

Supplementary Materials

Synthesis of polyfunctionalized 4*H*-pyrans

Manisha Bihani, Pranjal P Bora and Ghanashyam Bez*

Department of Chemistry, North Eastern Hill University, Shillong-793022, India

bez@nehu.ac.in; ghanashyambez@yahoo.com

CONTENT

General remarks	page S1
¹H and ¹³CNMR spectra of new compounds	page S2-S15

General remarks

IR spectra were recorded on a Perkin–Elmer Spectrum One FTIR spectrometer. ¹H and ¹³C NMR spectra were recorded on a Bruker (300 MHz and 400 MHz) spectrometer using TMS as internal reference. Chemical shifts for ¹H NMR spectra are reported with CDCl₃ and (CD₃)₂SO as solvents. ¹³C NMR spectra were recorded at 100 MHz. Chemical shifts for ¹³C NMR spectra are reported with CDCl₃ and (CD₃)₂SO as solvents. Mass spectra were obtained from Waters ZQ 4000 mass spectrometer by the ESI method, while the elemental analyses of the complexes were performed on a Perkin–Elmer-2400 CHN/S analyzer. TLC plates were visualized by exposing in iodine chamber, UV-lamp or spraying with KMnO₄ and heating.

¹H, M-147, DMSOd6, 07/06/12 SAIF, NEHU, DRD

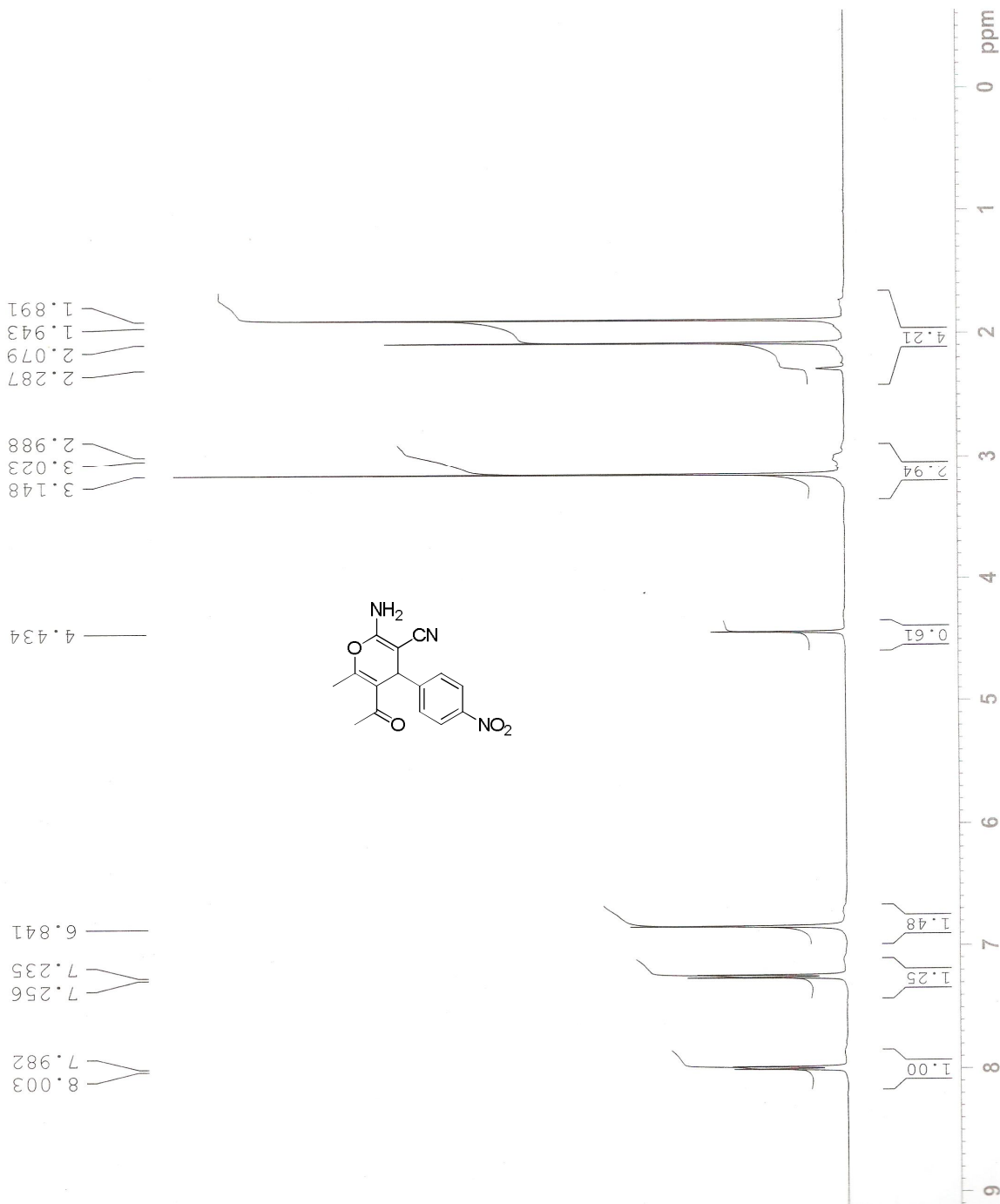


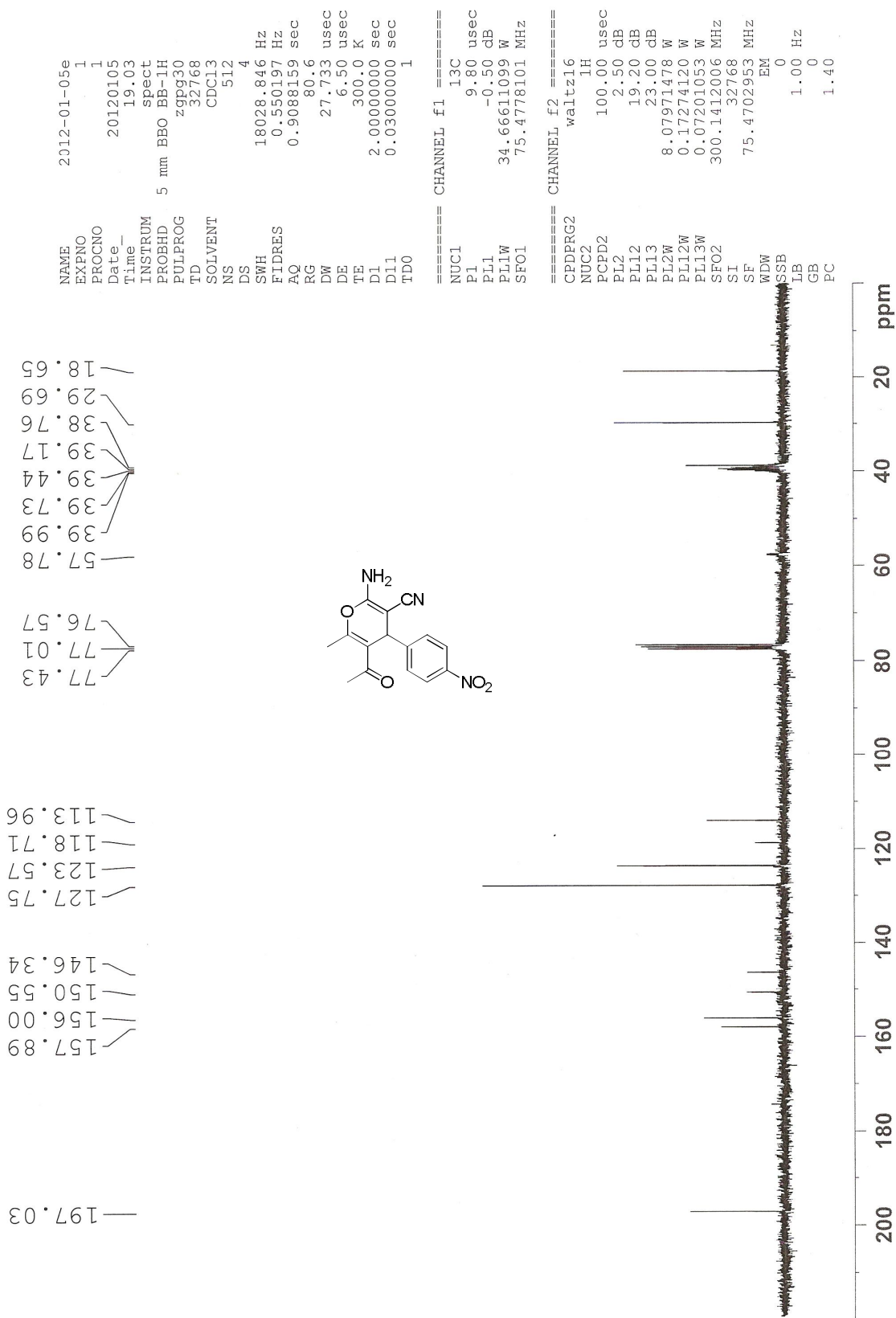
Current Data Parameters
NAME M-147
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120607
Time_ 12.15
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT DMSO
NS 8
DS 0
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 12.7
DW 41.600 use
DE 6.00 use
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 ¹H
P1 14.00 use
PL1 -1.00 dB
SFO1 400.1316005 MHz

F2 - Processing parameters
SI 16384
SF 400.1300824 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00





¹H M-150, DMSO-d₆, 22/06/12, SAIF, NEHU, DRW

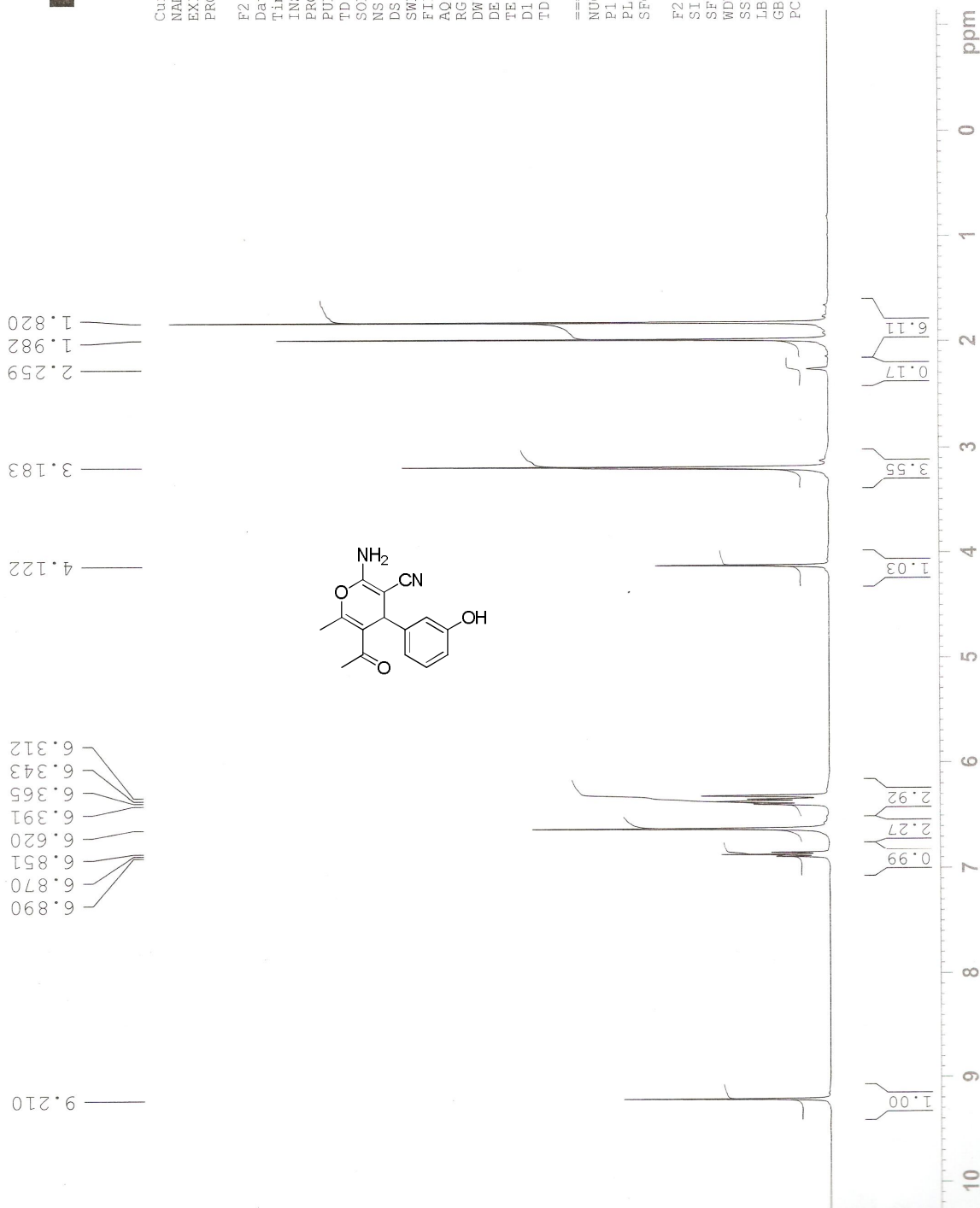


Current Data Parameters
NAME M-150
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120622
Time 11.59
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT DMSO
NS 8
DS 0
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 12.7
DW 41.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 ¹H
P1 14.00 usec
PL1 -1.00 dB
SFO1 400.1316005 MHz

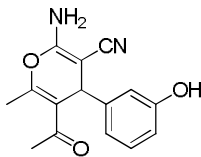
F2 - Processing parameters
SI 16384
SF 400.1300947 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



13C M-150, DMSOd6, 22/06/12, DRW, SAI7, NEHU

198.464
158.203
157.592
154.542
145.955
129.675
119.850
117.724
114.922
114.001
113.864
57.706
39.992
39.783
39.574
39.365
39.156
38.948
38.736
38.632
29.683
18.352

114.001
113.864



Current Data Parameters
NAME 13C M-150
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120622
Time 12.47
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg
TD 262144
SOLVENT DMSO
NS 525
DS 0
SWH 40760.871 Hz
FIDRES 0.155490 Hz
AQ 3.2156830 sec
RG 161
DW 12.267 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

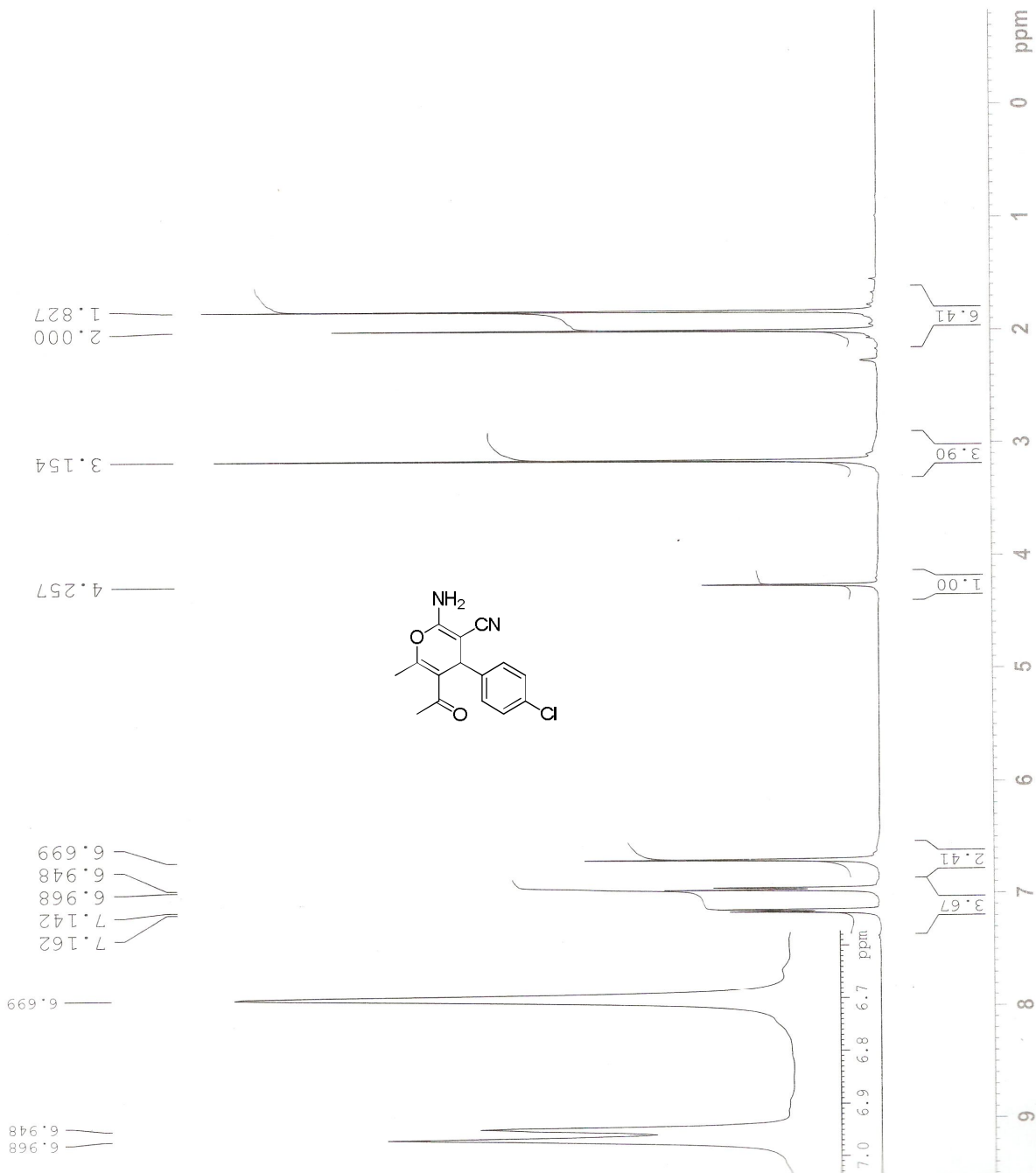
===== CHANNEL f1 =====
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
SFO1 100.6208180 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 14.00 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 131072
SF 100.6128193 MHz
EM
WDW 0
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

ppm

1H Z-163, DMSOd6, 21/06/12, SALT, NEHU, DRW



Current Data Parameters
NAME Z-163
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120621
Time_ 11.19
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT DMSO
NS 8
DS 0
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 101
DW 41.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
SFO1 400.1316005 MHz
F2 - Processing parameters
SI 16384
SF 400.1300930 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

¹³C M-149, DMSO-d₆, 22/06/12, DRW, SAIF, NEHU



Current Data Parameters
NAME 13C M-149
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120622
Time_ 13.29
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg
TD 262144
SOLVENT DMSO
NS 437
DS 0
SWH 40760.871 Hz
FIDRES 0.155490 Hz
AQ 3.2156830 sec
RG 161
DW 12.267 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 ¹³C
P1 8.50 usec
PL1 -2.00 dB
SFO1 100.6208180 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ¹H
PCPD2 80.00 usec
PL12 14.00 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 131072
SF 100.6128193 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40



1H PGB-3, DMSOd6, 02/12/11, SAIF, NEHU, DRW



Current Data Parameters
NAME PGB-3
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111202
Time_ 14.21
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT DMSO
NS 8
DS 0
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 64
DW 41.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
PL 14.00 usec
PL1 -1.00 dB
SFO1 400.1316005 MHz

F2 - Processing parameters
SI 16384
SF 400.1300643 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

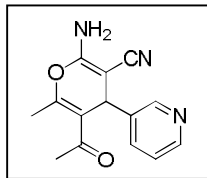


13C PGB-5, DMSOd6, 3/12/11, DRW, SAIF, NEHU

197.875

158.456
155.860
148.324
148.138
140.032
134.828
123.986
119.559
114.397

56.853
40.032
39.824
39.614
39.406
39.197
38.987
38.778
36.271
30.027
18.677



Current Data Parameters
NAME 13C PGB-5
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111203
Time 12.42
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg
TD 262144
SOLVENT DMSO
NS 837
DS 0
SWH 40760.871 Hz
FIDRES 0.135490 Hz
AQ 3.2156830 sec
RG 256
DE 12.267 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
DELTA 0.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.50 usec
PL1 -2.00 dB
SFO1 100.6208180 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 14.00 dB
PL13 18.00 dB
PL2 -1.00 dB
SFO2 400.1320007 MHz

F2 - Processing parameters
SI 131072
SF 100.6128193 MHz
EM 0
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

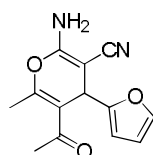
ppm

1H, M-108, DMSOd6, 10.01.2012, SAIF, NEHU, DRW

7.548
7.543
7.015
6.361
6.355
6.353
6.348
6.140
6.132
4.616
3.310
2.498
2.493
2.489
2.191
2.183
2.100

7.548
7.543

7.015



6.361
6.355
6.353
6.348

6.140
6.132

2.498
2.493
2.489

2.191
2.183
2.100

7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2 ppm

2.5 2.4 2.3 2.2 ppm

1.00

2.68

1.23

1.25

1.27

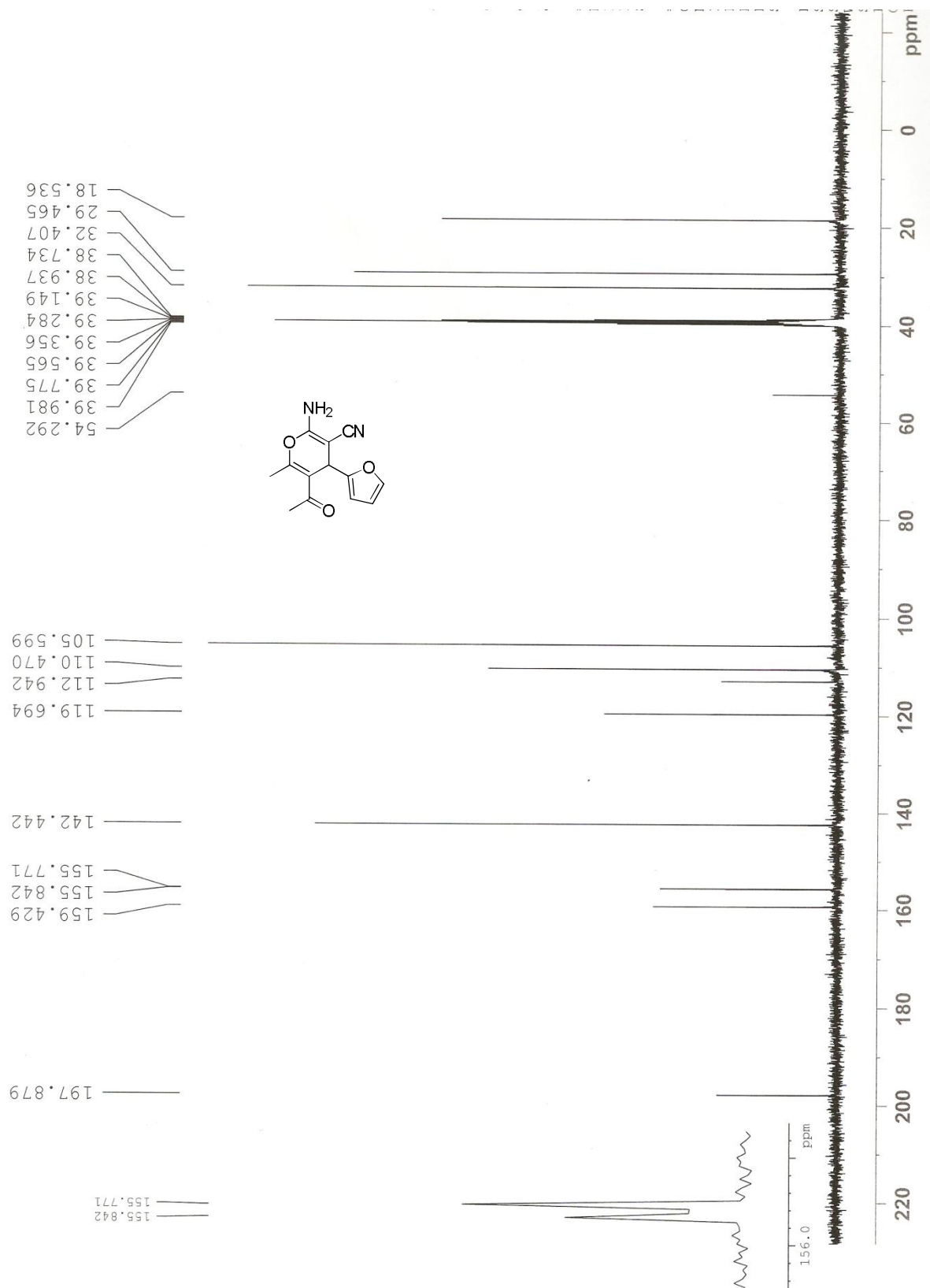
0.43

0.82

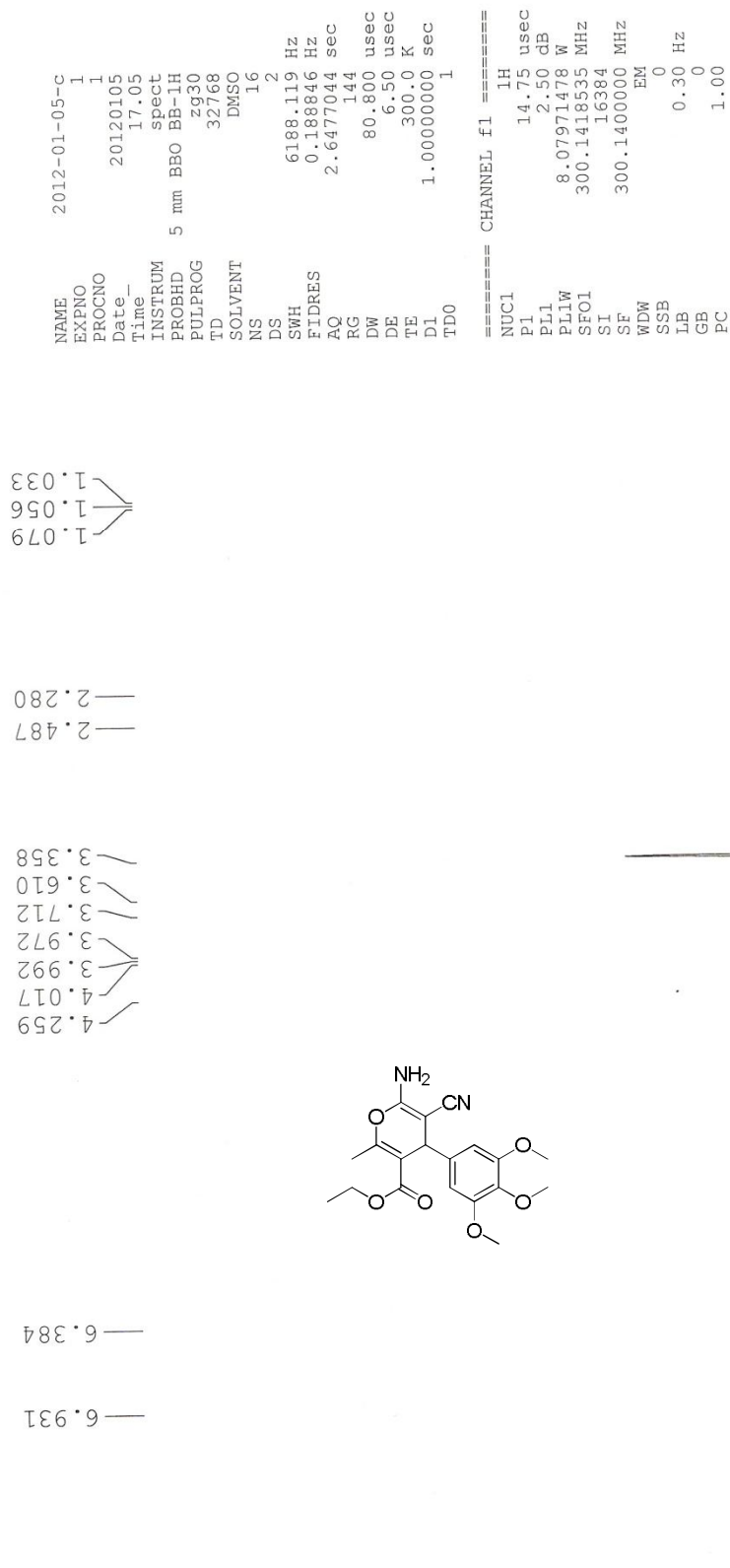
7.95

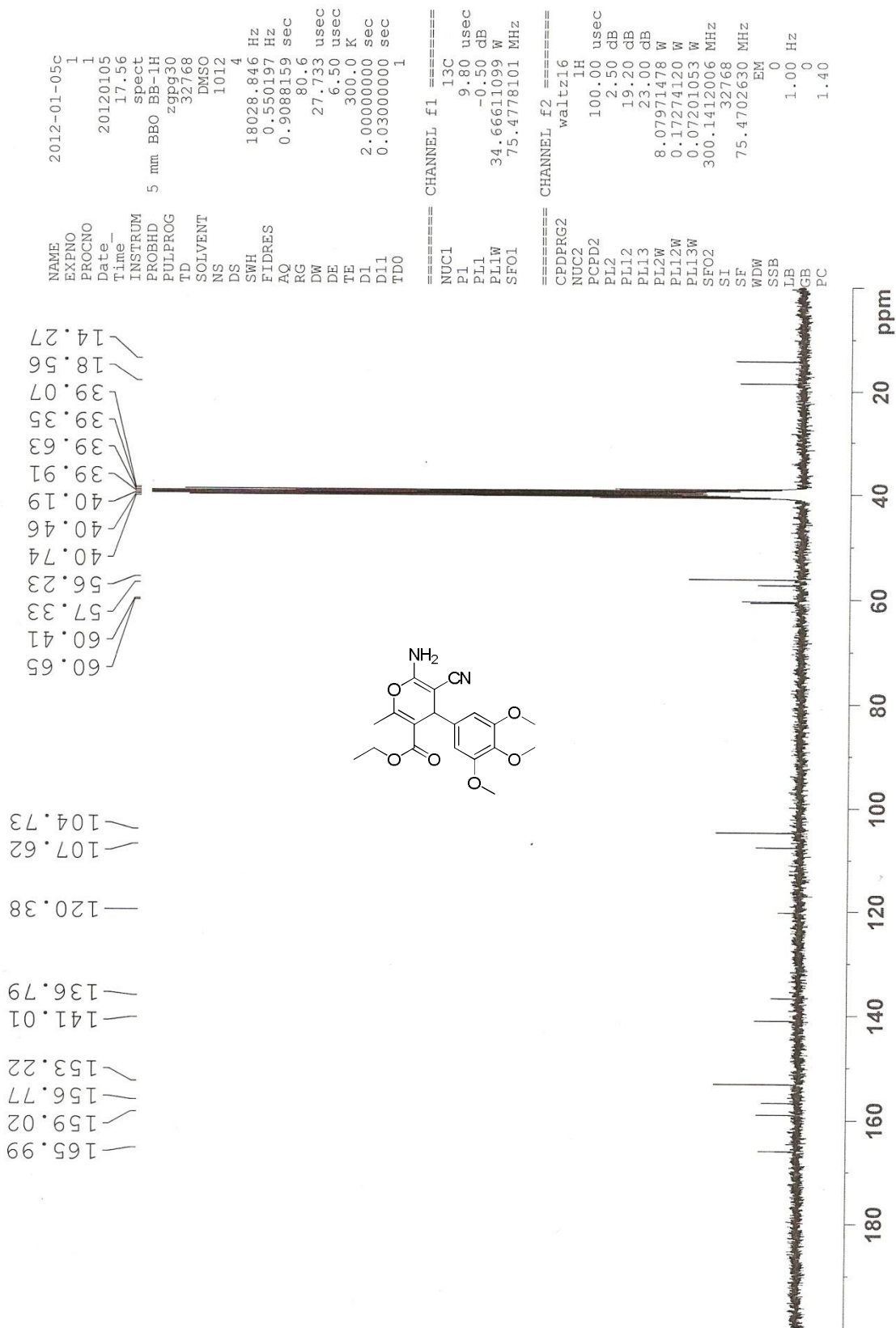
10 9 8 7 6 5 4 3 2 1 0 ppm

¹³C M-108, DMSO-d₆, 10.01.2012, SAIF, NEHU, DRW



MET-





1H PGB-4, DMSOd6, 08/12/11, SAIF, NEHU, DRW

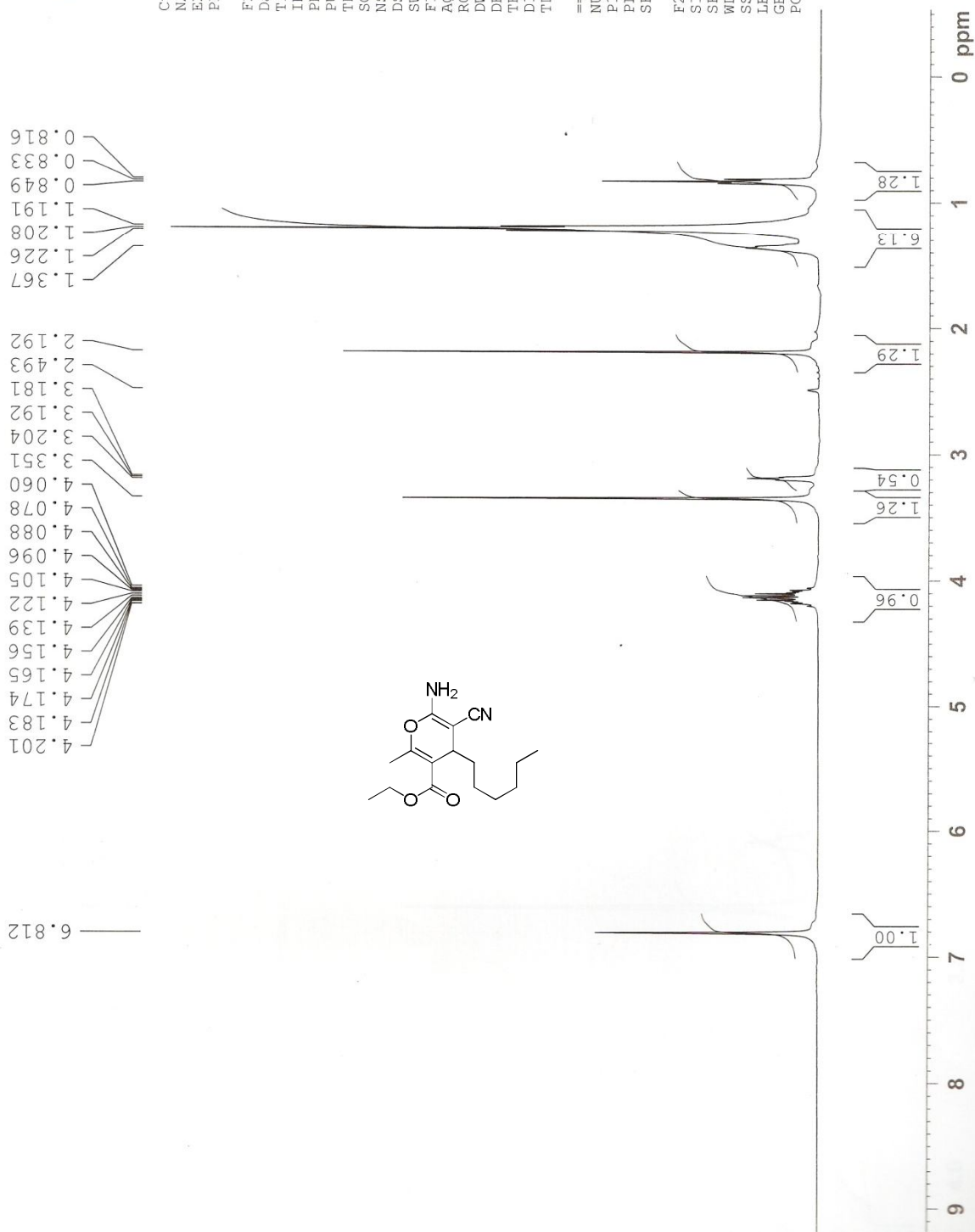


Current Data Parameters
NAME PGB-4
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111208
Time_ 15.11
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT DMSO
NS 8
DS 0
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 57
DW 41.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
SFO1 400.1316005 MHz

F2 - Processing parameters
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00



PGB-4

