

Supplementary Information (SI)

Solvent-Free 1*H*-tetrazole, 1,2,5,6-tetrahydronicotinonitrile and pyrazole synthesis using Quinoline Based Ionic Fluoride Salts (QuFs): Thermal and Theoretical Studies

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^a H.E.J. Research Institute of Chemistry, International Center for Chemical and Biological Sciences, University of Karachi, Karachi-75270, Pakistan

^b Department of Chemistry, Forman Christian College (A Chartered University), Ferozepur Road-54600, Lahore, Pakistan.

^c Department of Chemistry, King Abdulaziz University, Jeddah, Saudi Arabia

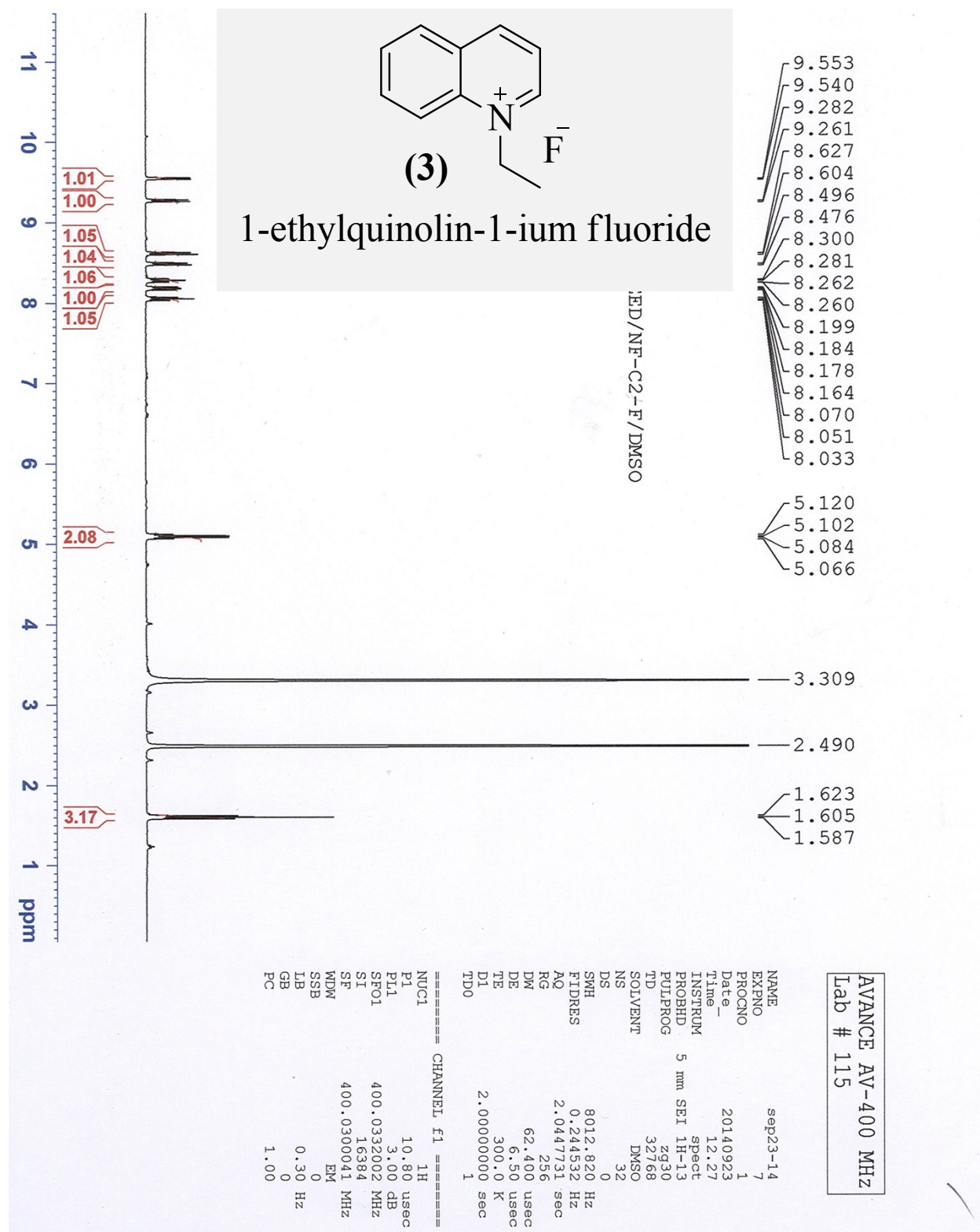
Corresponding author:

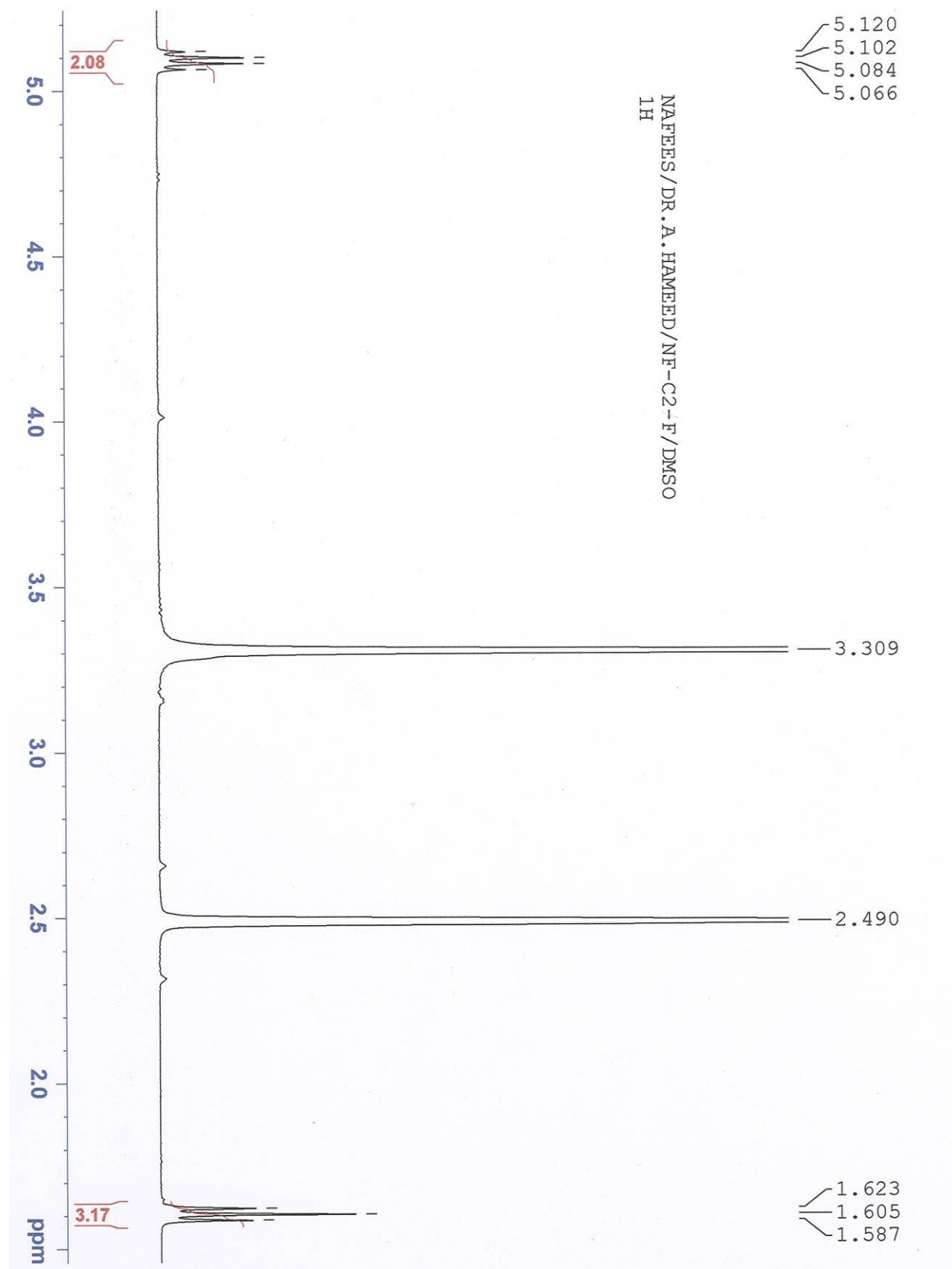
** A.H., Tel.: +92-219-9261701-2; Fax: +92-213-4819018

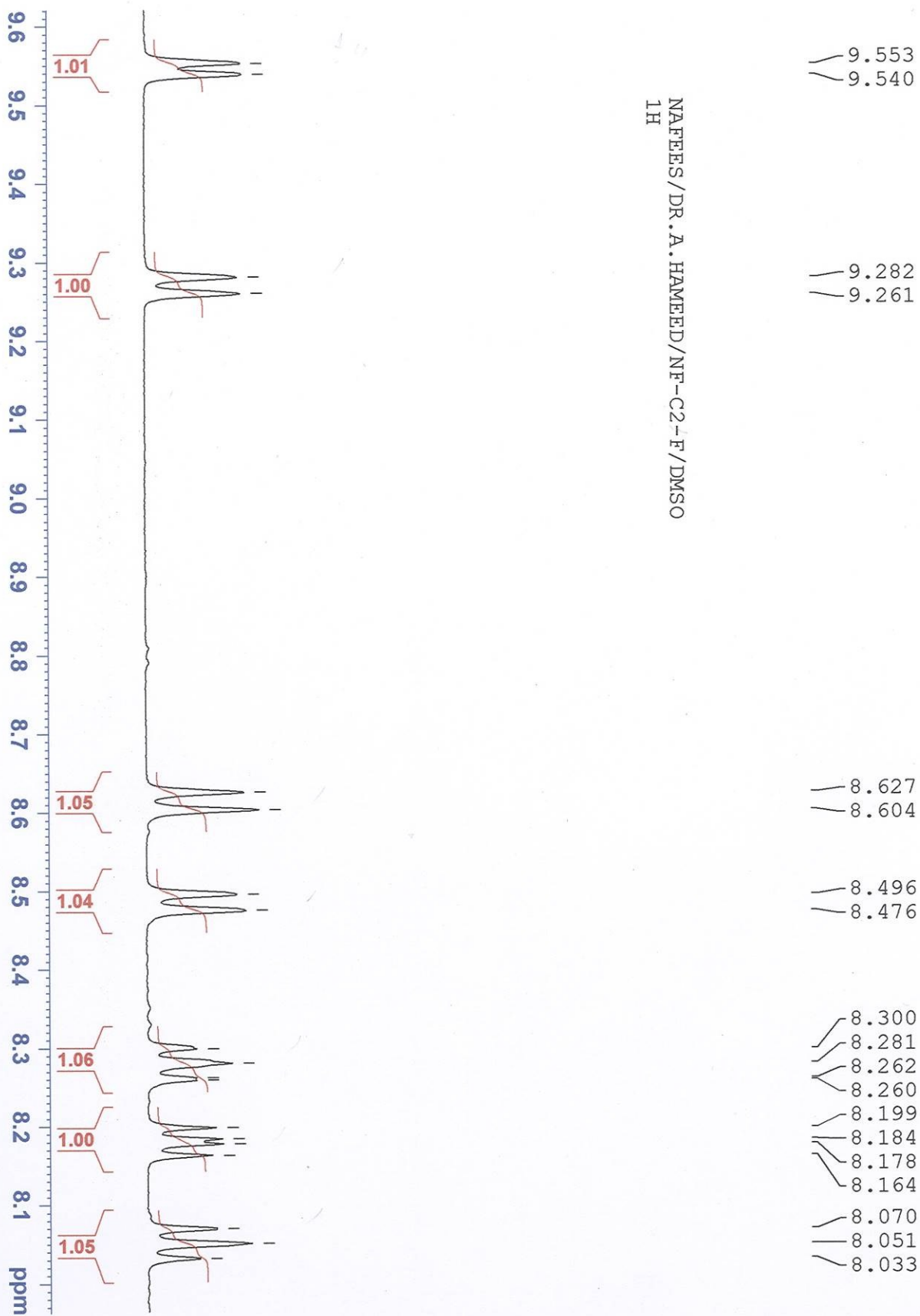
* S.T.M, Email: tarique.syed@iccs.edu

* A.H., Email: abdul.hameed@iccs.edu

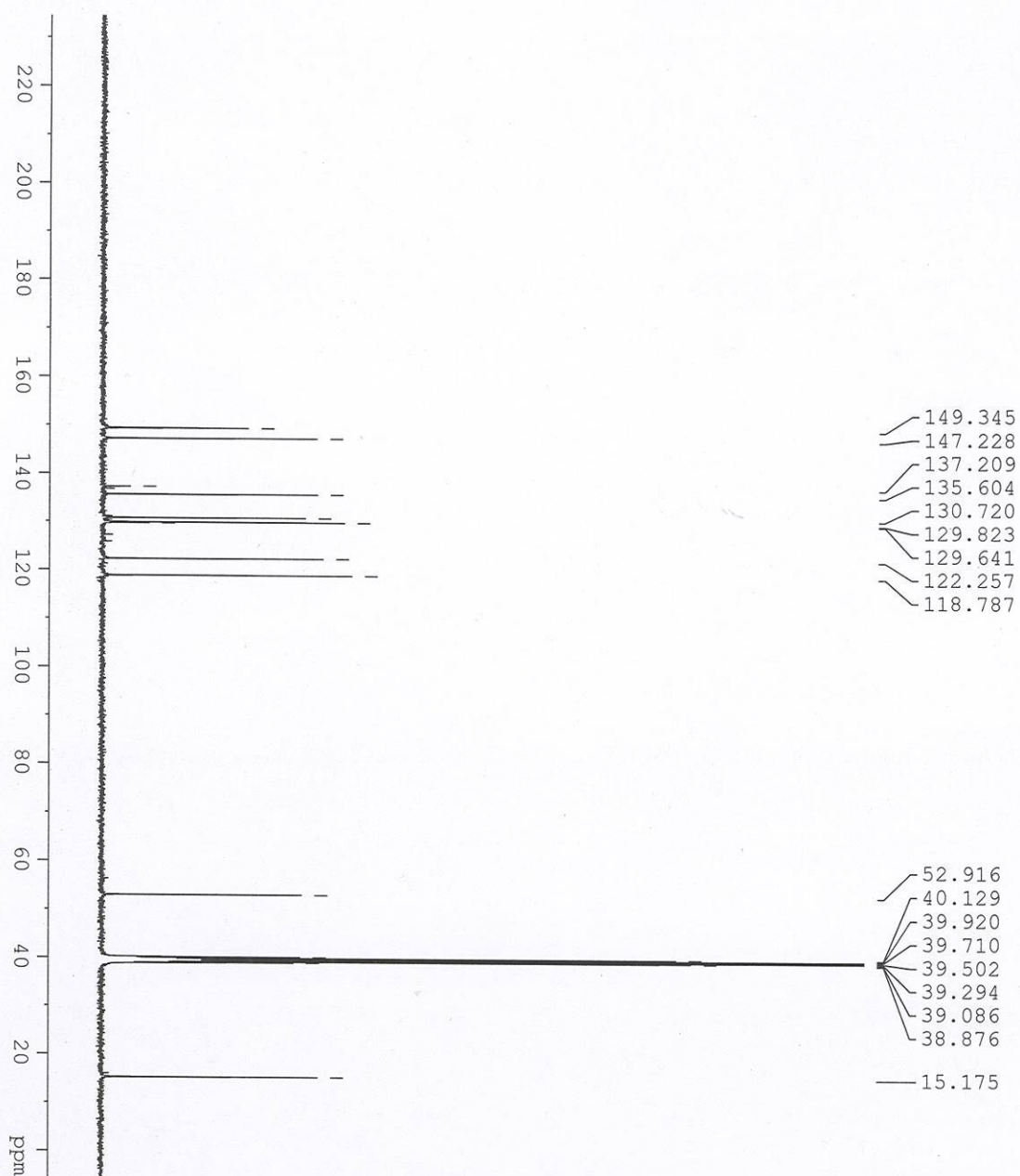
Spectral Data







NAFEES IQBAL/DR, ABDUL HAMEED/
NF-C2-F/BB



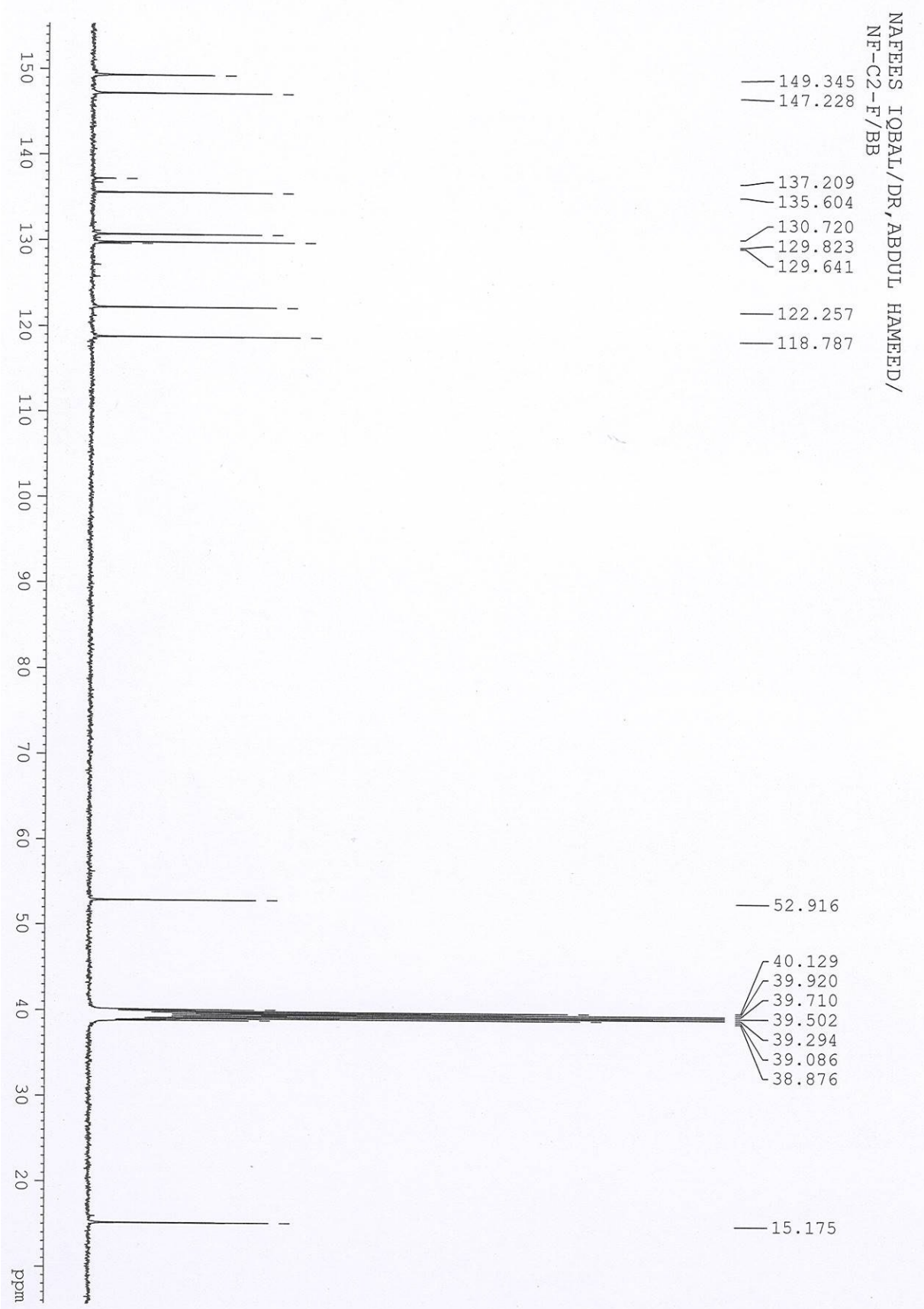
AVANCE 400
LAB NO 117

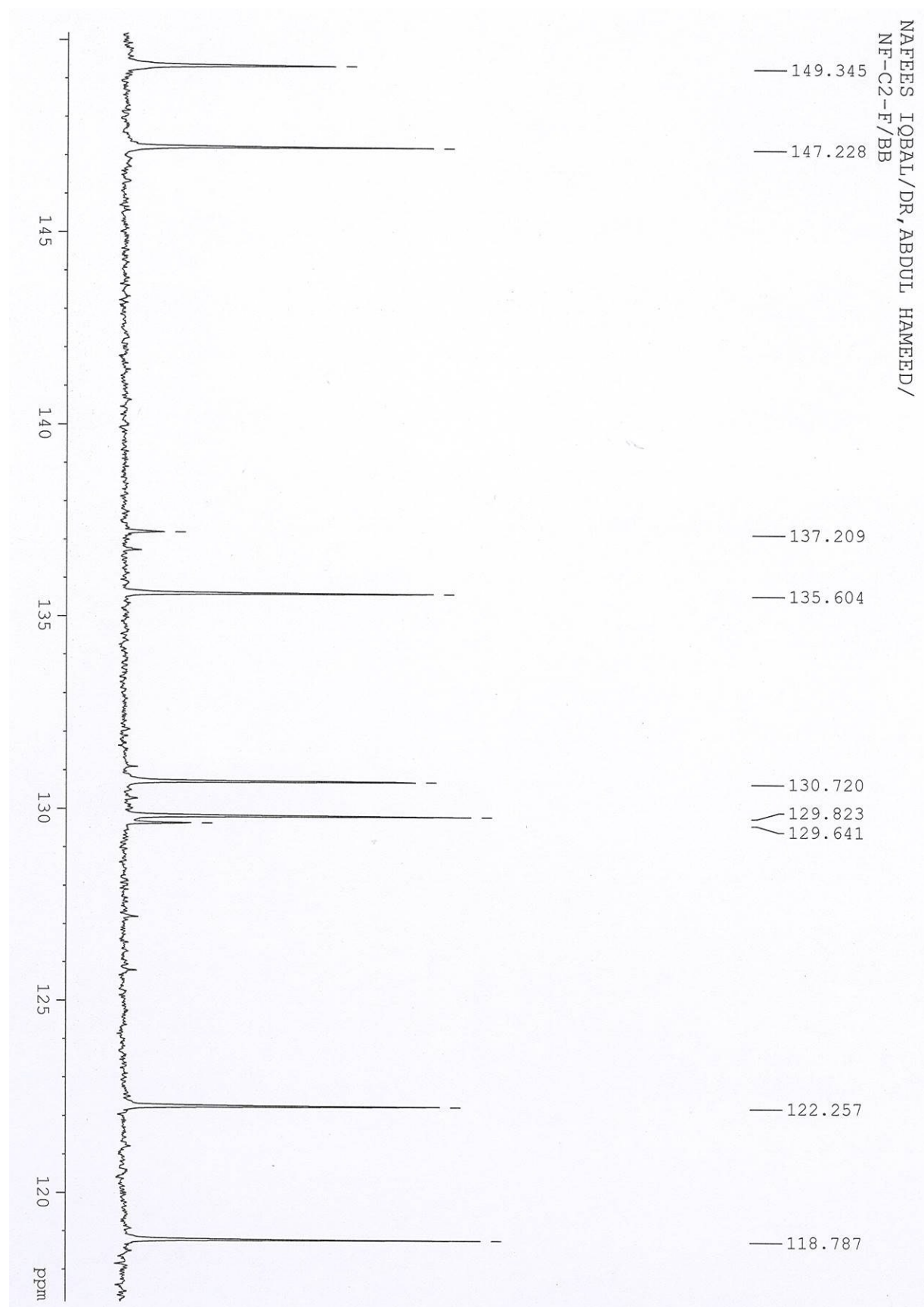
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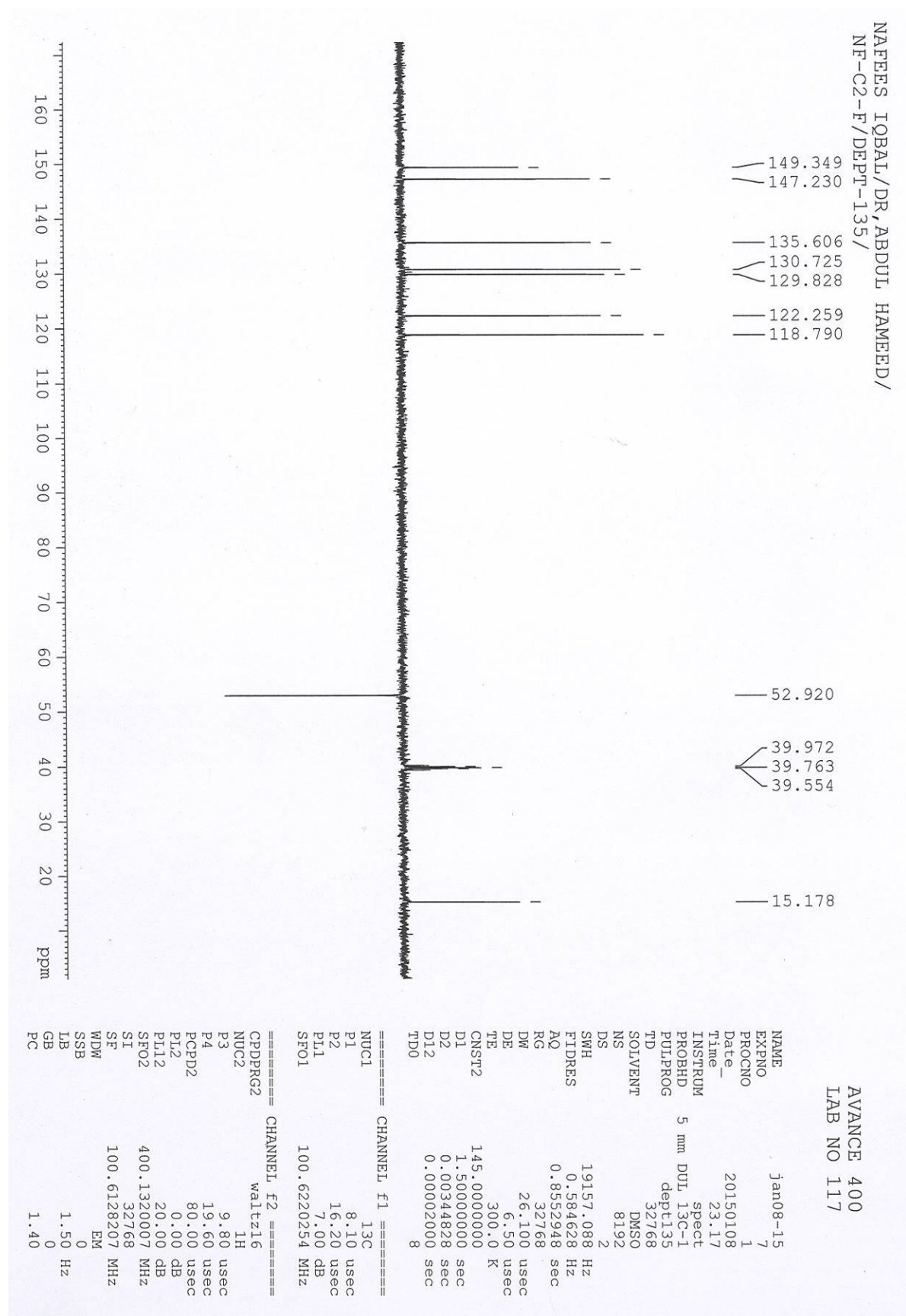
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EXPNO         6
PROCNO        1
Date_         20150108
Time_         11.46
INSTRUM       spect
PROBHD        5 mm DUL
PULPROG       zgpg
TD            32768
SOLVENT       DMSO
NS            18432
DS            2
SWH           24154.590 Hz
FIDRES        0.737140 Hz
AQ            0.6783476 sec
RG            32768
DM           20.700 usec
DE           6.50 usec
TE            300.0 K
D1            1.50000000 sec
D11           0.03000000 sec
TD0           18

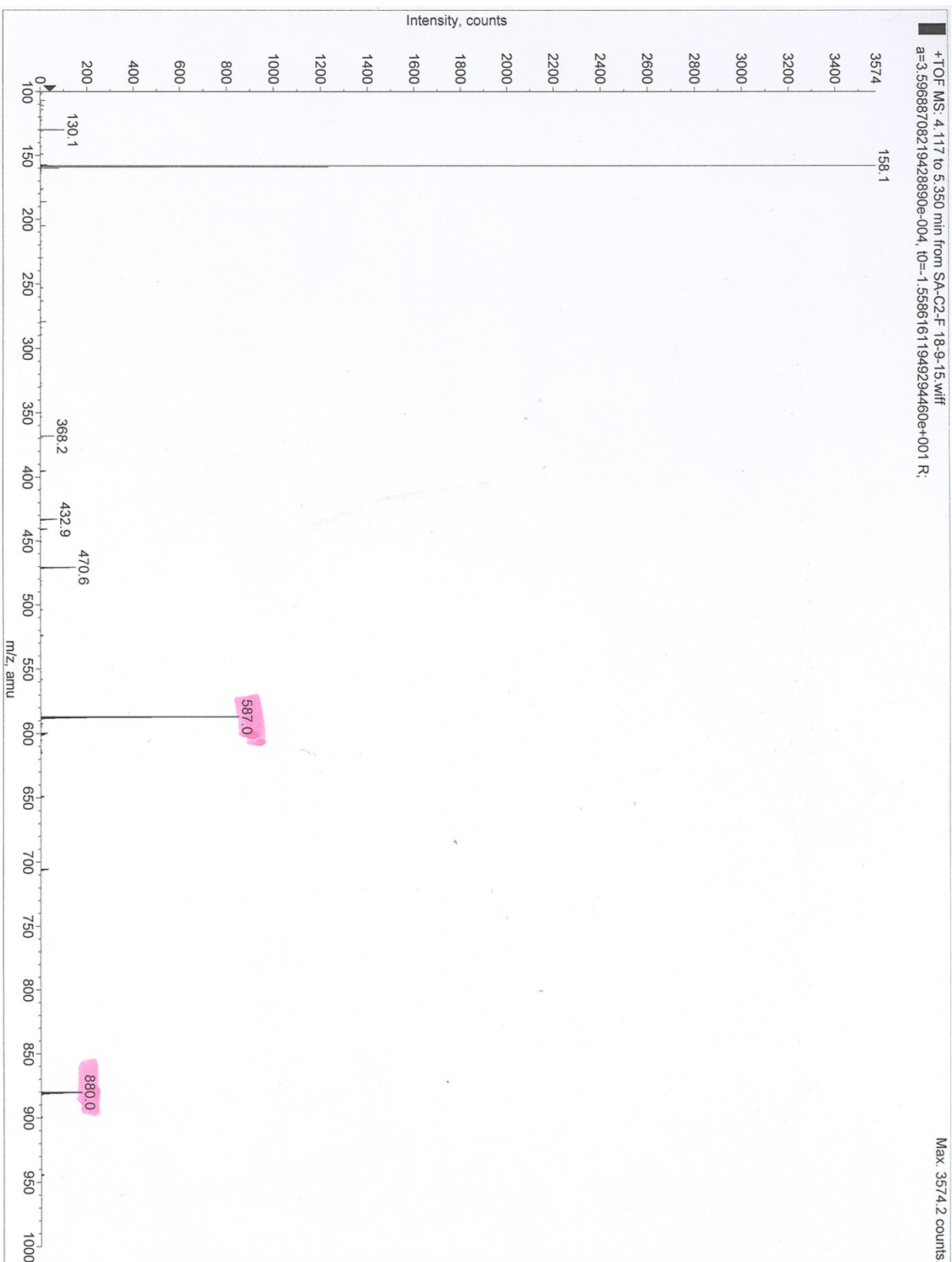
===== CHANNEL f1 =====
NUC1          13C
P1            8.10 usec
PL1           7.00 dB
SFO1          100.6243395 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           0.00 dB
PL12          20.00 dB
PL13          22.00 dB
SFO2          400.1324008 MHz
SI            16384
SF            100.6128207 MHz
WDW           EM
SSB           0
LB            1.50 Hz
GB            0
PC            1.00
  
```







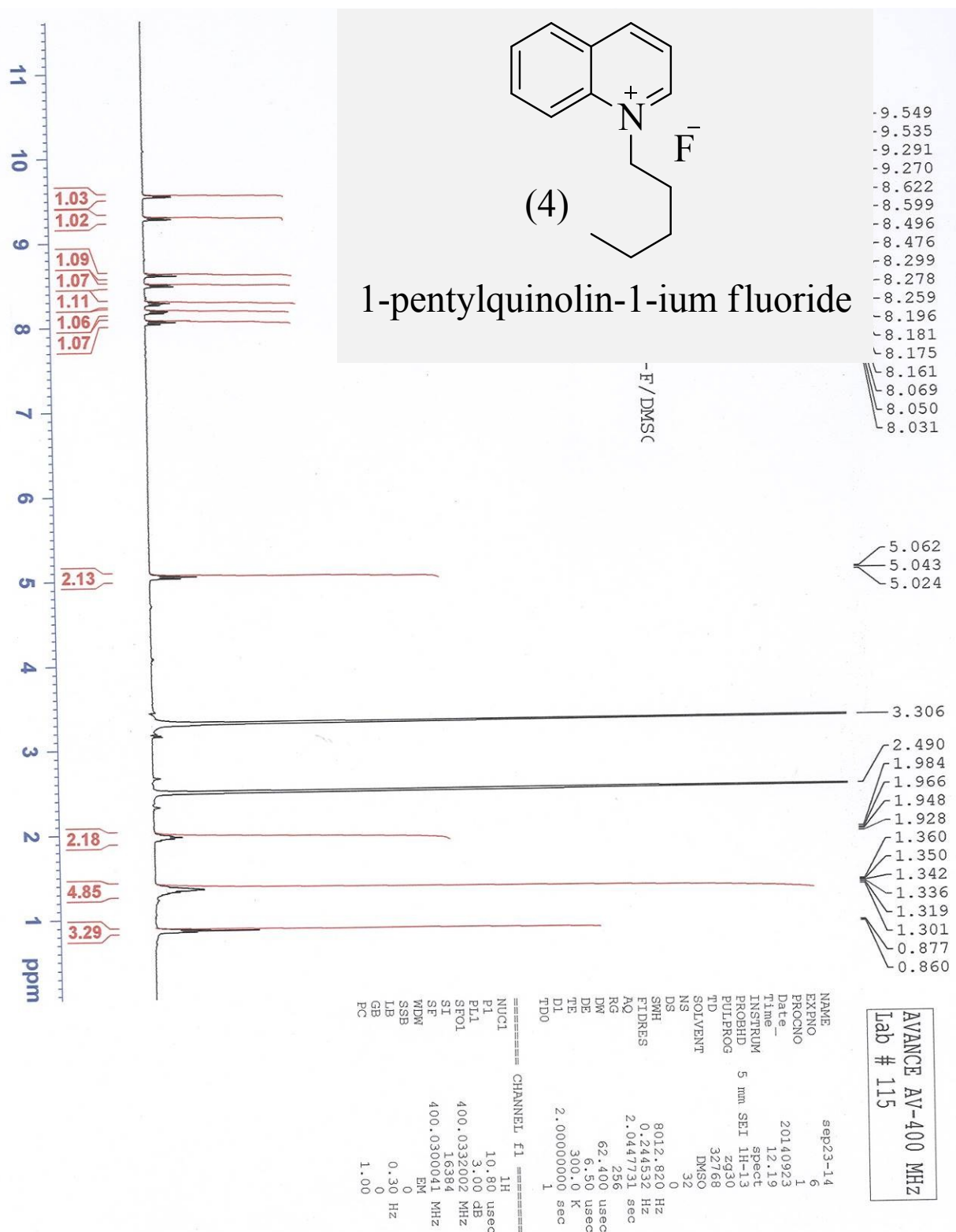


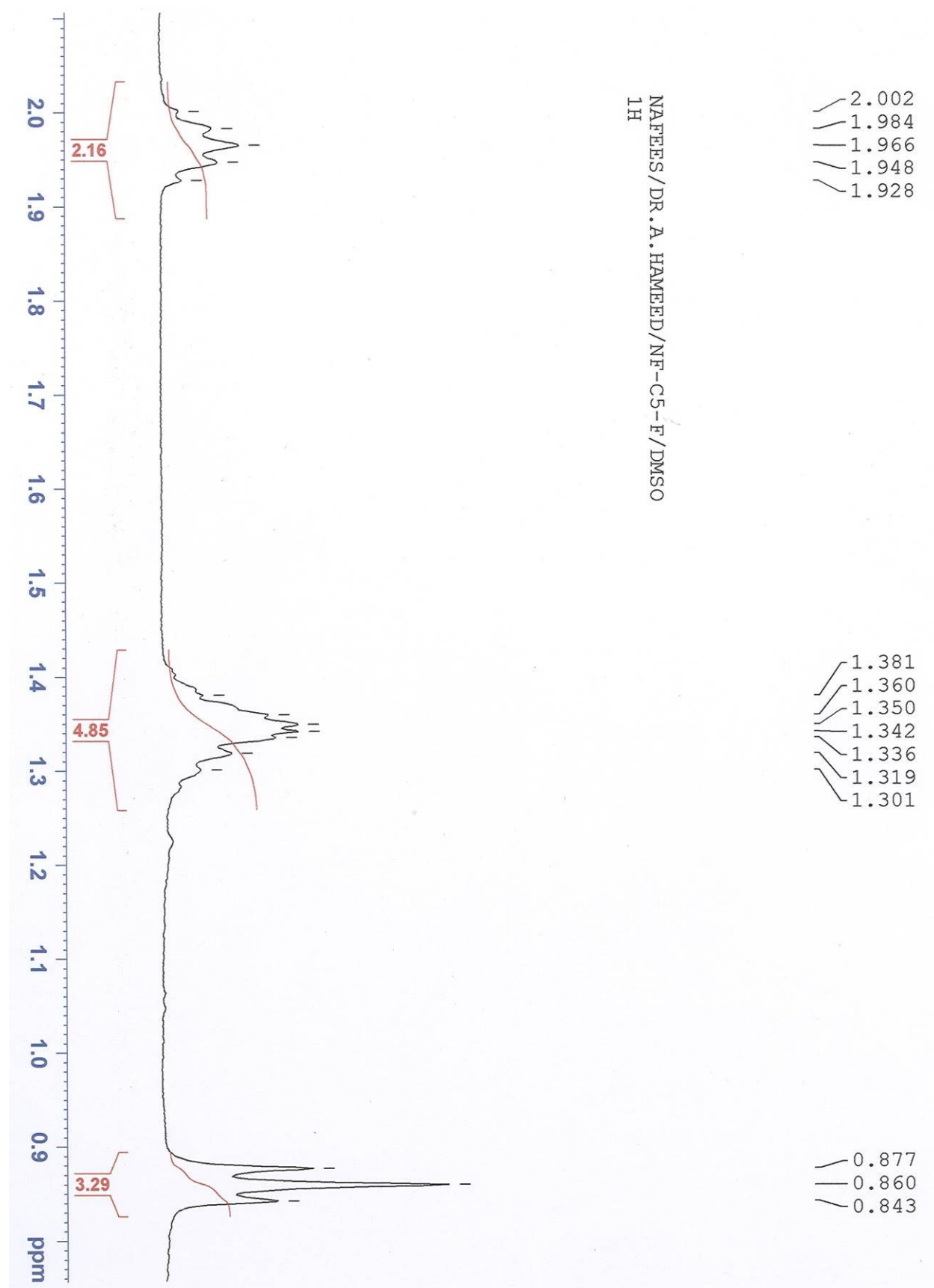
Elemental composition calculator

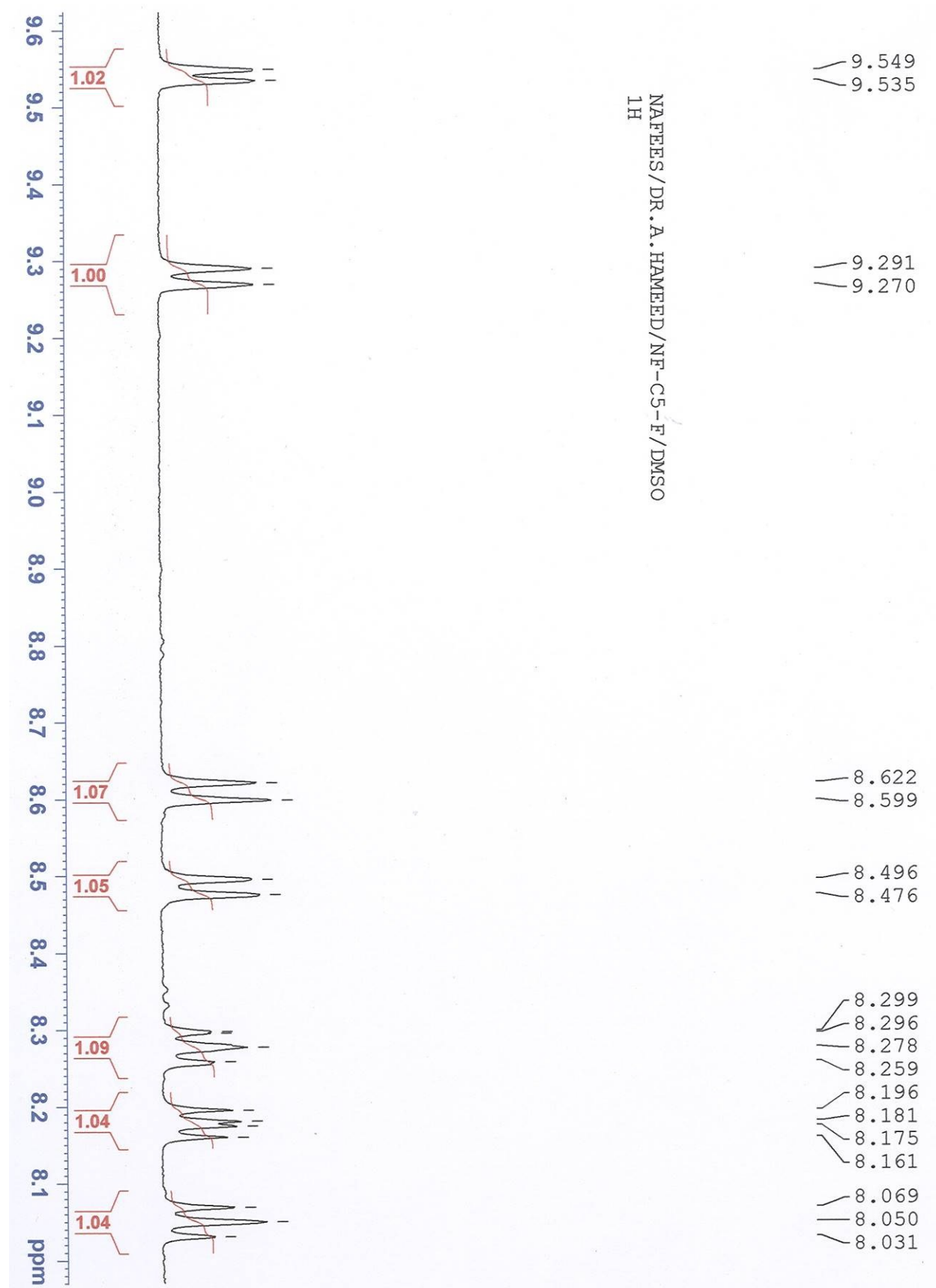
Target m/z: +158.0956 amu
 Tolerance: +10.0000 ppm
 Result type: Elemental
 Max num of results: 10
 Min DBE: -0.5000 Max DBE: +100.0000
 Electron state: OddAndEven
 Num of charges: 0
 Add water: N/A
 Add proton: N/A
 File Name: SA-C2-F 18-9-15.wiff

	Elements	Min Number	Max Number
1	C	0	18
2	F	0	1
3	H	0	30
4	N	0	2

	Formula	Calculated m/z (amu)	mDa Error	PPM Error	DBE
1	C11 H12 N	158.0969	-1.3744	-8.6940	6.5



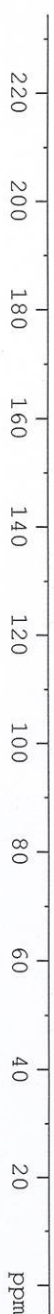




NAFEES/DR.A.HAMEED/NF-C5-F
ICCBS,U.O.K/BB

149.599
147.314
137.280
135.610
130.683
129.792
129.618
122.099
118.960

57.173
39.926
39.718
39.510
39.300
39.091
29.215
27.773
21.604
13.681



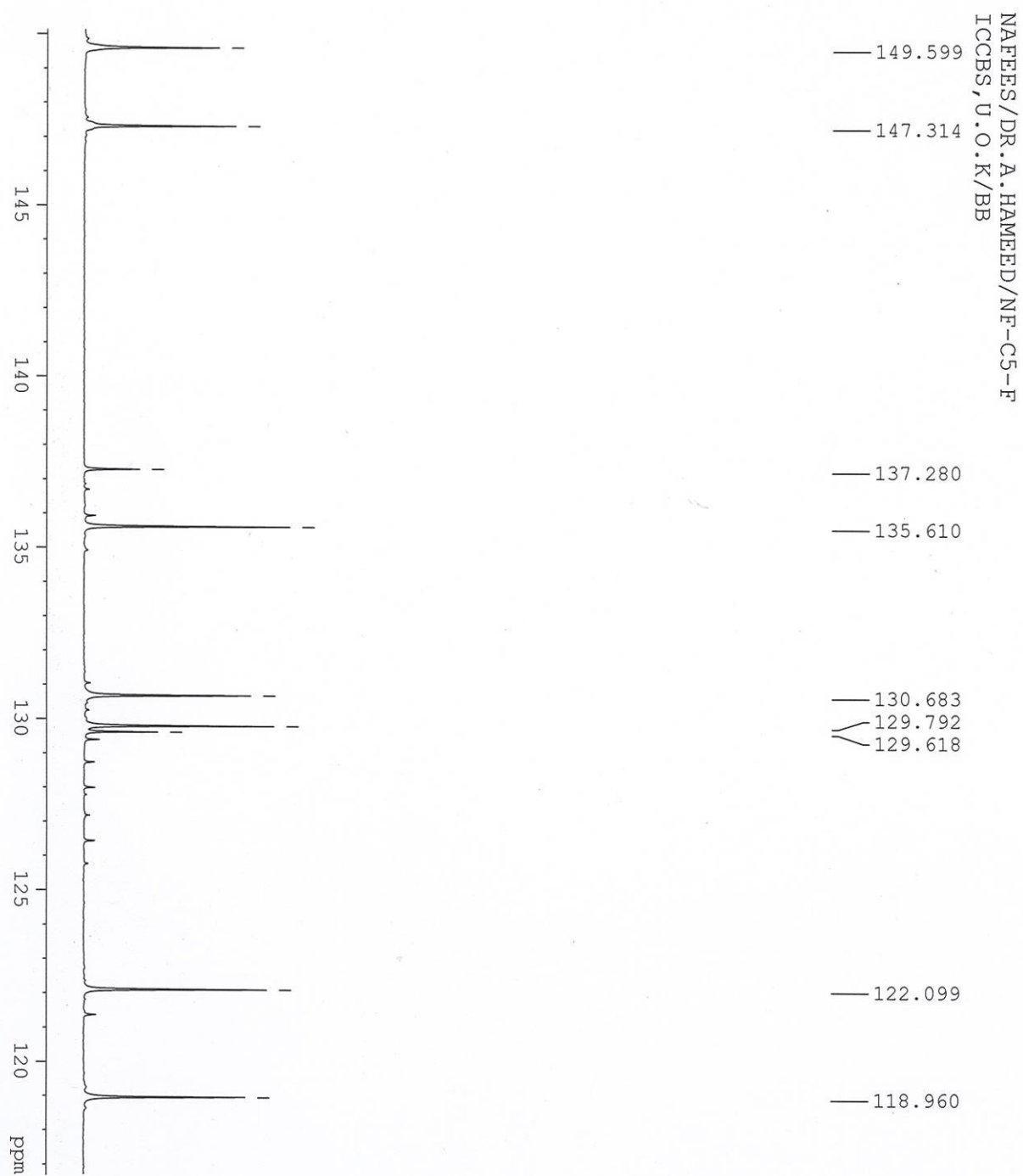
AVANCE 400
LAB NO 117

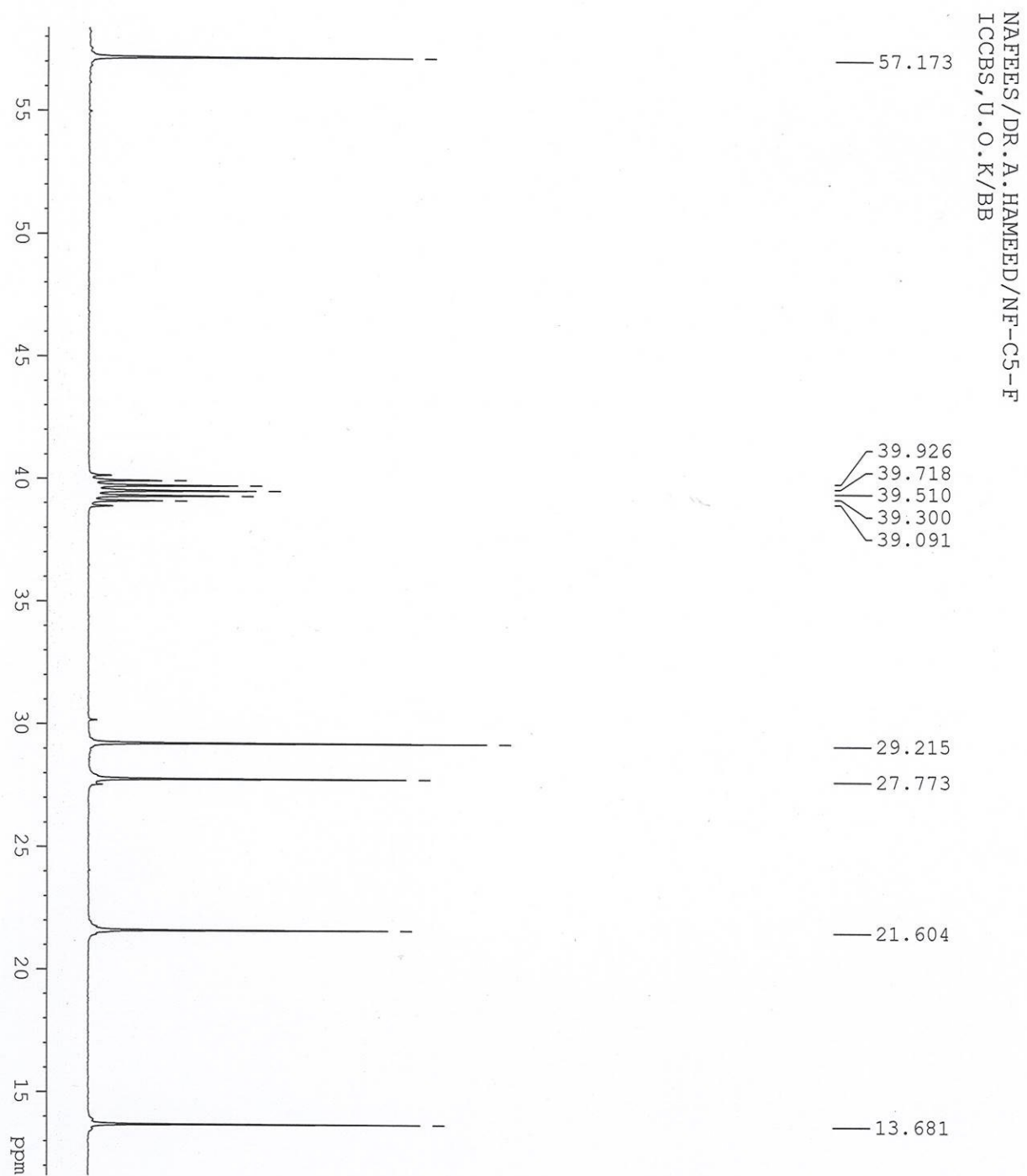
Scanned

NAME	feb15-15
EXPNO	1
PROCNO	1
Date_	20150215
Time	8.55
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg
TD	32768
SOLVENT	DMSO
NS	12288
DS	2
SWH	24154.590 Hz
FIDRES	0.737140 Hz
AQ	0.6783476 sec
RG	32768
DW	20.700 usec
DE	6.50 usec
TE	300.0 K
D1	1.50000000 sec
D11	0.03000000 sec
TD0	12

===== CHANNEL f1 =====	
NUC1	13C
P1	8.10 usec
PL1	7.00 dB
SFO1	100.6243395 MHz

===== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	0.00 dB
PL12	20.00 dB
PL13	22.00 dB
SFO2	400.1324008 MHz
SI	16384
SF	100.6128207 MHz
WDW	EM
SSB	0
LB	1.50 Hz
GB	0
PC	1.00

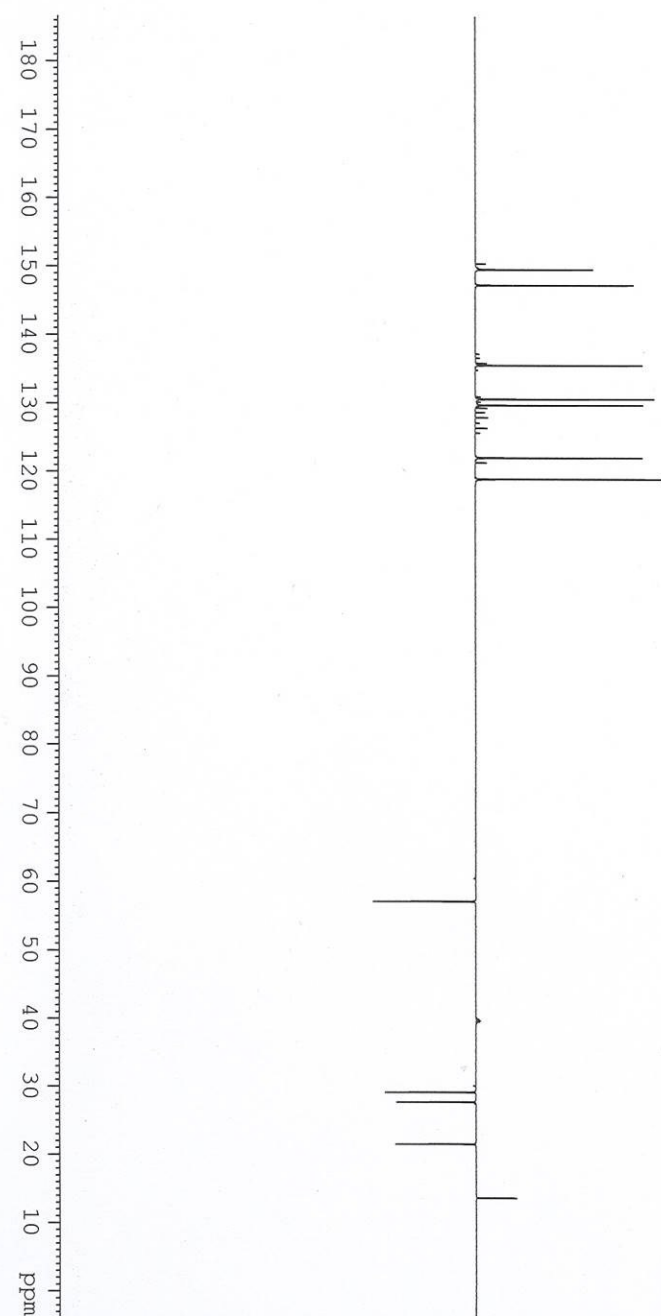




NAFEES/DR.A.HAMEED/NF-C5-F
DEPT-135/

149.602
147.317
135.933
135.615
130.685
129.798
122.104
118.962

57.178
39.980
39.770
39.560
29.222
27.776
21.607
13.686



NAME EXPNO feb15-15
PROCNO 2
Date_ 20150215
Time_ 16.37
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG dept135
TD 32768
SOLVENT DMSO
NS 6144
DS 2
SWH 19157.088 Hz
FIDRES 0.584628 Hz
AQ 0.8552948 sec
RG 32768
DM 26.100 usec
DE 6.50 usec
TE 300.0 K
CNSR2 145.0000000
D1 1.50000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
TD0 6

===== CHANNEL f1 =====
NUC1 13C
P1 8.10 usec
P2 16.20 usec
PL1 7.00 dB
SFO1 100.6220254 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P3 9.80 usec
P4 19.60 usec
PCPD2 80.00 usec
PL2 0.00 dB
PL12 20.00 dB
SFO2 400.1320007 MHz
SI 32768
SF 100.6128207 MHz
WDW EM
SSB 0
LB 1.50 Hz
GB 0
PC 1.40

AVANCE 400
LAB NO 117

870 KF Titrino Plus	15183	5.870.0023
2015-05-26 11:48:31		870 KF
User name		IAC

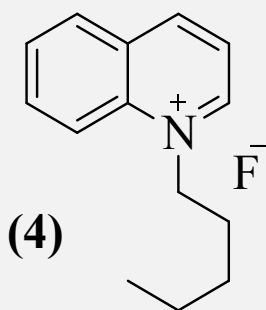
RESULT REPORT

Method	KFT Ip01
Determination time	2015-05-26 11:46:34
Duration of determination	39.2 s
Sample number	8
ID1	NF-C5
Sample size	0.0211 g

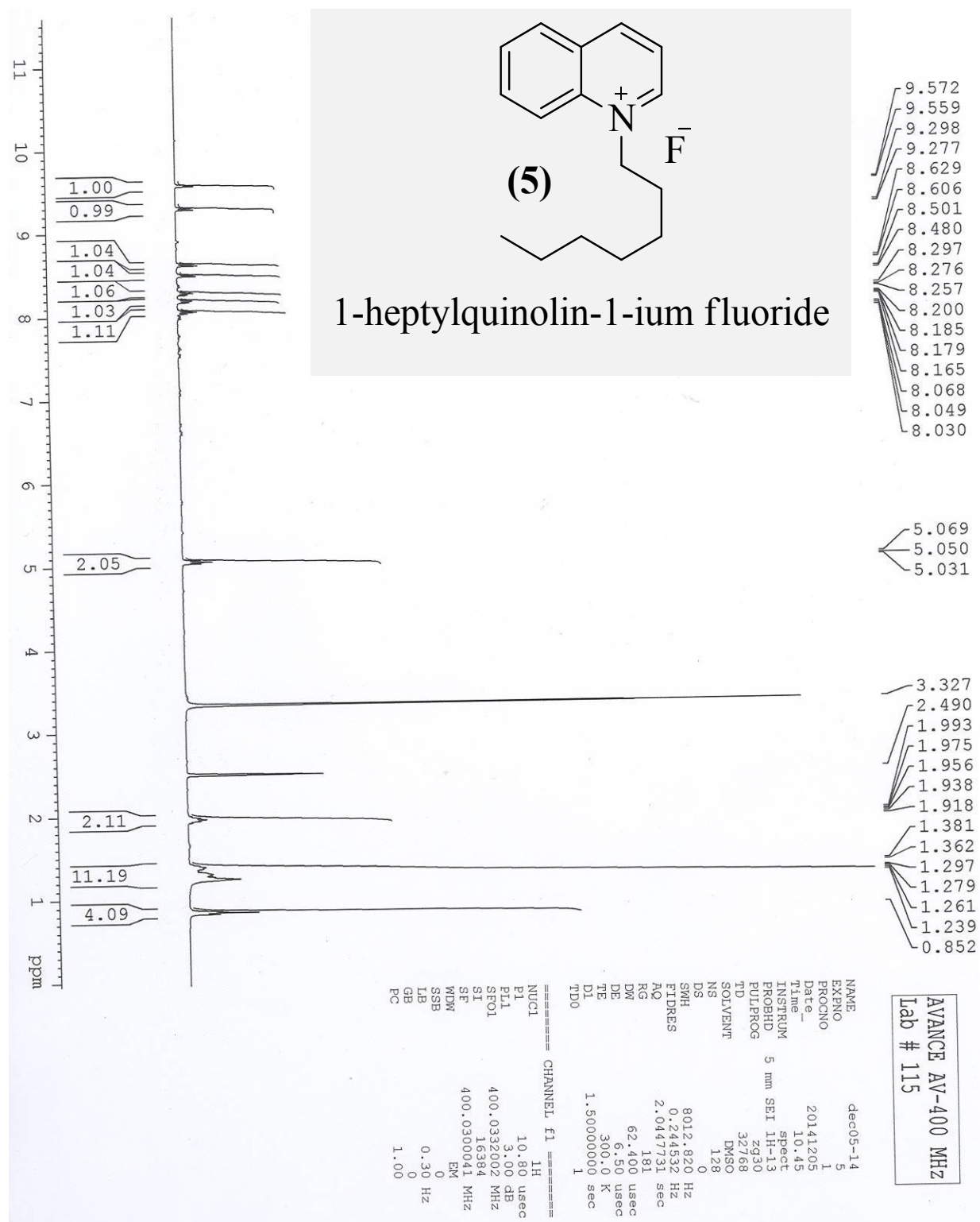
EP1	0.5670 mL
Regular stop	
Water	12.32 %

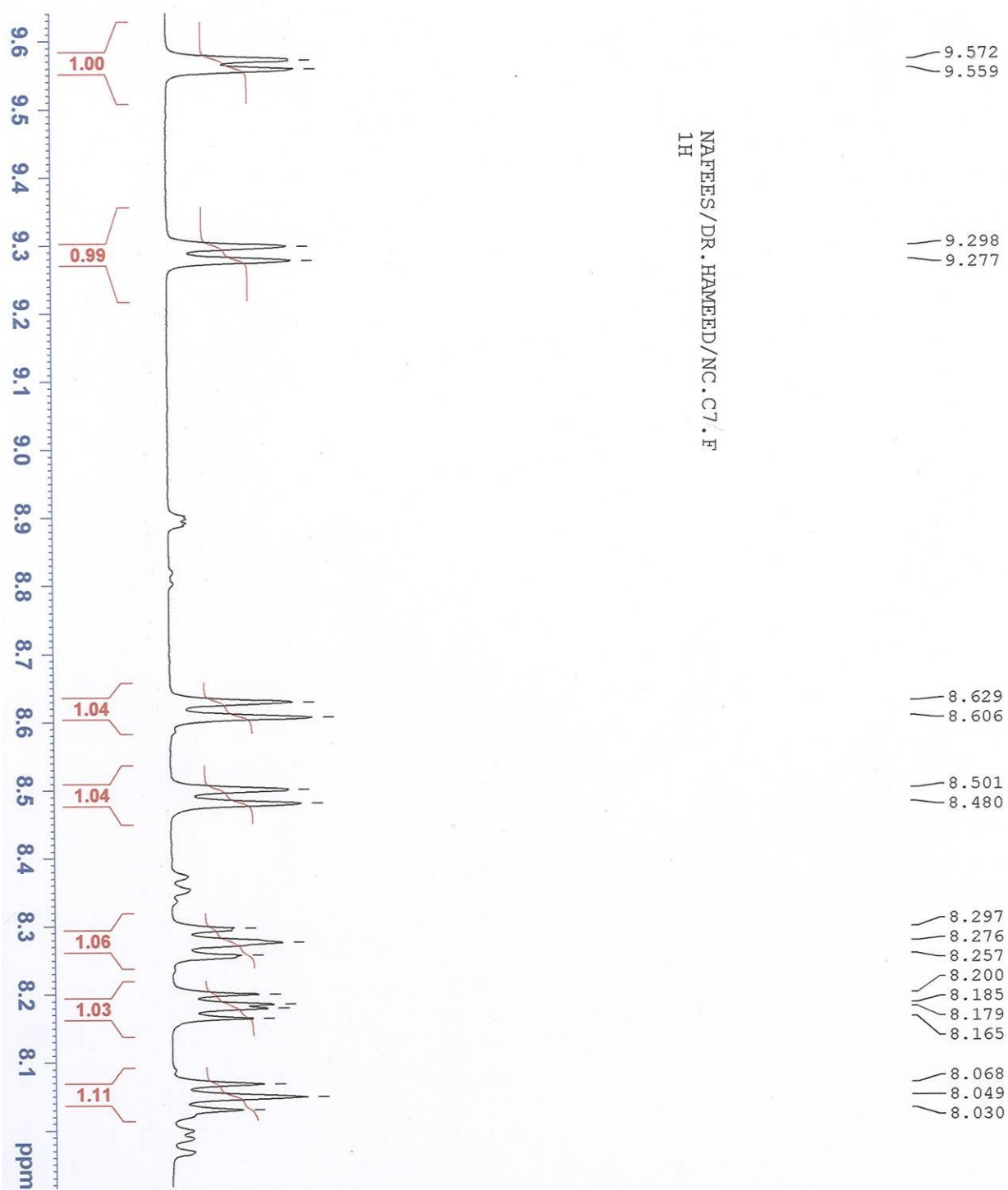
CALCULATIONS

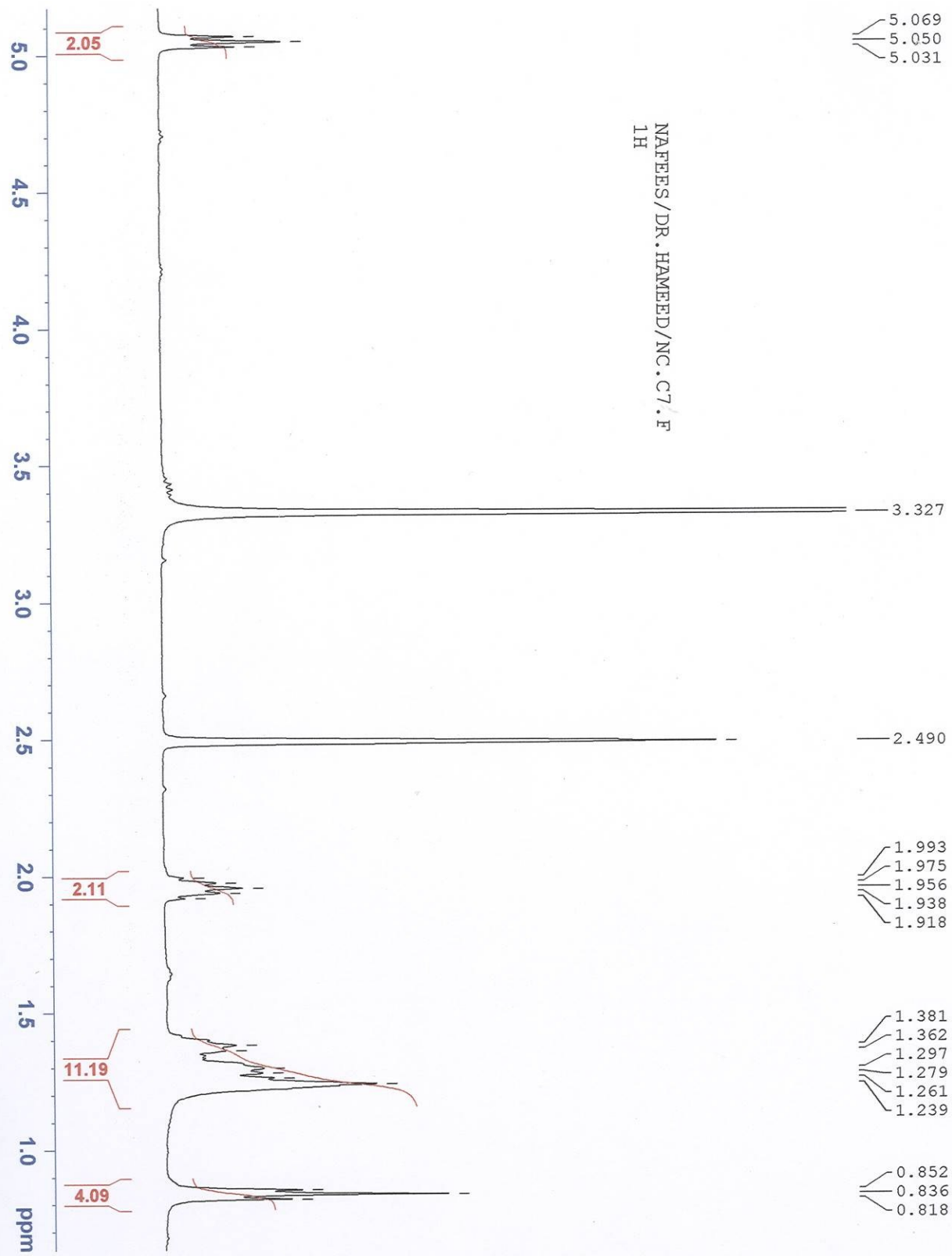
Method	KFT Ip01
Determination time	2015-05-26 11:46:34
Water=EP1*TITER*FCT/(C00*DIV)	
EP1	5.670000314712524E-01
TITER	4.586400000000000E+00
FCT	1.000000000000000E-01
C00	2.110000000000000E-02
DIV	1.000000000000000E+00



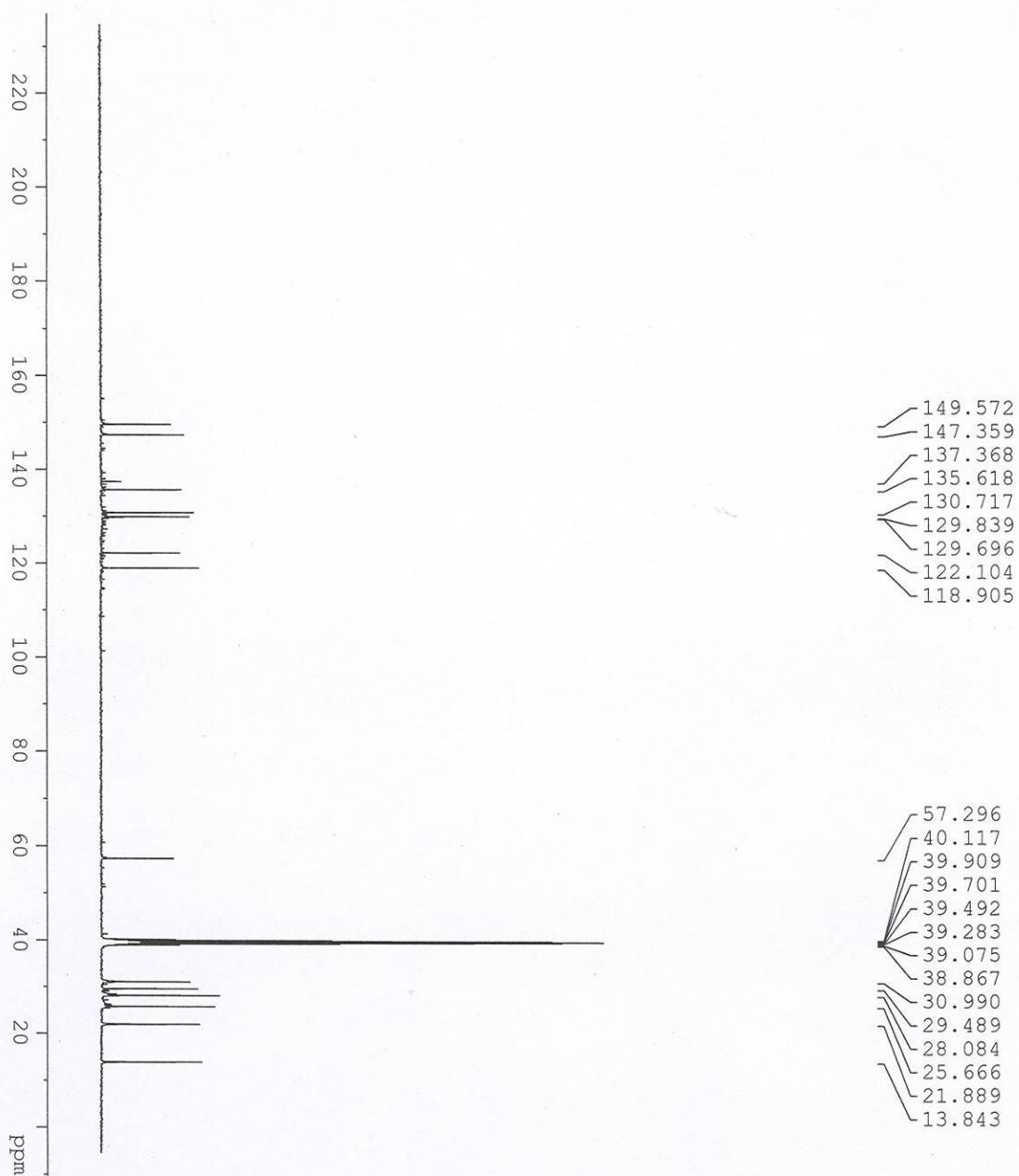
1-pentylquinolin-1-ium fluoride







NAFEES/DR.HAMEED/NF.C7.F
BB



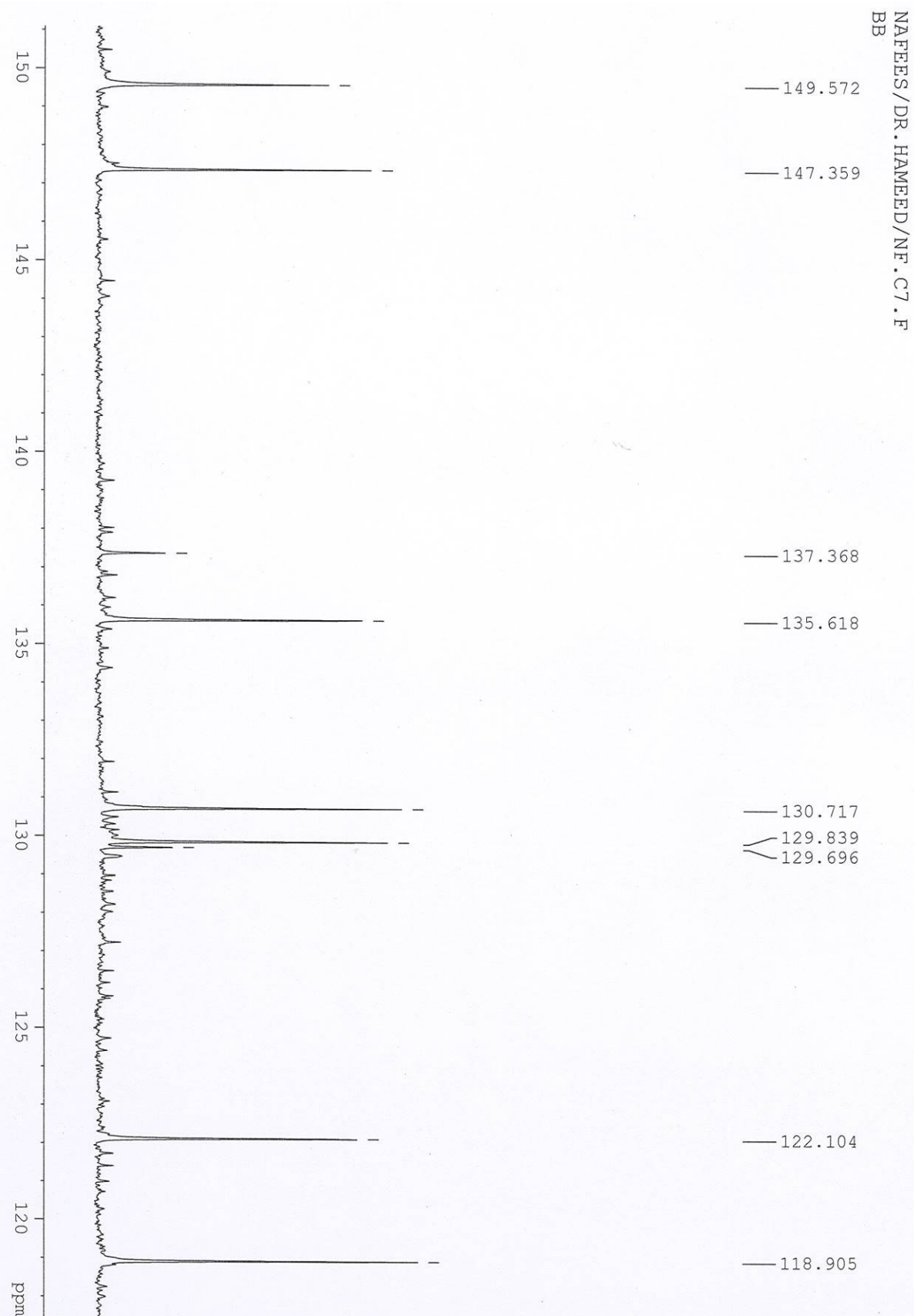
AVANCE 400
LAB NO 117

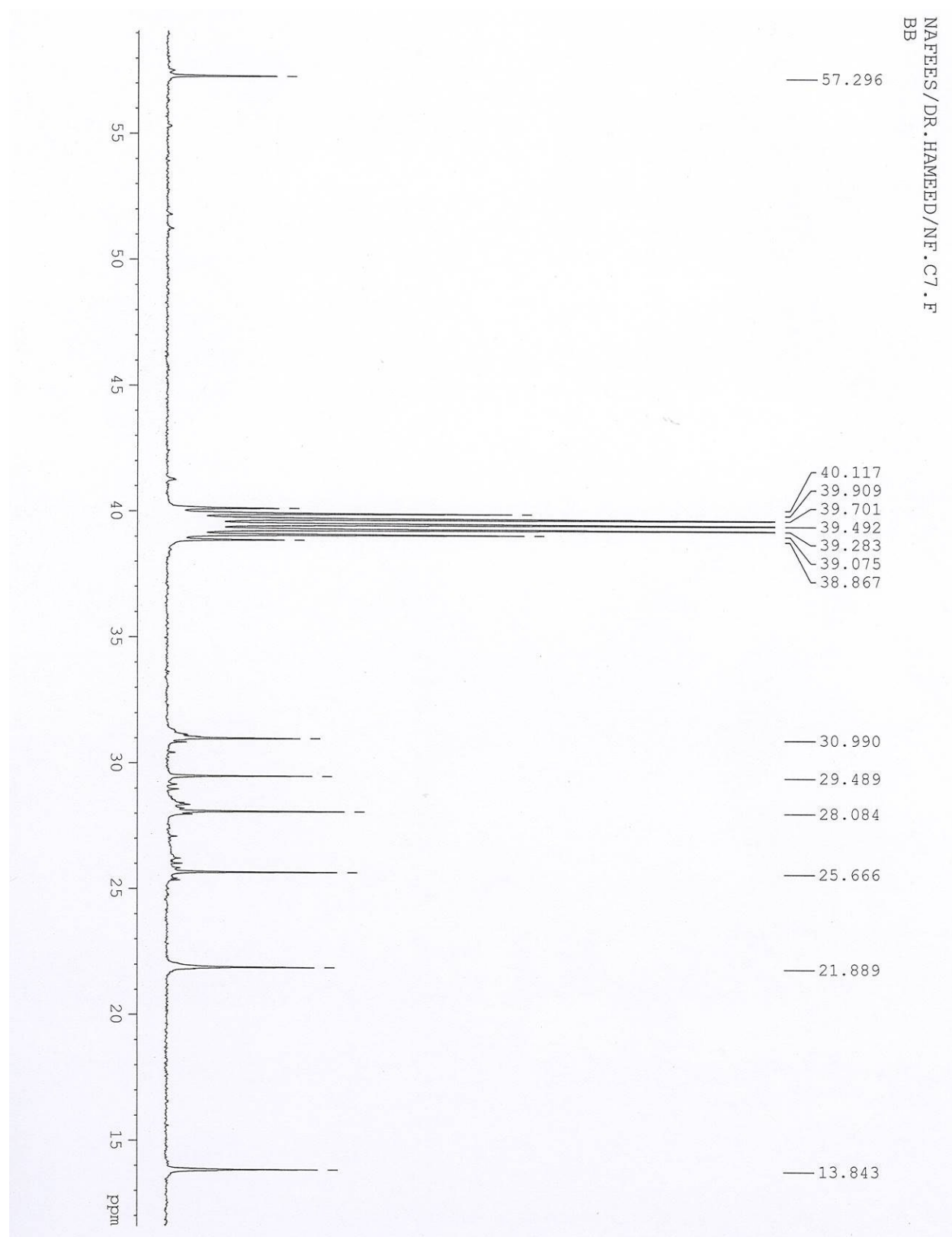
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EXPNO     1
PROCNO    1
Date_     20141227
Time      10.26
INSTRUM   spect
PROBHD    5 mm DUL 13C-1
PULPROG   zgpg
TD         32768
SOLVENT    DMSO
NS         18432
DS         2
SMH        24154.590 Hz
FIDRES     0.737140 Hz
AQ         0.6783476 sec
RG         32768
DE         20.700 usec
TE         300.0 K
D1         1.50000000 sec
D11        0.03000000 sec
TD0        18

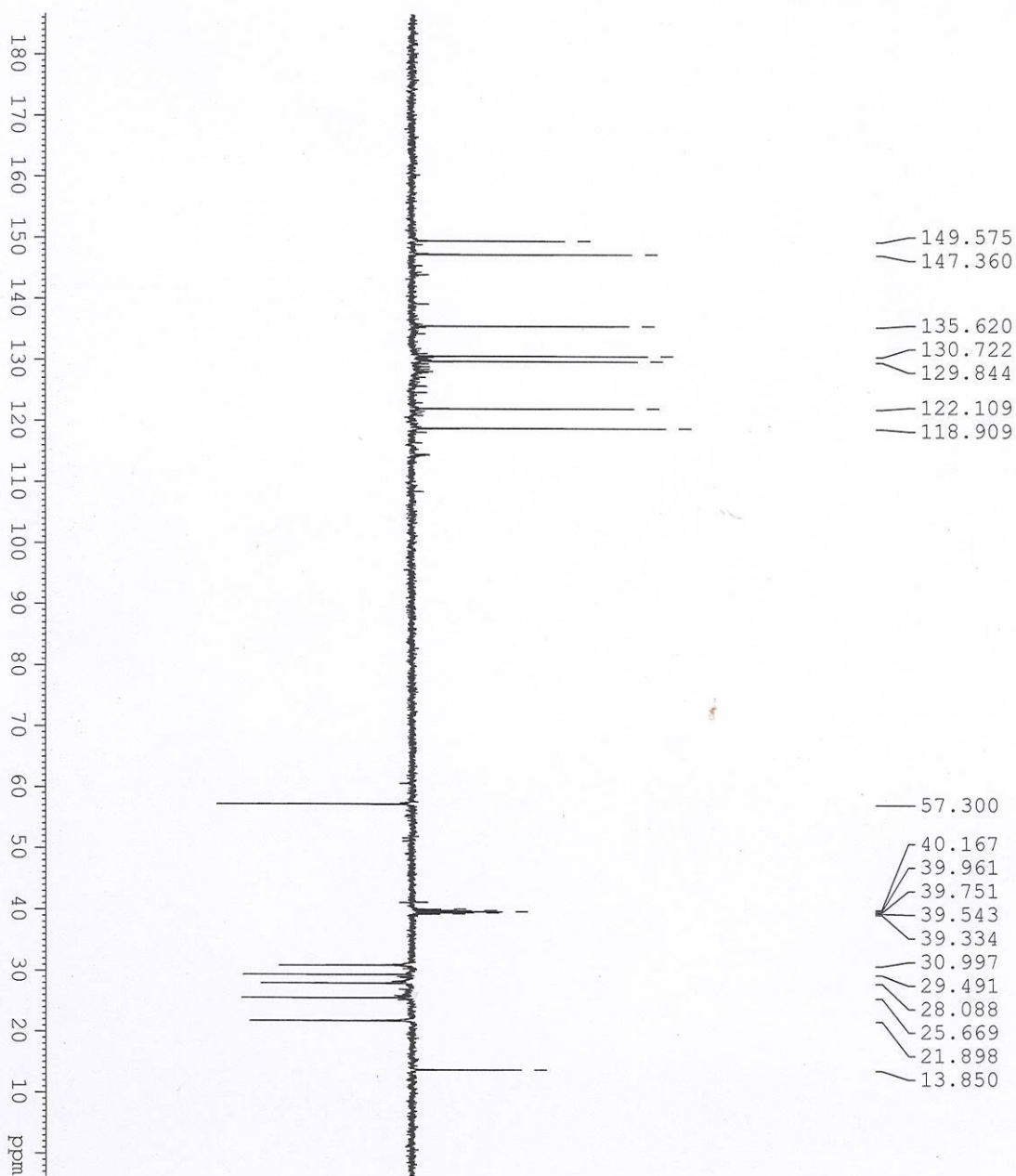
===== CHANNEL F1 =====
NUC1       13C
P1         8.10 usec
PL1        7.00 dB
SFO1       100.6243395 MHz

===== CHANNEL F2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        0.00 dB
PL12       20.00 dB
PL13       22.00 dB
SFO2       400.1324008 MHz
SI         16384
SF         100.6128207 MHz
WDW         EM
SSB         0
LB         1.50 Hz
GB         0
PC         1.00
  
```





NAFEES/DR.HAMEED/NF.C7.F
DEPT 135



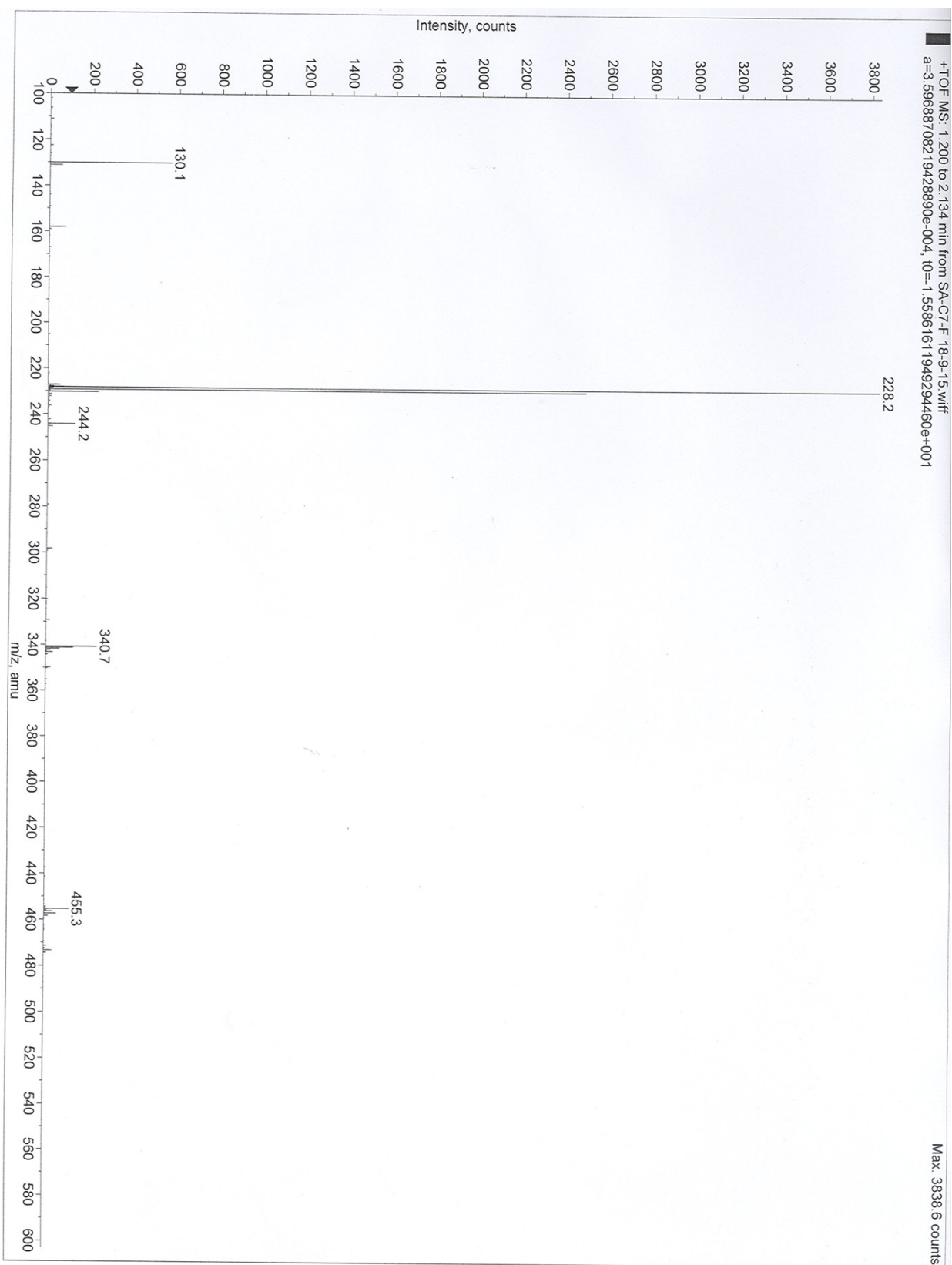
AVANCE 400
LAB NO 117

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NAME          dec27-14
EXPNO         2
PROCNO        1
Date_         20141227
Time          21.58
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       dept135
TD            32768
SOLVENT       DMSO
NS            9216
DS            2
SWH           19157.088 Hz
FIDRES        0.584628 Hz
AQ            0.8552948 sec
RG            32768
DW            26.100 usec
DE            6.50 usec
TE            300.0 K
CNST2         145.0000000
D1            1.500000000 sec
D2            0.00344828 sec
D12           0.00002000 sec
TD0           9

===== CHANNEL f1 =====
NUC1          13C
P1            8.10 usec
P2            16.20 usec
PL1           7.00 dB
SFO1          100.6220254 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
P3            9.80 usec
P4            19.60 usec
PCPD2         80.00 usec
PL2           0.00 dB
PL12          20.00 dB
SFO2          400.1320007 MHz
SI            32768
SF            100.6128207 MHz
WDW           EM
SSB           0
LB            1.50 Hz
GB            0
PC            1.40
  
```

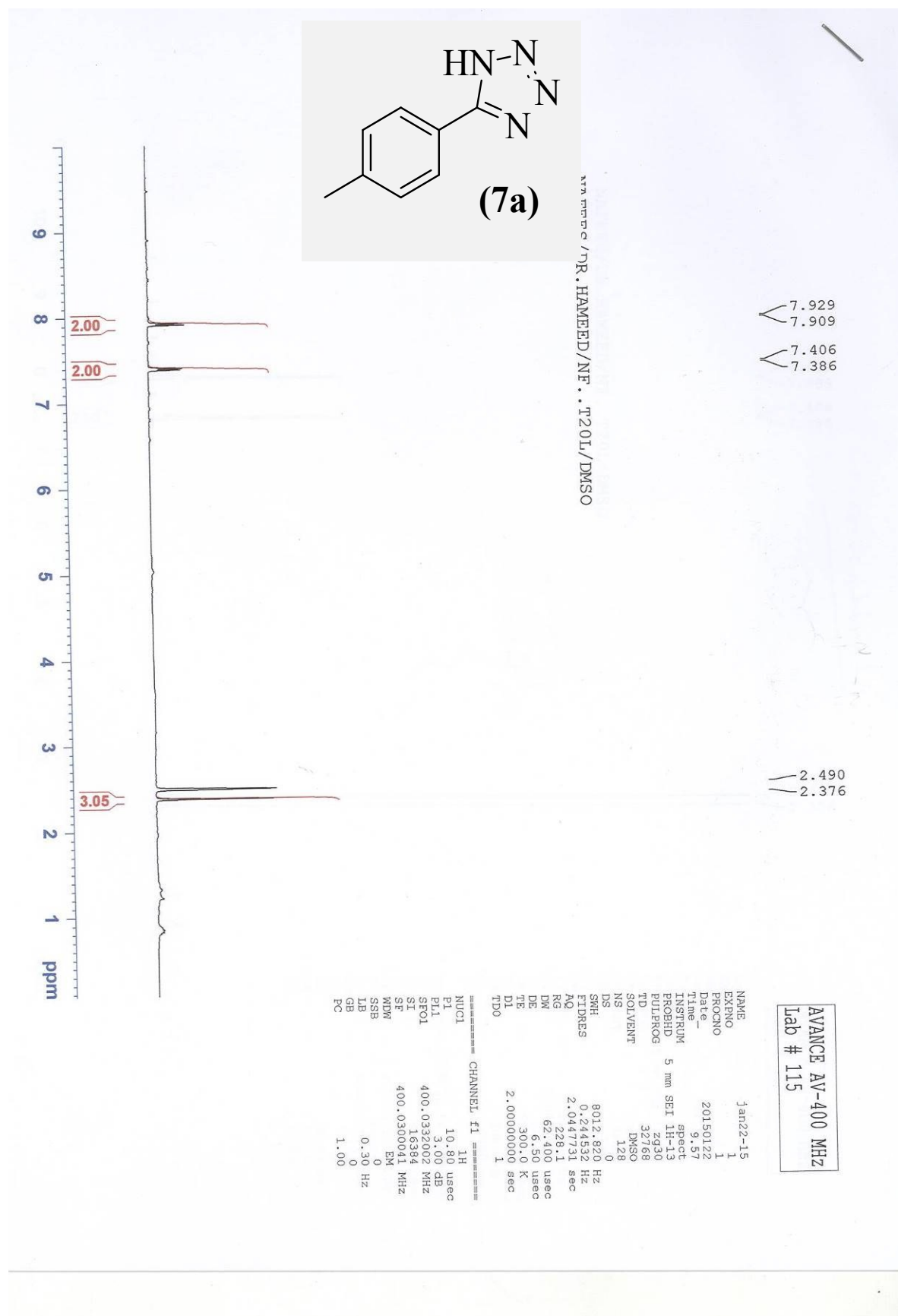



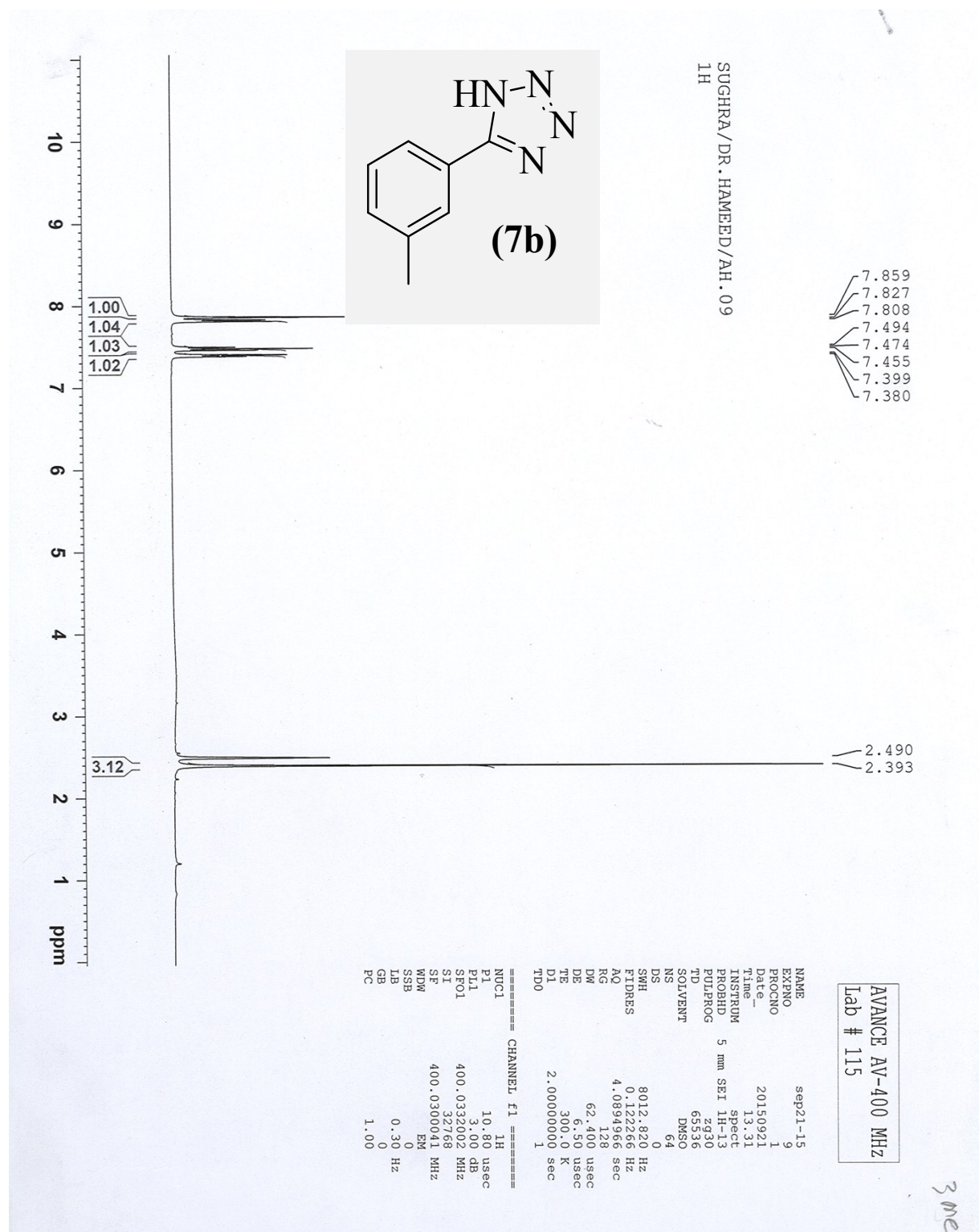
Elemental composition calculator

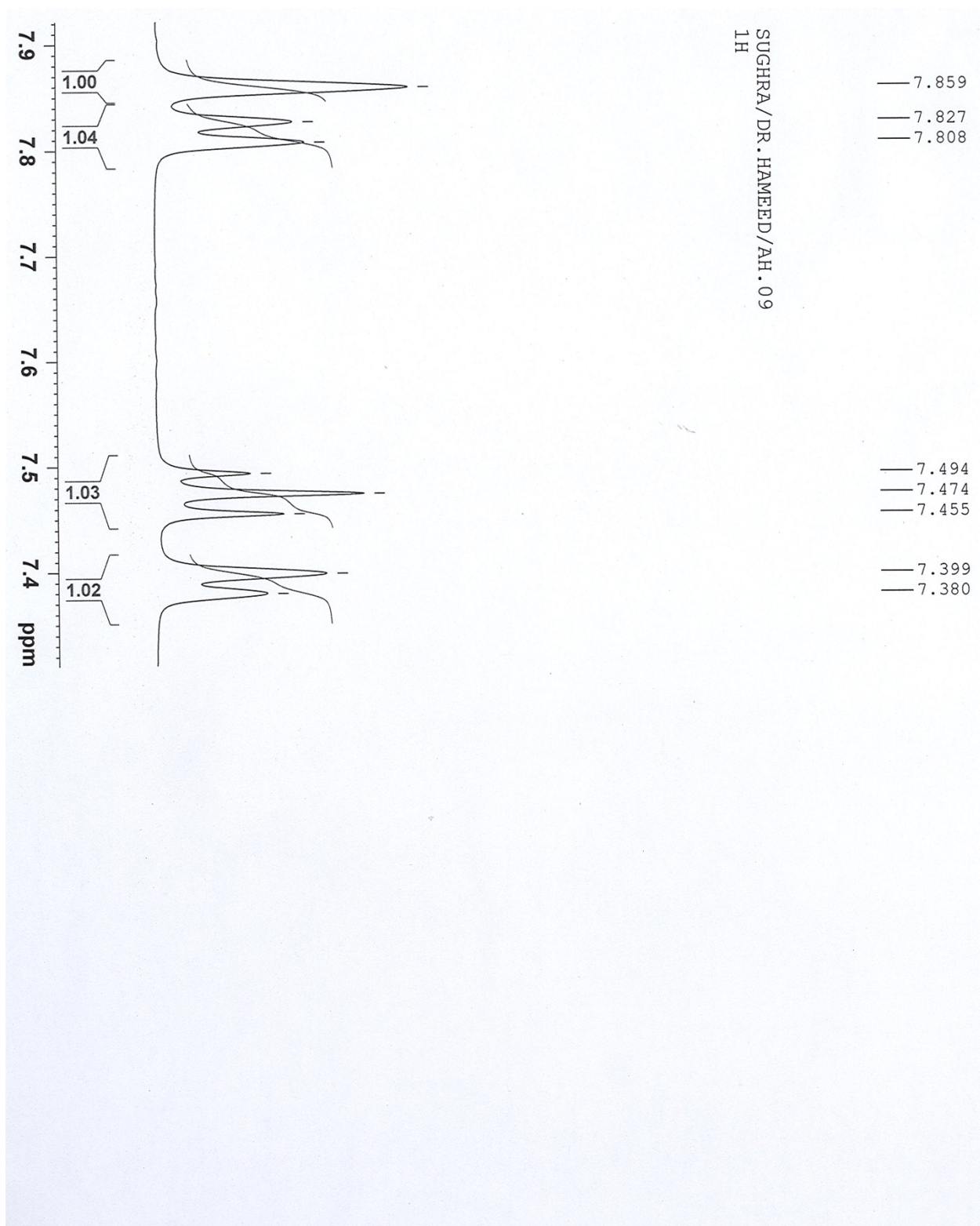
Target m/z: +228.1752 amu
 Tolerance: +10.0000 ppm
 Result type: Elemental
 Max num of results: 10
 Min DBE: -0.5000 Max DBE: +100.0000
 Electron state: OddAndEven
 Num of charges: 0
 Add water: N/A
 Add proton: N/A
 File Name: SA-C7-F 18-9-15.wiff

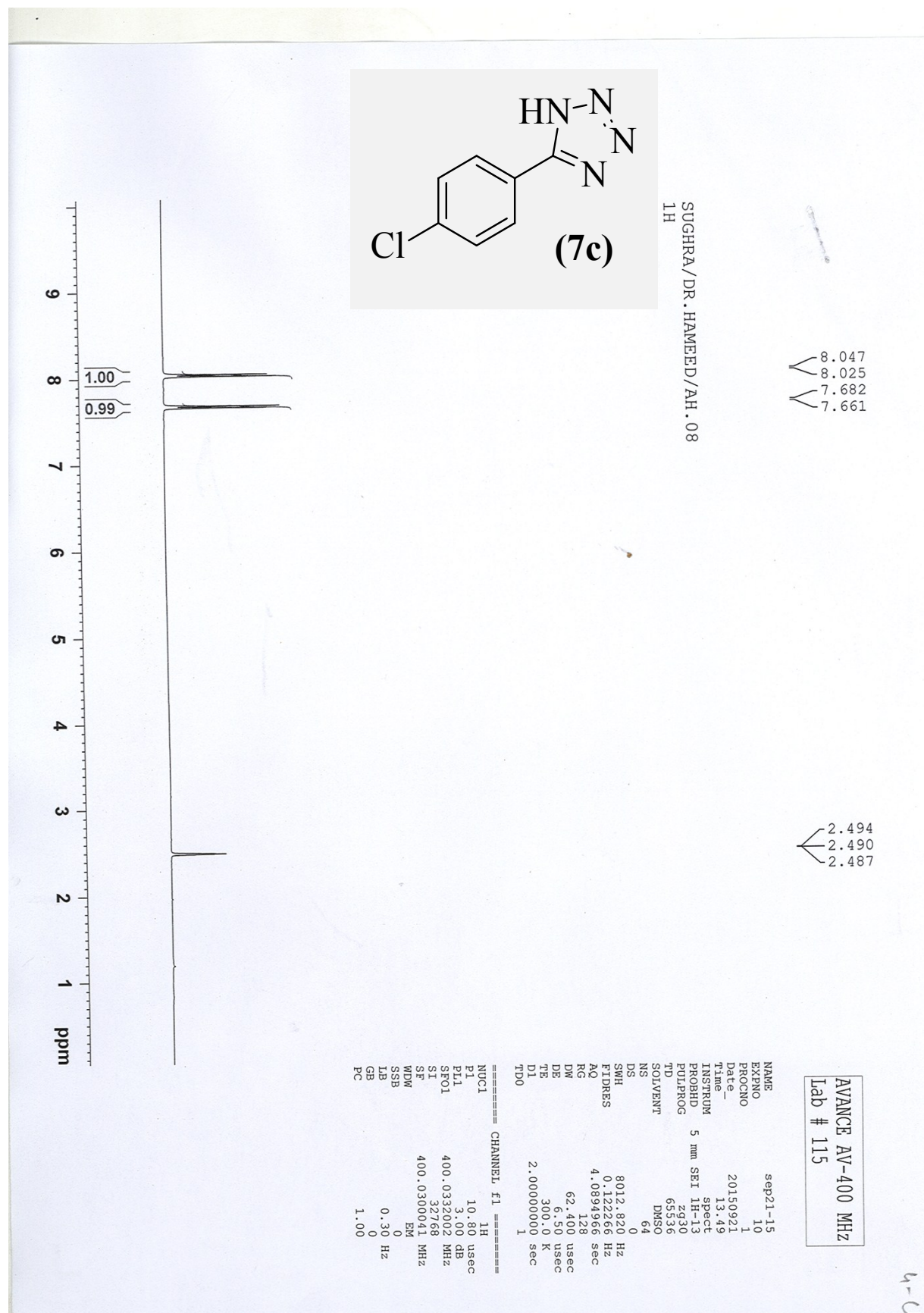
	Elements	Min Number	Max Number
1	C	0	18
2	F	0	1
3	H	0	30
4	N	0	2

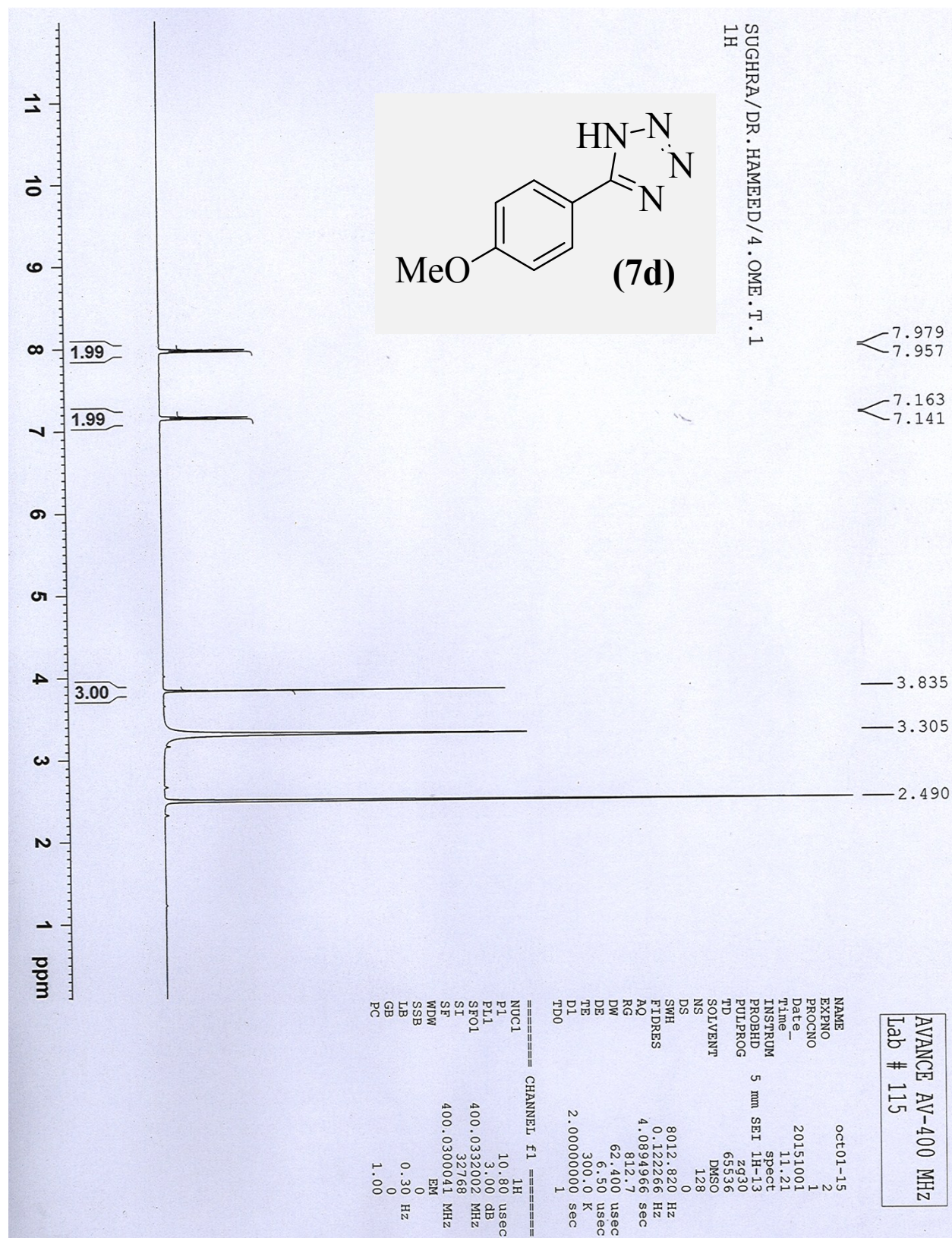
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1	C16 H22 N	228.1752	-0.0248	-0.1090	6.5

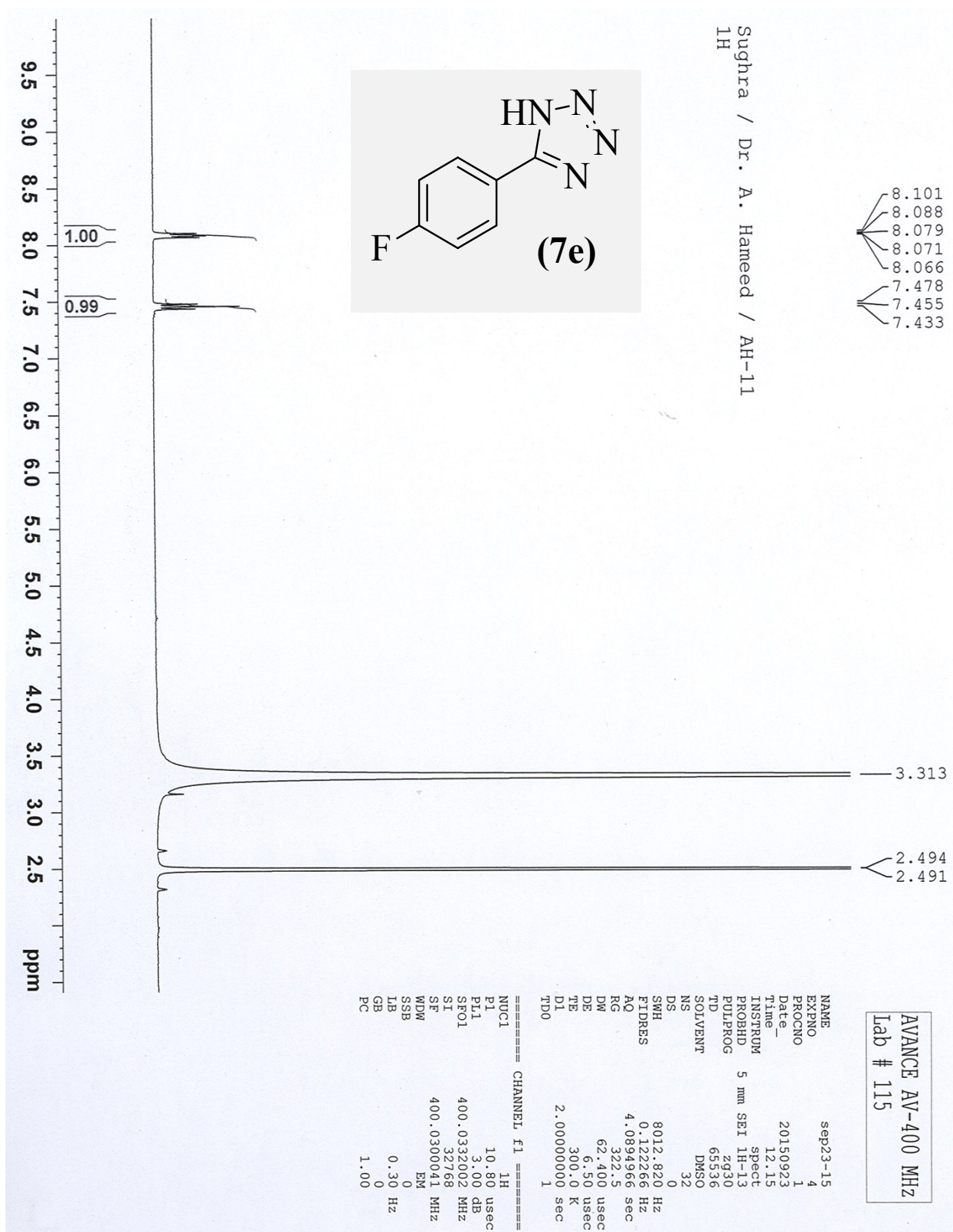


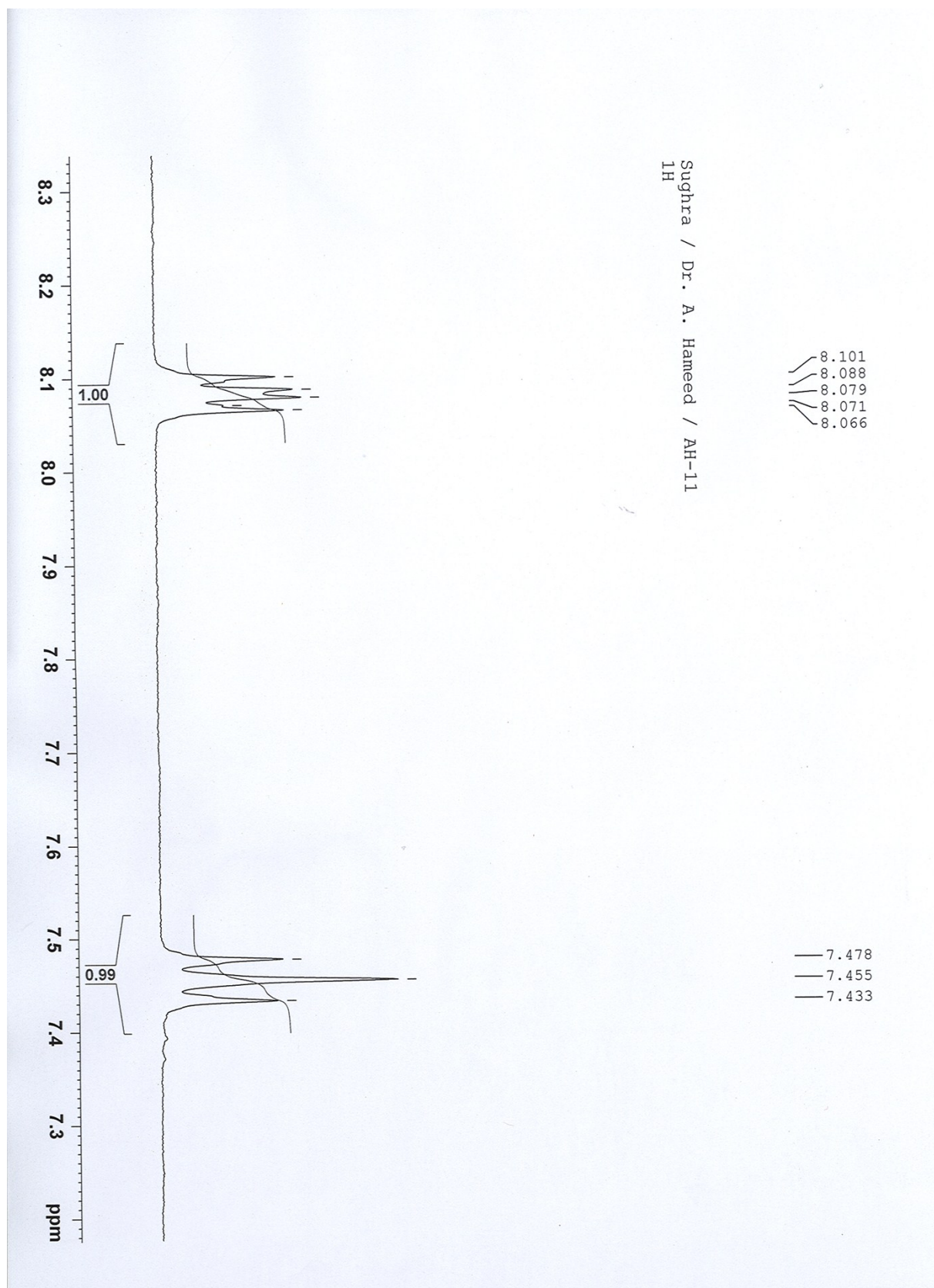


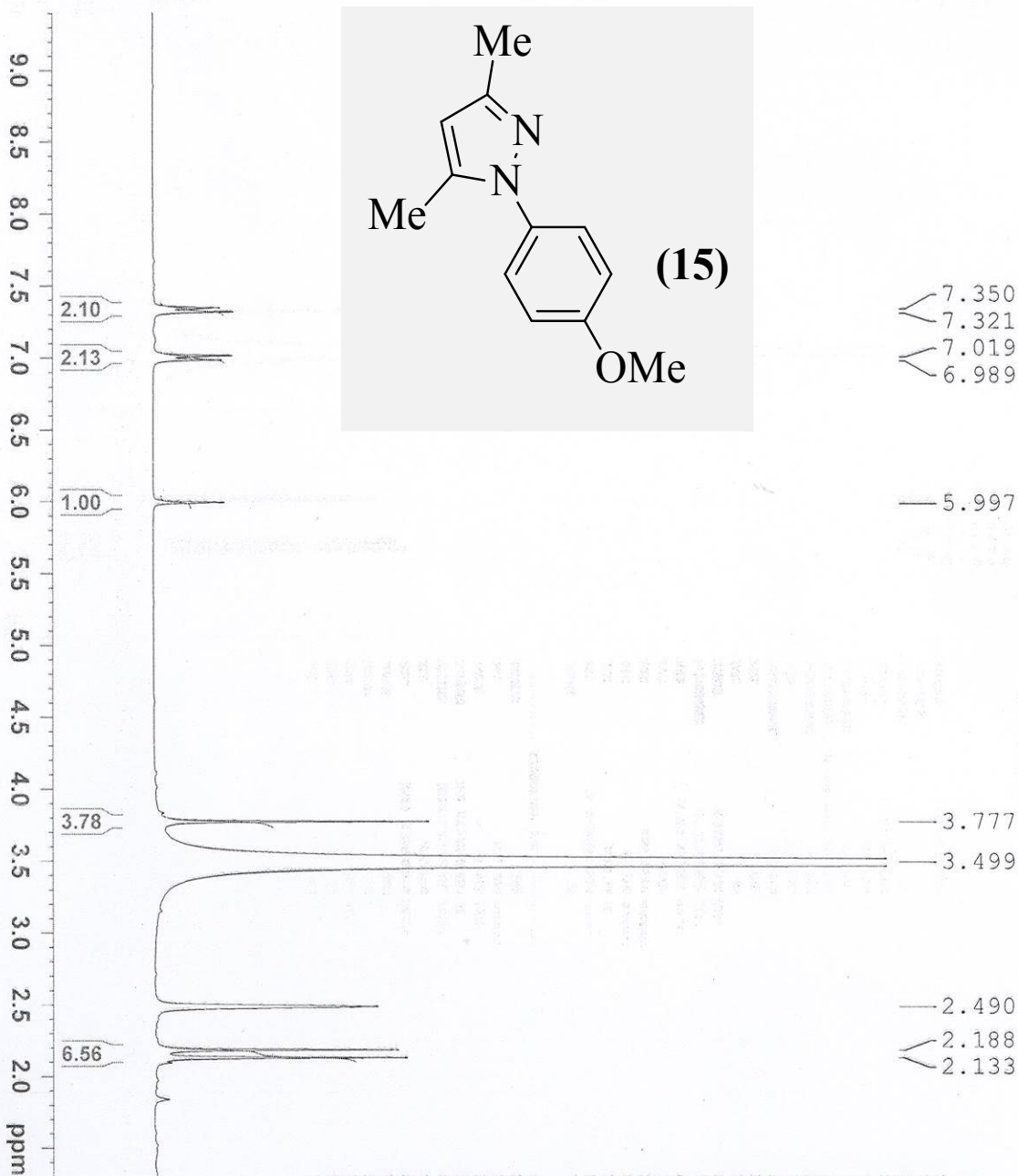
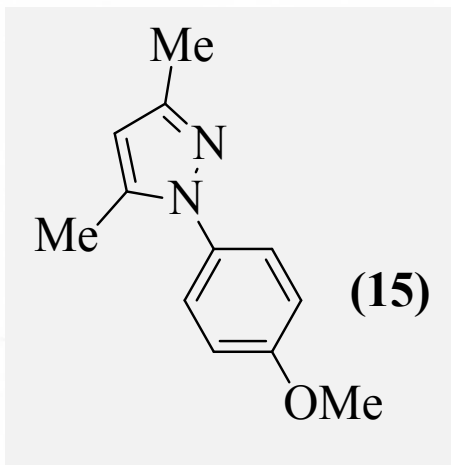








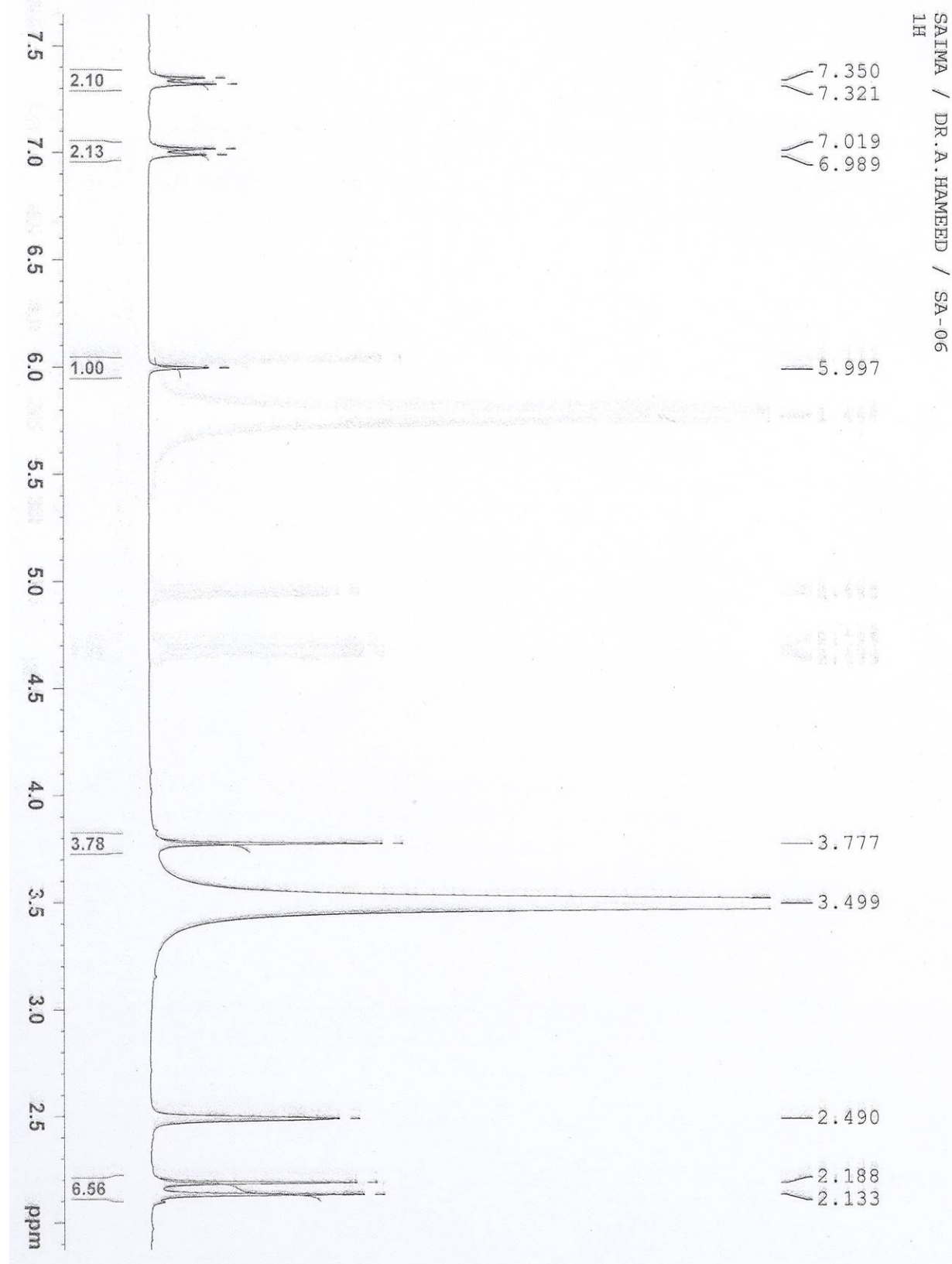


SAIMA / DR. A. HAMEED / SA-06
1HAVANCE AV - III
300 MHz, LAB # 116

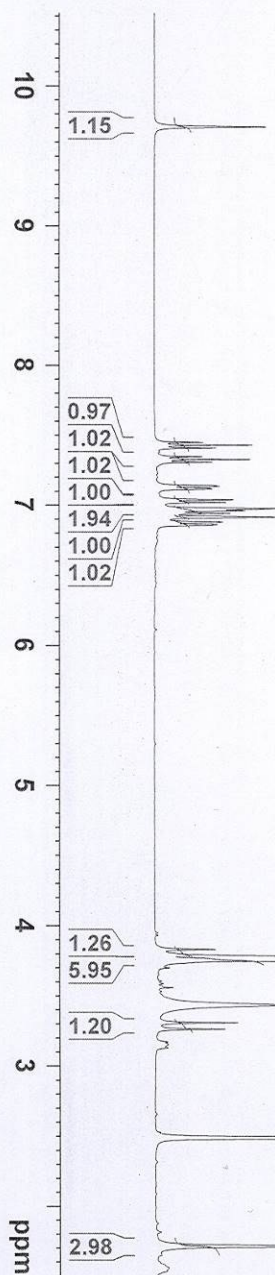
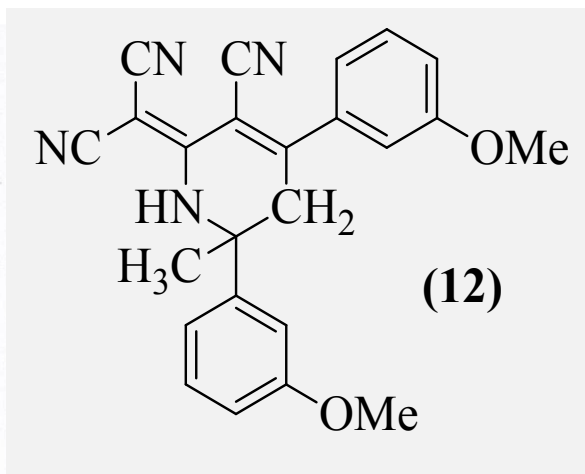
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NAME          may19-15
EXPNO         1
PROCNO        1
Date_         20150518
Time_         23.20
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            32768
SOLVENT       DMSO
NS            64
DS            0
SMH           6186.119 Hz
FIDRES        0.168846 Hz
AQ            2.6477044 sec
RG            203
DM            80.800 usec
DE            6.50 usec
TE            300.0 K
D1            2.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            12.50 usec
PL1           0.00 dB
PL1W          13.16228485 W
SFO1          300.1318534 MHz
SI            32768
SF            300.1300042 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.00
  
```

S. Tazeen / Dr. A. Hameed / TZ-DIL-IL
¹H

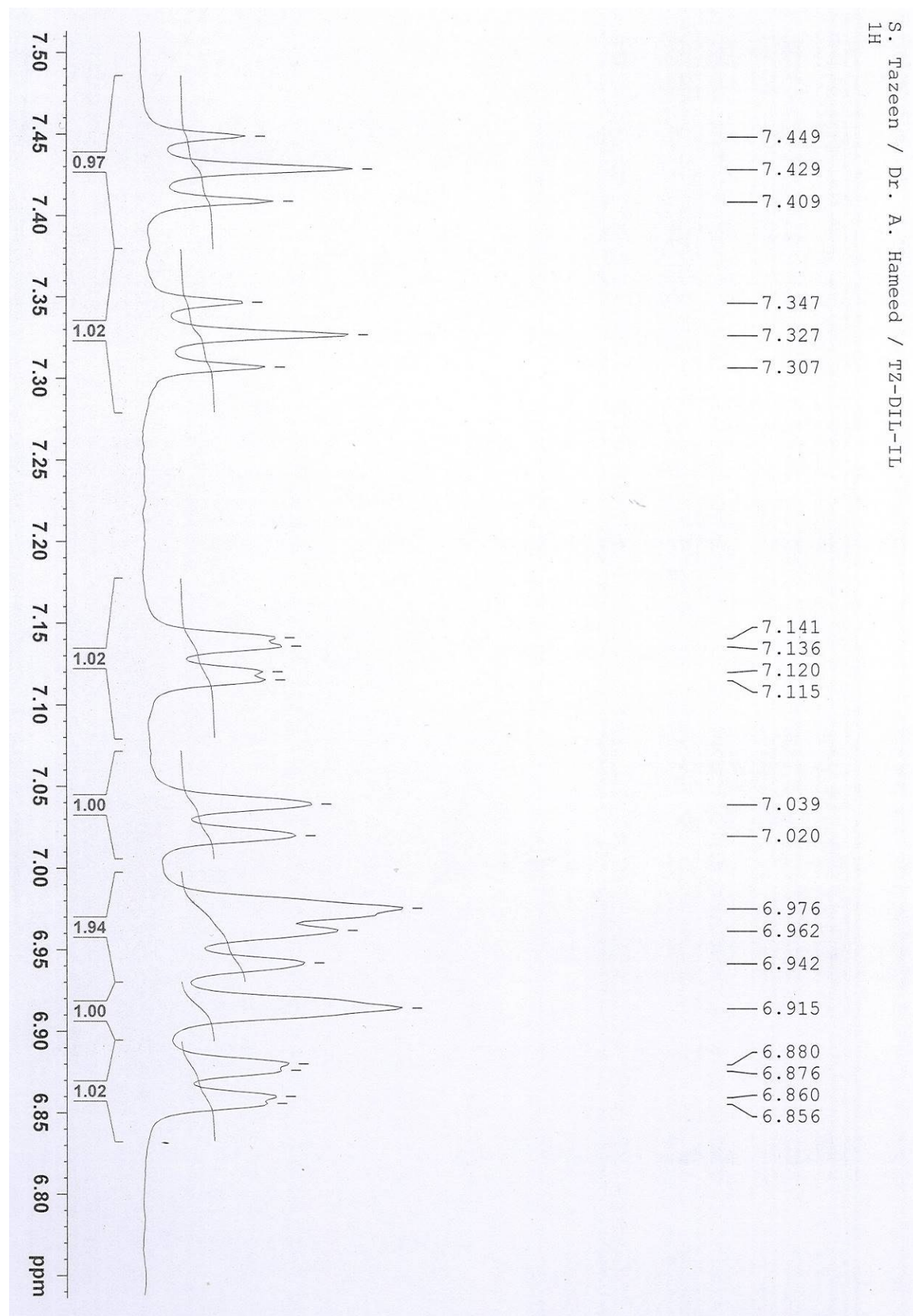


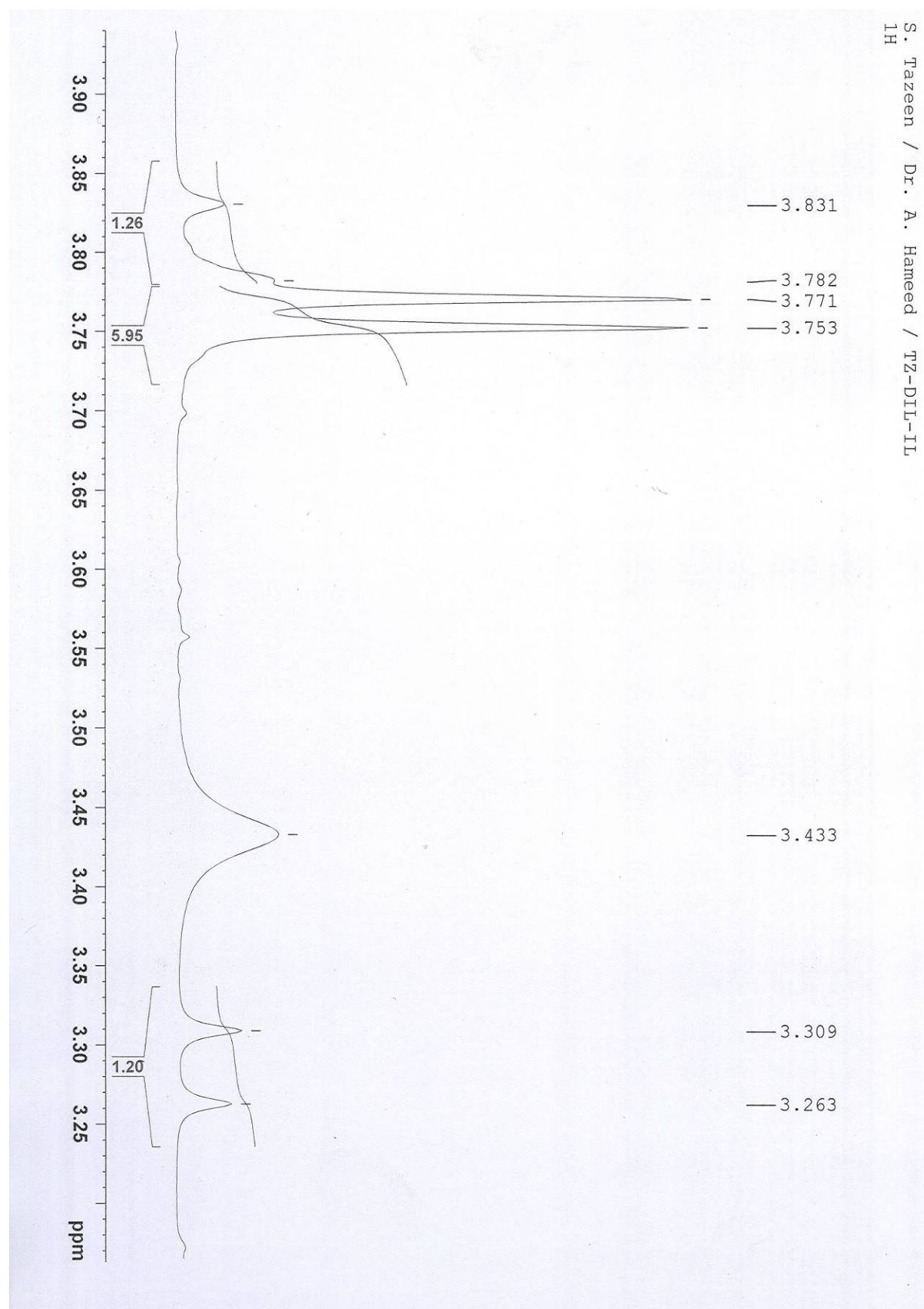
AVANCE-III
 AV-400 MHz (A)
 LAB # 109

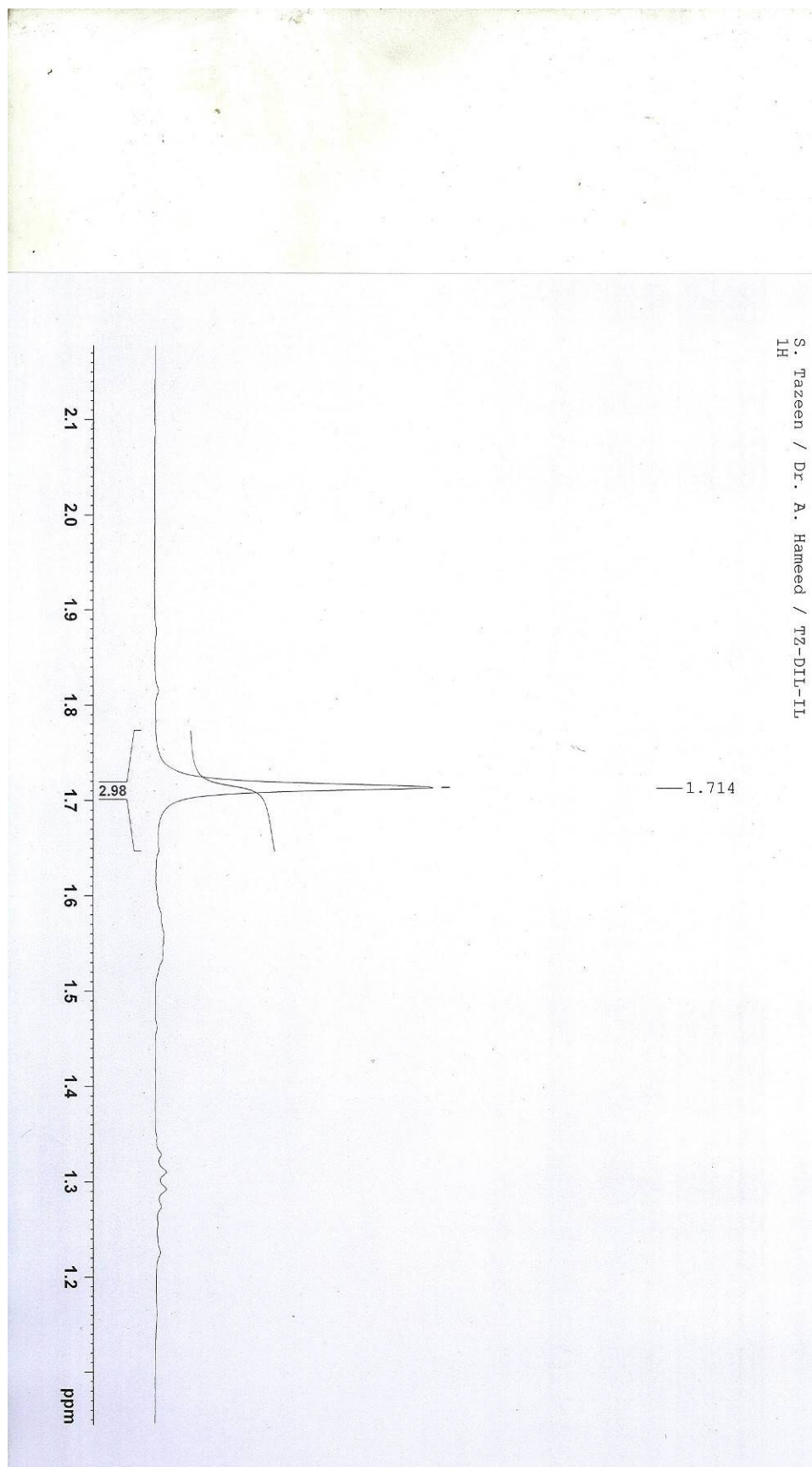
Current Data Parameters
 NAME jun12-15
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150612
 Time 15.22
 INSTRUM spect
 PROBD 5 mm PABBO BB/
 PULPROG zg30
 TD 32768
 SOLVENT DMSO
 NS 16
 DS 0
 SWH 8223.685 Hz
 FIDRES 0.250967 Hz
 AQ 1.9923444 sec
 RG 99.58
 DW 60.800 usec
 DE 6.50 usec
 TE 302.1 K
 D1 2.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 ¹H
 P1 9.70 usec
 PL1 17.00000000 W
 SFO1 400.2332018 MHz
 F2 - Processing parameters
 SI 16384
 SF 400.2300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00







S. Tazeen / Dr. A. Hameed / TZ-DIL-IL
BB

AVANCE -III
AV-400 MHz (A)
LAB # 109

168.61
159.45
159.09
157.72
144.60
136.75
130.11
129.77
120.44
117.61
117.11
115.41
114.80
113.60
113.44
112.45
111.46
101.18
56.95
55.29
55.14
49.47
43.79
40.13
39.92
39.71
39.50
39.29

Current Data Parameters
NAME jun12-15
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters

Date_ 20150613
Time 14.53
INSTRUM 5 mm PABBO BB/
PROBHD spect
PULPROG zgpg
TD 32768
SOLVENT DMSO
NS 20480
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 196.51
DW 20.800 usec
DE 6.50 usec
TE 302.9 K
D1 1.5000000 sec
D11 0.0300000 sec
TDO 20

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

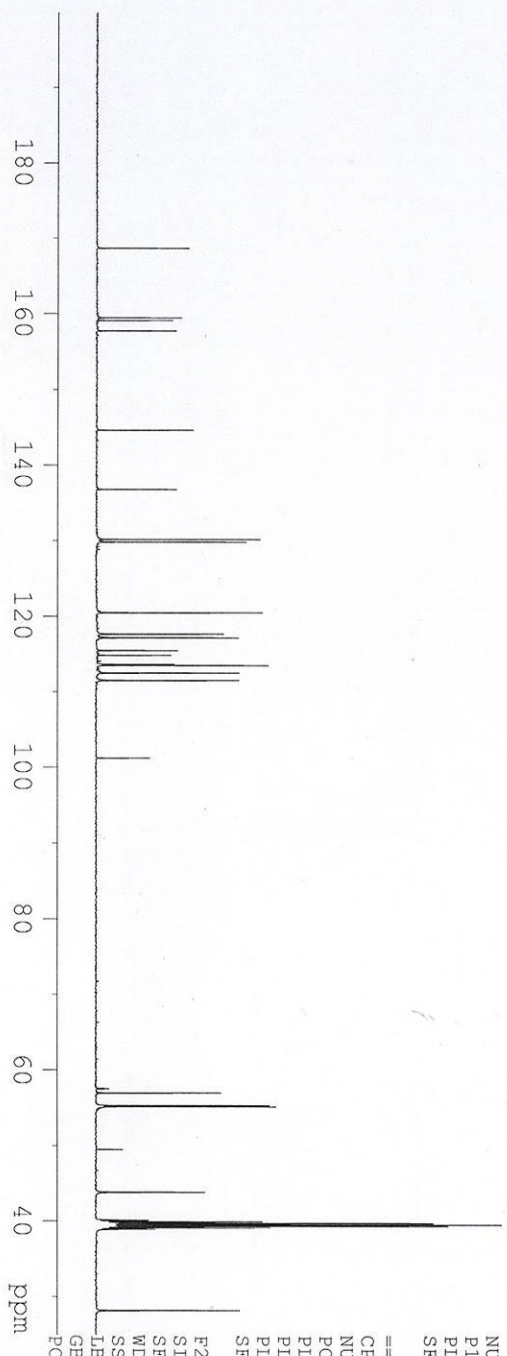
NUC1 13C
P1 10.20 usec
PLW1 70.0000000 W
SFO1 100.6494874 MHz

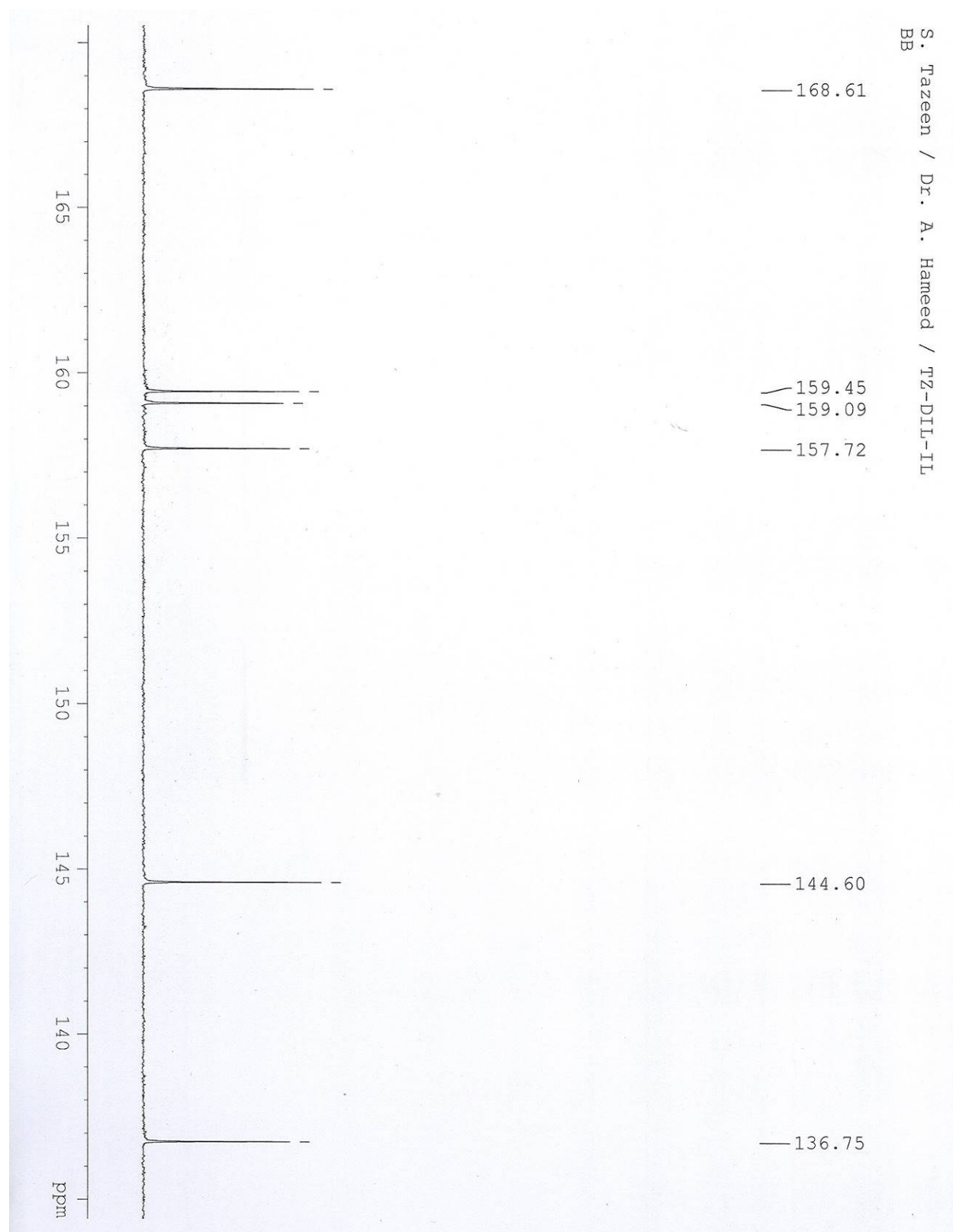
===== CHANNEL f2 =====

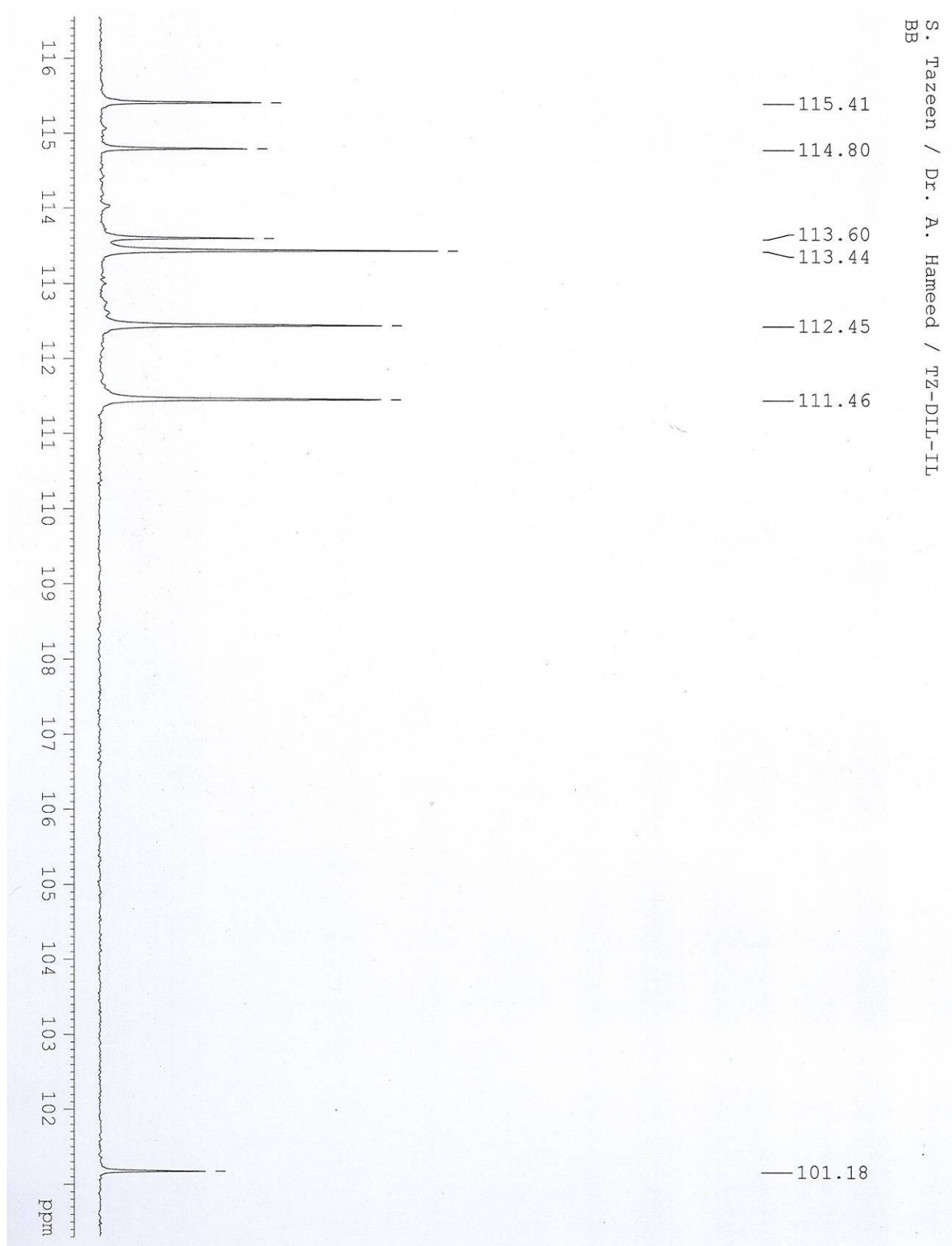
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 17.0000000 W
PLW12 0.1974699 W
PLW13 0.1599500 W
SFO2 400.2316009 MHz

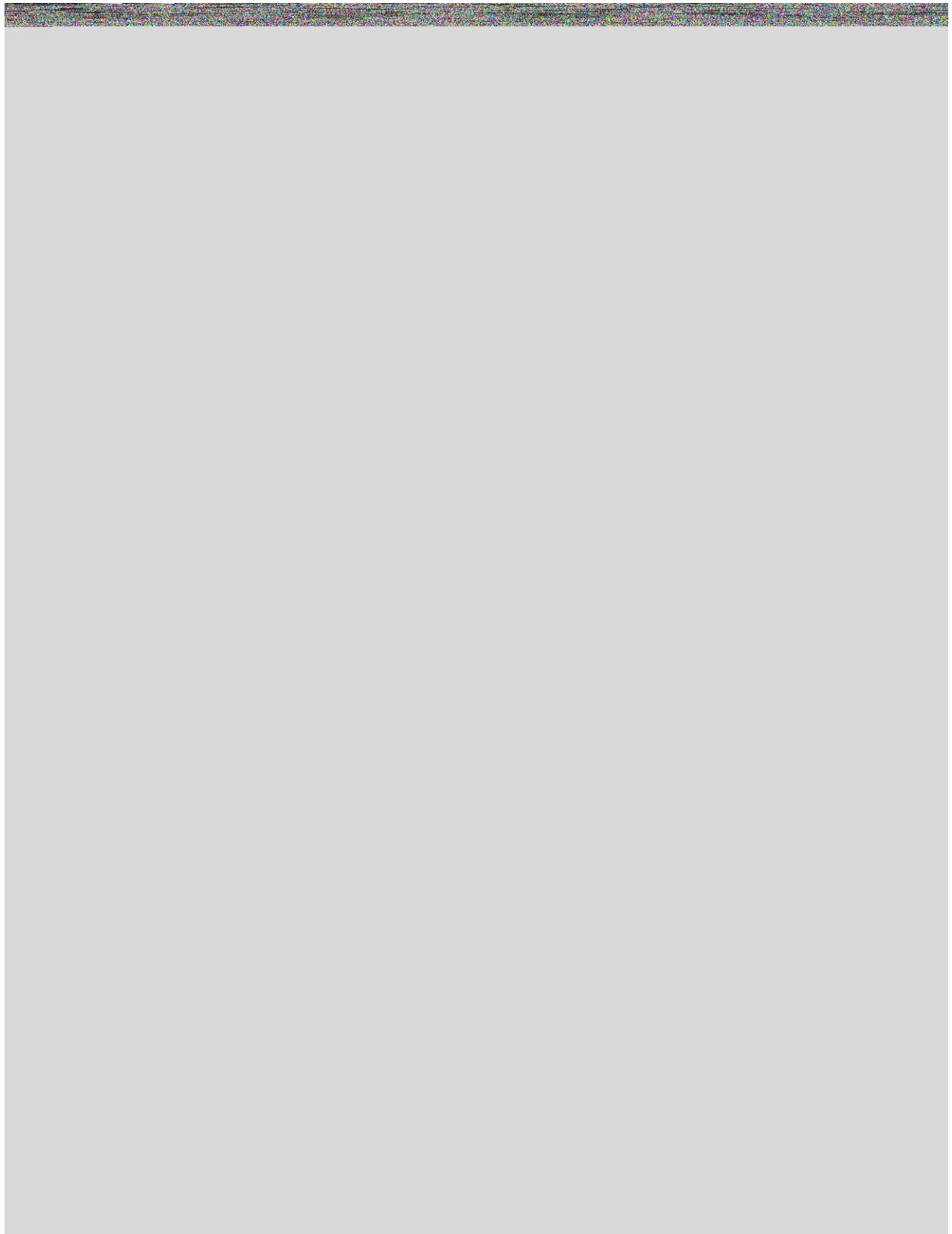
F2 - Processing parameters

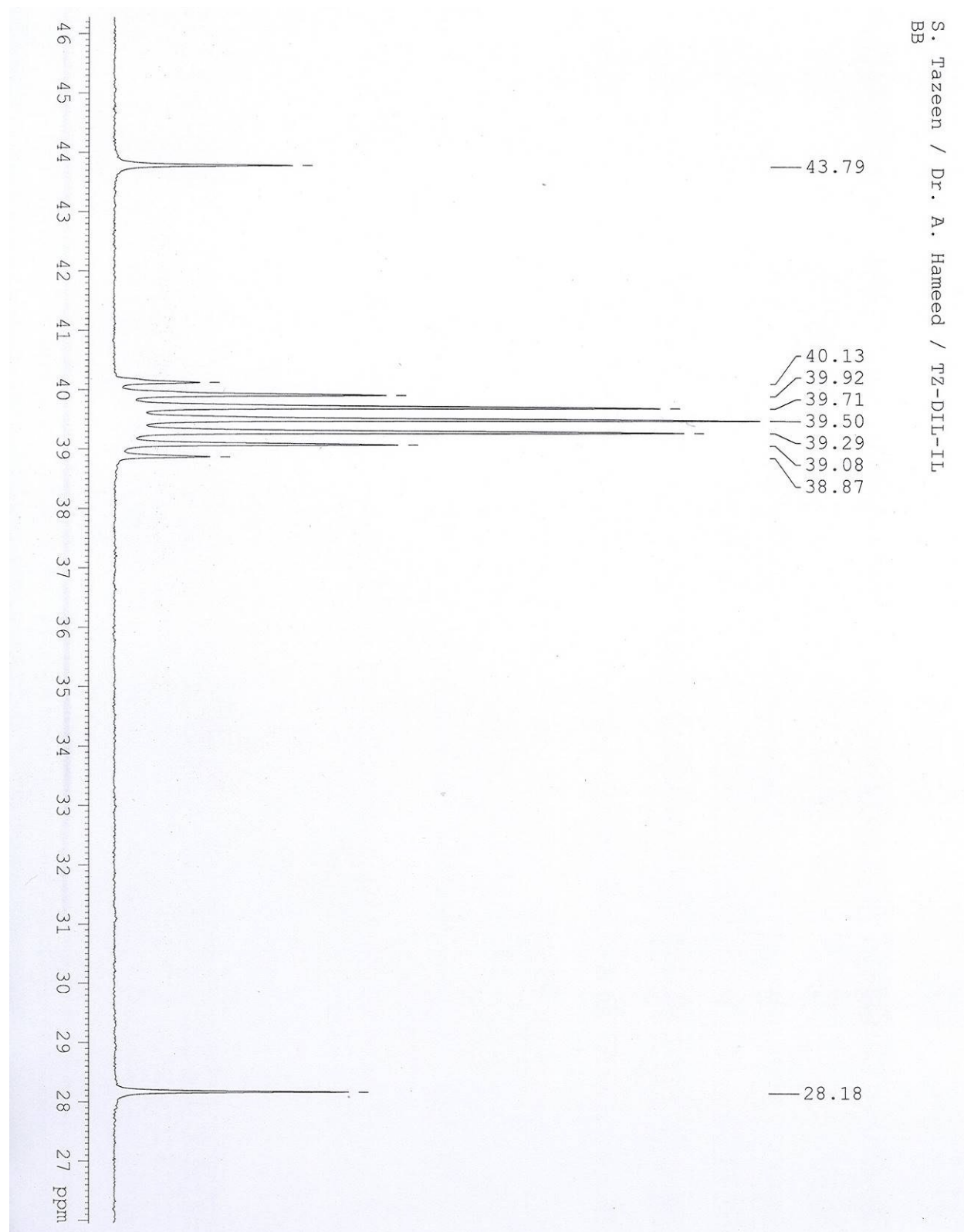
SI 32768
SF 100.6379667 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40











S. Tazeen / Dr. A. Hameed / TZ-DIL-IL
DEPT 135

130.10
129.76
120.43
117.61
117.11
113.44
112.44
111.46

55.29
55.13
43.79
39.96
39.76
39.55
28.18

AVANCE-III
AV-400 MHz (A)
LAB # 109

Current Data Parameters
NAME Jun12-15
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150614

Time 3.44

INSTRUM spect

PROBHD 5 mm PABBO BB/

PULPROG dept135

TD 32768

SOLVENT DMSO

NS 10240

DS 8

SWH 18115.941 Hz

FIDRES 0.552855 Hz

AQ 0.9044468 sec

RG 196.51

DW 27.600 usec

DE 6.50 usec

TE 302.2 K

CNST2 145.0000000

D1 1.50000000 sec

D2 0.00344828 sec

D12 0.00002000 sec

TD0 10

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

NUC1 13C

P1 10.20 usec

P2 20.40 usec

PLM1 70.00000000 W

SFO1 100.6469714 MHz

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

CPDPRG2 waltz16

NUC2 1H

P3 9.70 usec

P4 19.40 usec

PCPD2 90.00 usec

PLW2 17.00000000 W

PLM12 0.19746999 W

SFO2 400.2312800 MHz

F2 - Processing parameters

SF 16384

WDW 100.6379667 MHz

SSB EM

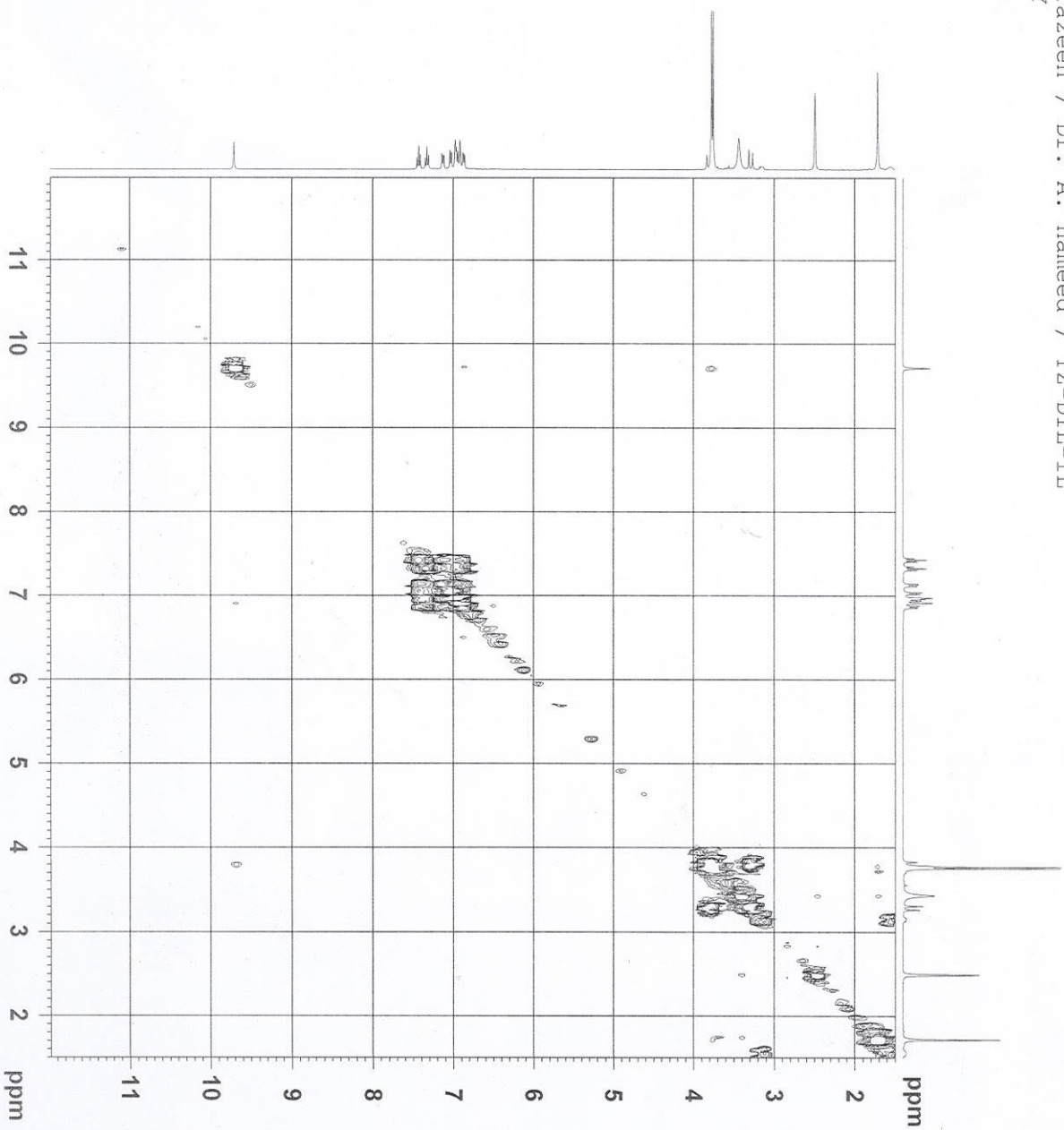
GB 0

LB 1.00 Hz

GB 0

170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

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COSY



AVANCE-III
AV-400 MHz (A)
LAB # 109

```

Current Data Parameters
NAME      jun12-15
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20150612
Time      15.22
INSTRUM   spect
PROBHD    5 mm PABBO BB/
PULPROG   cosyppqf
TD         1024
SOLVENT   DMSO
NS         16
DS         2
SWH        4807.692 Hz
FIDRES     4.65012 Hz
AQ         0.1065460 sec
RG         196.51
DE         104.000 usec
TE         302.1 K
DO         0.00000300 sec
D1         1.50000000 sec
D13        0.0000400 sec
D16        0.0002000 sec
INO        0.00020800 sec

===== CHANNEL f1 =====
NUC1       1H
P0         9.70 usec
PL1        9.70 usec
PLW1       17.00000000 W
SFO1       400.2324014 MHz

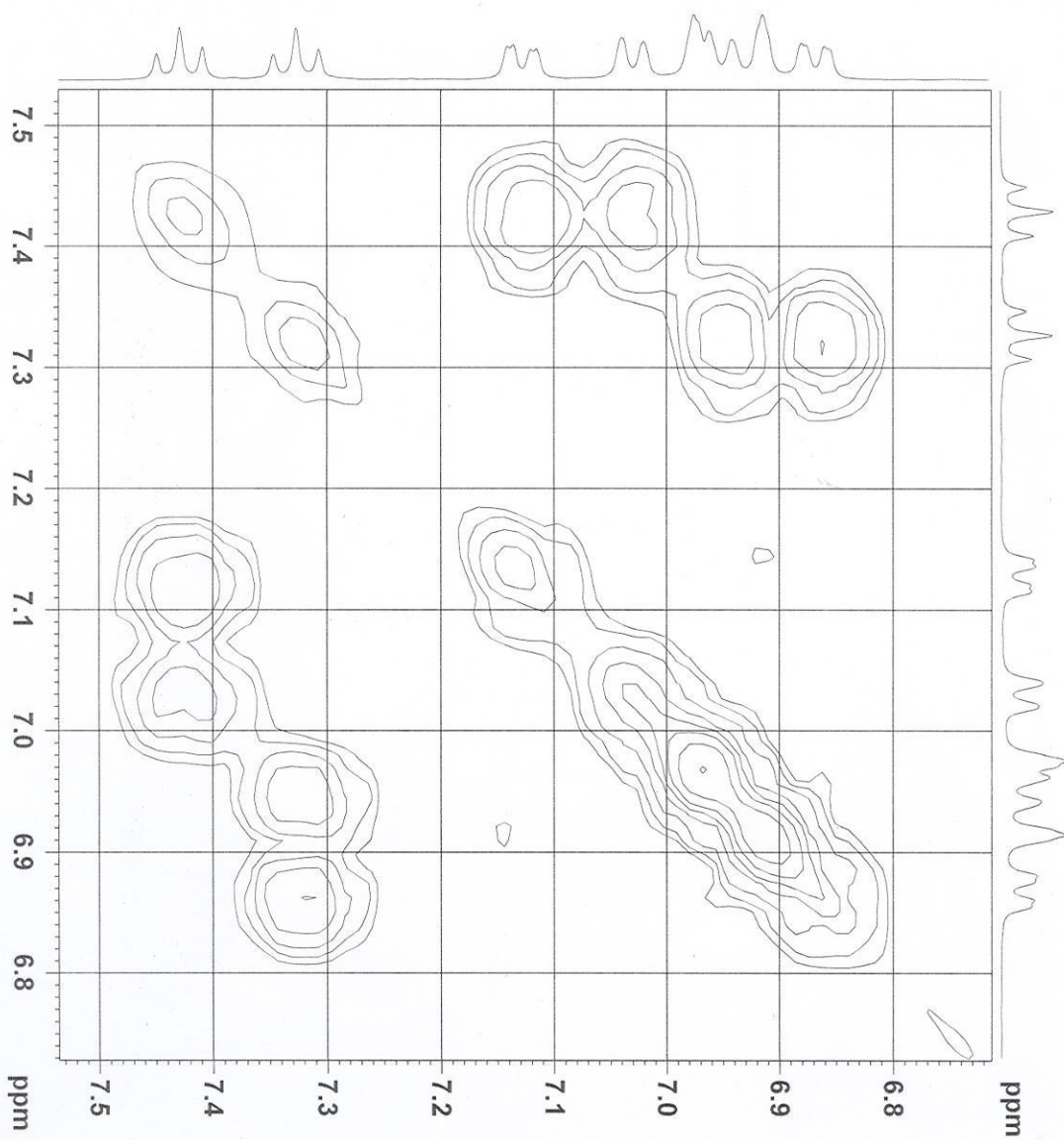
===== GRADIENT CHANNEL =====
GPRAM1     SMS210.100
GPZ1       10.00 %
PL6        1000.00 usec

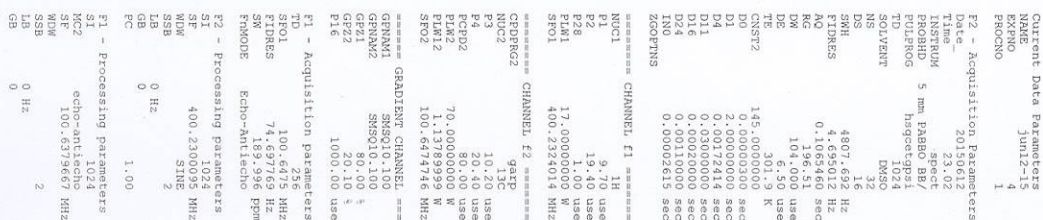
F1 - Acquisition parameters
TD          256
SFO1        400.2324 MHz
FIDRES      18.780046 Hz
SW          12.012 ppm
FMODE       QF

F2 - Processing parameters
SI          1024
SF          400.2300065 MHz
WDW         QSLINE
SSB         0 Hz
LB          0
GB          0
PC          1.00

F1 - Processing parameters
SI          128
MC2         QF
SF          400.2300095 MHz
WDW         States-TpPl
SSB         0 Hz
LB          0
GB          0
  
```

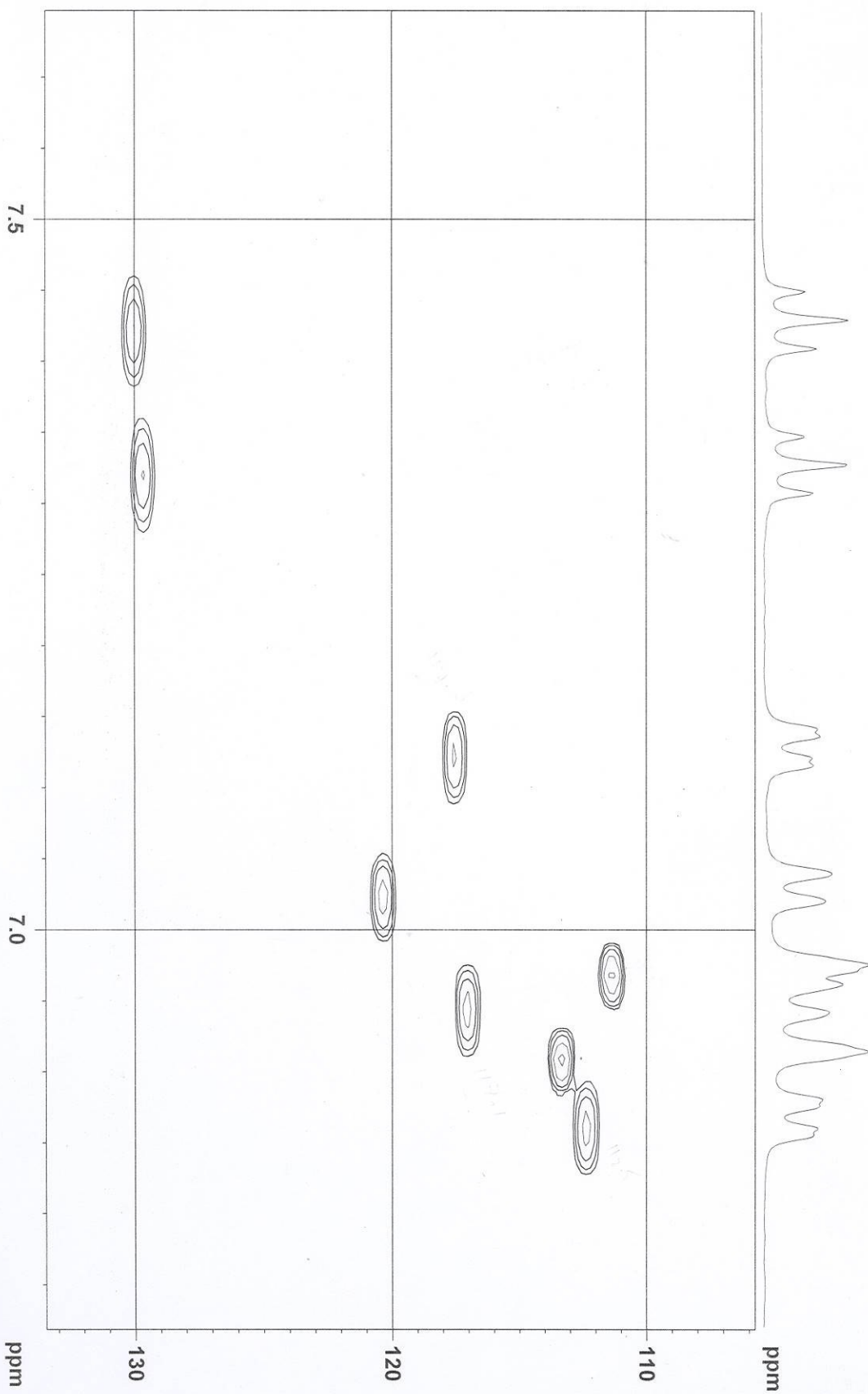
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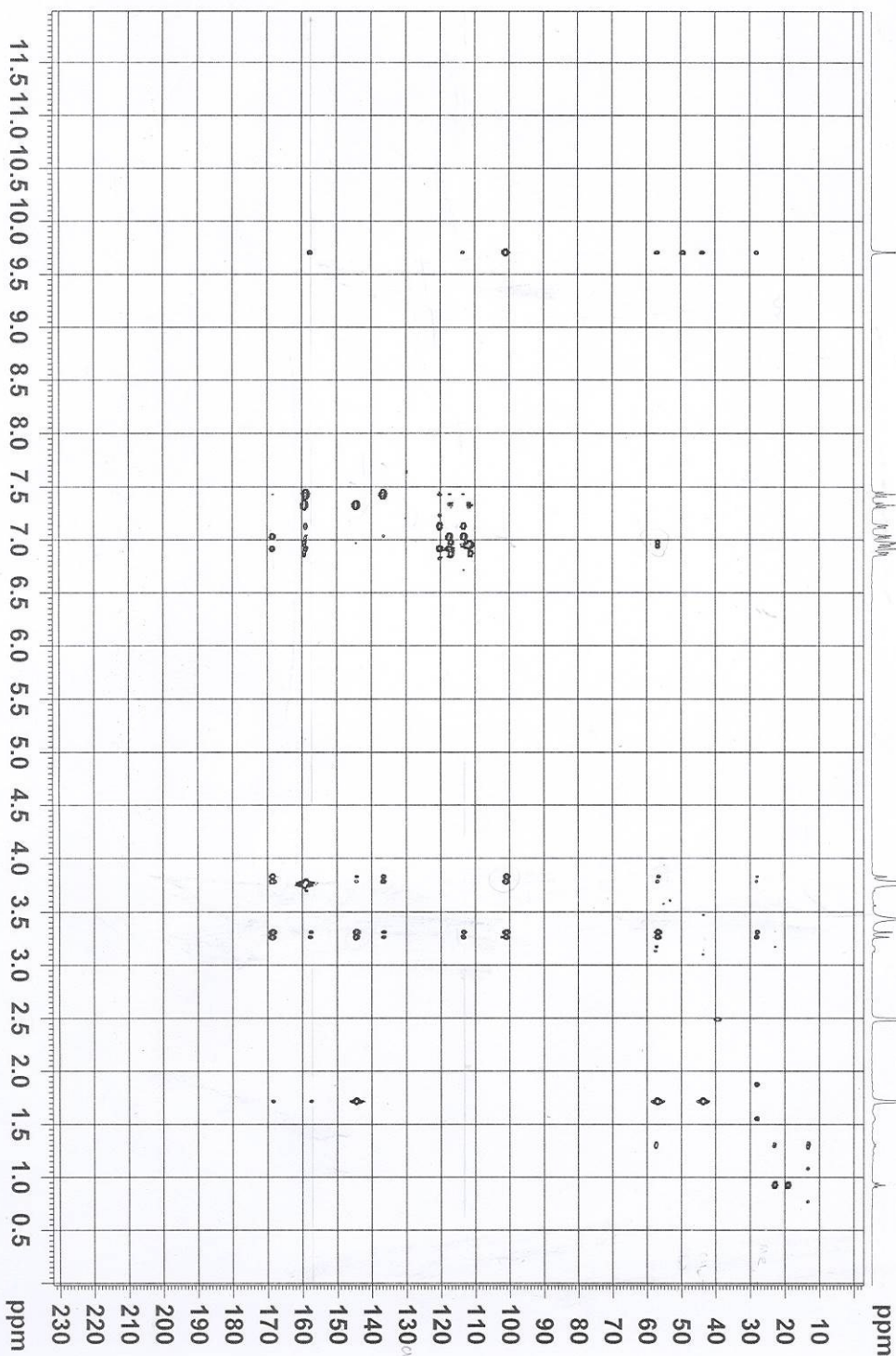


49

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HSQC



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HMBC



AVANCE-III
AV-400 MHz (A)
LAB # 109

Current Data Parameters
NAME Jun12-15
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Time 3.45
Date_ 20130925
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG hmcpgp1pdat
TD 2048
SOLVENT DMSO
NS 654
DS 16
SMH 4807.692 Hz
FIDRES 2.347506 Hz
AQ 0.2136420 sec
RG 0.00344828 sec
RG 104.000 usec
DE 6.50 usec
TE 302.0 K
CNS12 145.0000000
NUC1 13C
NUC2 1H
P1 9.70 usec
P2 13.40 usec
FAM1 17.0000000 W
SFO1 400.2324014 MHz
F3 10.20 usec
FAM2 70.0000000 W
SFO2 100.6249874 MHz

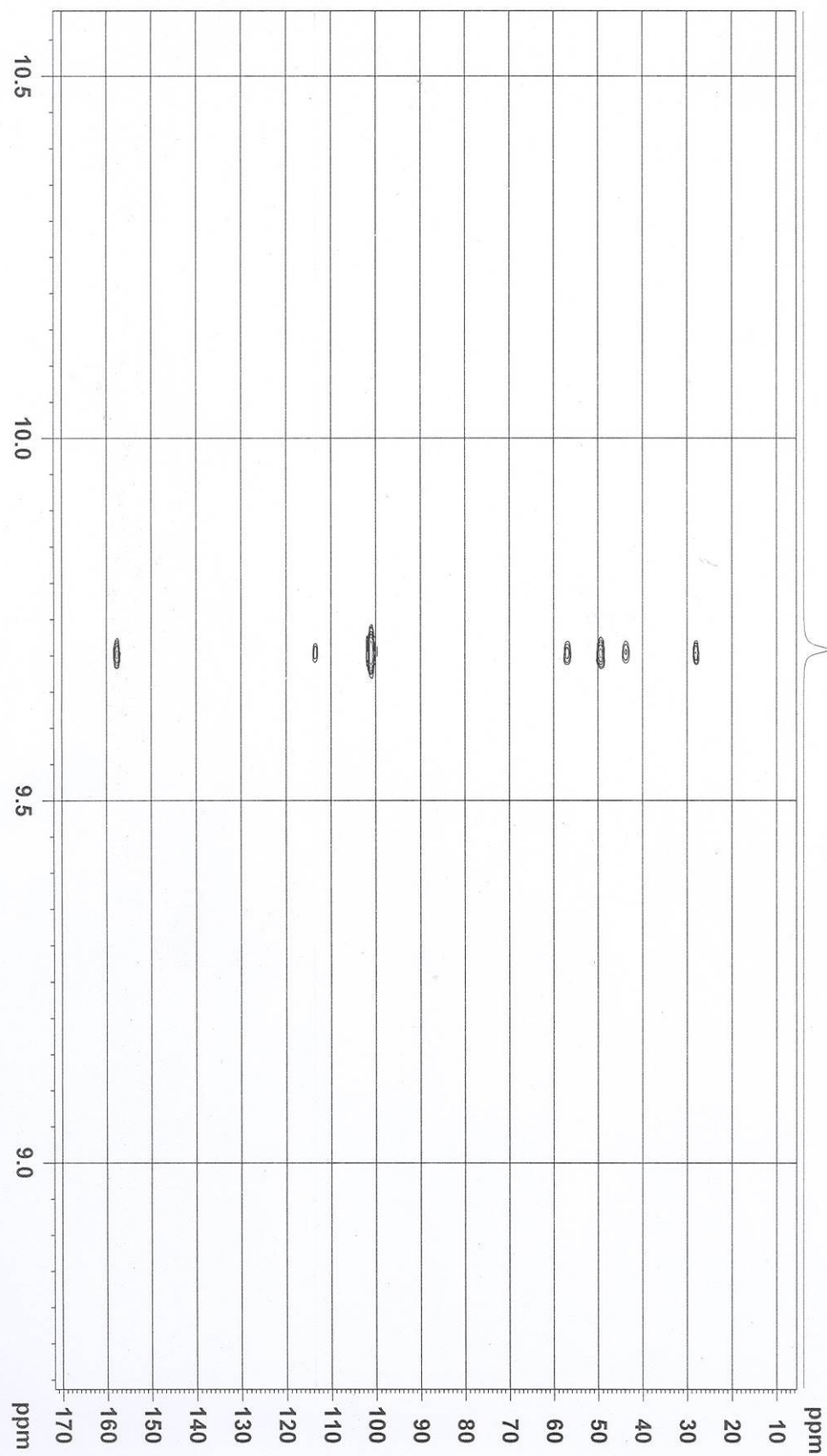
GRADIENT CHANNEL
GPM1 SMCQ10.100
GPM2 SMCQ10.100
GPM3 SMCQ10.100
GPM4 SMCQ10.100
GPM5 SMCQ10.100
GPM6 SMCQ10.100
GPM7 SMCQ10.100
GPM8 SMCQ10.100
GPM9 SMCQ10.100
GPM10 SMCQ10.100
GPM11 SMCQ10.100
GPM12 SMCQ10.100
GPM13 SMCQ10.100
GPM14 SMCQ10.100
GPM15 SMCQ10.100
GPM16 SMCQ10.100
GPM17 SMCQ10.100
GPM18 SMCQ10.100
GPM19 SMCQ10.100
GPM20 SMCQ10.100
GPM21 SMCQ10.100
GPM22 SMCQ10.100
GPM23 SMCQ10.100
GPM24 SMCQ10.100
GPM25 SMCQ10.100
GPM26 SMCQ10.100
GPM27 SMCQ10.100
GPM28 SMCQ10.100
GPM29 SMCQ10.100
GPM30 SMCQ10.100
GPM31 SMCQ10.100
GPM32 SMCQ10.100
GPM33 SMCQ10.100
GPM34 SMCQ10.100
GPM35 SMCQ10.100
GPM36 SMCQ10.100
GPM37 SMCQ10.100
GPM38 SMCQ10.100
GPM39 SMCQ10.100
GPM40 SMCQ10.100
GPM41 SMCQ10.100
GPM42 SMCQ10.100
GPM43 SMCQ10.100
GPM44 SMCQ10.100
GPM45 SMCQ10.100
GPM46 SMCQ10.100
GPM47 SMCQ10.100
GPM48 SMCQ10.100
GPM49 SMCQ10.100
GPM50 SMCQ10.100
GPM51 SMCQ10.100
GPM52 SMCQ10.100
GPM53 SMCQ10.100
GPM54 SMCQ10.100
GPM55 SMCQ10.100
GPM56 SMCQ10.100
GPM57 SMCQ10.100
GPM58 SMCQ10.100
GPM59 SMCQ10.100
GPM60 SMCQ10.100
GPM61 SMCQ10.100
GPM62 SMCQ10.100
GPM63 SMCQ10.100
GPM64 SMCQ10.100
GPM65 SMCQ10.100
GPM66 SMCQ10.100
GPM67 SMCQ10.100
GPM68 SMCQ10.100
GPM69 SMCQ10.100
GPM70 SMCQ10.100
GPM71 SMCQ10.100
GPM72 SMCQ10.100
GPM73 SMCQ10.100
GPM74 SMCQ10.100
GPM75 SMCQ10.100
GPM76 SMCQ10.100
GPM77 SMCQ10.100
GPM78 SMCQ10.100
GPM79 SMCQ10.100
GPM80 SMCQ10.100
GPM81 SMCQ10.100
GPM82 SMCQ10.100
GPM83 SMCQ10.100
GPM84 SMCQ10.100
GPM85 SMCQ10.100
GPM86 SMCQ10.100
GPM87 SMCQ10.100
GPM88 SMCQ10.100
GPM89 SMCQ10.100
GPM90 SMCQ10.100
GPM91 SMCQ10.100
GPM92 SMCQ10.100
GPM93 SMCQ10.100
GPM94 SMCQ10.100
GPM95 SMCQ10.100
GPM96 SMCQ10.100
GPM97 SMCQ10.100
GPM98 SMCQ10.100
GPM99 SMCQ10.100
GPM100 SMCQ10.100

F1 - Acquisition parameters
SFO1 100.6195 MHz
FIDRES 92.391670 Hz
SW 234.996 ppm
FMODE QF

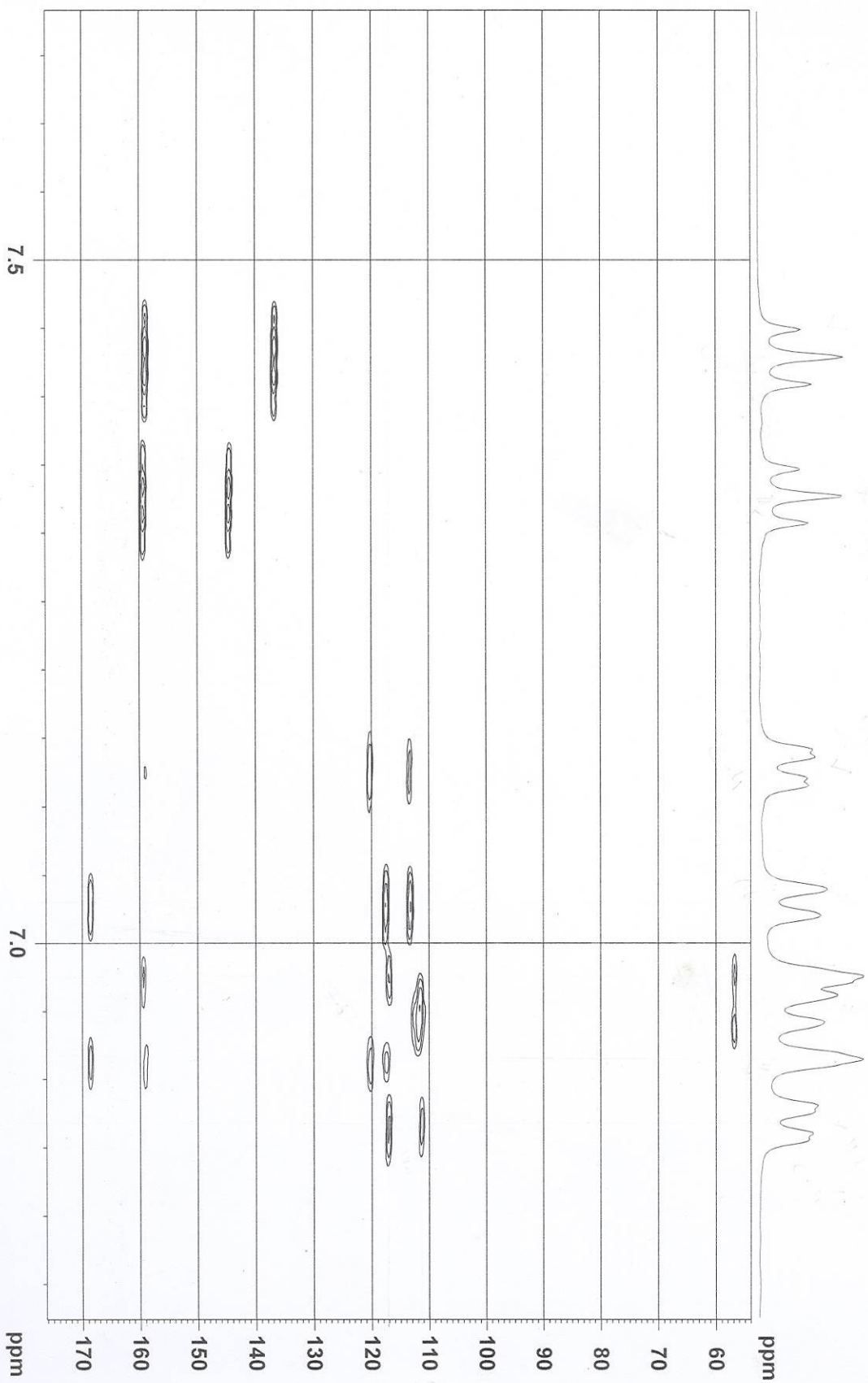
F2 - Processing parameters
SI 1024
SF 400.230095 MHz
WDW SINE
SSB 0 Hz
LB 0 Hz
GB 0 Hz
PC 1.00

F1 - Processing parameters
SI 1024
SF 100.627965 MHz
WDW SINE
SSB 0 Hz
GB 0 Hz

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HMBC



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HMBC



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HMBc

