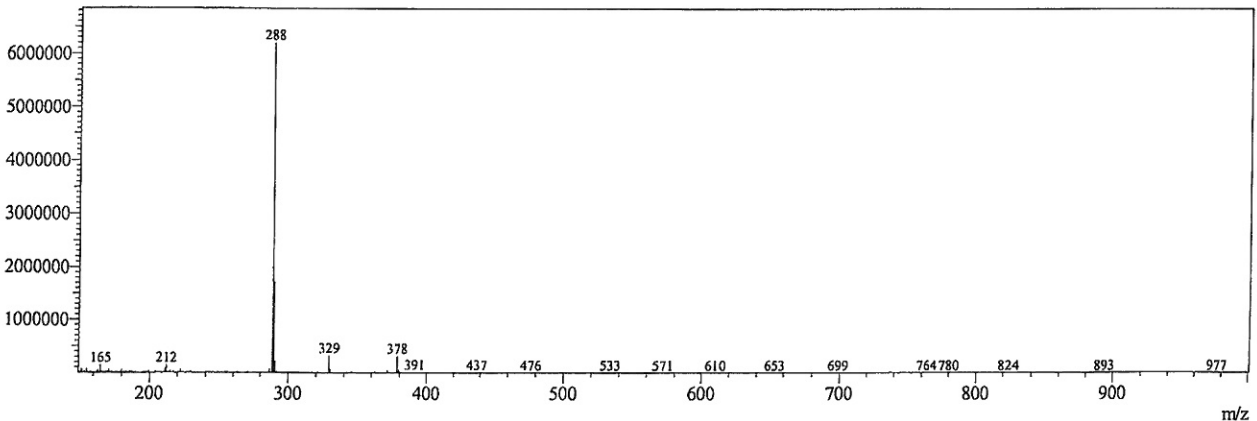
<Chromatogram>



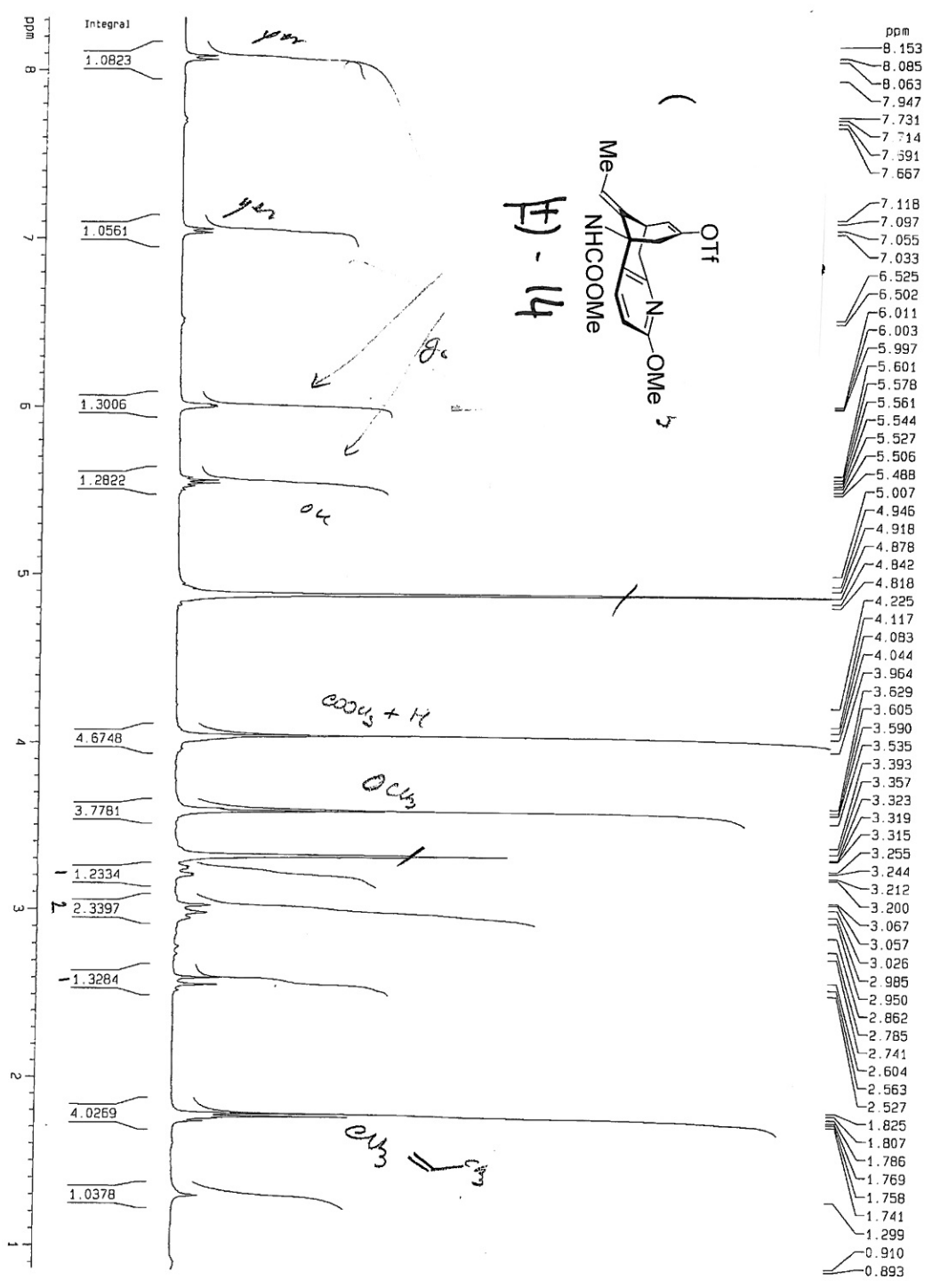
<Spectrum>

Retention Time: 7.100 (Scan#: 373)  
Max Peak: 851 Base Peak: 288.25 (6202411)  
Spectrum: Averaged 7.075-7.167 (370-381)  
Background: None Polarity: Pos Segment 1 - Event 1

*benzoin ok*



Sample: ~~100~~ (+)14  
 Solvent: MeOD  
 Standard 1-D Survey Procton NMR Experiment  
 Bruker DPX-400 NMR Spectrometer  
 October 14, 2004 / O. Lankin



Current Data Parameters  
 NAME: Marcus  
 EXPNO: 128  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_: 20101119  
 Time: 10:44  
 INSTNAME: spect  
 PROBHD: 5 mm GNP 1H/1  
 PULPROG: zg30  
 ID: g5536  
 SOLVENT: MeOD  
 NS: 15  
 DS: 2  
 SMH: 8278.146 Hz  
 FIDRES: 0.126314 Hz  
 AQ: 3.9584243 sec  
 RG: 406.4  
 DM: 60.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 D1: 1.00000000 sec  
 MCREST: 0.00000000 sec  
 MCKMR: 0.01500000 sec

===== CHANNEL f1 =====  
 NUC1: 1H  
 P1: 12.00 usec  
 PL1: 0.00 dB  
 SFO1: 400.1324710 MHz

F2 - Processing parameters  
 SI: 262144  
 SF: 400.1300037 MHz  
 WDW: EM  
 SSB: 0  
 LB: 0.30 Hz  
 GB: 0  
 PC: 1.00

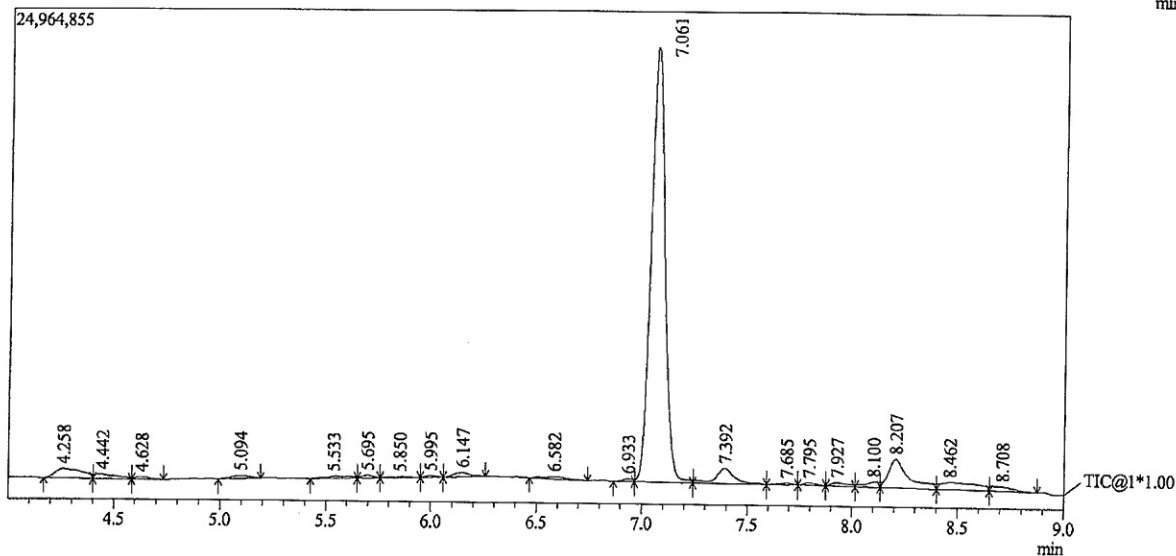
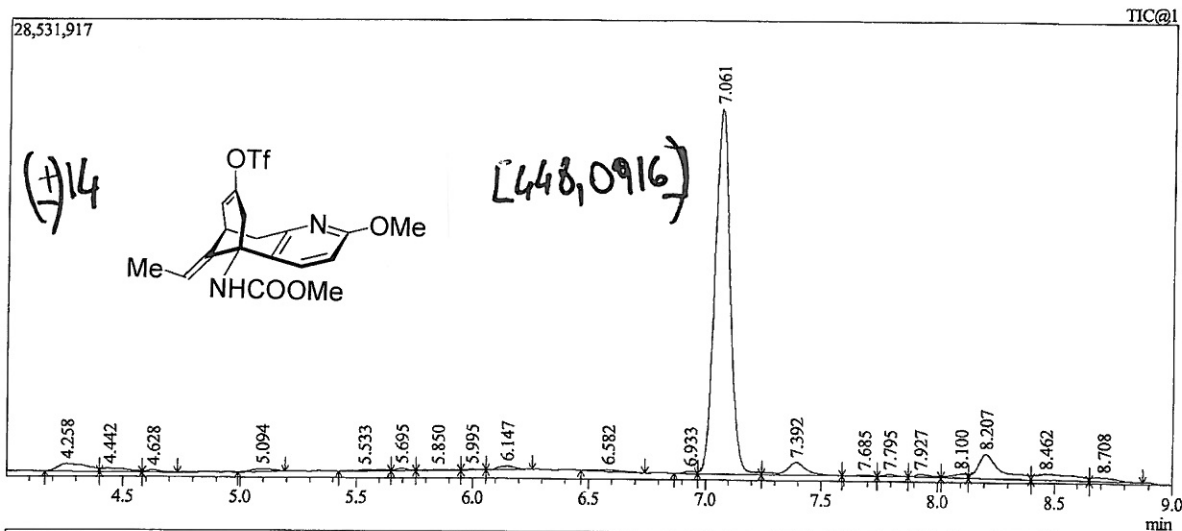
1D NMR plot parameters  
 CX: 28.00 cm  
 CY: 19.00 cm  
 F1P: 8.316 ppm  
 F1: 3327.57 Hz  
 F2P: 0.862 ppm  
 F2: 344.89 Hz  
 PPMCM: 0.26622 ppm/cm  
 HZCM: 105.52425 Hz/cm



(+) 14

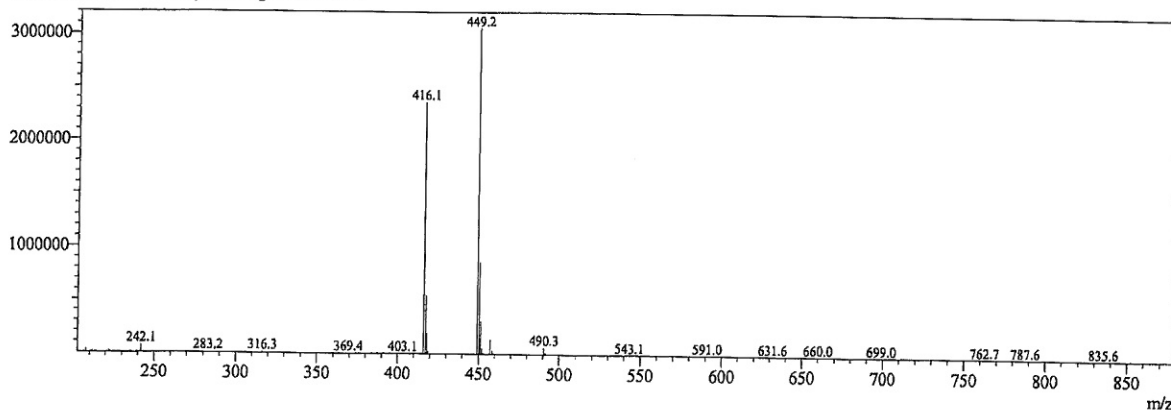
==== Shimadzu LCMsSolution Data Report ====

<Chromatogram>

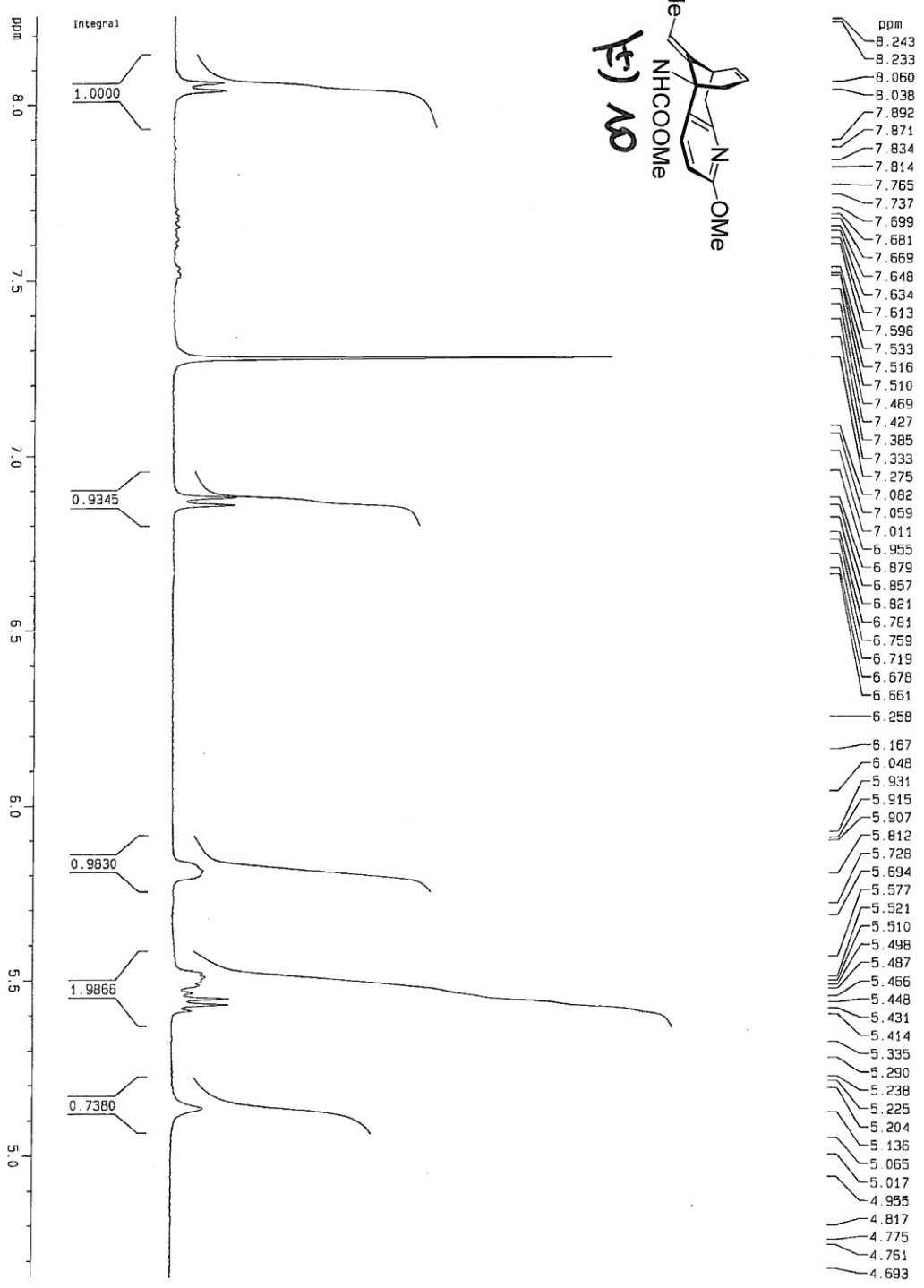


<Spectrum>

Retention Time: 7.058(Scan#:368)  
Max Peak: 851 Base Peak: 449.15(3054161)  
Spectrum: Averaged 6.992-7.192(360-384)  
Background: None Polarity: Pos Segment1 - Event1



Sample: ~~100-10~~ (F) NO CDU3  
 Standard 1-D Survey Proton NMR Experiment  
 Bruker DPX-400 NMR Spectrometer  
 October 14, 2004 / O. Lankin



- 8.243
- 8.233
- 8.060
- 8.038
- 7.892
- 7.871
- 7.834
- 7.814
- 7.765
- 7.737
- 7.699
- 7.681
- 7.659
- 7.648
- 7.634
- 7.613
- 7.596
- 7.533
- 7.516
- 7.510
- 7.469
- 7.427
- 7.385
- 7.333
- 7.275
- 7.082
- 7.059
- 7.011
- 6.955
- 6.879
- 6.857
- 6.821
- 6.781
- 6.759
- 6.719
- 6.678
- 6.661
- 6.258
- 6.167
- 6.048
- 5.931
- 5.915
- 5.907
- 5.812
- 5.728
- 5.694
- 5.577
- 5.521
- 5.510
- 5.498
- 5.487
- 5.466
- 5.448
- 5.431
- 5.414
- 5.335
- 5.290
- 5.238
- 5.225
- 5.204
- 5.136
- 5.065
- 5.017
- 4.955
- 4.817
- 4.775
- 4.761
- 4.693

Current Data Parameters  
 NAME: marcus  
 EXPNO: 69  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_: 20100217  
 Time: 17.47  
 INSTRUM: spect  
 PROBHD: 5 mm QNP 1H/1  
 PULPROG: zg30  
 TD: 65536  
 SOLVENT: CDCl3  
 NS: 16  
 DS: 2  
 SMH: 8278.145 Hz  
 FIDRES: 0.126314 Hz  
 AQ: 3.9584243 sec  
 RG: 812.7  
 DW: 60.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 D1: 1.00000000 sec  
 MDELTA: 0.00000000 sec  
 MCWRR: 0.01500000 sec

===== CHANNEL f1 =====  
 NUC1: 1H  
 P1: 12.00 usec  
 PL1: 0.00 dB  
 SFO1: 400.1324710 MHz

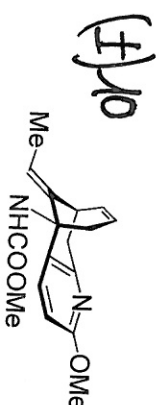
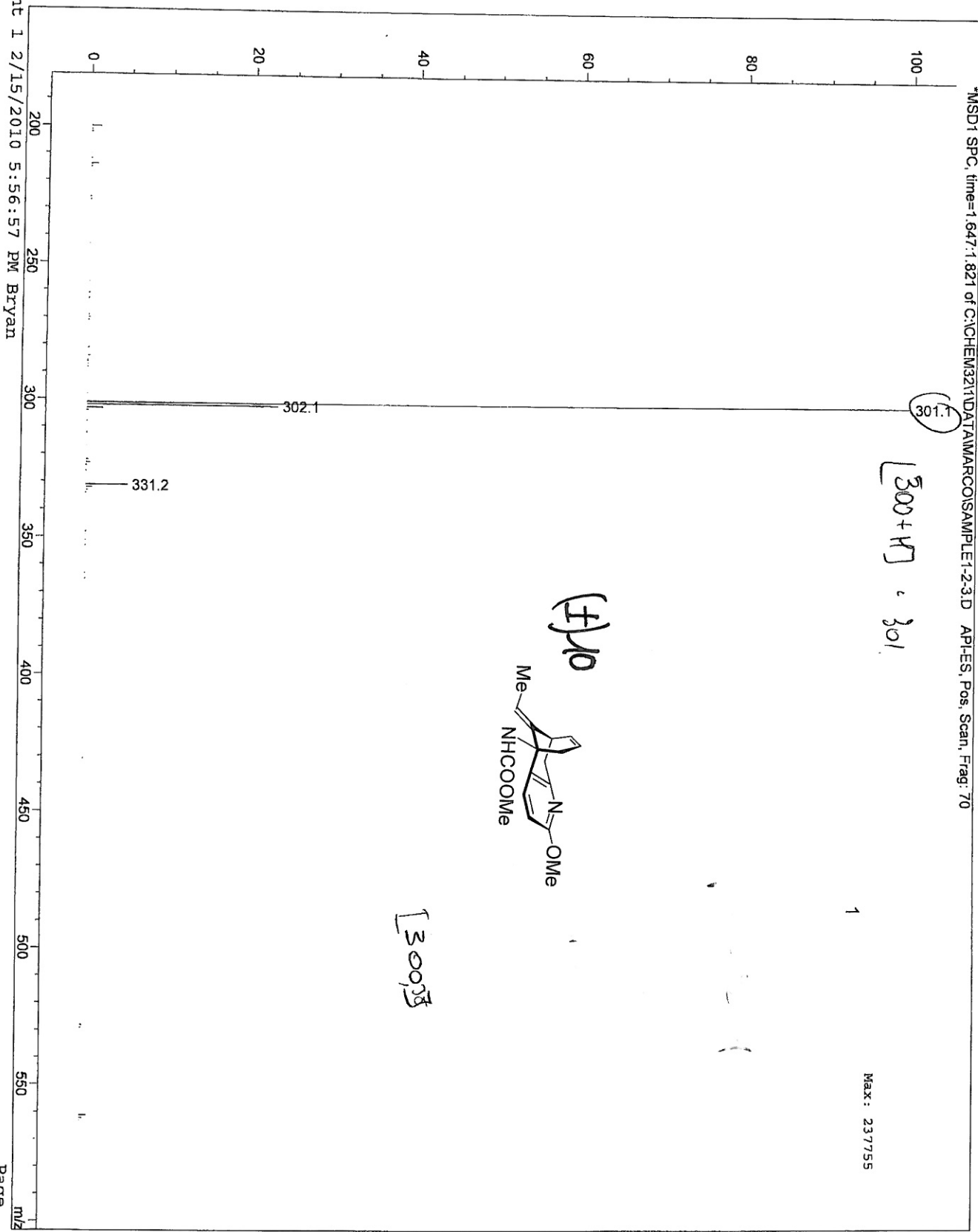
F2 - Processing parameters  
 SI: 262144  
 SF: 400.130037 MHz  
 WDW: EM  
 SSB: 0  
 LB: 0.30 Hz  
 GB: 0  
 PC: 1.00

1D NMR plot parameters  
 CX: 28.00 cm  
 CY: 10.00 cm  
 F1P: 8.254 ppm  
 F1: 3302.73 Hz  
 F2P: 4.654 ppm  
 F2: 1862.21 Hz  
 PPMCM: 0.12859 ppm/cm  
 HZCM: 51.44730 Hz/cm

(+) 10

MS Spectrum

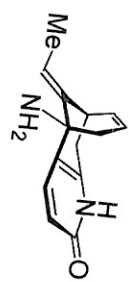
\*MSD1 SPC, time=1.647:1.821 of C:\CHEM32\1\DATA\MARCO\ISAMPLE1-2-3.D APLE5, Pos, Scan, Frag: 70



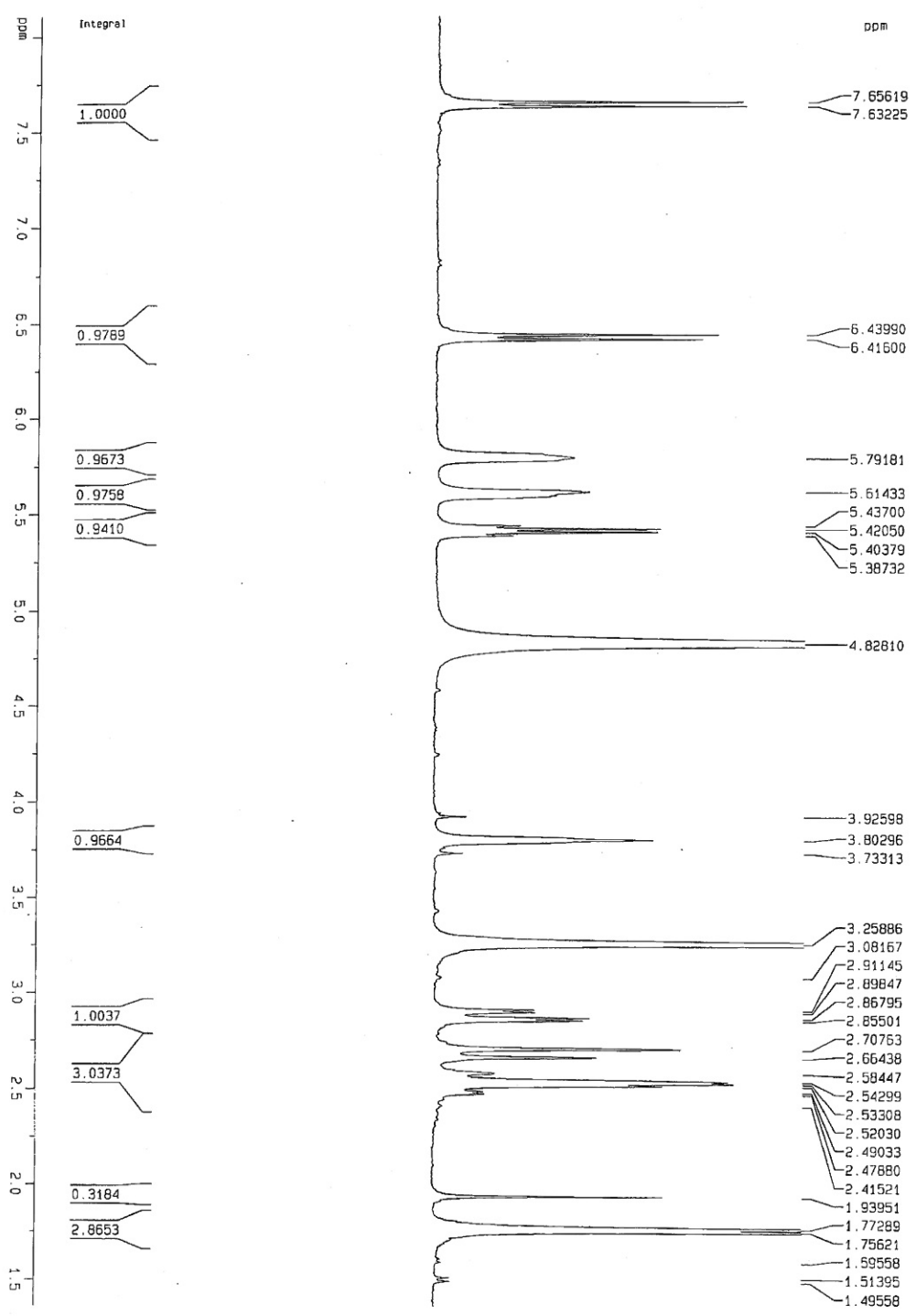
[300]3

Sample: ~~1000000000~~  
 Solvent: MeOD  
 Survey 1-D 4PT Experiment  
 C-13-200 / C-13-200  
 Bruker DPX-400 NMR Spectrometer  
 June 8, 2005 / D. Lemstra

(+)



MeOD



Current Data Parameters  
 NAME: marcus  
 EXPNO: 314  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_ 20101220  
 Time 15.35  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H/1  
 PULPROG zg30  
 TD 65536  
 SOLVENT MeOD  
 NS 16  
 DS 2  
 SWH 8278.148 Hz  
 FIDRES 0.126314 Hz  
 AQ 3.9584243 sec  
 RG 512  
 DM 60.400 usec  
 DE 5.00 usec  
 TE 298.2 K  
 D1 1.00000000 sec  
 ACQRES 0.00000000 sec  
 KMCHK 0.01500000 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NU1C1 1H  
 P1 12.00 usec  
 PL1 0.00 dB  
 SF01 400.1324710 MHz

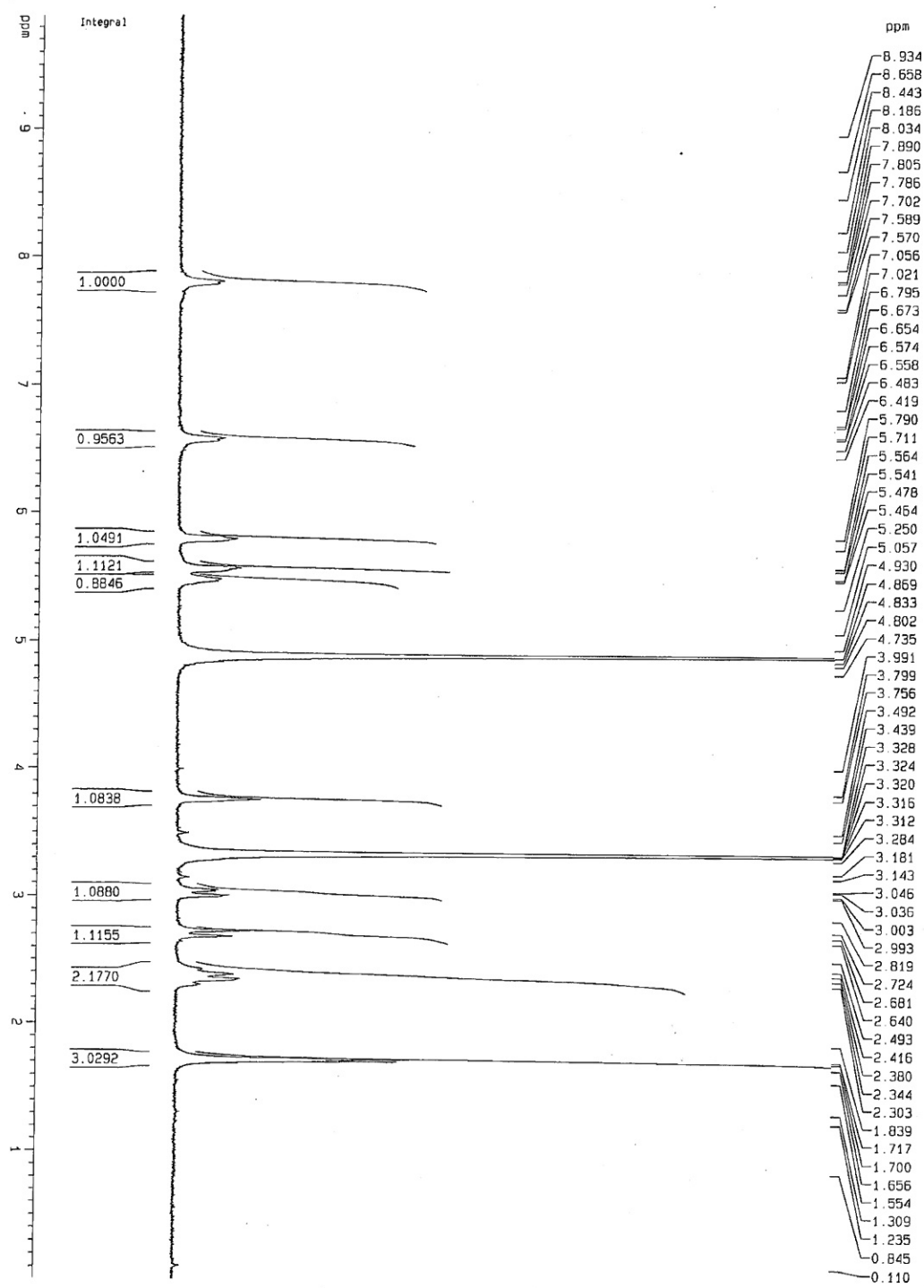
F2 - Processing parameters  
 S1 65536  
 SF 400.1300260 MHz  
 MDW EN  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

1D NMR plot parameters  
 CX 28.00 cm  
 CY 50.00 cm  
 F1P 8.113 ppm  
 F1 3246.43 Hz  
 F2P 1.364 ppm  
 F2 545.94 Hz  
 PPMCKM 0.24104 ppm/cm  
 HZCKM 96.44616 Hz/cm

Sample: **MWASST**  
 Solvent: **MeOD + D<sub>2</sub>O**  
 Standard 1-0 Survey Proton NMR Experiment  
 Bruker DPX-400 NMR Spectrometer  
 October 14, 2004 / D. Lankin

**MWASST**  
**MeOD + D<sub>2</sub>O**

**(+)-5 MeOD + D<sub>2</sub>O**



Current Data Parameters  
 NAME Marcus  
 EXPNO 316  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20110110  
 Time 10.29  
 INSTRUM spect  
 PROSD 5 mm QNP 1H/1  
 PULPROG zg30  
 TD 65536  
 SOLVENT MeOD  
 NS 16  
 DS 2  
 SMH 8278.146 Hz  
 FIDRES 0.126314 Hz  
 AQ 3.958243 sec  
 RG 724.1  
 DW 60.400 usec  
 DE 6.00 usec  
 TE 298.2 K  
 D1 1.00000000 sec  
 MPROG 0.00000000 sec  
 MCWK 0.01500000 sec

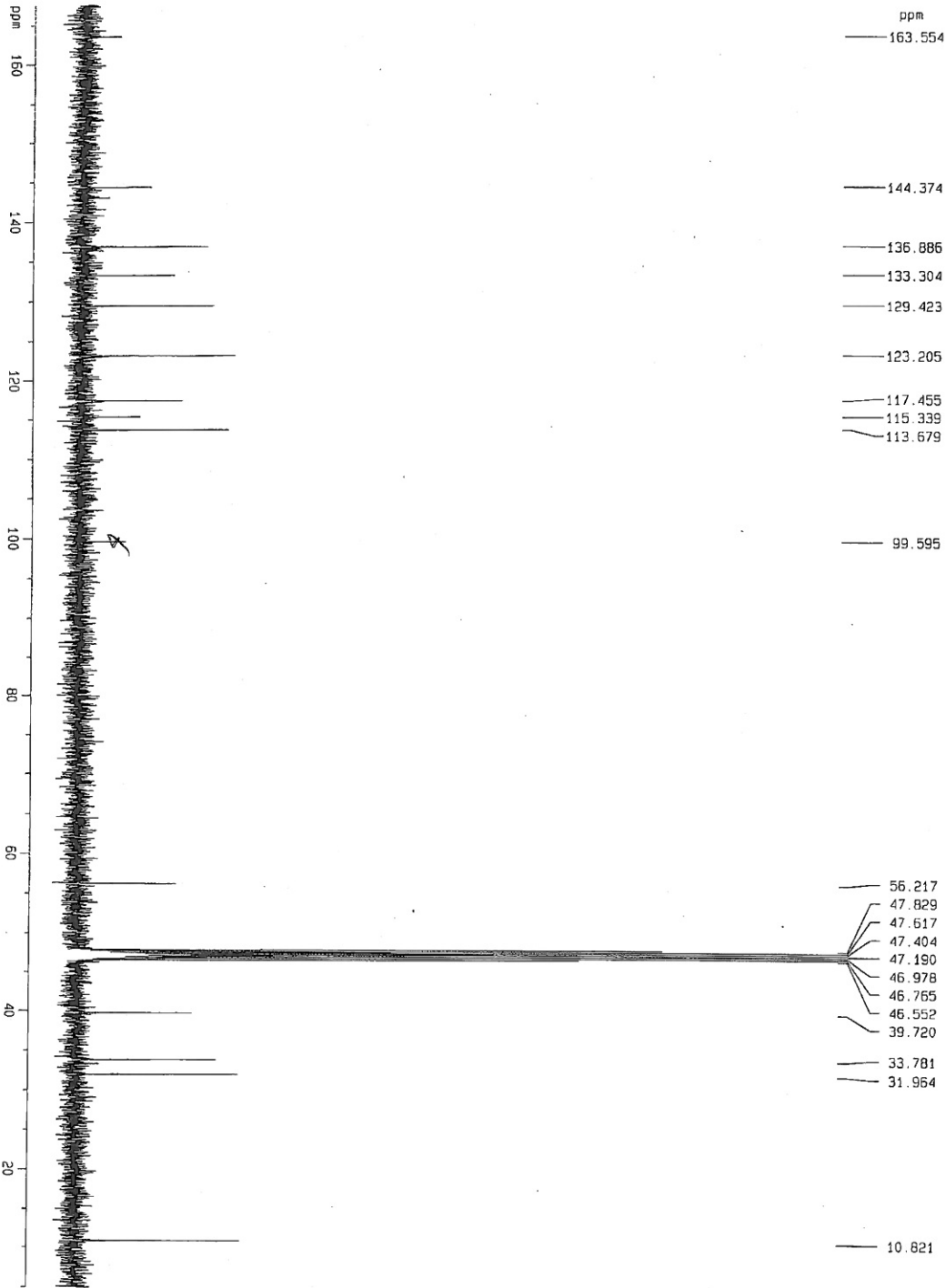
\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1 1H  
 P1 12.00 usec  
 PL1 0.00 dB  
 SF01 400.1324710 MHz

F2 - Processing parameters  
 SI 262144  
 SF 400.1300037 MHz  
 MDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

1D NMR plot parameters  
 CX 28.00 cm  
 CY 80.00 cm  
 F1P 9.888 ppm  
 F1 3956.36 Hz  
 F2P 0.006 ppm  
 F2 2.37 Hz  
 PPMCK 0.35292 ppm/cm  
 HZCM 141.21402 Hz/cm

Sample: **MONA**  
 Solvent: **MeOD**  
 1-D Carbon-13 NMR Spectrum  
 with Data for Auto Decoupling  
 Bruker Date: 18/10/2004  
 October 14, 2004 / D. Larkin

**(+)-5**  
**MeOD**



Current Data Parameters  
 NAME: marcus  
 EXPNO: 326  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_: 20110119  
 Time: 13.33

INSTRUM: spect  
 PROBRD: 5 mm GNP 1H/1  
 PULPROG: zgpg30  
 TO: 65536  
 SOLVENT: MeOD  
 NS: 1289

DS: 4  
 SMH: 23980.814 Hz  
 FIDRES: 0.365918 Hz  
 AQ: 1.3664756 sec

RG: 2298.8  
 DW: 20.850 usec  
 DE: 5.00 usec  
 TE: 299.2 K

D1: 1.00000000 sec  
 d11: 0.03000000 sec  
 DELTA: 0.89999998 sec  
 MCREST: 0.00000000 sec  
 MCHKK: 0.01500000 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1: 13C  
 P1: 10.00 usec  
 PL1: -3.00 dB  
 SF01: 100.628303 MHz

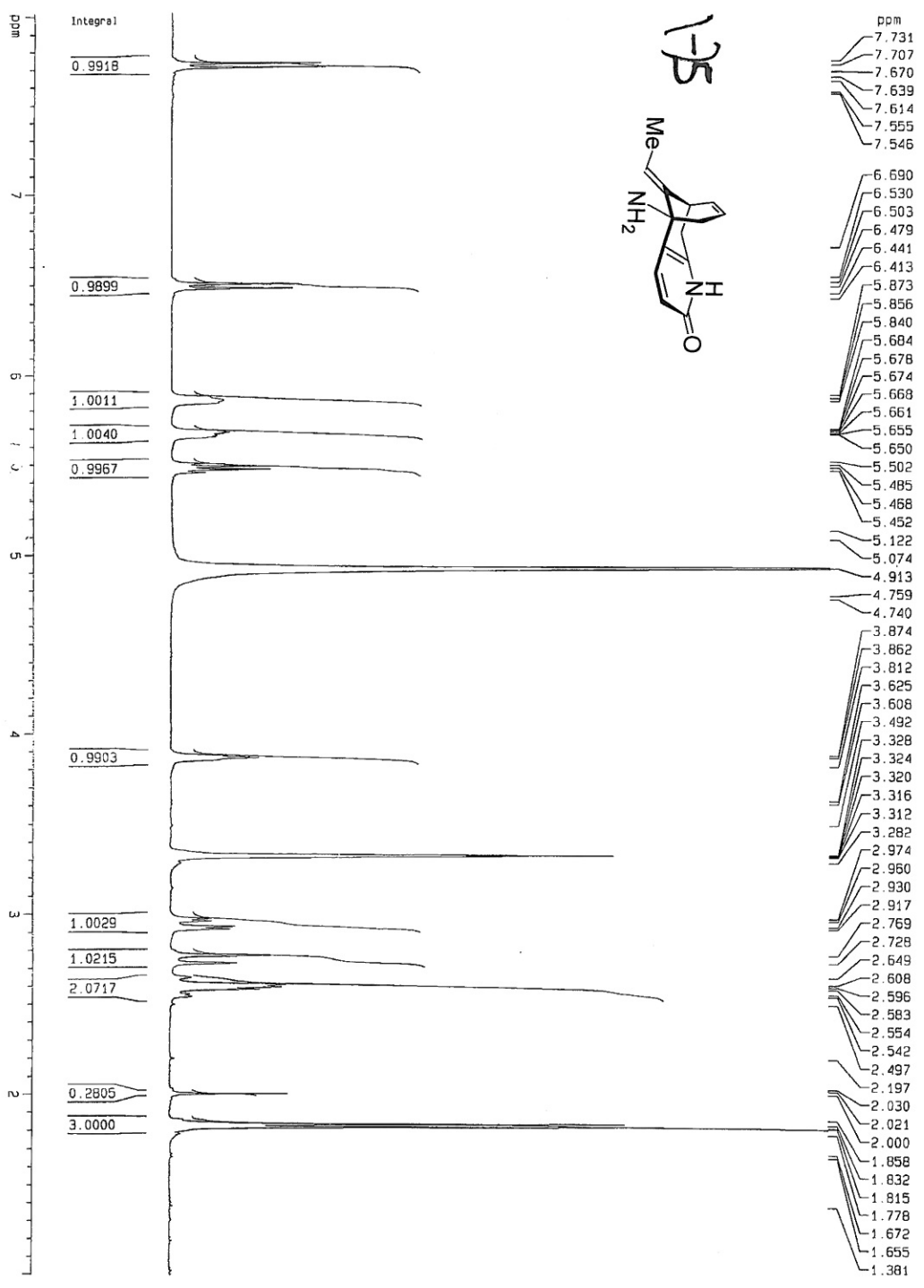
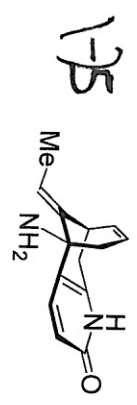
\*\*\*\*\* CHANNEL f2 \*\*\*\*\*  
 CPDPRG2: waltz16  
 NUC2: 1H  
 PCDP2: 80.00 usec  
 PL2: 0.00 dB  
 PL12: 17.00 dB  
 PL13: 17.00 dB  
 SF02: 400.1316005 MHz

F2 - Processing parameters  
 SI: 32768  
 SF: 100.6128095 MHz  
 WDW: EM  
 SSB: 0  
 LB: 1.00 Hz  
 GB: 0  
 PC: 1.40

1D NMR plot parameters  
 CX: 28.00 cm  
 CY: 85.00 cm  
 F1P: 167.633 ppm  
 F1: 16866.07 Hz  
 F2P: 4.979 ppm  
 F2: 500.99 Hz  
 PPGICH: 5.80907 ppm/cm  
 HZCM: 584.46716 Hz/cm

Sample: ~~135~~  
 SOLVENT: MeOD  
 Standard 1-D Survey Proton NMR Experiment  
 Bruker DPX-400 NMR Spectrometer  
 October 14, 2004 / D. Lankin

135  
 MeOD



Current Data Parameters  
 NAME: marcus  
 EXPNO: 319  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_: 20110110  
 Time: 10.55  
 INSTRUM: spect  
 PROBHD: 5 mm QNP 1H/1  
 PULPROG: zg30  
 TD: 65536  
 SOLVENT: MeOD  
 NS: 16  
 DS: 2  
 SWH: 8278.146 Hz  
 FIDRES: 0.126314 Hz  
 AQ: 3.9584243 sec  
 RG: 382  
 DK: 60.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 O1: 1.00000000 sec  
 MCREST: 0.00000000 sec  
 MCWRR: 0.01500000 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1: 1H  
 P1: 12.00 usec  
 PL1: 0.00 dB  
 SFO1: 400.1324710 MHz

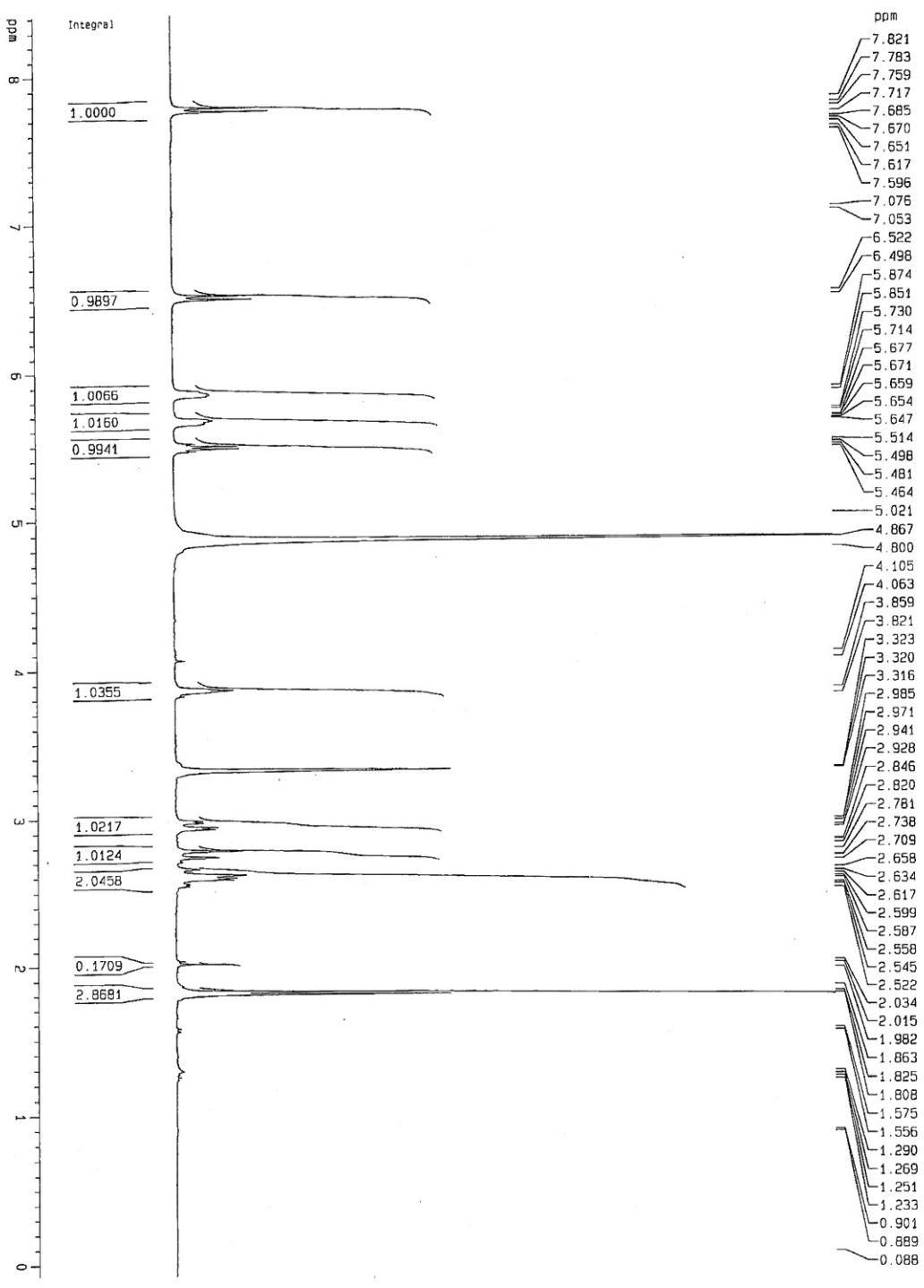
F2 - Processing parameters  
 SI: 262144  
 SF: 400.130037 MHz  
 WDW: EM  
 SSB: 0  
 LB: 0.30 Hz  
 GB: 0  
 PC: 1.00

1D NMR plot parameters  
 CX: 28.00 cm  
 CY: 19.00 cm  
 F1P: 8.016 dbm  
 F1: 3207.62 Hz  
 F2P: 0.994 dbm  
 F2: 397.77 Hz  
 PPMCH: 0.25080 ppm/cm  
 HZCM: 100.35209 Hz/cm

Sample: Standard 1-D Survey Proton NMR Experiment  
 SOLVENT: Bruker DPX-400 NMR Spectrometer  
 October 14, 2004 / D. Lankin

*Handwritten:* ~~MeOD~~ D<sub>2</sub>O

*Handwritten:* MeOD + D<sub>2</sub>O



Current Data Parameters  
 NAME: marcus  
 EXPNO: 327  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_: 20110119  
 Time: 14.30  
 INSTRUM: spect  
 PROBHD: 5 mm QNP 1H/1  
 PULPROG: zg30  
 TD: 65536  
 SOLVENT: MeOD  
 NS: 15  
 DS: 2  
 SMH: 8278.146 Hz  
 FIDRES: 0.126314 Hz  
 AQ: 3.9584243 sec  
 R6: 322.5  
 DM: 60.400 usec  
 DE: 6.00 usec  
 TE: 299.2 K  
 D1: 1.00000000 sec  
 MCREST: 0.00000000 sec  
 MCWRR: 0.01500000 sec

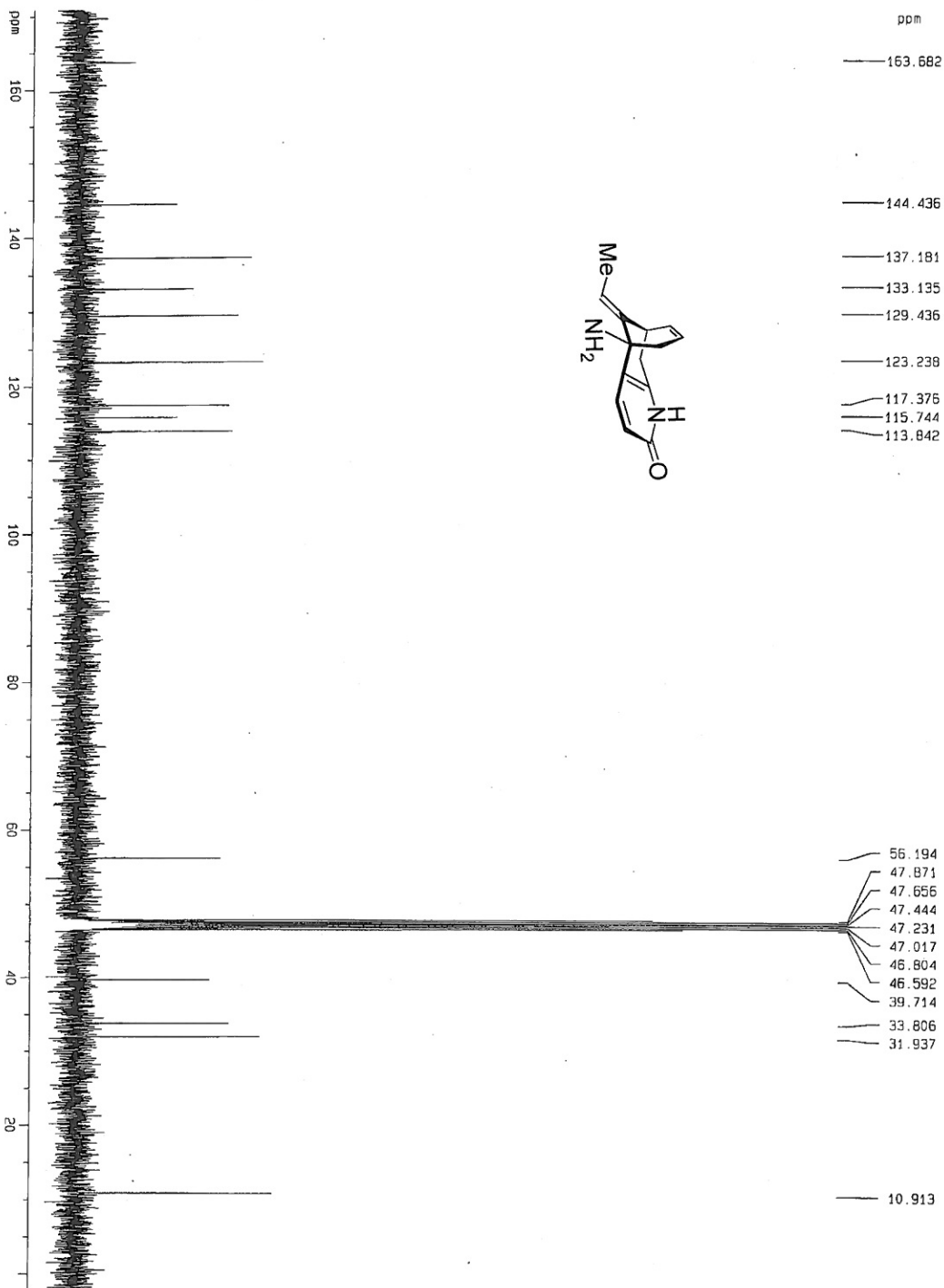
\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1: 1H  
 P1: 12.00 usec  
 PL1: 0.00 dB  
 SF01: 400.1324710 MHz

F2 - Processing parameters  
 SI: 262144  
 SF: 400.130037 MHz  
 MDW: EM  
 SSB: 0  
 LB: 0.30 Hz  
 GB: 0  
 PC: 1.00

1D NMR plot parameters  
 CX: 28.00 cm  
 CY: 19.00 cm  
 F1P: 8.416 ppm  
 F1: 3367.47 Hz  
 F2P: -0.078 ppm  
 F2: -31.28 Hz  
 PPMCKM: 0.30356 ppm/cm  
 HZCM: 121.38396 Hz/cm

(-)-5 MeOD

Sample:   
 Solvent:   
 1-D Carbon-13 NMR Spectrum   
 with H<sub>2</sub>O-16 (CD<sub>3</sub>)<sub>2</sub> Decoupling   
 Bruker DPX-400 NMR Spectrometer   
 October 14, 2004 / O. Lankin



Current Data Parameters   
 NAME marcus   
 EXPNO 332   
 PROCNO 1

F2 - Acquisition Parameters   
 Date\_ 20110119   
 Time 19 28

INSTRUM spect   
 PROBRD 5 mm QNP 1H/1   
 PULPROG zgpg30   
 TO 65536   
 SOLVENT MeOD   
 NS 1338   
 DS 4

SMH 23980.814 Hz   
 FIDRES 0.365918 Hz   
 AQ 1.3664756 sec   
 R6 2048   
 DM 20.850 usec   
 DE 5.00 usec   
 TE 299.2 K

D1 1.0000000 sec   
 d11 0.0300000 sec   
 DELTA 0.8999998 sec   
 MCREST 0.0000000 sec   
 MCMRK 0.0150000 sec

===== CHANNEL f1 =====   
 NUC1 13C   
 P1 10.00 usec   
 PL1 -3.00 dB   
 SF01 100.6282603 MHz

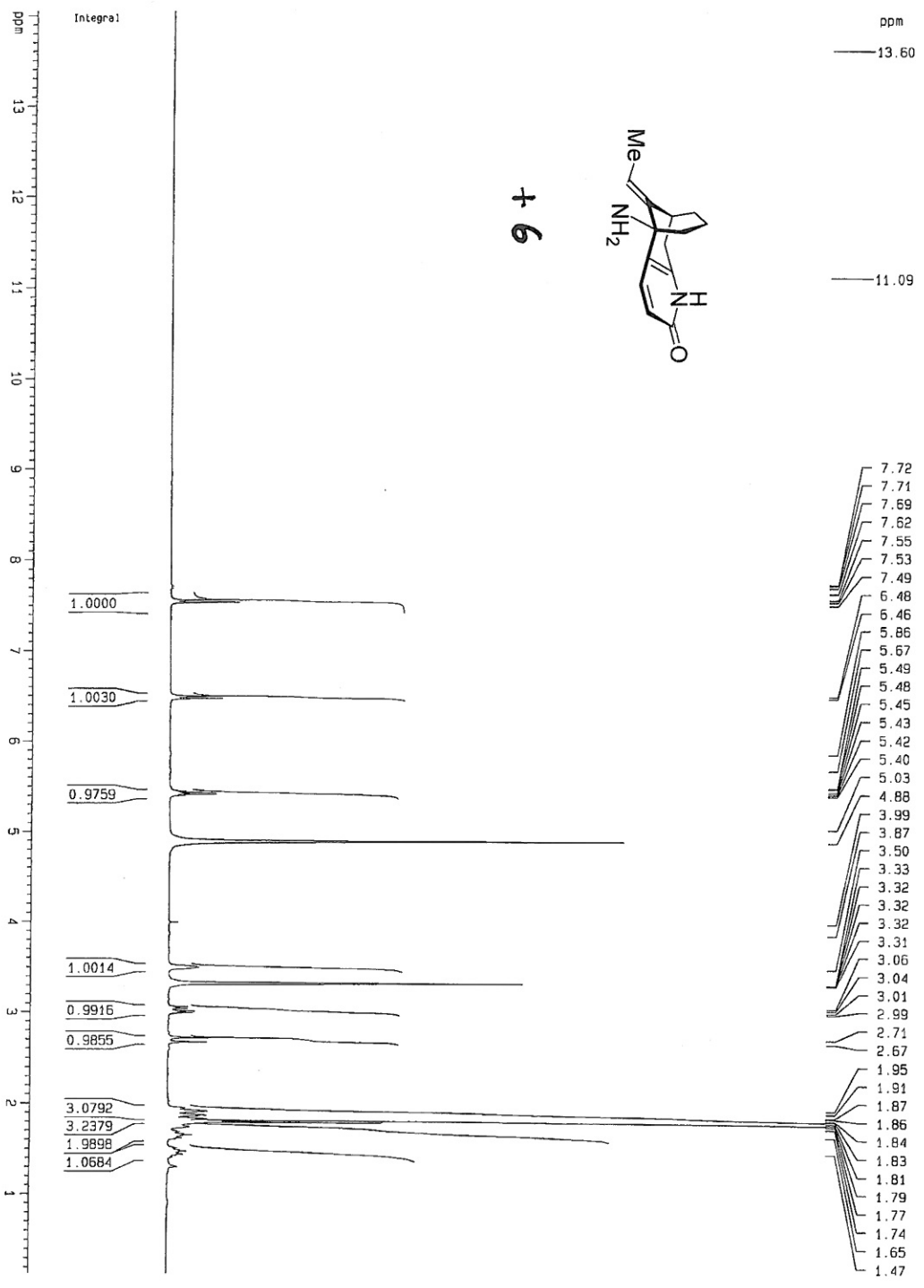
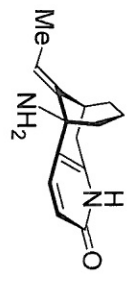
===== CHANNEL f2 =====   
 DPRORG2 waltz16   
 NUC2 1H   
 PCPD2 80.00 usec   
 PL2 0.00 dB   
 PL12 17.00 dB   
 PL13 17.00 dB   
 SF02 400.1316005 MHz

F2 - Processing parameters   
 SI 32768   
 SF 100.6128096 MHz   
 WDM Ek   
 SSS 0   
 LB 1.00 Hz   
 GB 0   
 PC 1.40

1D NMR plot parameters.   
 CX 28.00 cm   
 CY 50.00 cm   
 F1P 170.810 ppm   
 F1 17185.64 Hz   
 F2P -2.138 ppm   
 F2 -215.08 Hz   
 PPRCM 6.17659 ppm/cm   
 HZCM 621.45404 Hz/cm

Sample: ~~MeOD~~  
 Solvent: MeOD  
 Standard 1-D Survey Proton NMR Experiment  
 Bruker DPX-400 NMR Spectrometer  
 October 14, 2004 / D. Lankin

(+) 6  
 MeOD



Current Data Parameters  
 NAME Marcus  
 EXPNO 101  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20101118  
 Time 18.57  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H/1  
 PULPROG zg30  
 TD 65536  
 SOLVENT MeOD  
 NS 16  
 DS 2  
 SWH 8278.146 Hz  
 FIDRES 0.126314 Hz  
 AQ 3.9584243 sec  
 RG 456.1  
 DN 60.400 usec  
 DE 6.00 usec  
 TE 298.2 K  
 D1 1.00000000 sec  
 MCREST 0.00000000 sec  
 MCMR 0.01500000 sec

\*\*\*\*\* CHANNEL f1 \*\*\*\*\*  
 NUC1 1H  
 P1 12.00 usec  
 PL1 0.00 dB  
 SF01 400.1324710 MHz

F2 - Processing parameters  
 SI 262144  
 SF 400.1300037 MHz  
 KW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

10 NMR plot parameters  
 CX 28.00 cm  
 CY 10.00 cm  
 F1P 14.051 ppm  
 F1 5622.08 Hz  
 F2P 0.132 ppm  
 F2 52.84 Hz  
 PPMCM 0.49705 ppm/cm  
 HZCM 198.90144 Hz/cm

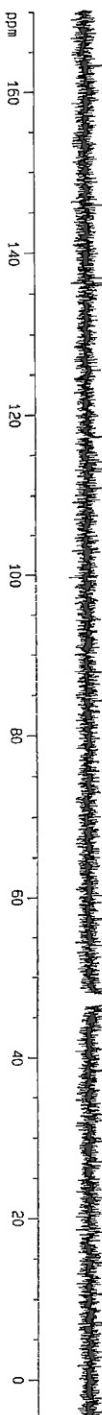
Sample: ~~1605~~  
 Solvent: ~~MeOD~~  
 1-D Carbon-13 NMR Spectrum  
 with Hantz-15 (C90) Decoupling  
 Bruker DPX-400 NMR Spectrometer  
 October 14, 2004 / D. Lemkin

(+)-6  
 MeOD



CH4  
 MeOD

ppm  
 163.182  
 145.817  
 136.145  
 135.935  
 117.192  
 114.034  
 113.198



57.337  
 47.825  
 47.611  
 47.399  
 47.187  
 46.973  
 46.760  
 46.548  
 38.343  
 32.873  
 32.129  
 29.534  
 18.182  
 10.630

Current Data Parameters  
 NAME: marcus  
 EXNO: 102  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_: 20101118  
 Time: 19.05

INSTRUM: spect  
 PROBHD: 5 mm QNP 1H/1  
 PULPROG: zgpg30  
 TD: 65536  
 SOLVENT: MeOD  
 NS: 1895  
 DS: 4

SWH: 23980.814 Hz  
 FIDRES: 0.365918 Hz  
 AQ: 1.3664756 sec  
 RG: 1625.5  
 DW: 20.850 usec  
 DE: 6.00 usec  
 TE: 298.2 K

D1: 1.00000000 sec  
 d11: 0.03000000 sec  
 DELTA: 0.89999998 sec  
 MCREST: 0.00000000 sec  
 MDNRK: 0.01500000 sec

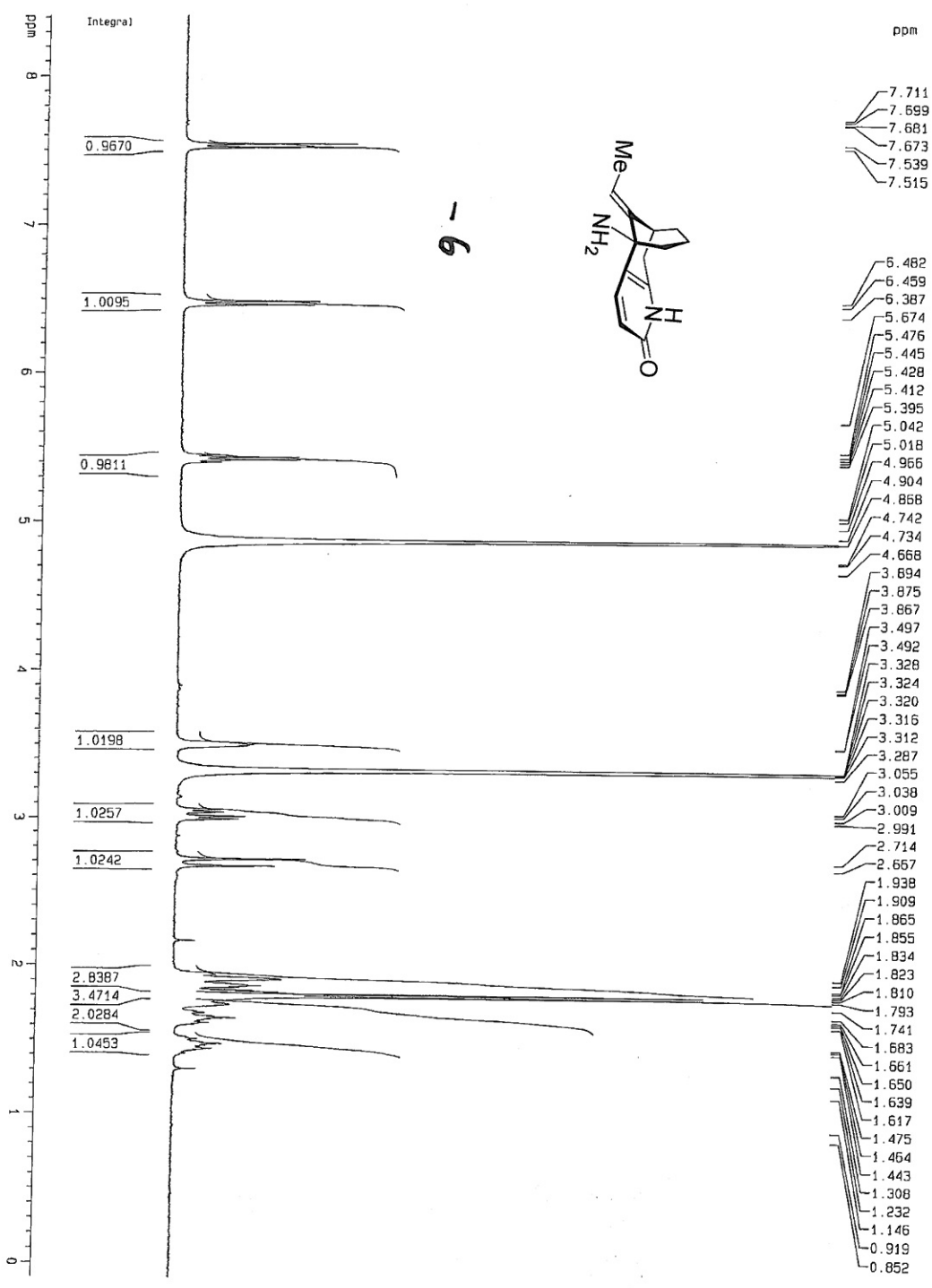
CHANNEL: f1  
 NUC1: 13C  
 P1: 10.00 usec  
 PL1: -3.00 dB  
 SFO1: 100.628303 MHz

CHANNEL: f2  
 CPDPRG2: waltz16  
 NUC2: 1H  
 PCPD2: 80.00 usec  
 PL2: 0.00 dB  
 PL12: 17.00 dB  
 PL13: 17.00 dB  
 SFO2: 400.1316005 MHz

F2 - Processing parameters  
 SI: 32768  
 SF: 100.6128096 MHz  
 MDW: EM  
 SSB: 0  
 LB: 1.00 Hz  
 GB: 0  
 PC: 1.40

10 NMR plot parameters  
 CX: 28.00 cm  
 CY: 100.00 cm  
 F1P: 170.053 ppm  
 F1: 17112.53 Hz  
 F2P: -4.802 ppm  
 F2: -483.15 Hz  
 PPMCM: 6.24590 ppm/cm  
 HZCM: 628.41711 Hz/cm

Sample: ~~121.58442~~ (-)6  
 SOLVENT: MeOD  
 Standard 1-D Survey Proton NMR Experiment  
 Bruker DPX-400 NMR Spectrometer  
 October 14, 2004 / D. Lenkin



Current Data Parameters  
 NAME: marcus  
 EXPNO: 108  
 PROCNO: 1

F2 - Acquisition Parameters  
 Date\_ Time: 20101119 7:00  
 INSTRUM: spect  
 PROBHD: 5 mm QNP 1H/1  
 PULPROG: zg30  
 TO: 65536  
 SOLVENT: MeOD  
 NS: 16  
 DS: 2  
 SWH: 8278.146 Hz  
 FIDRES: 0.126314 Hz  
 AQ: 3.9584243 sec  
 RG: 645.1  
 DW: 60.400 usec  
 DE: 6.00 usec  
 TE: 298.2 K  
 D1: 1.00000000 sec  
 WCOREST: 0.00000000 sec  
 MCKMRK: 0.01500000 sec

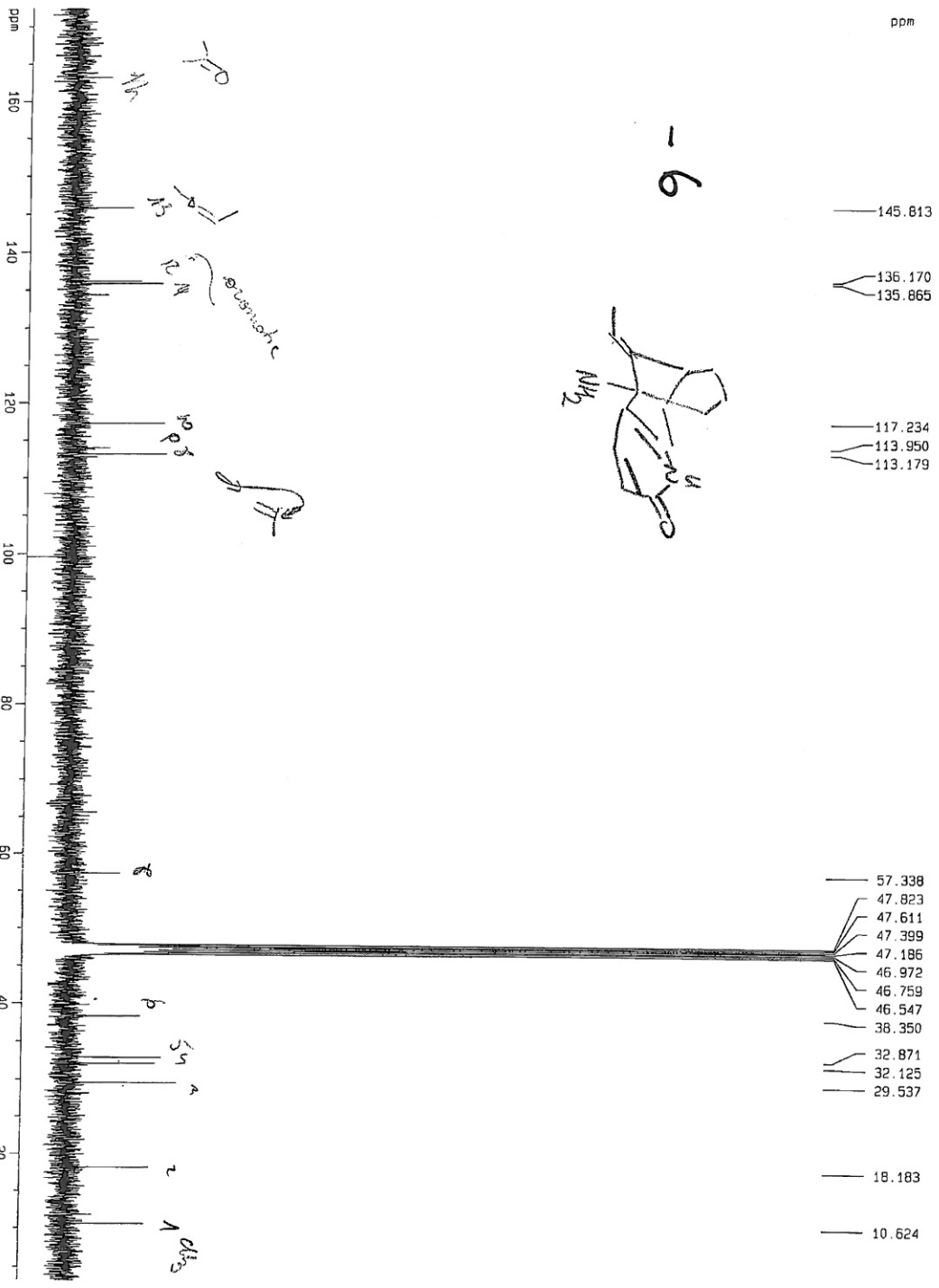
===== CHANNEL f1 =====  
 NUC1: 1H  
 P1: 12.00 usec  
 PL1: 0.00 dB  
 SFO1: 400.1324710 MHz

F2 - Processing parameters  
 SI: 262144  
 SF: 400.130037 MHz  
 WDW: EM  
 SSB: 0  
 LB: 0.30 Hz  
 GB: 0  
 PC: 1.00

1D NMR Plot parameters  
 CX: 28.00 cm  
 CY: 50.00 cm  
 F1P: 8.416 ppm  
 F1: 3357.47 Hz  
 F2P: -0.099 ppm  
 F2: -39.70 Hz  
 PPMCM: 0.30411 ppm/cm  
 HZCM: 121.58442 Hz/cm

Sample: ~~111111~~  
 Solvent: MeOD  
 1-D Carbon-13 NMR Spectrum  
 with Waltz-16 (CD) Decoupling  
 Bruker DPX-400 NMR Spectrometer  
 October 14, 2004 / D. Linkin

**p(-)6**  
**MeOD**



- 145.813
- 136.170
- 135.865
- 117.234
- 113.950
- 113.179
- 57.338
- 47.823
- 47.611
- 47.399
- 47.186
- 46.972
- 46.759
- 46.547
- 38.350
- 32.871
- 32.125
- 29.537
- 18.183
- 10.624

```

Current Data Parameters
NAME          Marcus
EXPNO        125
PROCNO       1

F2 - Acquisition Parameters
Date_        20101119
Time         8.36
INSTRUM     spect
PROBHD      5 mm DNP 1H/1
PULPROG     zgpg30
TD           65536
SOLVENT     MeOD
NS           3125
DS           4
SMH          23980.814 Hz
FIDRES       0.365918 Hz
AQ           1.3664756 sec
RG           3251
DM           20.850 usec
DE           6.00 usec
TE           299.2 K
D1           1.00000000 sec
d11          0.03000000 sec
DELTA        0.89999988 sec
MCREST       0.00000000 sec
MCNMR        0.01500000 sec

===== CHANNEL f1 =====
NUC1          13C
P1           10.00 usec
PL1          -3.00 dB
SFO1         100.6228303 MHz

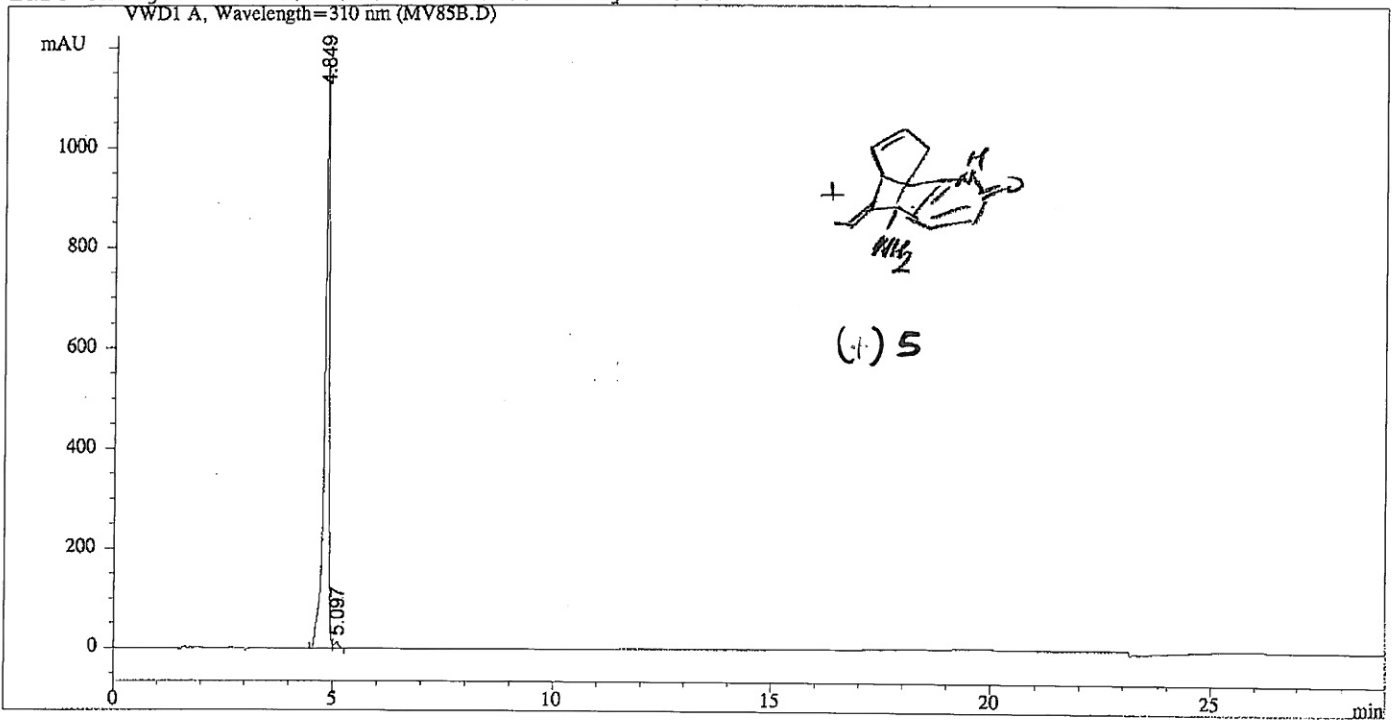
===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           0.00 dB
PL12         17.00 dB
PL13         17.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI           32768
SF           100.6128056 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40

1D NMR plot parameters
CX           28.00 cm
CY           150.00 cm
F1P          172.505 DPM
F1           17356.23 Hz
F2P          3.191 DPM
F2           321.08 Hz
PPMCH        6.04653 ppm/cm
HZCM         608.39825 Hz/cm
  
```

```

=====
Injection Date : 1/20/2011 8:05:11 PM      Seq. Line : 8
Sample Name    : m-v-85                Location  : Vial 72
Acq. Operator  : Andrea                    Inj      : 1
                                                Inj Volume : 5 µl
                                                Actual Inj Volume : 15 µl
Different Inj Volume from Sequence !
Method         : C:\HPCHEM\1\METHODS\3AQHUP.M
Last changed   : 1/20/2011 5:16:33 PM by Andrea
    
```



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

```

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

Signal 1: VWD1 A, Wavelength=310 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU*s | Height [mAU] | Area %  |
|--------|---------------|------|-------------|------------|--------------|---------|
| 1      | 4.849         | BV   | 0.0904      | 7734.23242 | 1168.01196   | 98.9103 |
| 2      | 5.097         | VV   | 0.0882      | 85.21005   | 13.23517     | 1.0897  |

Totals : 7819.44247 1181.24714

Results obtained with enhanced integrator!

=====  
 \*\*\* End of Report \*\*\*

UIC  
University of Illinois at Chicago  
Research Resources Center  
Mass Spectrometry Laboratory  
www.rrc.uic.edu

# Mass Spectrometry SINGLE ANALYSIS REPORT

Room 109A SES, M/C 337  
845 W. Taylor St.  
Chicago, IL 60607-7058  
Office: (312) 355-2124  
Art@uic.edu Lab: (312) 996-6849

MSL Log# 024430

Logged in 01/13/2011 01:32:24 PM

RRC Account No YG3340

UserName Marco Pieroni

Affiliation UIC

Phone

FAX

Email piero@uic.edu

SampleLabel MP-V-85

ProjectName

Formula C<sub>14</sub>H<sub>16</sub>N<sub>2</sub>O<sub>1</sub>

Mono-isotopic Mass 228.12626

M.W. 228.29

Instrument Shimadzu LCMSITTOF

Source, deg C 220

Gas Ar

Acc. Volt. 9100

Resolution 10,300

ScanRange 100-1000

Solvent MeOH

Matrix

Ref. dG

Advisor Gaysin  
Department Medicinal Chemistry and

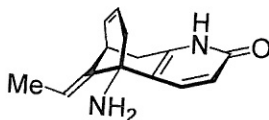
| Analysis Requested |               |
|--------------------|---------------|
| Analysis Type      | Accurate Mass |
| Resolution         | HR            |
| LR spect attach?   | No            |
| Chromatography     | None          |
| Ionization         | ESI           |
| Polarity           | Positive      |
| m/z Range          | 150 -2000     |
| OtherAnalysis      | None          |

Operator Comments

| Ion   | Expected  | Observed | ppm Error |
|-------|-----------|----------|-----------|
| M - H | 227.11899 | 227.1190 | 0.06      |

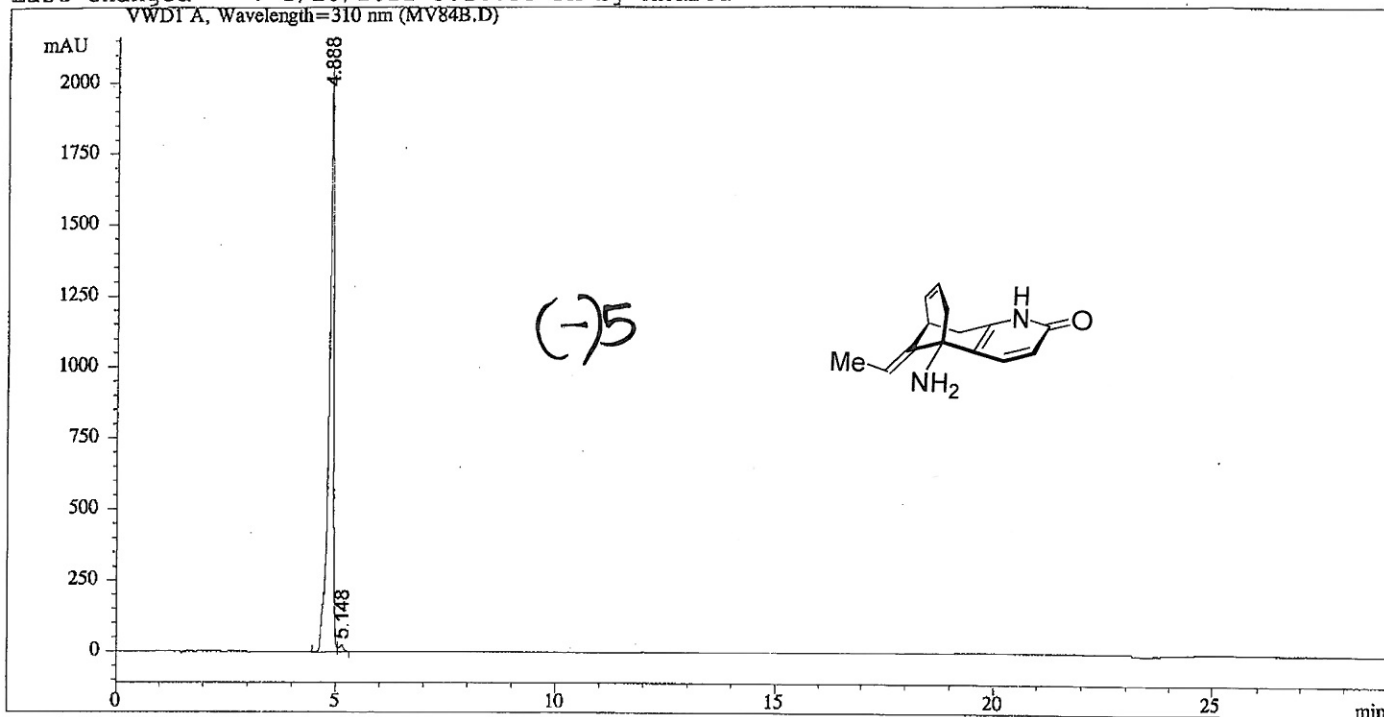
| MSL Log# | Analyst            | Date      | Analysis Performed | Rate    | Quantity | Total   | N/C |
|----------|--------------------|-----------|--------------------|---------|----------|---------|-----|
| 024430   | Bryan M. Zahakaylo | 1/18/2011 | HRESI              | \$35.00 | 1        | \$35.00 |     |

(+) 5



```

=====
Injection Date : 1/20/2011 7:02:05 PM      Seq. Line :    6
Sample Name    : m-v-84                    Location  : Vial 71
Acq. Operator  : Andrea                    Inj      :    1
                                           Inj Volume: 5 µl
                                           Actual Inj Volume: 15 µl
Different Inj Volume from Sequence !
Method         : C:\HPCHEM\1\METHODS\3AQHUP.M
Last changed   : 1/20/2011 5:16:33 PM by Andrea
    
```



Area Percent Report

```

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

Signal 1: VWD1 A, Wavelength=310 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU *s | Height [mAU] | Area %  |
|--------|---------------|------|-------------|-------------|--------------|---------|
| 1      | 4.888         | VV   | 0.0949      | 1.41962e4   | 2062.13892   | 98.7996 |
| 2      | 5.148         | VV   | 0.0955      | 172.47617   | 24.41029     | 1.2004  |

Totals : 1.43687e4 2086.54921

Results obtained with enhanced integrator!

\*\*\* End of Report \*\*\*

UIC  
University of Illinois at Chicago  
Research Resources Center  
Mass Spectrometry Laboratory  
www.rrc.uic.edu

# Mass Spectrometry SINGLE ANALYSIS REPORT

Room 109A SES, M/C 337  
845 W. Taylor St.  
Chicago, IL 60607-7058  
Office: (312) 355-2124  
Art@uic.edu Lab: (312) 996-6849

MSL Log# 024429

Logged in 01/13/2011 01:31:16 PM

RRC Account No YG3340

UserName Marco Pieroni

Affiliation UIC

Phone

FAX

Email piero@uic.edu

SampleLabel MP-V-86

ProjectName

Formula C14 H16 N2 O1

Mono-isotopic Mass 228.12626

M.W. 228.29

Instrument Shimadzu LCMSITTOF

Source, deg C 220

Gas Ar

Acc. Volt. 9100

Resolution 10,300

ScanRange 100-1000

Solvent MeOH

Matrix

Ref. dG

Advisor Gaysin

Department Medicinal Chemistry and

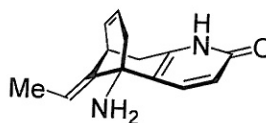
| Analysis Requested |               |
|--------------------|---------------|
| Analysis Type      | Accurate Mass |
| Resolution         | HR            |
| LR spect attach?   | No            |
| Chromatography     | None          |
| Ionization         | ESI           |
| Polarity           | Positive      |
| m/z Range          | 150 -2000     |
| OtherAnalysis      | None          |

Operator Comments

| Ion   | Expected  | Observed | ppm Error |
|-------|-----------|----------|-----------|
| M - H | 227.11899 | 227.1201 | 4.90      |

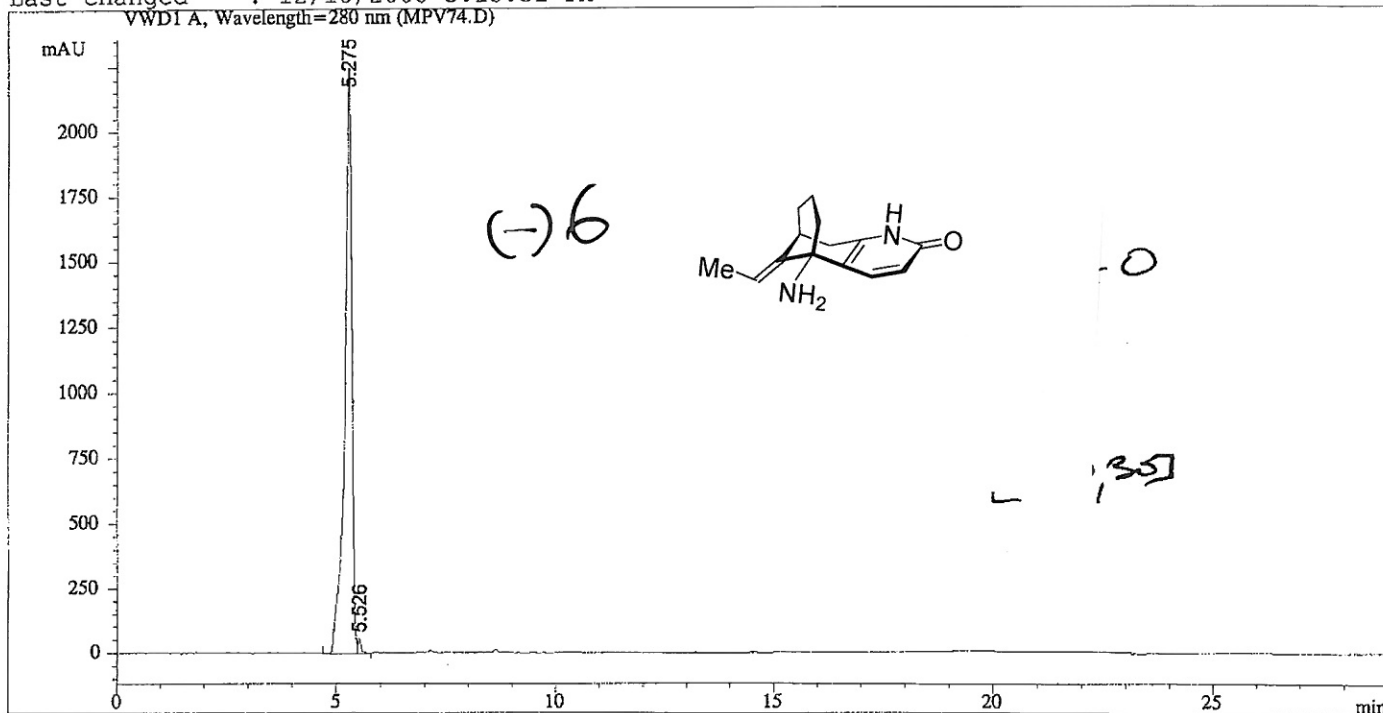
| MSL Log# | Analyst            | Date      | Analysis Performed | Rate    | Quantity | Total   | N/C |
|----------|--------------------|-----------|--------------------|---------|----------|---------|-----|
| 024429   | Bryan M. Zahakaylo | 1/18/2011 | HRESI              | \$35.00 | 1        | \$35.00 |     |

~~Me~~  
(-)-5



```

=====
Injection Date : 10/22/2010 1:36:15 PM      Seq. Line : 1
Sample Name    : mpv74                      Location  : Vial 21
Acq. Operator  : Andrea                    Inj      : 1
                                           Inj Volume: 5 µl
                                           Actual Inj Volume: 20 µl
Different Inj Volume from Sequence !
Method         : C:\HPCHEM\1\METHODS\3AQ280.M
Last changed  : 12/18/2008 5:15:31 PM
    
```



Area Percent Report

```

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

Signal 1: VWD1 A, Wavelength=280 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU *s | Height [mAU] | Area %  |
|--------|---------------|------|-------------|-------------|--------------|---------|
| 1      | 5.275         | VV   | 0.1569      | 2.49399e4   | 2250.98779   | 98.7195 |
| 2      | 5.526         | VV   | 0.0768      | 323.48666   | 59.97657     | 1.2805  |

Totals : 2.52634e4 2310.96436

Results obtained with enhanced integrator!

\*\*\* End of Report \*\*\*

# Mass Spectrometry SINGLE ANALYSIS REPORT

MSL Log# 024428

Logged in 01/13/2011 01:30:26 PM

RRC Account No YG3340

UserName Marco Pieroni

Affiliation UIC

Phone

FAX

Email piero@uic.edu

SampleLabel MP-V-74

ProjectName

Formula C14 H18 N2 O1

Mono-isotopic Mass 230.14191

M.W. 230.31

Instrument Shimadzu LCMSITTOF

Source, deg C 220

Gas Ar

Acc. Volt. 9100

Resolution 10,300

ScanRange 100-1000

Solvent MeOH

Matrix

Ref. dG

Advisor Gaysin

Department Medicinal Chemistry and

### Analysis Requested

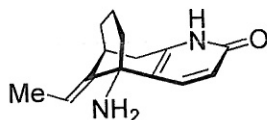
| Analysis Type    | Accurate Mass |
|------------------|---------------|
| Resolution       | HR            |
| LR spect attach? | No            |
| Chromatography   | None          |
| Ionization       | ESI           |
| Polarity         | Positive      |
| m/z Range        | 150 -2000     |
| OtherAnalysis    | None          |

### Operator Comments

| Ion   | Expected  | Observed | ppm Error |
|-------|-----------|----------|-----------|
| M + H | 231.14919 | 231.1495 | 1.34      |

| MSL Log# | Analyst            | Date      | Analysis Performed | Rate    | Quantity | Total   | N/C |
|----------|--------------------|-----------|--------------------|---------|----------|---------|-----|
| 024428   | Bryan M. Zahakaylo | 1/18/2011 | HRESI              | \$35.00 | 1        | \$35.00 |     |

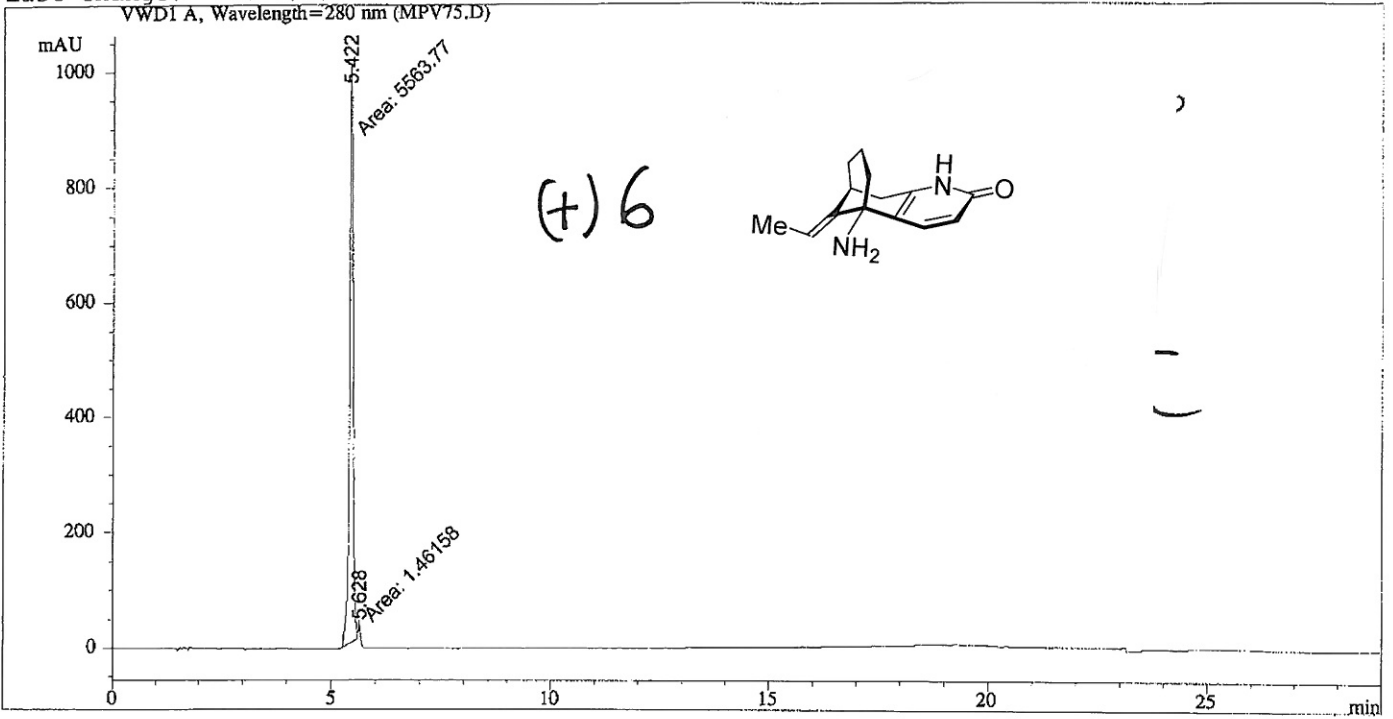
(-) 6



```

=====
Injection Date : 11/16/2010 11:17:21 AM      Seq. Line : 2
Sample Name    : mpv75                        Location  : Vial 2
Acq. Operator  : Andrea                       Inj       : 1
                                           Inj Volume: 5 µl
                                           Actual Inj Volume: 10 µl
Different Inj Volume from Sequence !
Acq. Method    : C:\HPCHEM\1\METHODS\3AQ280.M
Last changed   : 12/18/2008 5:15:31 PM
Analysis Method: C:\HPCHEM\1\METHODS\1AQ100MM.M
Last changed   : 8/8/2008 12:31:58 PM
=====

```



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

```

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

```

Signal 1: VWD1 A, Wavelength=280 nm

| Peak # | RetTime [min] | Type | Width [min] | Area mAU *s | Height [mAU] | Area %  |
|--------|---------------|------|-------------|-------------|--------------|---------|
| 1      | 5.422         | MM   | 0.0923      | 5563.77197  | 1004.79272   | 99.9737 |
| 2      | 5.628         | MM   | 0.0113      | 1.46158     | 2.15683      | 0.0263  |

Totals : 5565.23355 1006.94955

Results obtained with enhanced integrator!

=====  
\*\*\* End of Report \*\*\*

UIC  
 University of Illinois at Chicago  
 Research Resources Center  
 Mass Spectrometry Laboratory  
 www.rrc.uic.edu

# Mass Spectrometry SINGLE ANALYSIS REPORT

Room 109A SES, M/C 337  
 845 W. Taylor St.  
 Chicago, IL 60607-7058  
 Office: (312) 355-2124  
 Art@uic.edu Lab: (312) 996-6849

MSL Log# 024427

Logged in 01/13/2011 01:28:33 PM

RRC Account No YG3340  
 UserName Marco Pieroni  
 Affiliation UIC  
 Phone  
 Email piero@uic.edu  
 SampleLabel MP-V-75  
 ProjectName  
 Formula C14 H18 N2 O1  
 Mono-isotopic Mass 230.14191

FAX

M.W. 230.31

Advisor Gaysin  
 Department Medicinal Chemistry and

| Analysis Requested |               |
|--------------------|---------------|
| Analysis Type      | Accurate Mass |
| Resolution         | HR            |
| LR spect attach?   | No            |
| Chromatography     | None          |
| Ionization         | ESI           |
| Polarity           | Positive      |
| m/z Range          | 150 -2000     |
| OtherAnalysis      | None          |

Instrument Shimadzu LCMSITTOF  
 Source, deg C 220 Gas Ar  
 Acc. Volt. 9100 Resolution 10,300  
 ScanRange 100-1000  
 Solvent MeOH  
 Matrix  
 Ref. dG

Operator Comments

| Ion   | Expected  | Observed | ppm Error |
|-------|-----------|----------|-----------|
| M + H | 231.14919 | 231.1495 | 1.34      |

| MSL Log# | Analyst            | Date      | Analysis Performed | Rate    | Quantity | Total   | N/C |
|----------|--------------------|-----------|--------------------|---------|----------|---------|-----|
| 024427   | Bryan M. Zahakaylo | 1/18/2011 | HRESI              | \$35.00 | 1        | \$35.00 |     |

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