Supplementary Information

Convenient syntheses of halo-dibenz[*b,f*]azepines and carbamazepine analogues via *N*-arylindoles

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1. Experimental details and characterisation

For general procedures, see the main MS.

_N_-Phenyl-1H-indole 23.[19(b)]

Found: m/z, 194.0693, $C_{14}H_{12}N$ (MH^+) requires m/z, 194.0964; δ_H 6.62 (1 H, dd, J = 3.3 and 0.7 Hz, 3-H), 7.11-7.19 (2 H, m, 5-H + 6-H), 7.20-7.25 (2 H, m, 2-H + 4′-H), 7.34-7.40 (4 H, m, 2′-H + 3′-H + 5′-H + 6′-H), 7.52 (1 H, m, 4-H) and 7.64-7.67 (1 H, m, 7-H); δ_C 103.9, 110.8, 120.7, 121.4, 122.6, 124.5, 126.6, 128.2, 129.6, 129.8, 136.1 and 140.0.

_N_-(4-Methoxy)phenylindole 31.

Mp. 71-72° C. Found: C, 80.5; H, 5.9; N, 6.2; m/z 224.0173. $C_{15}H_{13}NO$ requires C, 80.7; H, 5.9; N, 6.3%; $C_{15}H_{14}NO$ (MH^+) requires m/z, 224.1070; δ_H 3.86 (3 H, s, CH_3O), 6.60 (1 H, dd, J = 3.2 and 0.6 Hz, 3-H), 7.01 and 7.38 (4 H, 2m, 2′-H + 3′-H + 5′-H + 6′-H), 7.12-7.25 (2 H, m, 5-H + 6-H), 7.27 (1 H, d, J = 3.2 Hz, 2-H), 7.45 (1 H, d, J= 8.4 Hz, 7-H) and 7.68 (1 H, d, J = 7 Hz, 4-H); δ_C 56.0, 103.3, 110.8, 115.2, 120.5, 121.4, 122.6, 126.4, 128.7, 129.4, 133.3, 136.8 and 158.7.

5-Methoxy-_N_-phenylindole 32.

Light red oil. Found: m/z, 224.1076. $C_{15}H_{14}NO$ (MH^+) requires m/z, 224.1070; δ_H 3.86 (3 H, s, CH_3O), 6.60 (1 H, dd, J = 3.2 and 0.8 Hz, 3-H), 6.87 (1 H, dd, J = 9.0 and 2.5 Hz, 6-H), 7.13 (1 H, d, J = 2.4 Hz, 4-H), 7.31 (1 H, d, J = 3.3 Hz, 2-H), 7.30-7.35 (1 H, m, ArH of N-Ph), 7.46 (1 H, d, J = 7 Hz, 7-H) and 7.46-7.52 (4 H, m, ArH of N-Ph); δ_C 56.3, 103.1, 103.7, 111.7, 112.9, 124.5, 126.7, 128.8, 130.0, 130.3, 131.5, 140.4 and 155.0.

6-Methoxy-_N_-phenylindole 33.

Light red oil. Found: m/z, 224.1074. $C_{15}H_{14}NO$ (MH^+) requires m/z, 224.1070; δ_H 3.83 (3 H, s, CH_3O), 6.60 (1 H, d, J = 3.2 Hz, 3-H), 6.84 (1 H, dd, J = 8.6 and 2.2 Hz, 5-H), 7.04 (1 H, d, J = 2.1 Hz, 7-H), 7.23 (1 H, d, J = 3.2 Hz, 2-H), 7.32-7.42 (2 H, m, ArH of N-Ph), 7.45-7.53 (3 H, m, ArH of N-Ph) and 7.55 (1 H, d, J = 8.6 Hz, 4-H); δ_C 56.2, 94.6, 103.8, 110.6, 122.0, 123.9, 124.7, 126.8, 127.4, 130.0, 137.0, 140.3 and 157.2.

***N*-(4-Chloro)phenylindole 35.**

Off-white solid. Found: C, 73.34; H, 4.4; N, 6.1; m/z, 228.0572. $C_{14}H_{10}ClN$ requires C, 73.85; H, 4.4; N, 6.15%; $C_{14}H_{11}ClN$ (MH^+) requires m/z, 228.0575; δ_H 6.69 (1 H, d, J = 3.2 Hz, 3-H), 7.16-7.26 (2 H, m, 5-H + 6-H), 7.29 (1 H, d, J = 3.2 Hz, 2-H), 7.42-7.49 (4 H, approx. dd, 2′-, 3′-, 5′- and 6′-H), 7.51 (1 H, d, J = 8 Hz, 7-H) and 7.69 (1 H, d, J = 7.6 Hz, 4-H); δ_C 104.5, 110.7, 121.0, 121.7, 123.0, 125.9, 128.1, 129.8, 130.2, 132.4, 136.2 and 138.8.

5-Chloro-*N*-phenylindole 36.

Found: m/z, 228.0573. $C_{14}H_{11}ClN$ (MH^+) requires m/z, 228.0575; δ_H 6.61 (1 H, dd, J = 3.2 and 0.5 Hz, 3-H), 7.16 (1 H, dd, J = 8.8 and 2.0 Hz, 6-H), 7.35 (1 H, d, J = 3.3 Hz, 2-H), 7.37 (1 H, m, ArH), 7.45 (1 H, d J = 8.8 Hz, 7-H), 7.43-7.48 (2 H, m, ArH), 7.49-7.54 (2 H, m, ArH) and 7.64 (1 H, d, J = 2.0 Hz, 4-H); δ_C 103.5, 112.0, 120.9, 123.0, 124.8, 126.4, 127.2, 129.7, 130.1, 130.7, 134.7 and 139.8.

***N*-(4-Bromo)phenylindole 38.**

Found: m/z, 272.0071. $C_{14}H_{11}NBr$ (MH^+ for ^{79}Br) requires m/z, 272.0069; δ_H 6.69 (1 H, dd, J = 3.3 and 0.8 Hz, 3-H), 7.15-7.26 (2 H, m, 5-H + 6-H), 7.23 (1 H, d, J = 3.3 Hz, 2-H), 7.35-7.40 and 7.60-7.65 (4 H, m, 2′-, 3′,- 5′- and 6′-H), 7.52 (1 H, dd, J =

8.4 and 1.0 Hz, 7-H) and 7.68 (1 H, dt, J = 8.4 and 0.7 Hz, 4-H); δ_C 104.6, 110.7, 120.1, 121.1, 121.7, 123.1, 126.2, 128.0, 129.8, 133.2, 136.1 and 139.3.

5-Bromo-*N*-phenylindole 39.

Mp. 39-41° C. Found: C, 61.45; H, 3.7; N, 4.9; m/z, 272.0065. $C_{14}H_{10}BrN$ requires C, 61.8; H, 3.7; N, 5.1%; $C_{14}H_{11}NBr$ (MH^+ for ^{79}Br) requires m/z, 272.0060; δ_H 6.60 (1 H, dd, J = 3.2 and 0.7 Hz, 3-H), 7.28 (1 H, dd, J = 8.8 and 1.9 Hz, 6-H), 7.32 (1 H, d, J = 3.2 Hz, 2-H), 7.40 (1 H, d, J = 8.8 Hz, 7-H), 7.34-7.39 (1 H, m, ArH), 7.43-7.48 (2 H, m, ArH), 7.49-7.55 (2 H, m, ArH) and 7.80 (1 H, d, J = 1.8 Hz, 4-H); δ_C 103.4, 112.4, 113.9, 124.0, 124.8, 125.6, 127.3, 129.5, 130.2, 131.4, 135.0 and 139.8.

5-Bromo-*N*-(4-bromo)phenylindole 40.

Mp. 72-74° C, softened 68-70°C. Found: C, 48.0; H, 2.6; N, 3.9. $C_{14}H_9Br_2N$ requires C, 47.9; H, 2.6; N, 4.0%; δ_H 6.62 (1 H, d, J = 3.3 Hz, 3-H), 7.28 (1 H, d, J = 3.4 Hz, 2-H), 7.30-7.37 (4 H, m, 6-H + 7-H + 3′-H + 5′-H), 7.61-7.67 (2 H, m, 2′-H + 6′-H) and 7.80 (1 H, d, J = 1.8 Hz, 4-H); δ_C 104.0, 112.1, 114.2, 120.7, 124.1, 125.9, 126.2, 129.2, 131.5, 133.3, 134.8 and 138.8.

3-Methoxy-5*H*-dibenz[*b*,*f*]azepine 48.[24]

Found: m/z, 224.1079. $C_{15}H_{14}NO$ requires m/z, 224.1070 (MH^+); δ_H 3.75 (3 H, s, CH_3O), 4.91 (1 H, br s, NH), 6.06 (1 H, d, J = 2.4 Hz, 4-H), 6.17, 6.24 (2 H, ABq, 10-H + 11-H), 6.36 (1 H, dd, J = 8.4 and 2.4 Hz, 2-H), 6.47 (1H, d, J = 7.6 Hz, 6-H), 6.76 (1 H, d, J = 8.4 Hz, 9-H), 6.79-6.85 (2 H, m, 7-H + 8-H) and 7.00 (1 H, ddd, J = 7.8, 6.1 and 2.8 Hz, 1-H); δ_C 55.3, 103.3, 105.6, 107.4, 119.2, 123.0, 129.1, 129.6, 129.9, 130.3, 131.7, 147.5, 149.6, 152.9 and 161.2; m/z (CI) 224 (MH^+, 100%).

2-Chloro-5*H*-dibenz[*b,f*]azepine 49.[9] δ_H 4.91 (1 H, br s, NH), 6.22, 6.35 (2 H, ABq, 10-H + 11-H), 6.43 (1 H, d, *J* = 8.4 Hz, 6-H), 6.49 (1 H, d, *J* = 7.8 Hz, 4-H), 6.83 (1H, d, *J* = 2.1 Hz, 1-H), 6.92 - 6.78 (2 H, m, 7-H + 8-H), 6.98 (1 H, dd, *J* = 8.3 and 2.3 Hz, 3-H) and 7.09 - 6.94 (1 H, m, 4-H); δ_C 119.3, 120.3,123.2, 127.9, 128.9, 129.3, 129.7, 129.9, 130.7, 131.3, 132.1, 133.4, 146.8 and 148.0.

2,8-Dichloro-5*H*-dibenz[*b,f*]azepine 50.[9] δ_H 6.99 (dd, *J*=8.4, 2.4 Hz, 2 H), 6.84 (d, *J*=2.5 Hz, 2 H), 6.42 (d, *J*=8.4 Hz, 2 H), 6.25 (s, 2 H) and 4.89 (br s, 1 H); δ_C146.6, 132.0, 131.0, 130.1, 129.2, 128.3 and 120.4.

2-Bromo-5*H*-dibenz[*b,f*]azepine 51.[9] Mp. 152-154°C; δ 7.09 (dd, *J*=8.3, 2.3 Hz, 1 H), 7.03 (ddd, *J*=7.9, 5.9, 3.1 Hz, 1 H), 6.95 (d, *J*=2.3 Hz, 1 H), 6.82 - 6.86 (m, 2 H), 6.46 (d, *J*=7.6 Hz, 1 H), 6.35 (s, 1 H), 6.30 (d, *J*=12.0 Hz, 1 H), 6.17 (d, *J*=11.6 Hz, 1 H) and 4.89 (br s, 1 H); ^{13}C NMR (101 MHz,$CDCl_3$) δ 148.3, 147.4, 133.4, 132.8, 131.8, 131.7, 130.7, 130.6, 129.8, 129.3, 123.3, 120.7, 119.3 and 115.3.

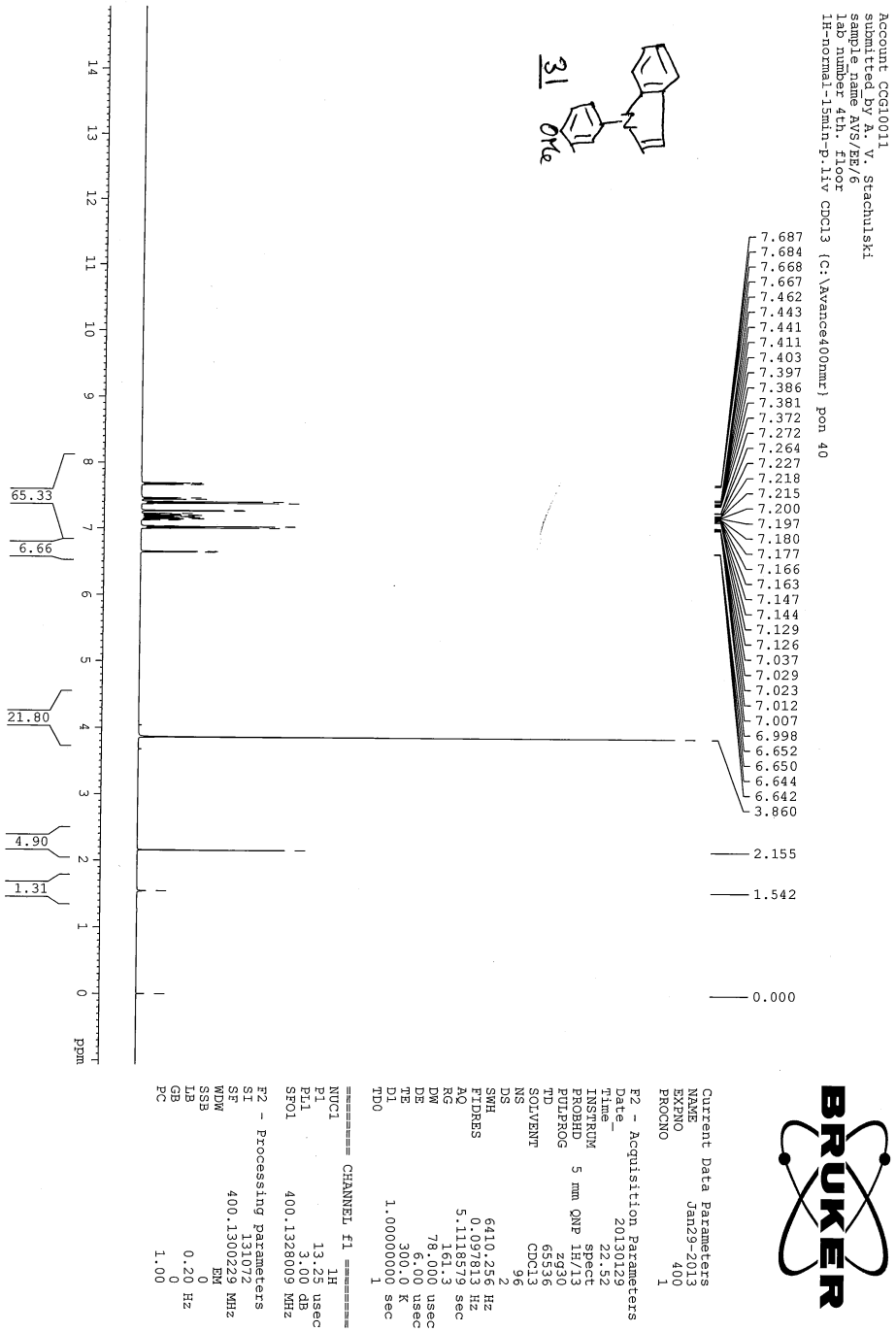
2,8-Dichloro-5*H*-dibenz[*b,f*]azepine-5-carboxamide 55. White solid; δ_H 4.46(2 H, br s, NH_2), 6.89 (2 H, s, 10-H + 11-H), 7.26 (2 H, s), 7.36 (2 H, d, *J*=1.8 Hz) and 7.40 - 7.42 (2 H, d, *J*=2.3 Hz); δ_C 121.7, 130.3, 132.4, 132.7, 136.5, 138.8 and 156.3.

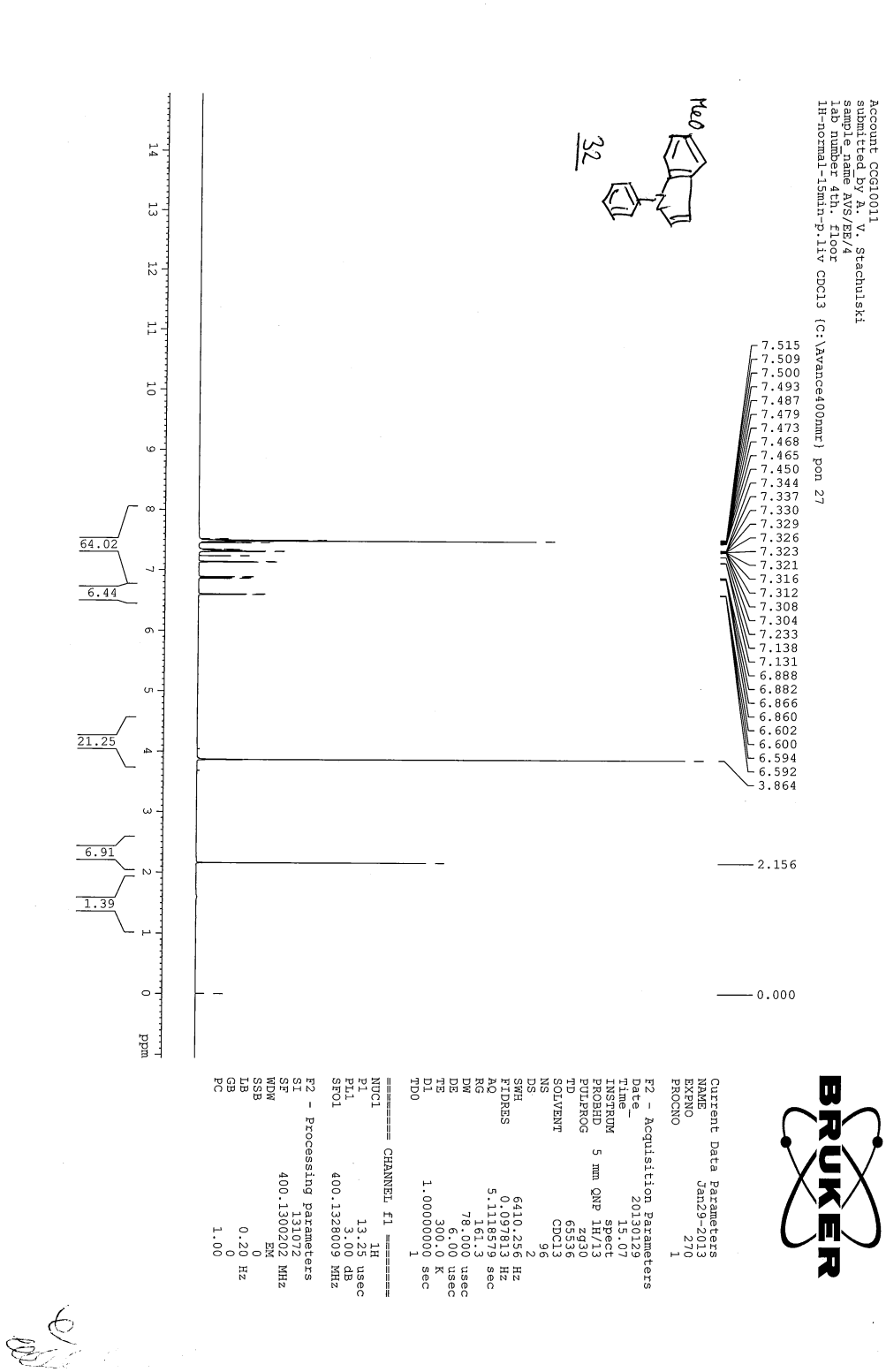
2,8-Dibromo-5*H*-dibenz[*b,f*]azepine-5-carboxamide 56. White solid; δ_H 4.58 (2 H, br s, NH_2), 6.87 (2 H, s, 10-H + 11-H), 7.32 (2 H, d, *J*=8.4 Hz), 7.51 (2 H, d, *J*=2.3 Hz) and 7.55 (2 H, dd, *J*=8.4 and 2.2 Hz, 3-H + 7-H); δ_C122.1, 130.7, 132.8, 133.1, 136.9, 139.2 and 156.7.

2. Photocopy NMR spectra

^{1}H: **31-33**, **35-40**, **47-51**, **55** and **56**.

^{13}C: **36**, **37**, **40**.

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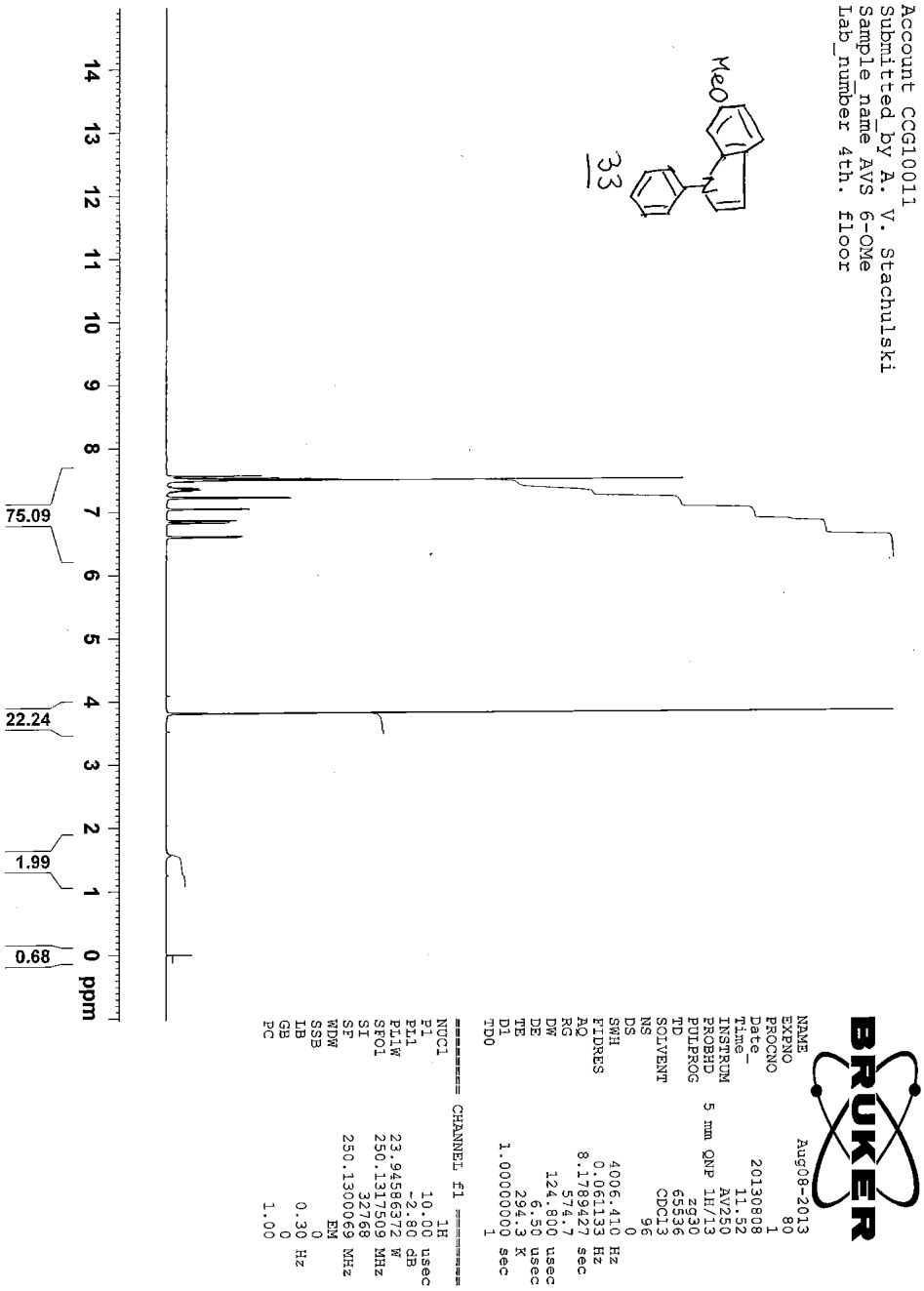
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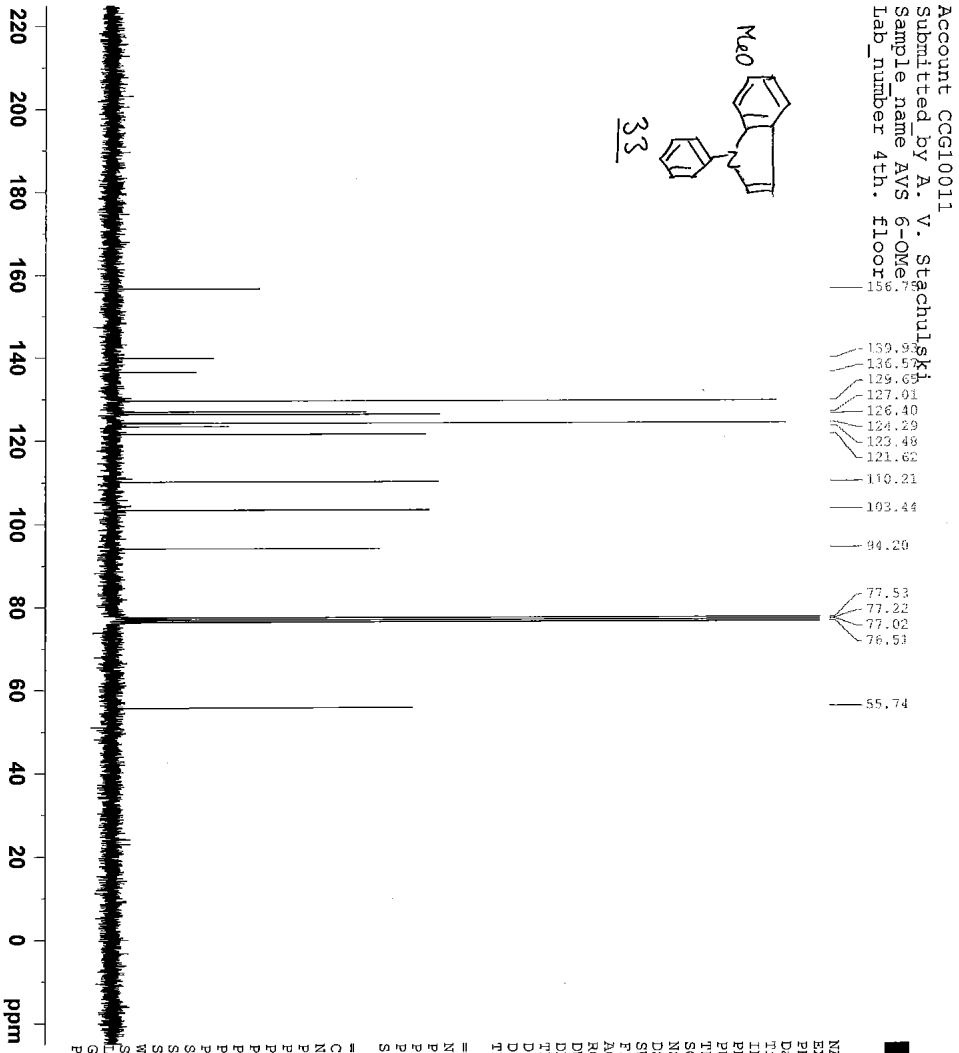

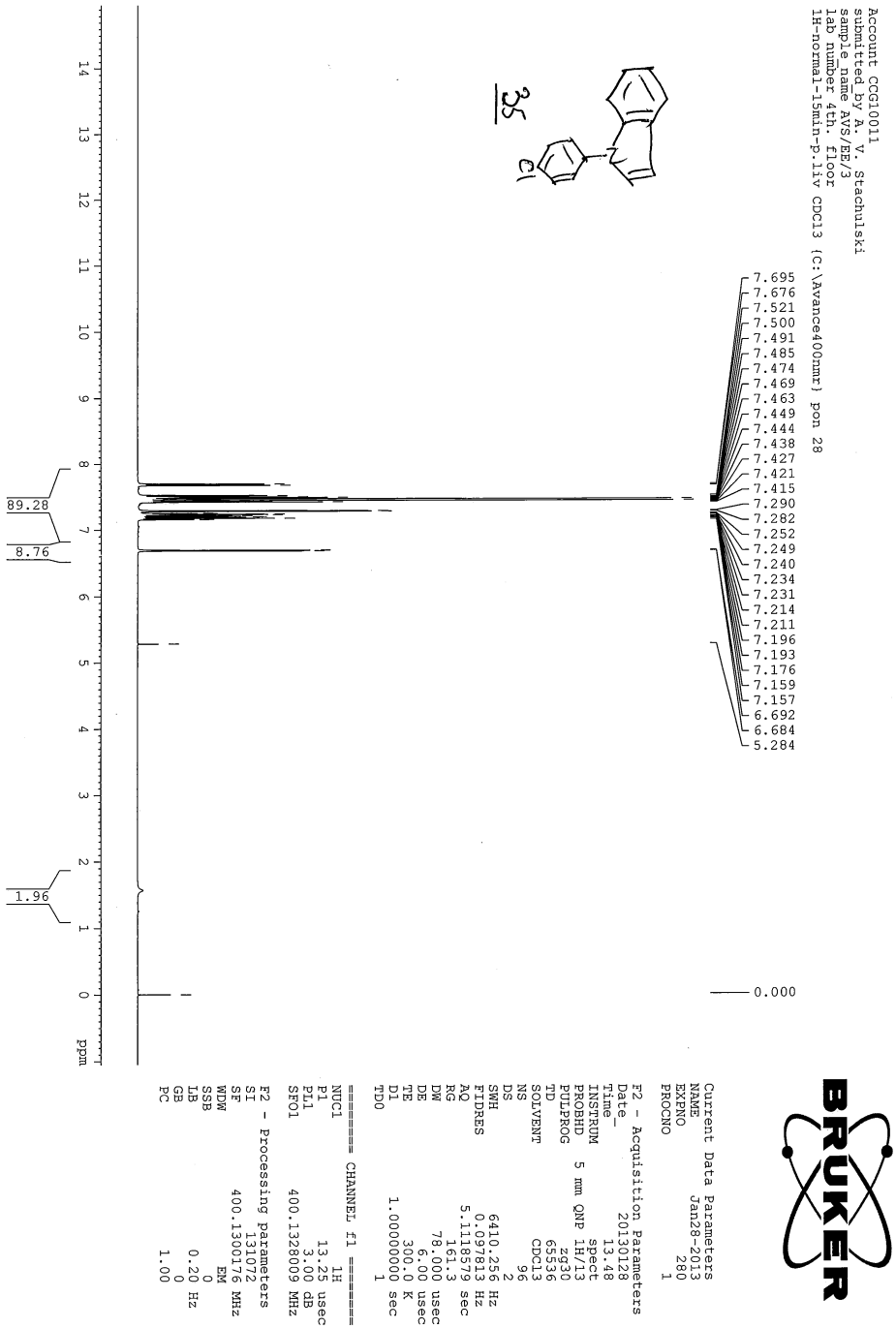
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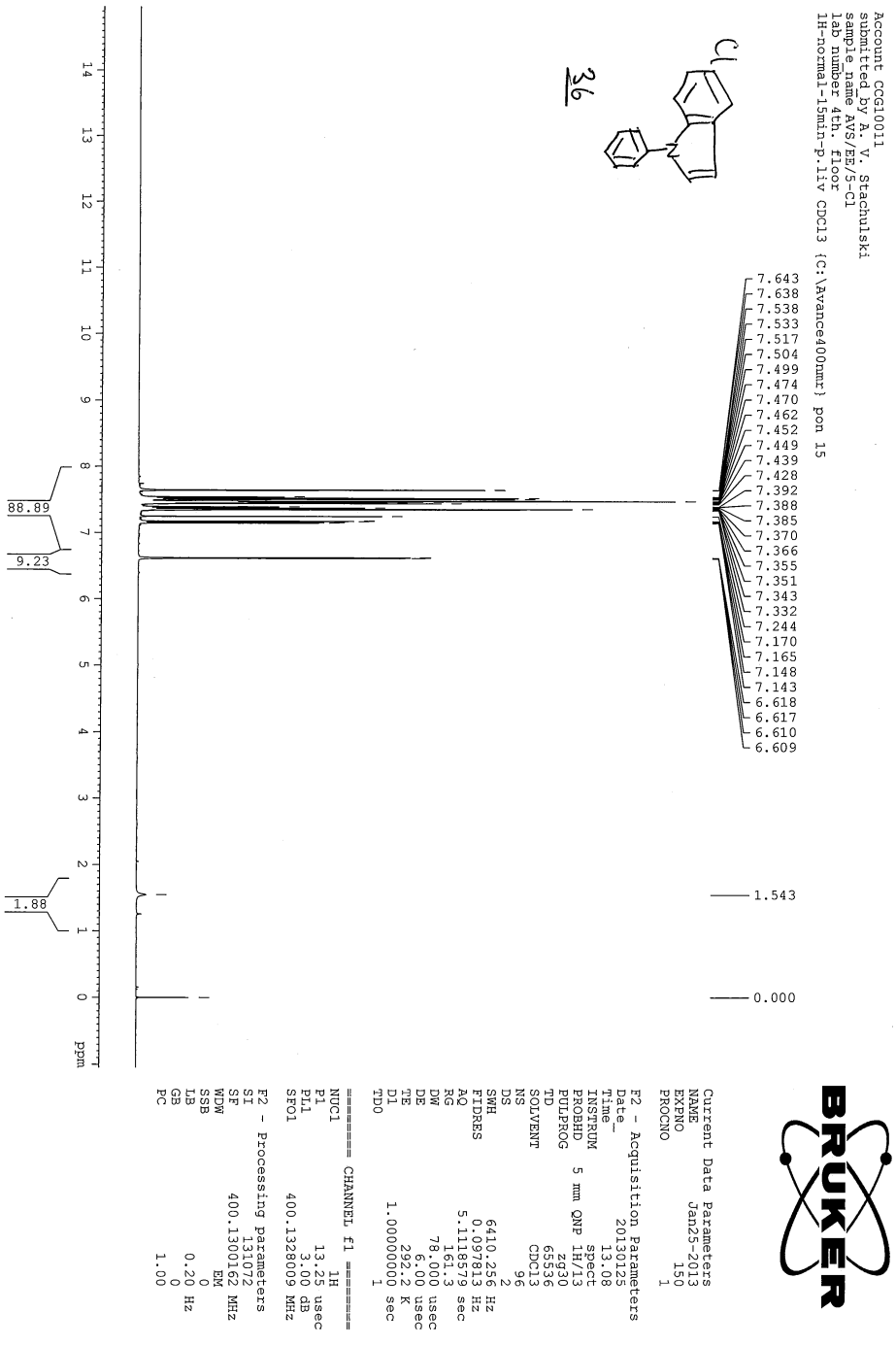
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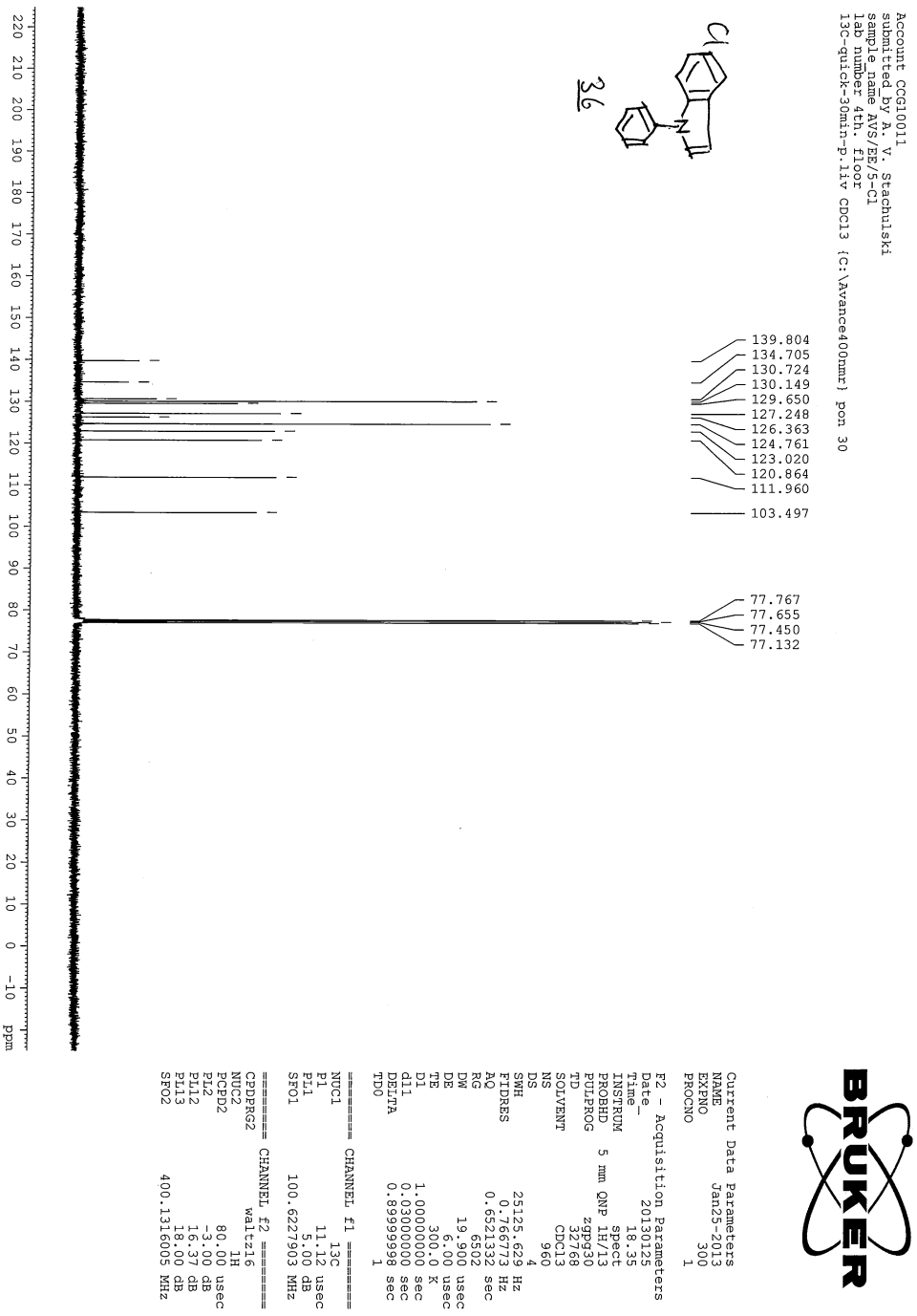
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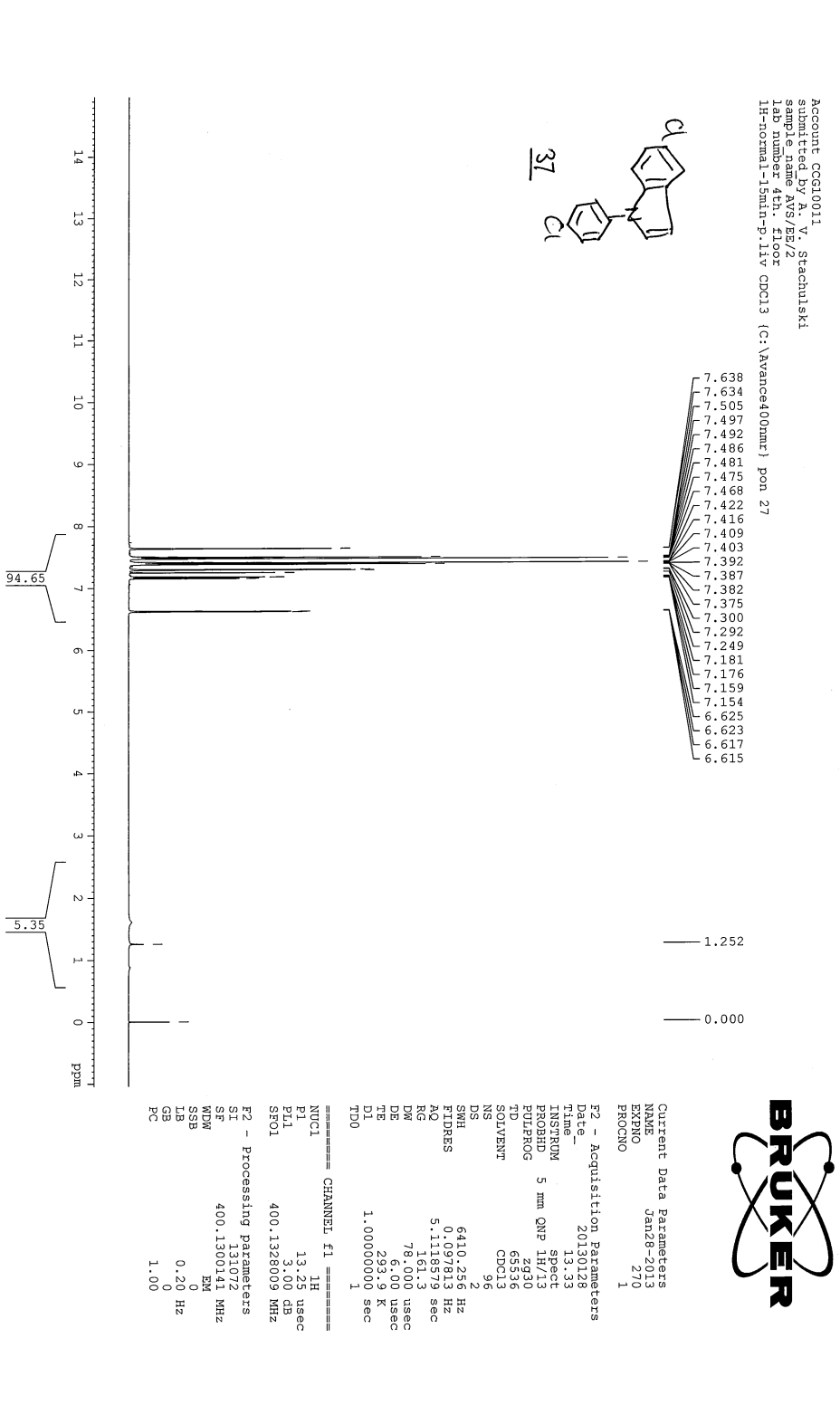
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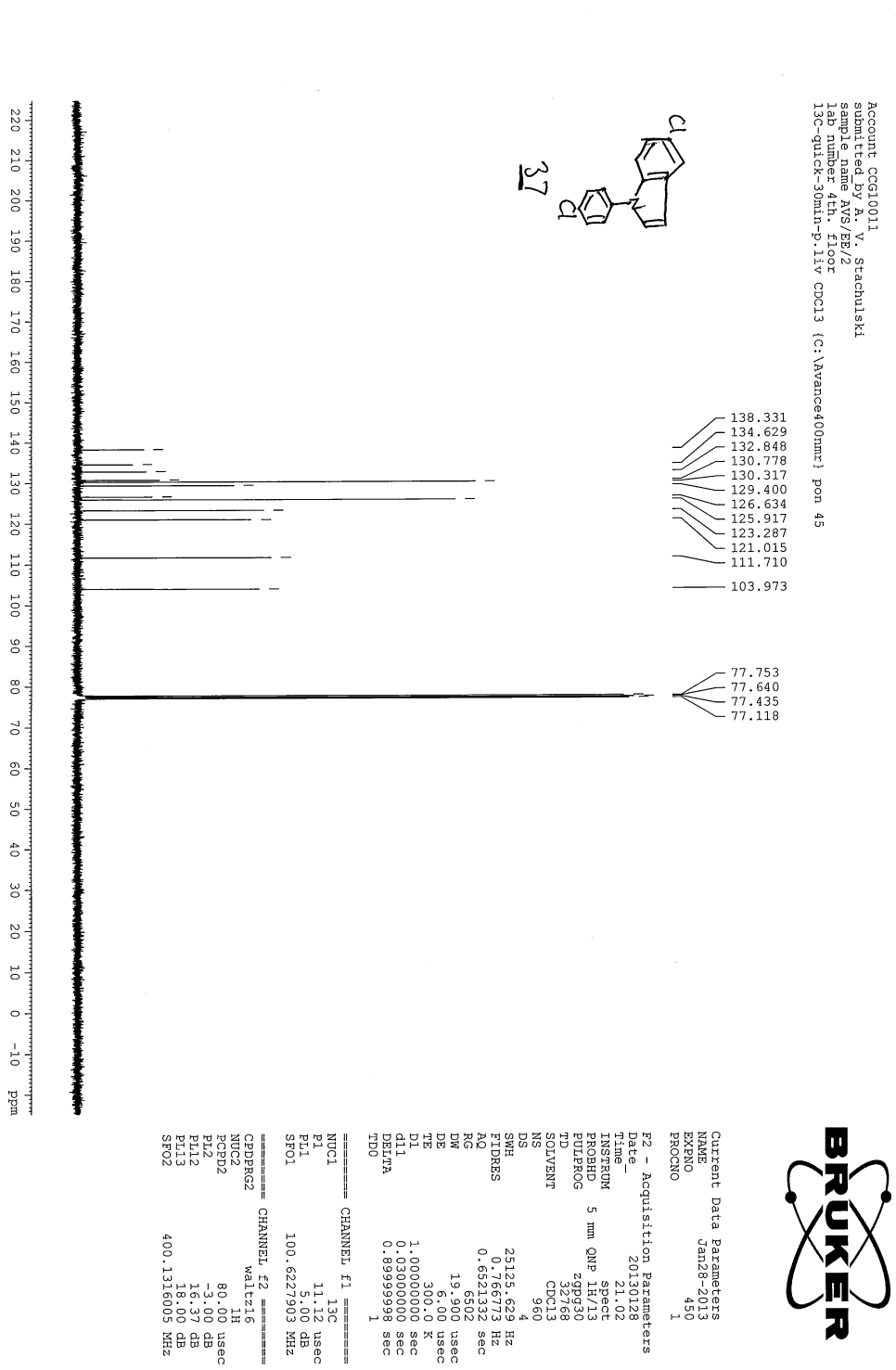
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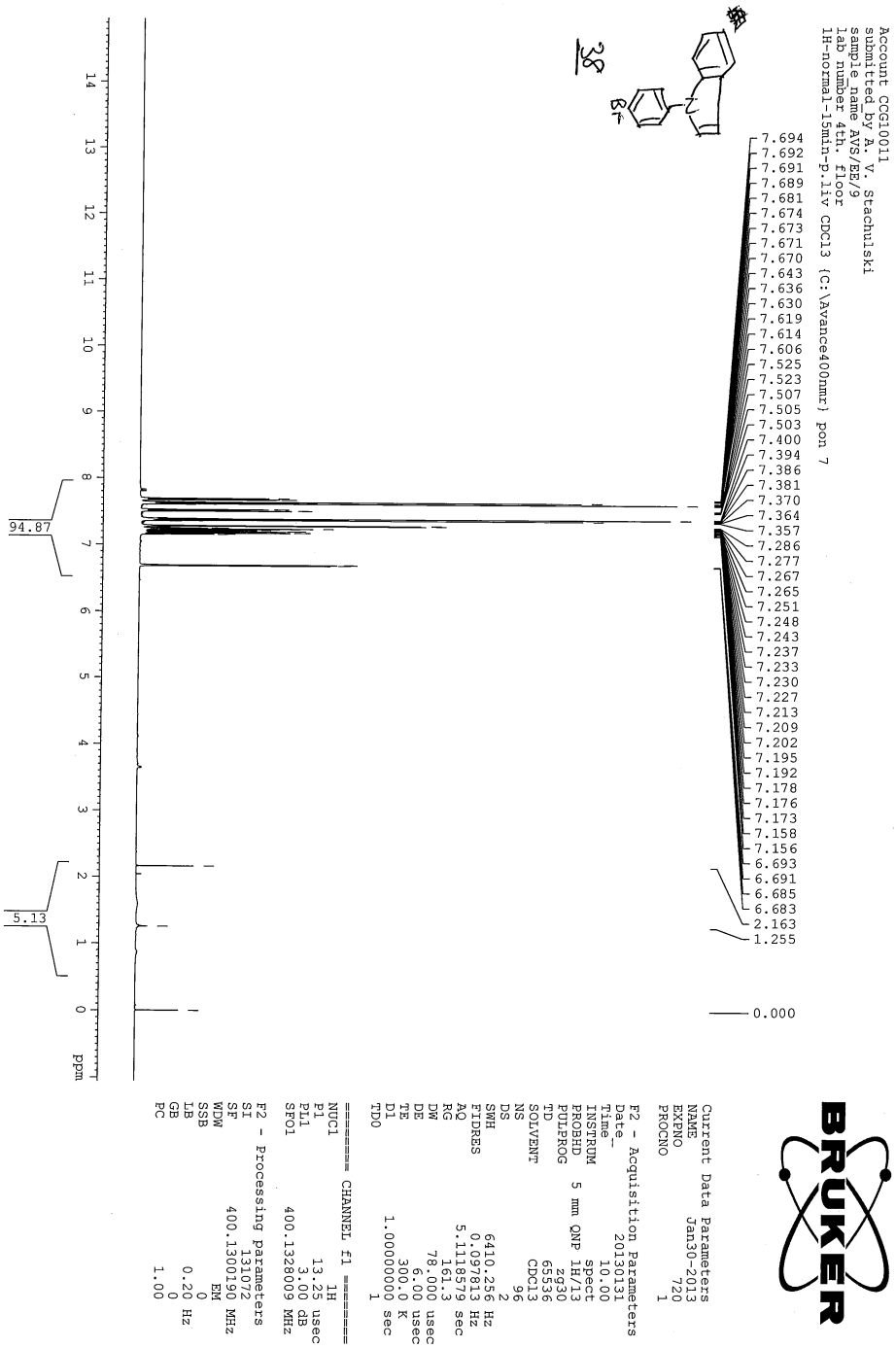
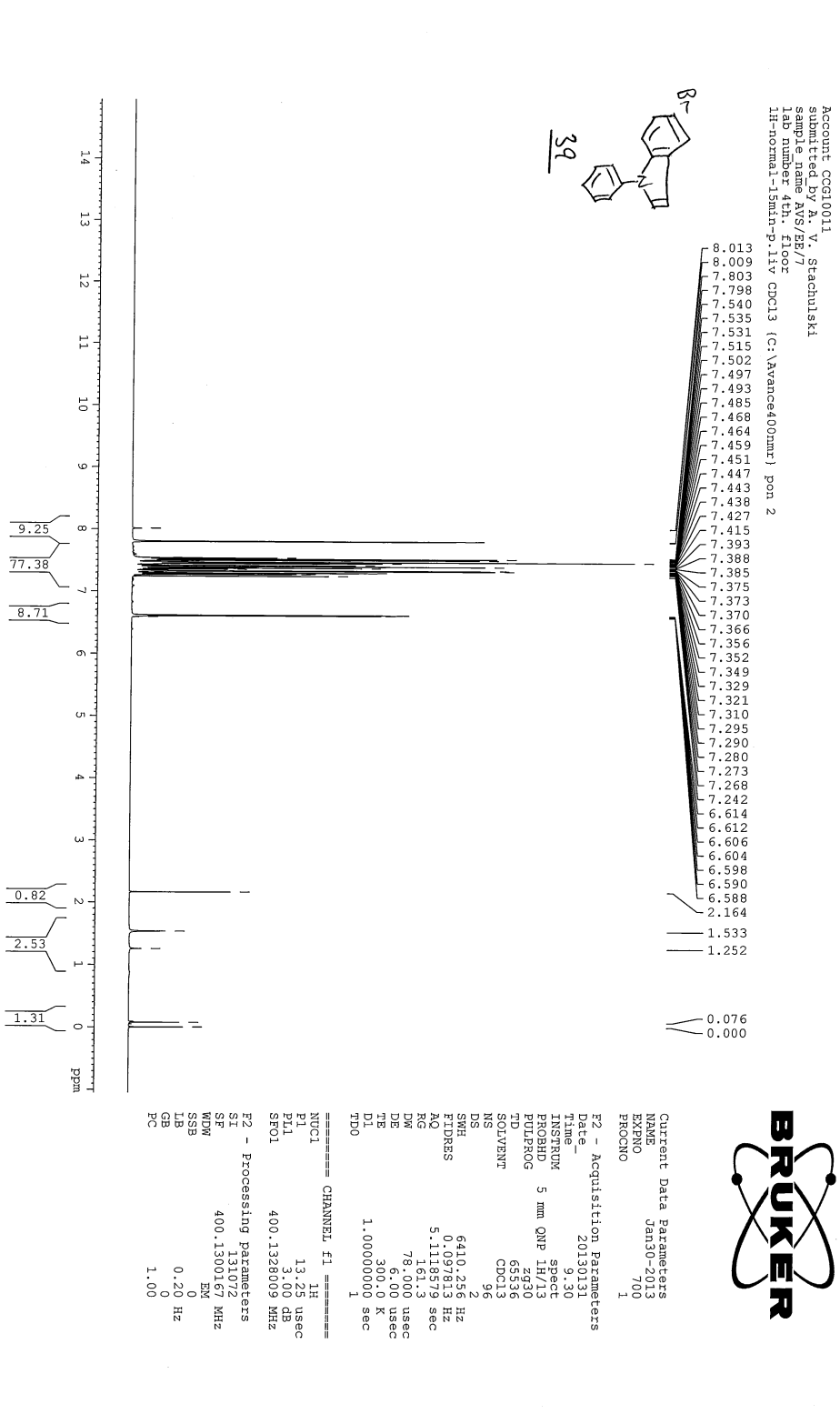
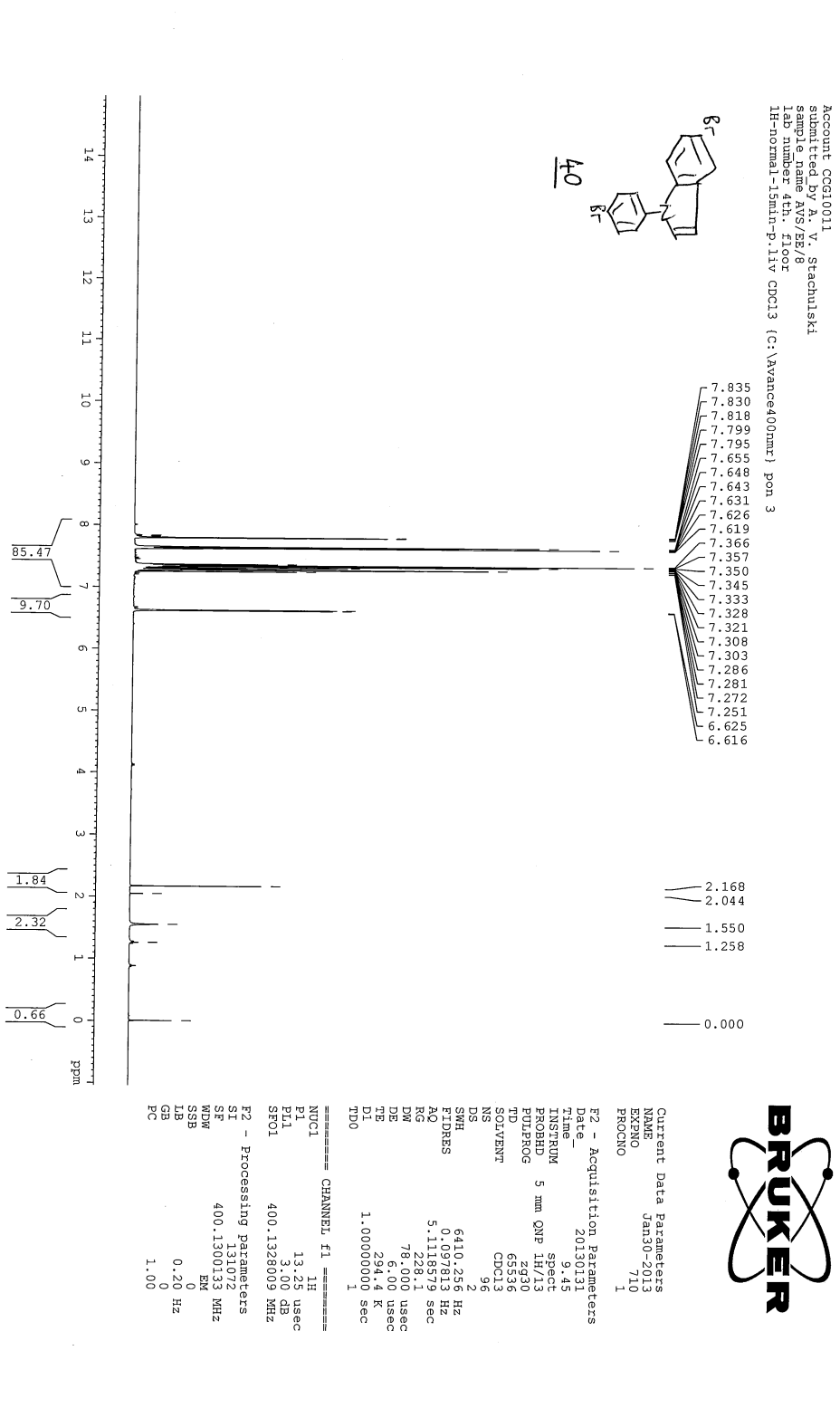
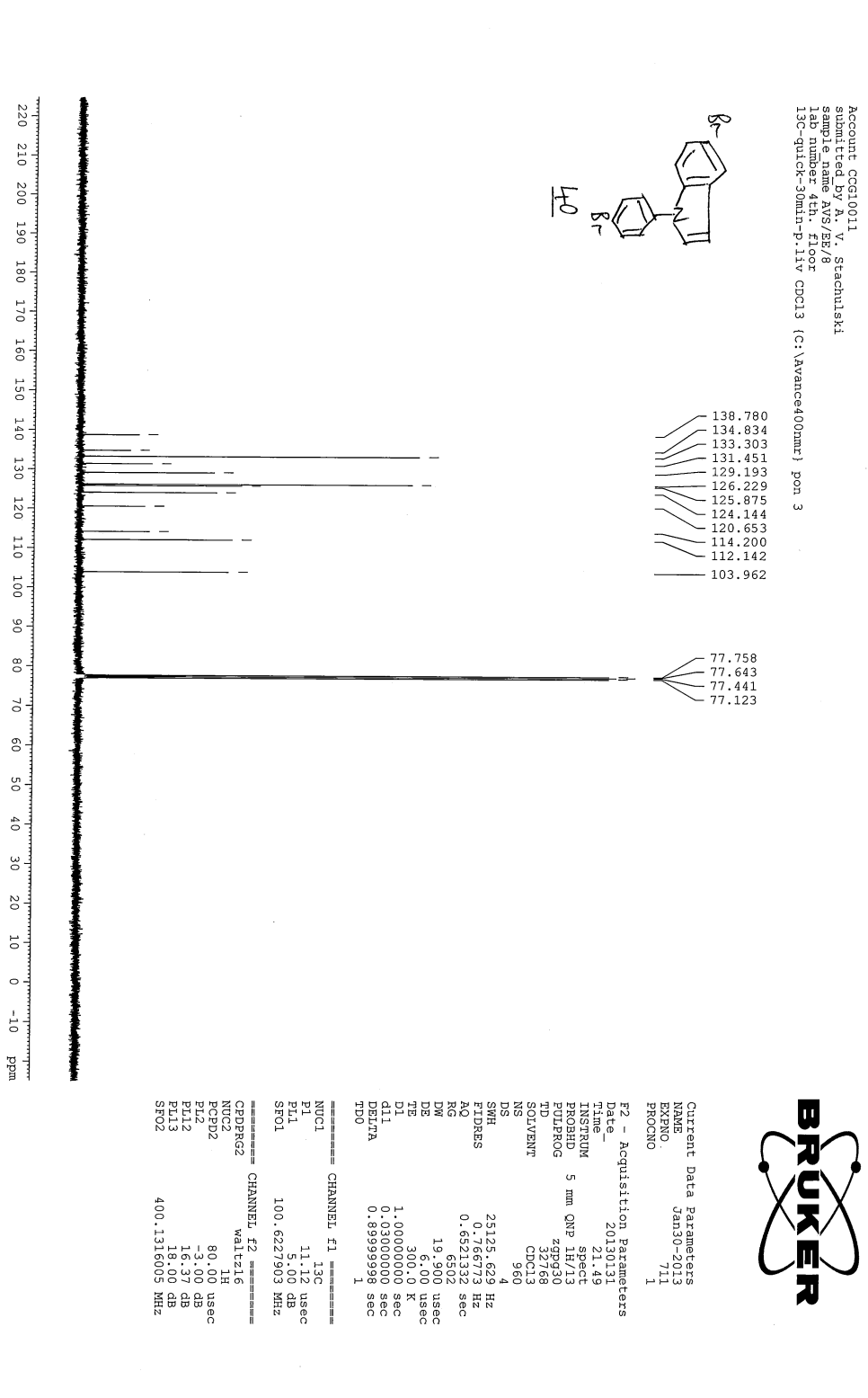
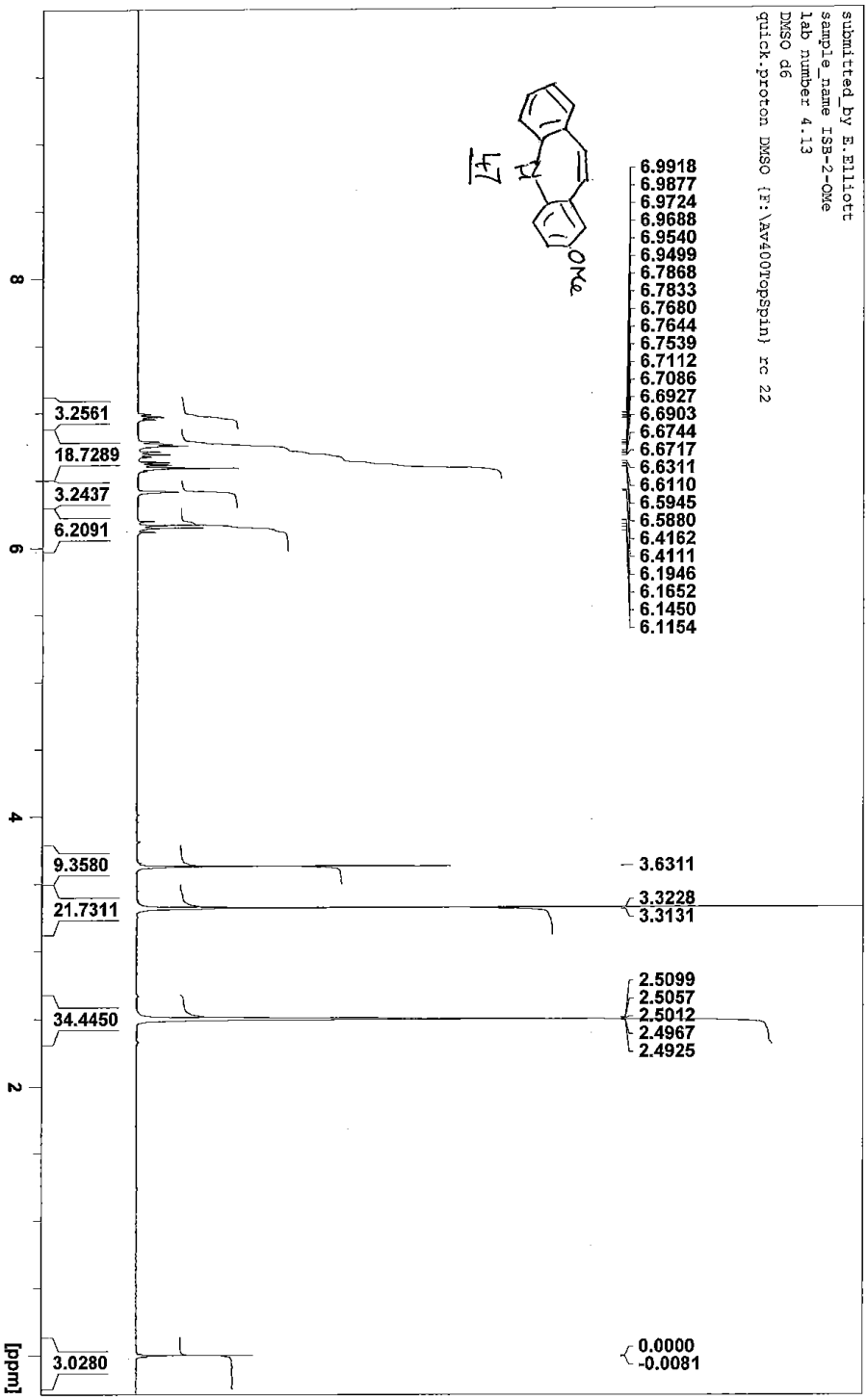
submitted by A. V. Stachulski
sample name AVS/EE/2
lab number 4th. floor
13C-quick-30min-p.liv CDCl3 {C:\Avance400nmr} pon 45
37
Cl
Cl
138.331
134.629
132.848
130.778
130.317
129.400
126.634
125.917
123.287
121.015
111.710
103.973
77.753
77.640
77.435
77.118
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm
BRUKER
Current Data Parameters
NAME Jan28-2013
EXPNO 450
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130128
Time_ 21.02
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 960
DS 4
SWH 25125.629 Hz
FIDRES 0.766773 Hz
AQ 0.65213328 sec
RG 6502
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.12 usec
PL1 5.00 dB
SFO1 100.6227903 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 16.37 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

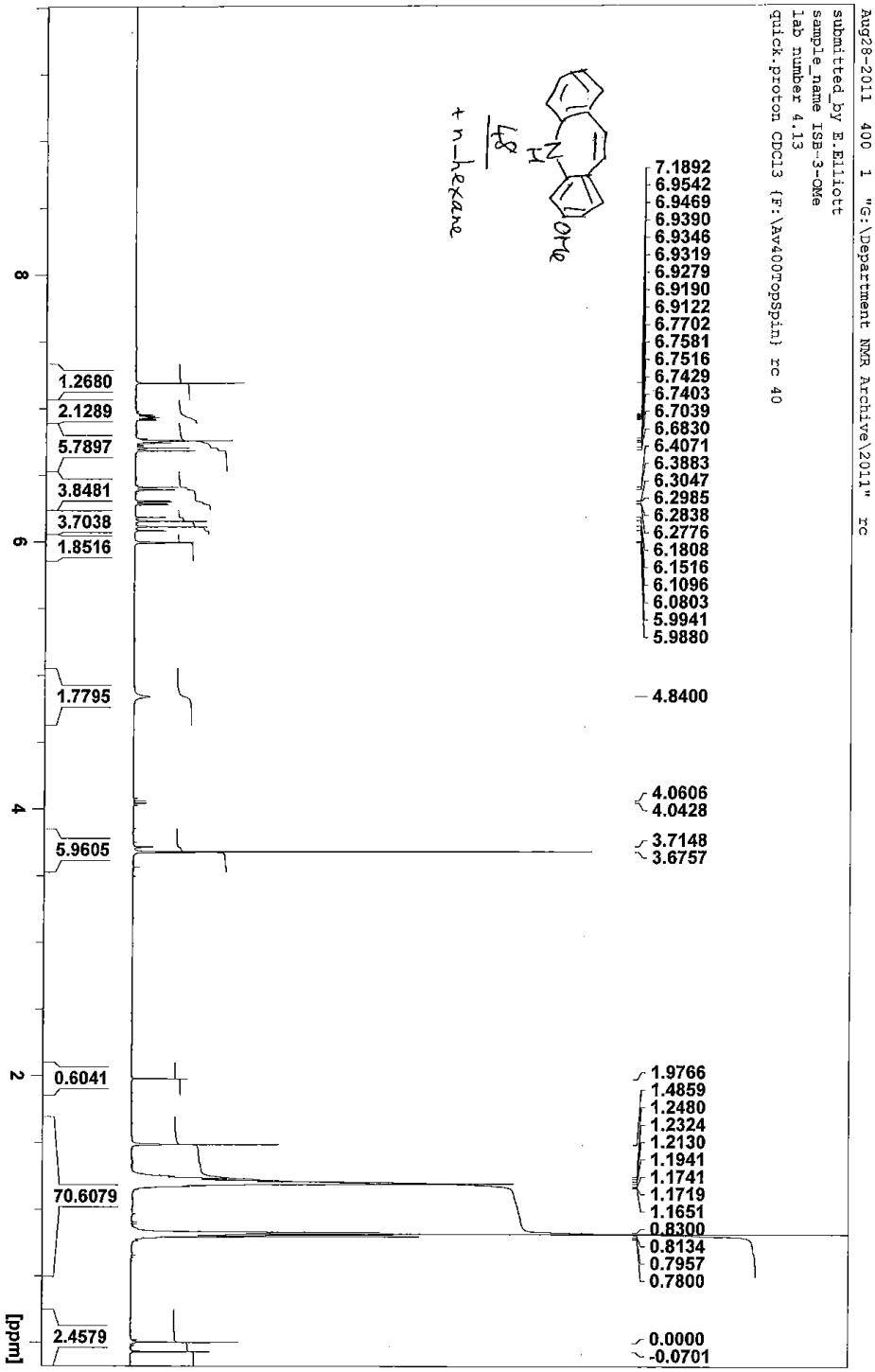

Account CCG10011
submitted_by A. V. Stachulski
sample_name AVS/EE/9
lab number 4th. floor
1H-normal-15min-p.liv CDCl3 {C:\Avance400nmr} pon 7
7.694
7.692
7.691
7.689
7.681
7.674
7.673
7.671
7.670
7.643
7.636
7.630
7.619
7.614
7.606
7.525
7.523
7.507
7.505
7.503
7.400
7.394
7.386
7.381
7.370
7.364
7.357
7.286
7.277
7.267
7.265
7.251
7.248
7.243
7.237
7.233
7.230
7.227
7.213
7.209
7.202
7.195
7.192
7.178
7.176
7.173
7.158
7.156
6.693
6.691
6.685
6.683
2.163
1.255
0.000
94.87
5.13
ppm
Current Data Parameters
NAME Jan30-2013
EXPNO 720
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130131
Time_ 10.00
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 96
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 161.3
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
========== CHANNEL f1 ==========
NUC1 1H
P1 13.25 usec
PL1 3.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 1310720
SF 400.1300190 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

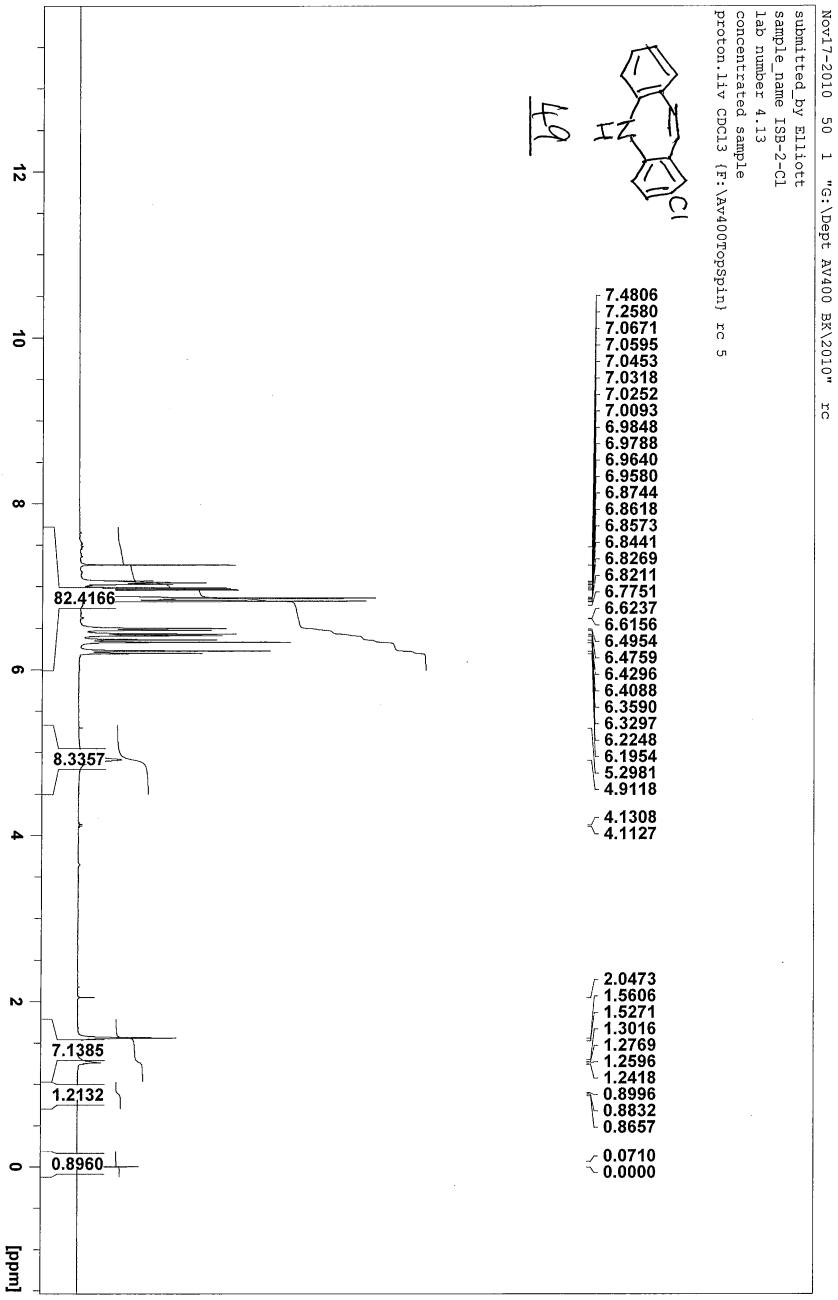

Account CCG10011
submitted by A. V. Stachulski
sample name AVS/BE/7
lab number 4th. floor
1H-normal-15min-p.liv CDCl3 (C:\Avance400nmr) pon 2
8.013
8.009
7.803
7.798
7.540
7.535
7.531
7.515
7.502
7.497
7.493
7.485
7.468
7.464
7.459
7.451
7.447
7.443
7.438
7.427
7.415
7.393
7.388
7.385
7.375
7.373
7.370
7.366
7.356
7.352
7.349
7.329
7.321
7.310
7.295
7.290
7.280
7.273
7.268
7.242
6.614
6.612
6.606
6.604
6.598
6.590
6.588
2.164
1.533
1.252
0.076
0.000
9.25
77.38
8.71
0.82
2.53
1.31
ppm
Current Data Parameters
NAME
EXPNO 700
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130131
Time_ 9.30
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 96
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 161.3
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.25 usec
PL1 3.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 131072
SF 400.1300167 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

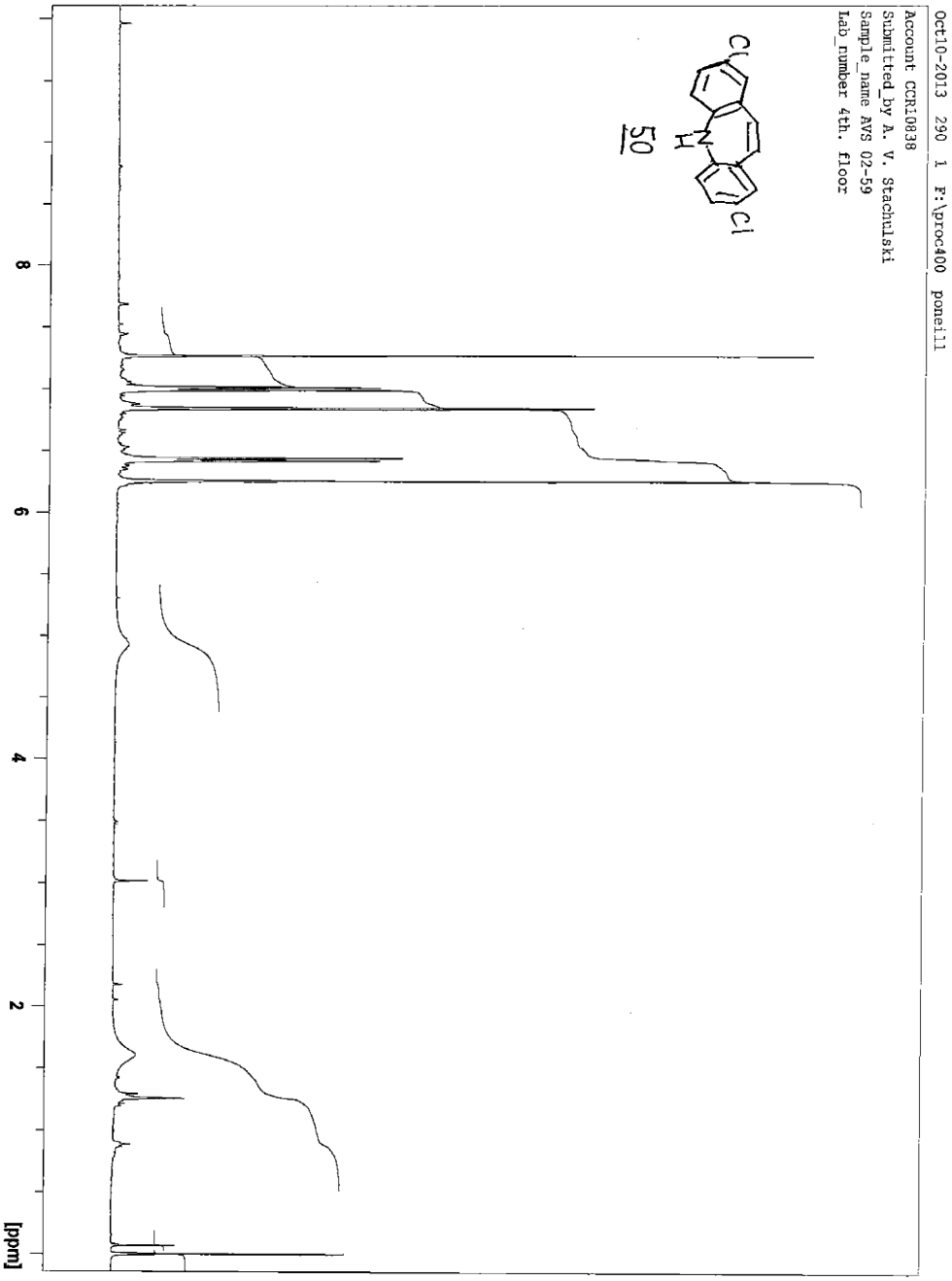

Account CCG10011
submitted by A. V. Stachulski
sample_name AVS/BE/8
lab number 4th. floor
1H-normal-15min-p.liv CDCl3 {C:\Avance400nmr} pon 3
40
Br
Br
7.835
7.830
7.818
7.799
7.795
7.655
7.648
7.643
7.631
7.626
7.619
7.366
7.357
7.350
7.345
7.333
7.328
7.321
7.308
7.303
7.286
7.281
7.272
7.251
6.625
6.616
2.168
2.044
1.550
1.258
0.000
85.47
9.70
1.84
2.32
0.66
ppm
Current Data Parameters
NAME
EXPNO 710
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130131
Time_ 9.45
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 96
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 228.1
DW 78.000 usec
DE 6.00 usec
TE 294.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.25 usec
PL1 3.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 131072
SF 400.1300133 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00
BRUKER


Account CCG10011
submitted by R. v. Stachulski
sample name AVS/EE/8
lab number 4th. floor
13C-quick-30min-p.liv CDCl3 {C:\Avance400nmr} pon 3
Br
Br
40
138.780
134.834
133.303
131.451
129.193
126.229
125.875
124.144
120.653
114.200
112.142
103.962
77.758
77.643
77.441
77.123
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm
Current Data Parameters
NAME Jan30-2013
EXPNO 711
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130131
Time 21.49
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 960
DS 4
SWH 25125.629 Hz
FIDRES 0.766773 Hz
AQ 0.6521332 sec
RG 6502
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.12 usec
PL1 5.00 dB
SFO1 100.6227903 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 16.37 dB
PL13 18.00 dB
SFO2 400.1316005 MHz


Jul26-2011 220 1 "G:\Department NMR Archive\2011" rc
submitted by E.Elliott
sample_name ISB-2-OMe
lab number 4.13
DMSO d6
quick.proton DMSO {F:\AV400TopSpin} rc 22
OMe
6.9918
6.9877
6.9724
6.9688
6.9540
6.9499
6.7868
6.7833
6.7680
6.7644
6.7539
6.7112
6.7086
6.6927
6.6903
6.6744
6.6717
6.6311
6.6110
6.5945
6.5880
6.4162
6.4111
6.1946
6.1652
6.1450
6.1154
3.6311
3.3228
3.3131
2.5099
2.5057
2.5012
2.4967
2.4925
0.0000
-0.0081
3.2561
18.7289
3.2437
6.2091
9.3580
21.7311
34.4450
3.0280
8
6
4
2
[ppm]




Nov17-2010 50 1 "G:\Dept AV400 BK\2010" rc
submitted_by Elliott
sample_name ISB-2-Cl
lab number 4.13
concentrated sample
proton.liv CDCl3 {F:\Av400TopSpin} rc 5
4g
Cl
H
N
7.4806
7.2580
7.0671
7.0595
7.0453
7.0318
7.0252
7.0093
6.9848
6.9788
6.9640
6.9580
6.8744
6.8618
6.8573
6.8441
6.8269
6.8211
6.7751
6.6237
6.6156
6.4954
6.4759
6.4296
6.4088
6.3590
6.3297
6.2248
6.1954
5.2981
4.9118
4.1308
4.1127
2.0473
1.5606
1.5271
1.3016
1.2769
1.2596
1.2418
0.8996
0.8832
0.8657
0.0710
0.0000
82.4166
8.3357
7.1385
1.2132
0.8960
12
10
8
6
4
2
0
[ppm]


Oct10-2013 290 1 F:\proc400 poneill
Account CCR10838
Submitted_by A. V. Stachulski
Sample_name AVS 02-59
Lab_number 4th. floor
50
Cl
N
H
Cl
8
6
4
2
[ppm]