

PBr₃-Mediated [5+1] Annulation of α -Alkenoyl- α -Carbamoyl Ketene-*S,S*-acetals: Access to Substituted Pyridine- 2,6(1*H*,3*H*)-diones

Liping Shi,^a Qian Zhang,^b Fengjiao Gan,^b Rui Zhang,^{*b} Yuanli Ding,^b Chun Liu,^a and Dewen Dong,^{*b}

^a College of Chemistry and Biology, Beihua University, Jilin, 132013, China..

^b Key Laboratory of Synthetic Rubber, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences,
Changchun 130022, China.

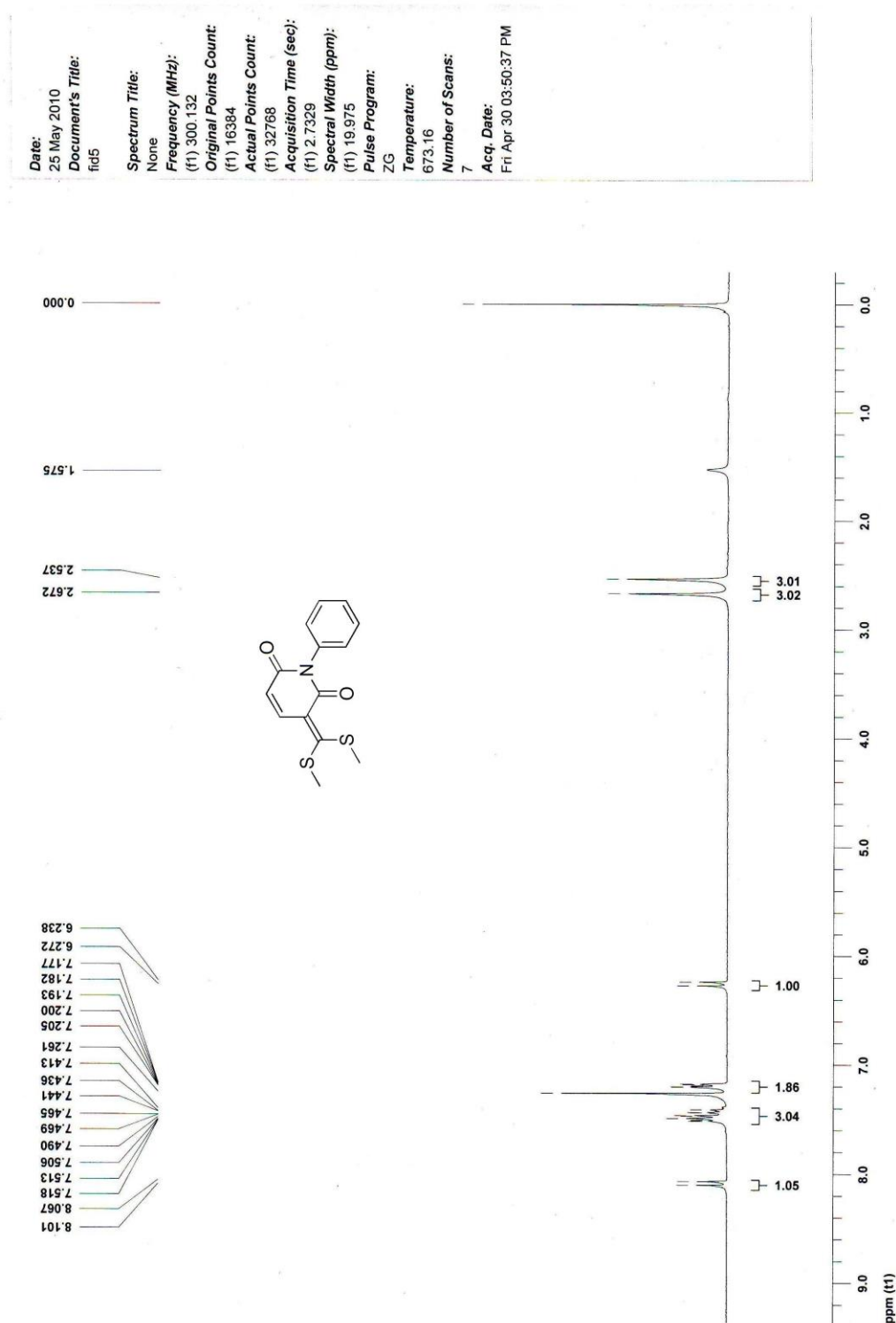
E-mail: ariel@ciac.ac.cn; dwdong@ciac.ac.cn

Electronic Supplementary Information

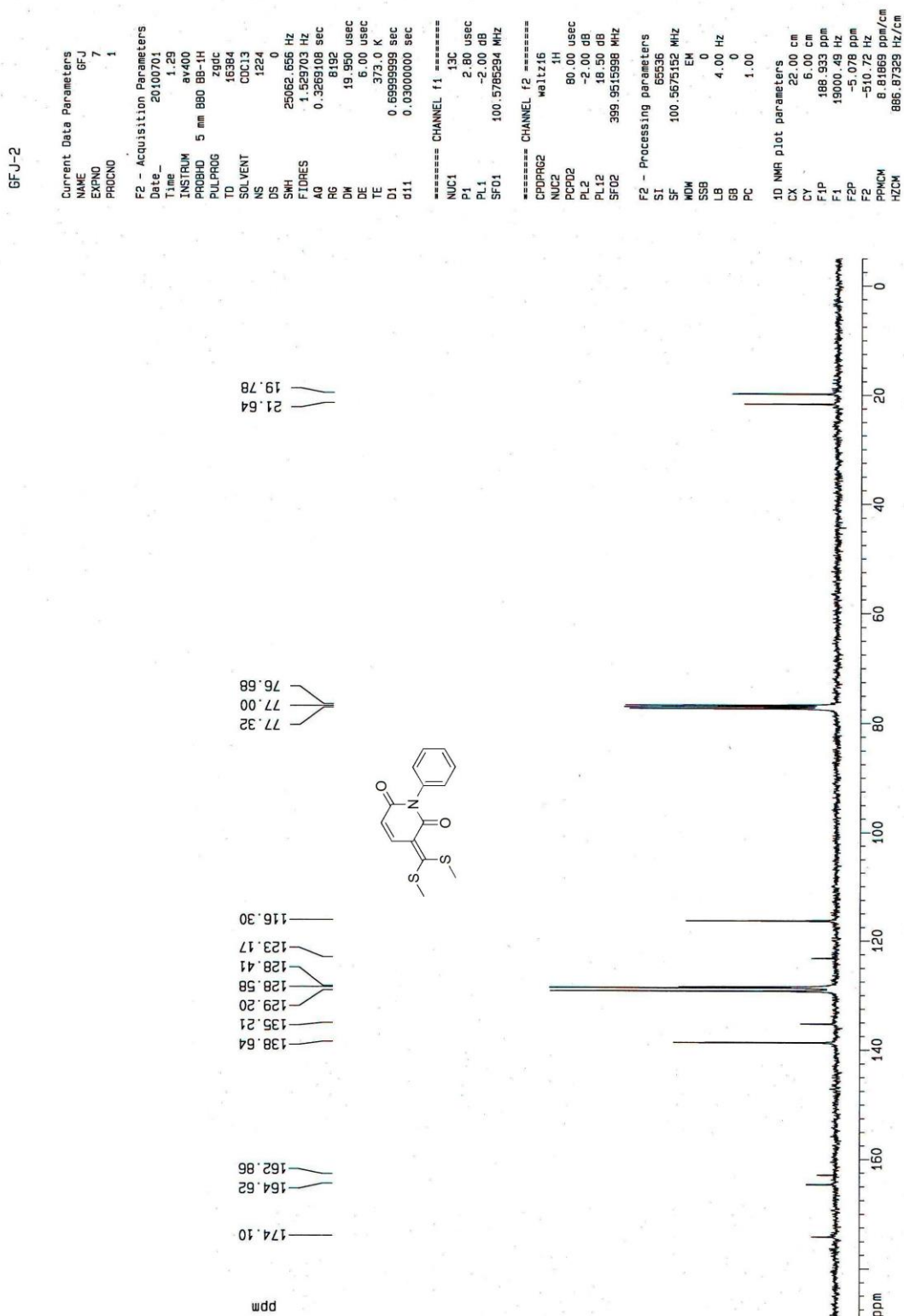
Table of contents.....	S1
I. Copies of NMR spectra for compounds 2	S2-27

I. Copies of NMR spectra for compounds 2

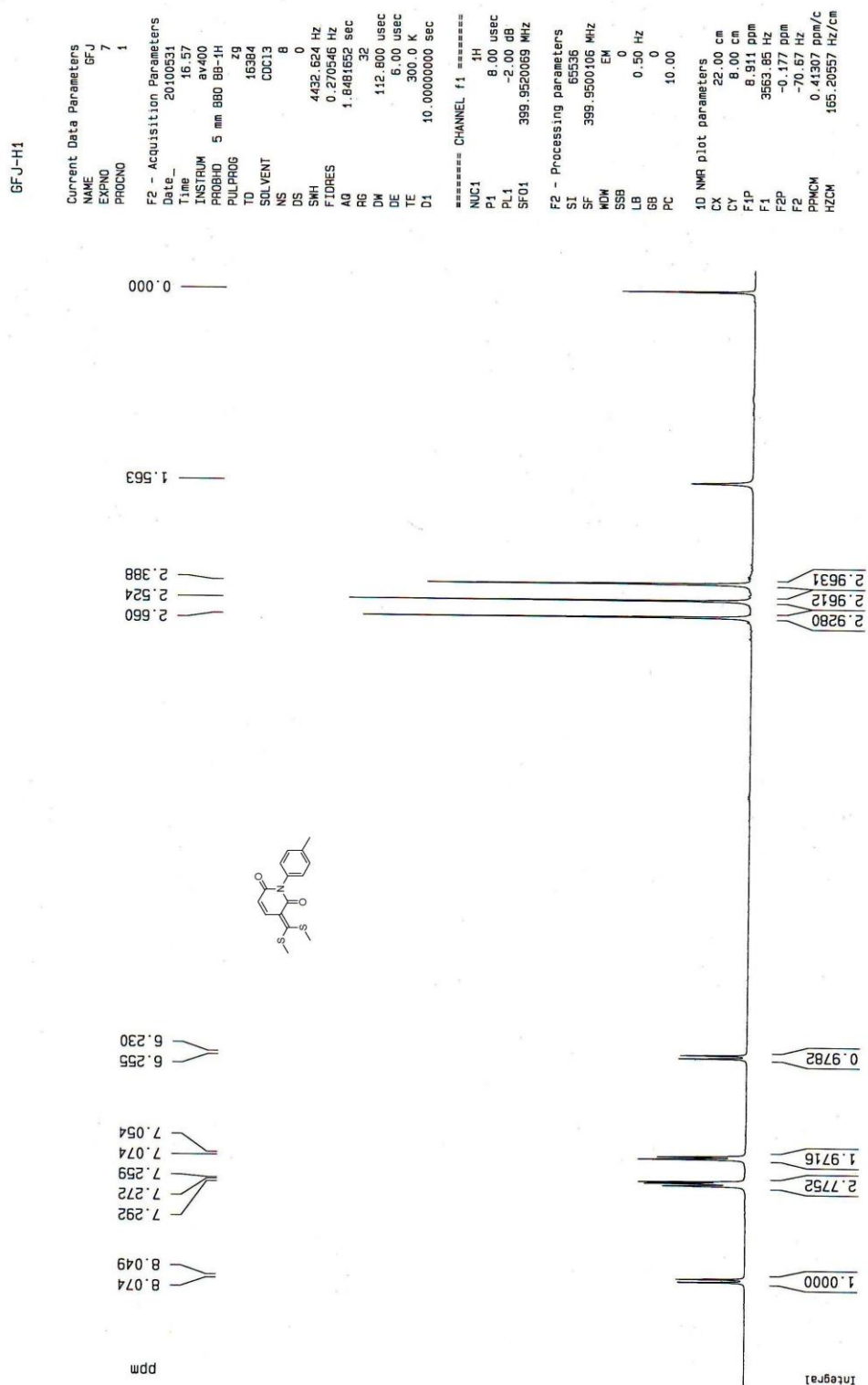
2a



2a

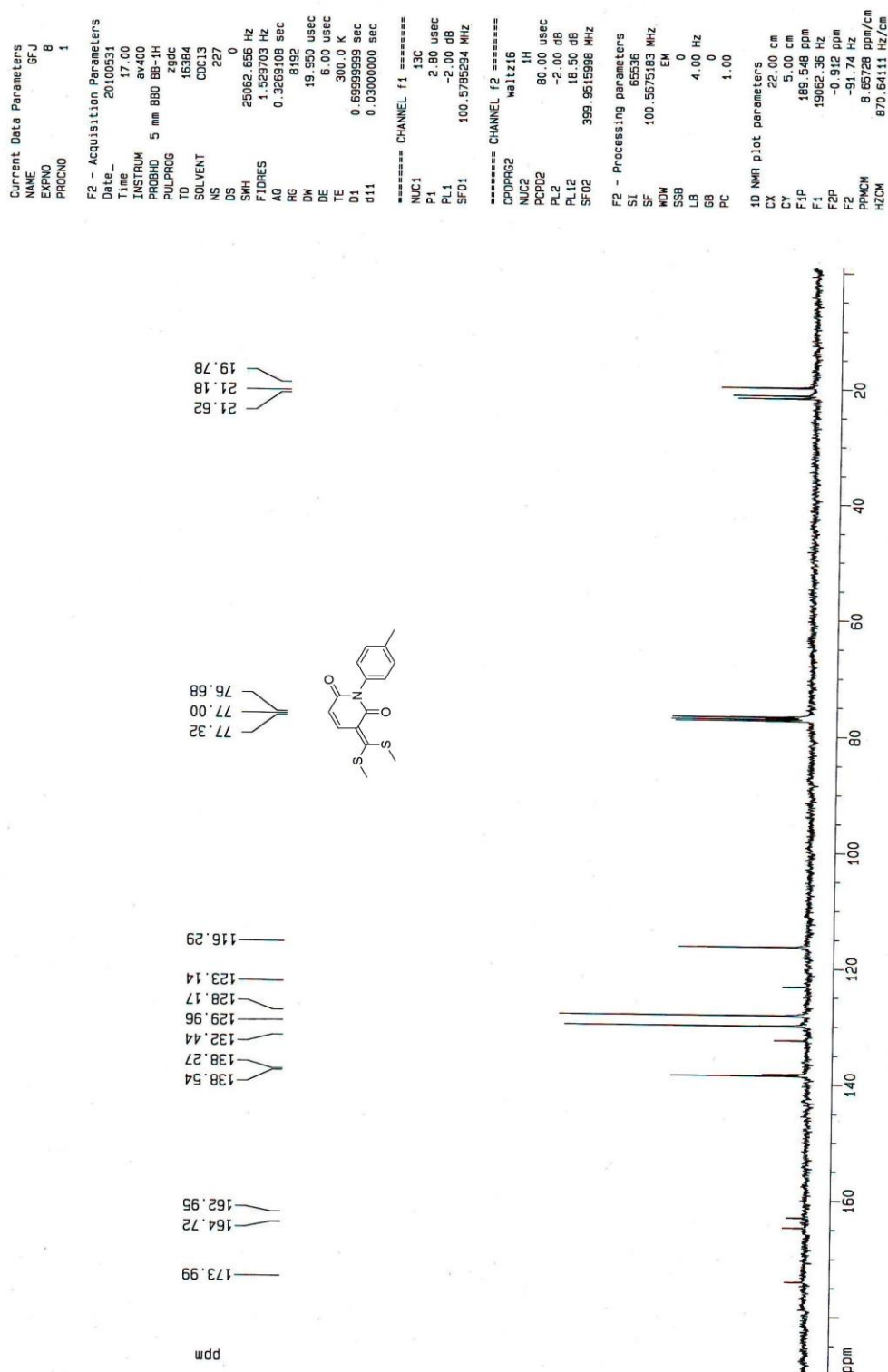


2b

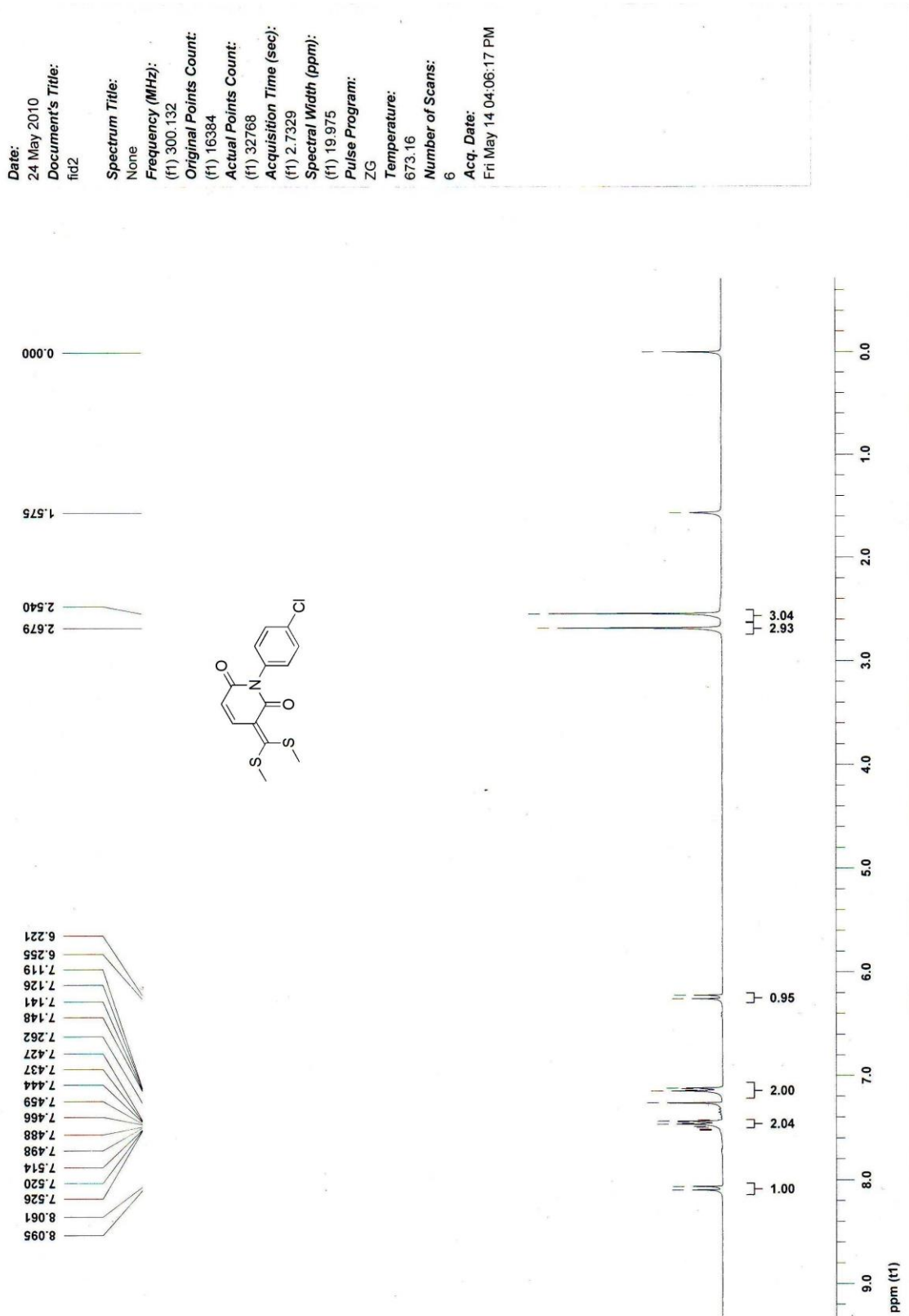


2b

6FJ-7



2c



2c

GFJ-1

Current Data Parameters
 NAME GFJ
 EXPNO 8
 PROCNO 1

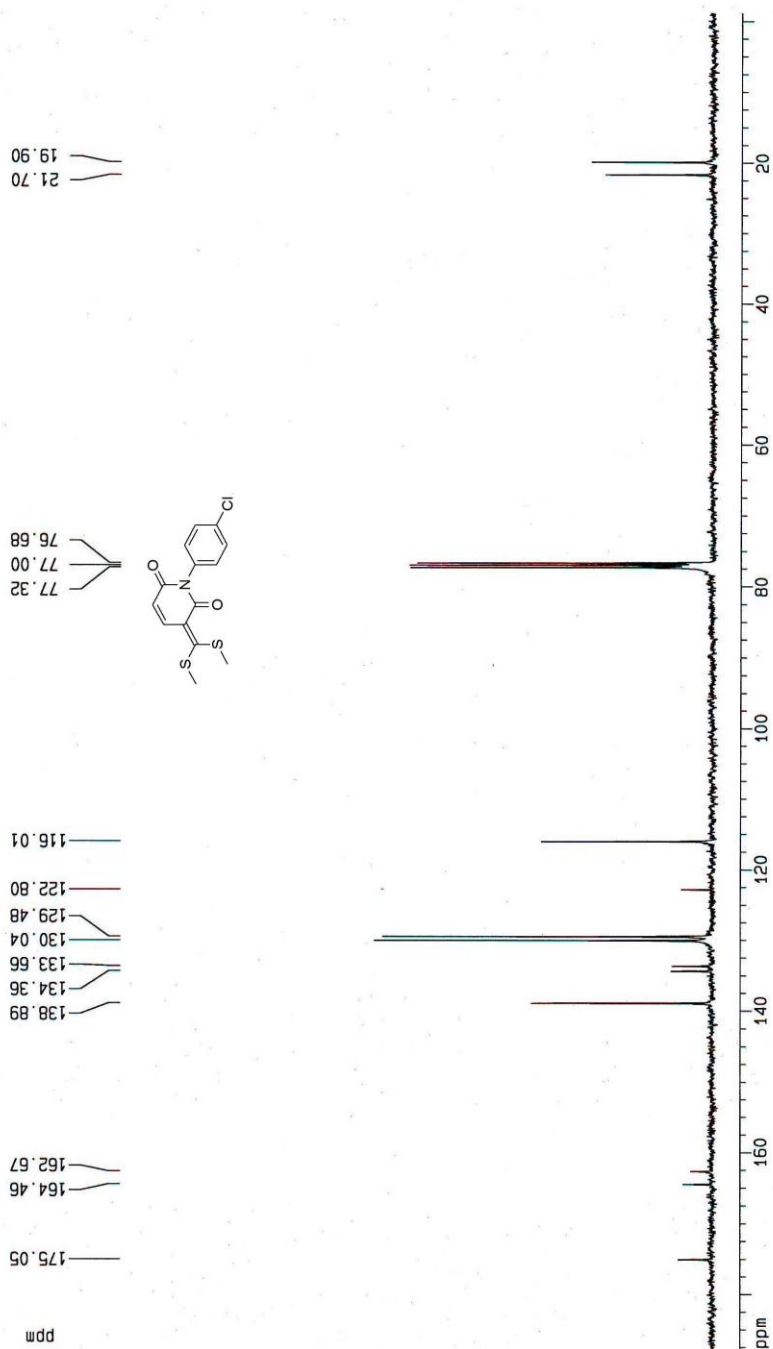
F2 - Acquisition Parameters
 Date_ 20100701
 Time 18.10
 INSTRUM av400
 PROBHD 5 mm BBO BB-H1
 PULPROG zgpg
 TD 16384
 SOLVENT CDCl3
 NS 1901
 DS 0
 SWH 25062.656 Hz
 FIDRES 1.529703 Hz
 AQ 0.3269108 sec
 RG 8192
 DM 19.950 usec
 DE 6.00 usec
 TE 373.0 K
 D1 0.69999999 sec
 d11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 2.80 usec
 PL1 -2.00 dB
 SF01 100.5785294 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 18.50 dB
 SF02 399.9515998 MHz

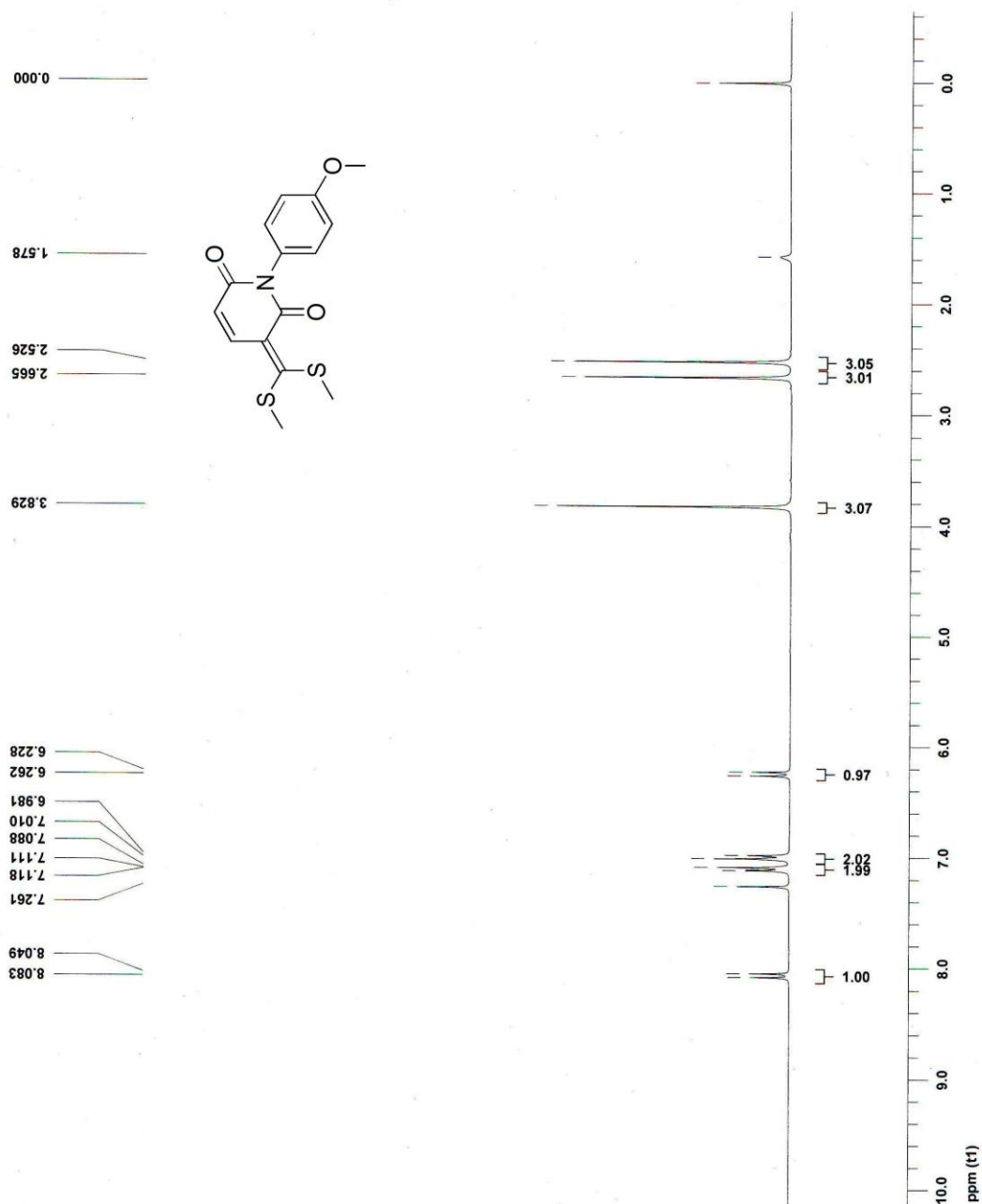
F2 - Processing parameters
 SI 65536
 SF 100.5675133 MHz
 WDW EM
 SSB 0
 LB 4.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 22.00 cm
 CY 6.00 cm
 F1P 187.660 ppm
 F1 18872.55 Hz
 F2P -1.186 ppm
 F2 -119.23 Hz
 PPMCM 8.58391 ppm/cm
 HZCM 863.26276 Hz/cm



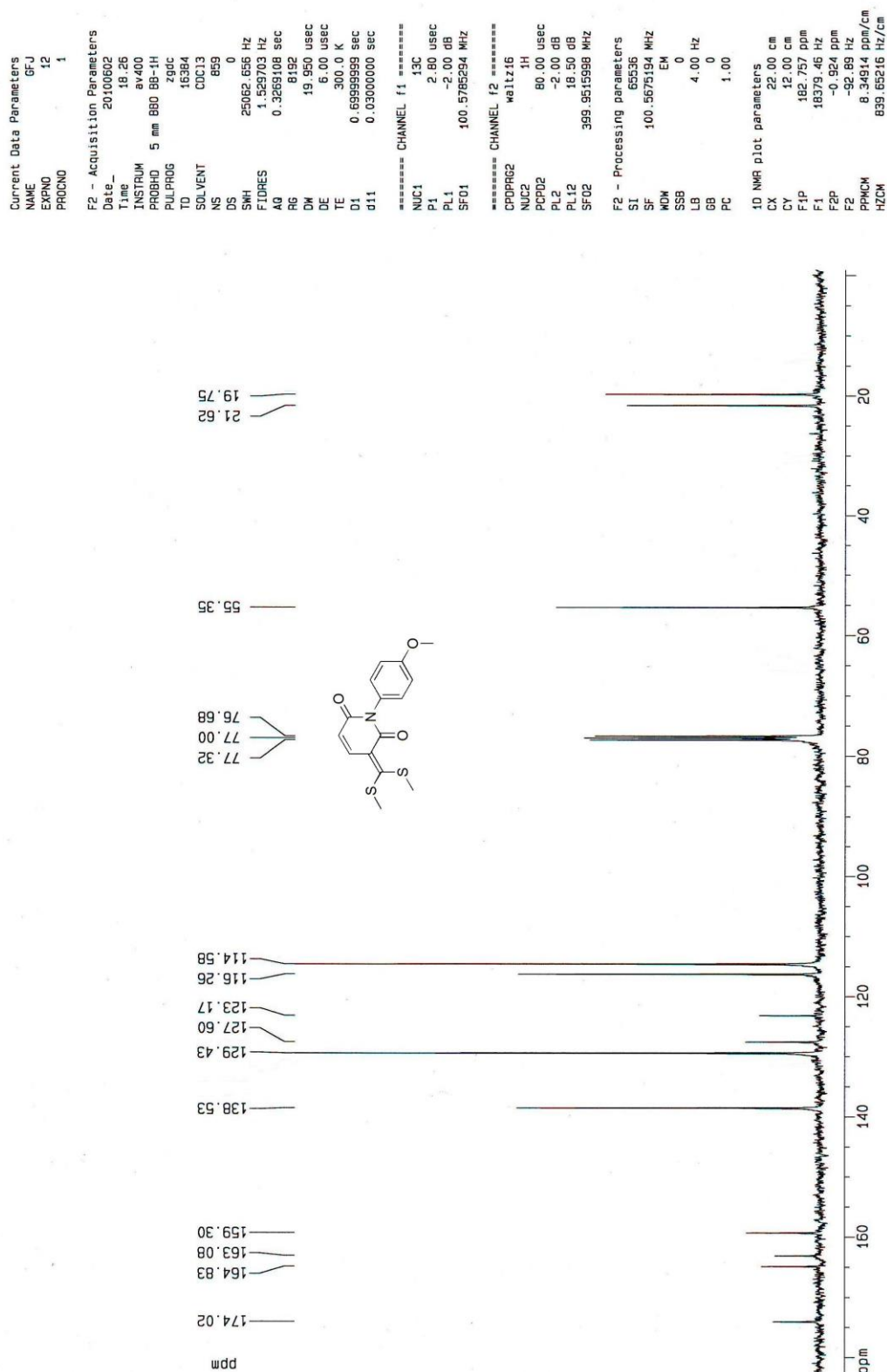
2d

Date: 30 May 2010
Document's Title: fid10
Spectrum Title: None
Frequency (MHz): (f1) 300.132
Original Points Count: (f1) 16384
Actual Points Count: (f1) 32768
Acquisition Time (sec): (f1) 2.7329
Spectral Width (ppm): (f1) 19.975
Pulse Program: ZG
Temperature: 673.16
Number of Scans: 4
Acq. Date: Fri May 28 04:17:33 PM



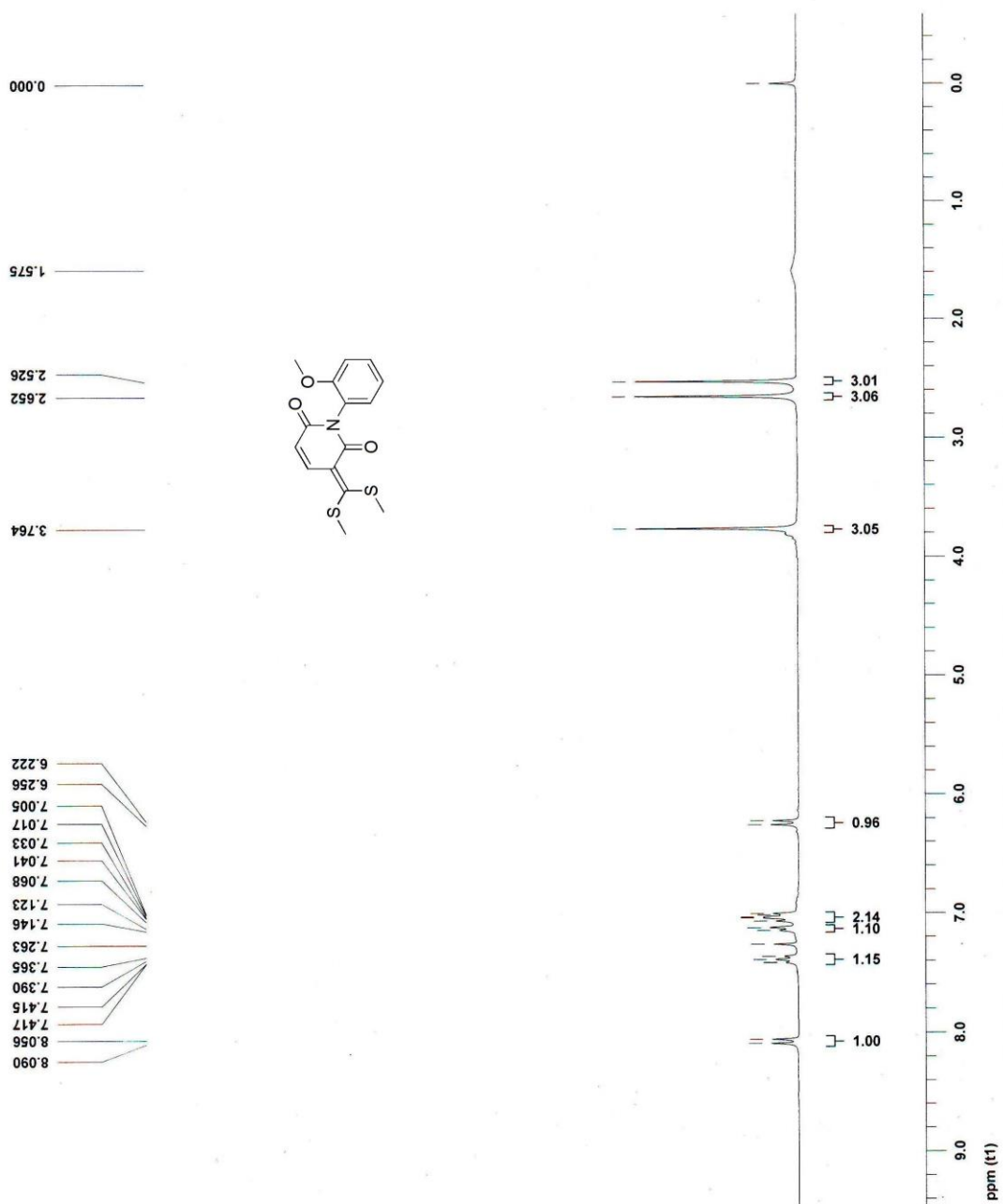
2d

GFJ-4

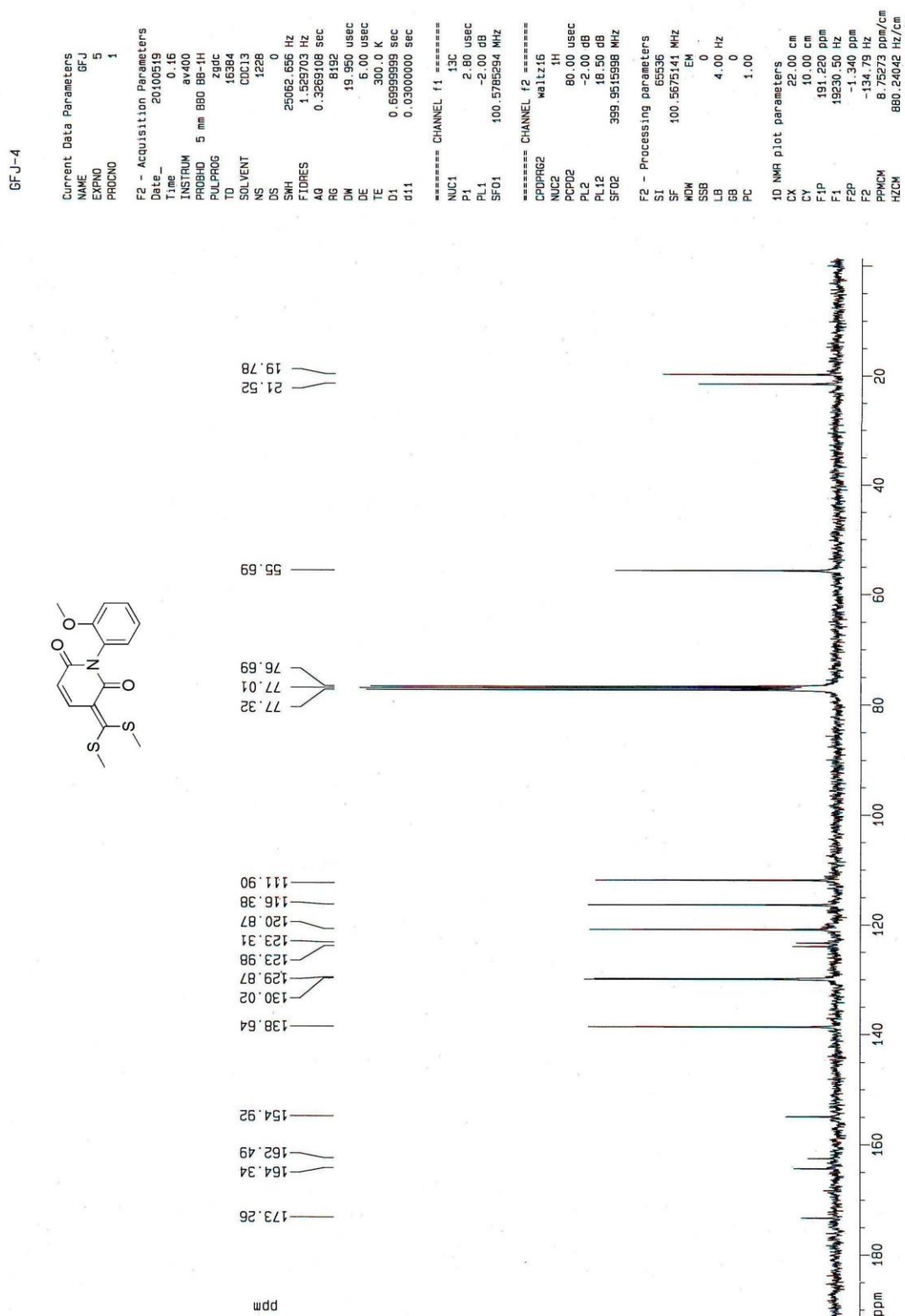


2e

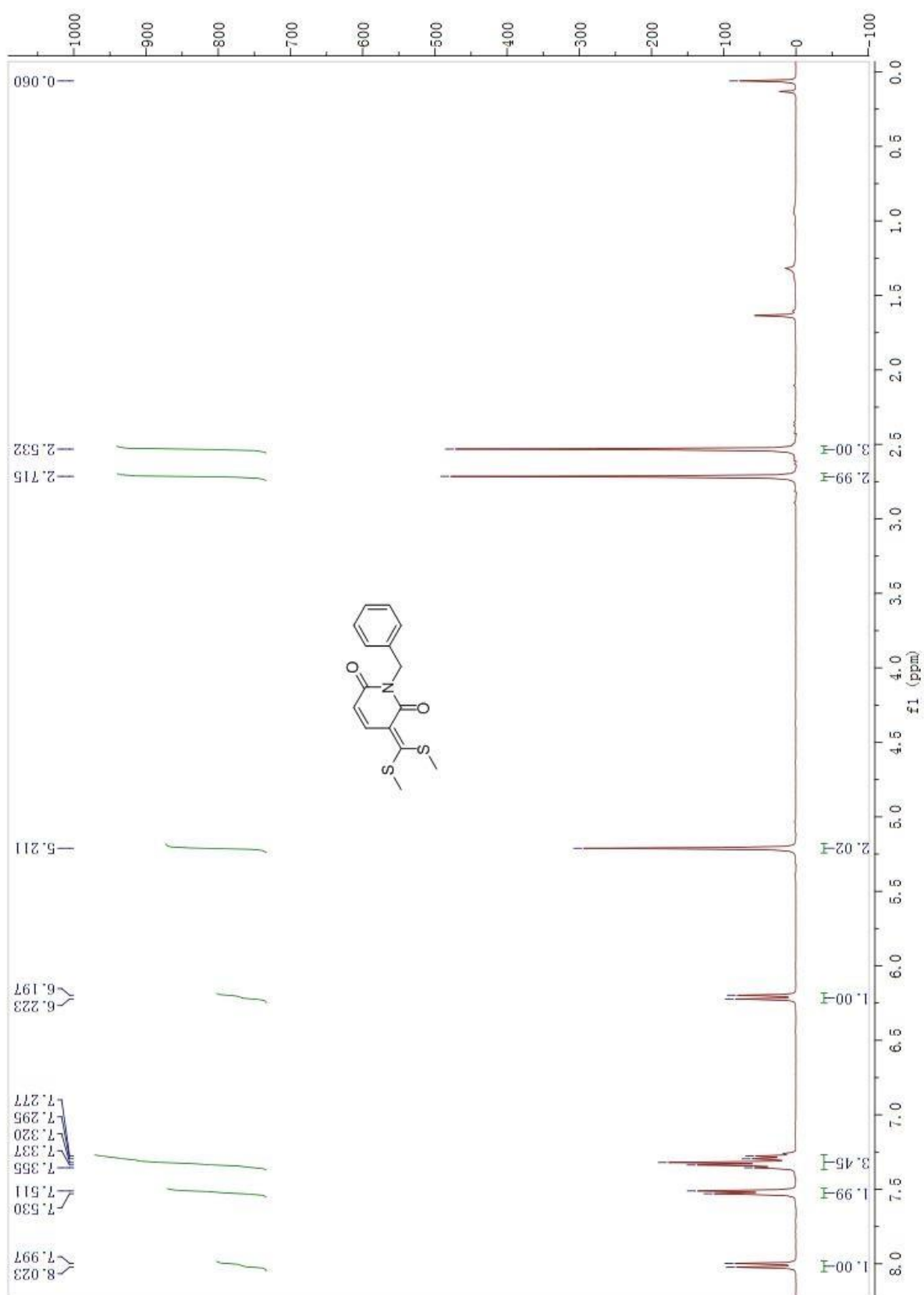
Date: 24 May 2010
 Document's Title: fid3
 Spectrum Title: None
 Frequency (MHz): (f1) 300.132
 Original Points Count: (f1) 16384
 Actual Points Count: (f1) 32768
 Acquisition Time (sec): (f1) 2.7329
 Spectral Width (ppm): (f1) 19.975
 Pulse Program: ZG
 Temperature: 673.16
 Number of Scans: 6
 Acq. Date: Fri May 14 04:12:11 PM



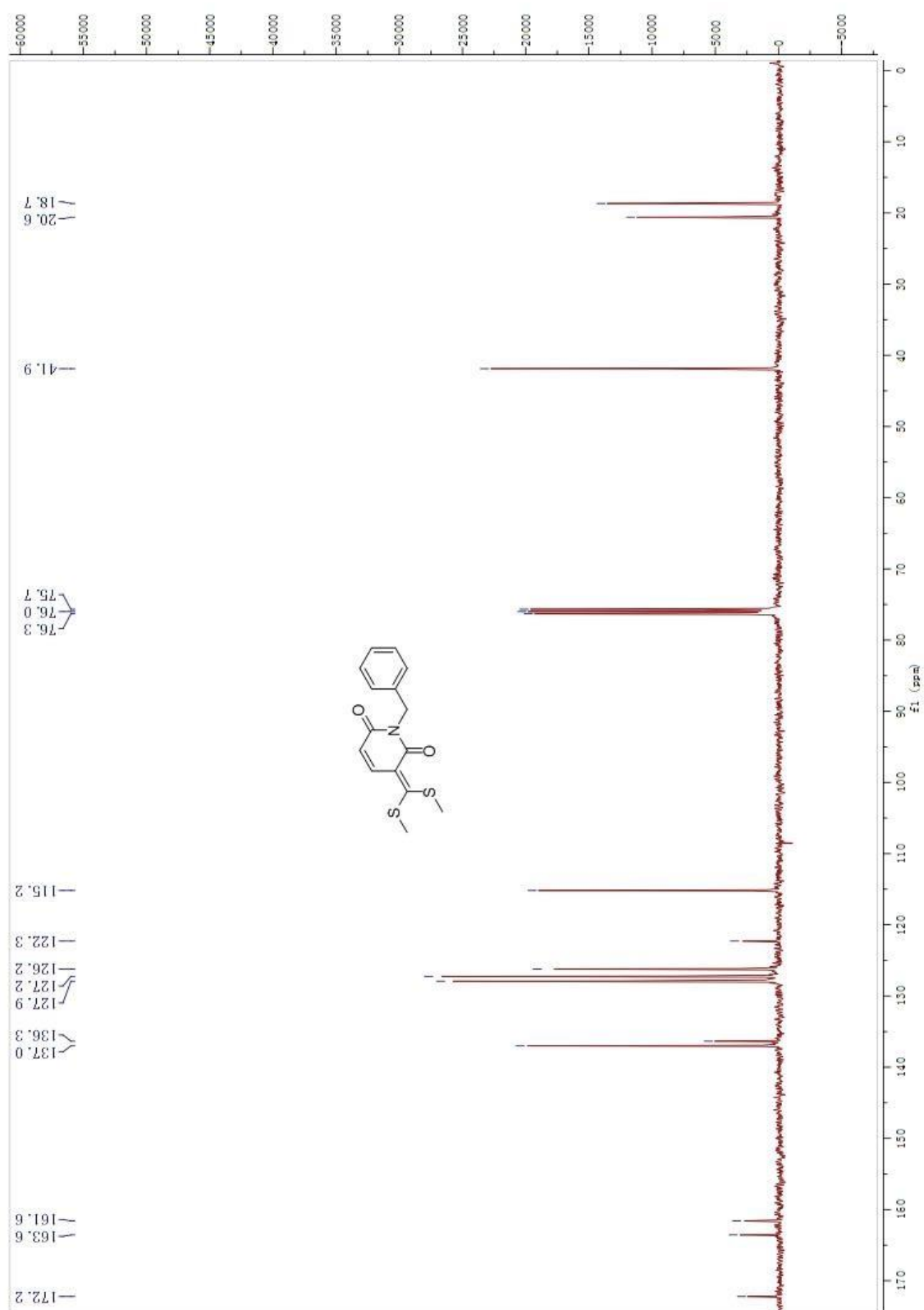
2e



2f

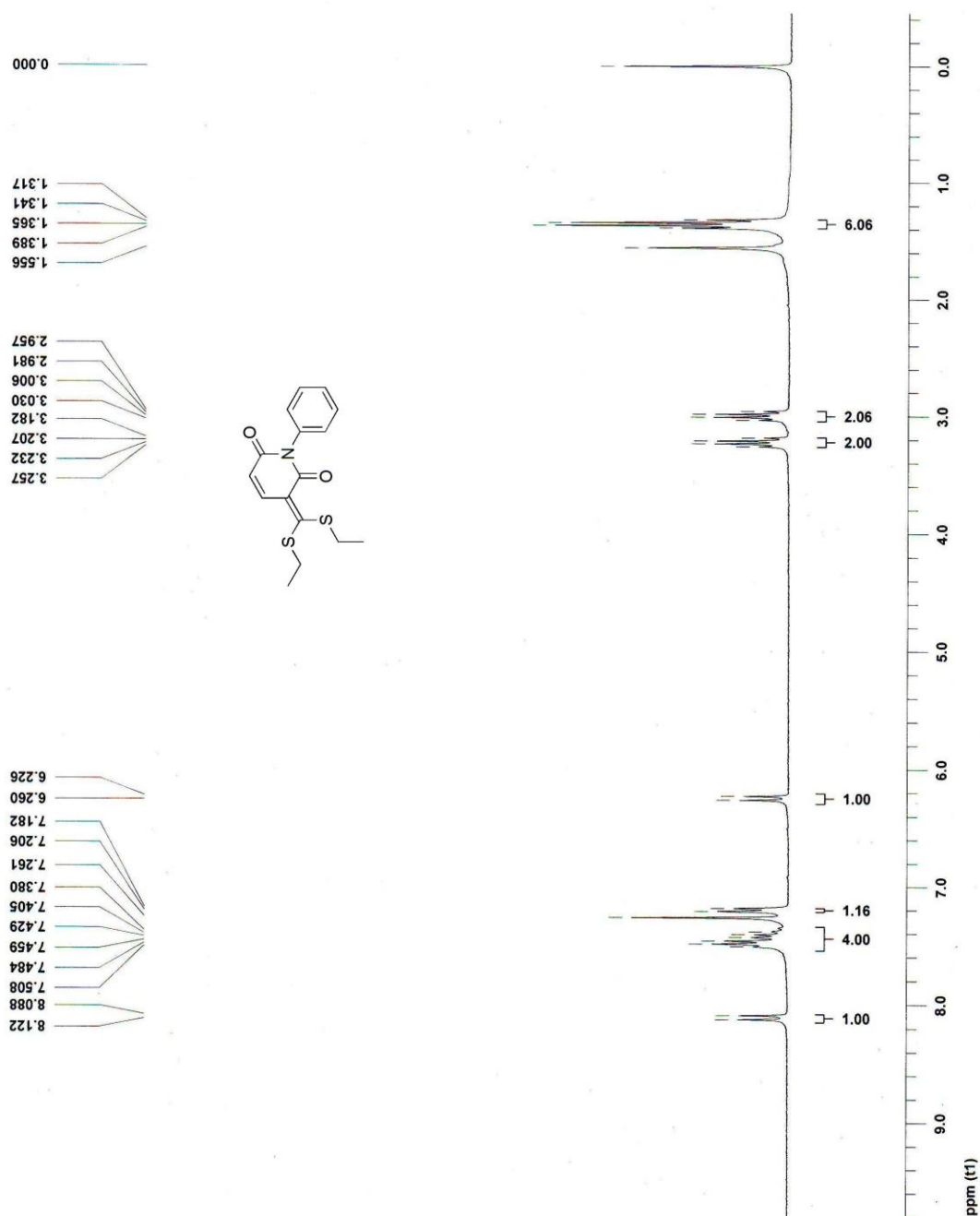


2f



2g

Date: 24 May 2010
 Document's Title: fid4
 Spectrum Title: None
 Frequency (MHz): (f1) 300.132
 Original Points Count: (f1) 16384
 Actual Points Count: (f1) 32768
 Acquisition Time (sec): (f1) 2.7329
 Spectral Width (ppm): (f1) 19.975
 Pulse Program: ZG
 Temperature: 673.16
 Number of Scans: 4
 Acq. Date: Fri Mar 19 04:01:12 PM



GFJ-2

Current Data Parameters
 NAME GFJ
 EXPNO 2
 PROCNO 1

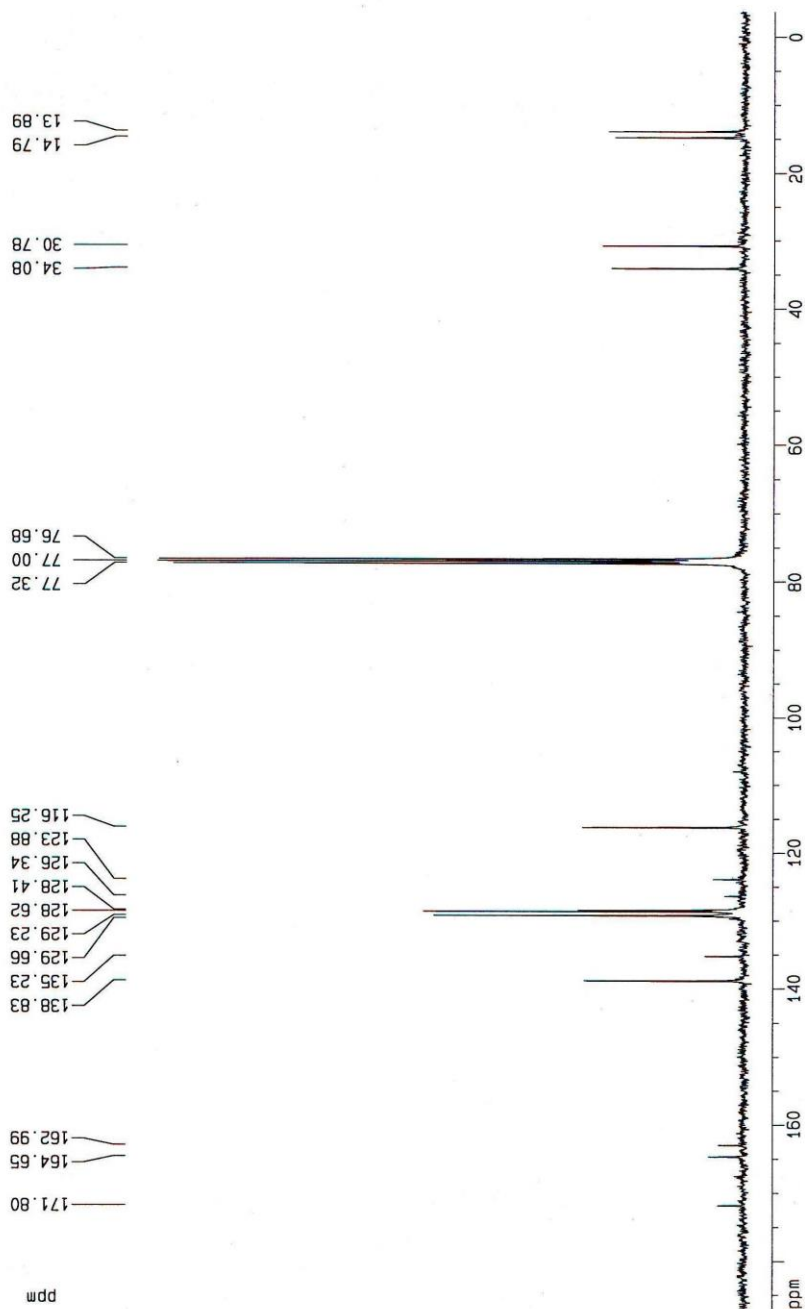
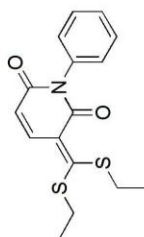
F2 - Acquisition Parameters
 Date_ 20100330
 Time 20.10
 INSTRUM av400
 PROBHD 5 mm BB-1H
 PULPROG zgpg30
 TD 16384
 SOLVENT CDCl3
 NS 4146
 DS 0
 SWH 25062.656 Hz
 FIDRES 1.52703 Hz
 AQ 0.3269108 sec
 RG 8192
 DW 19.950 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.69999999 sec
 d11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 2.80 usec
 PL1 -2.00 dB
 SF01 100.5785294 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 18.50 dB
 SF02 399.9515998 MHz

F2 - Processing parameters
 SI 65536
 SF 100.5675118 MHz
 NQW EM
 SSB 0
 LB 4.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 22.00 cm
 CY 10.00 cm
 FIP 187.021 ppm
 F1 18808.25 Hz
 F2 -3.738 ppm
 FZ -375.91 Hz
 PPMCM 8.67086 ppm/cm
 HZCM 872.00690 Hz/cm



2h

STANDARD PROTON PARAMETERS

Archive directory: /export/home/11uy/vnmrsys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

File: f712

INOVA-500 "NENUS00"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 8164.1 Hz

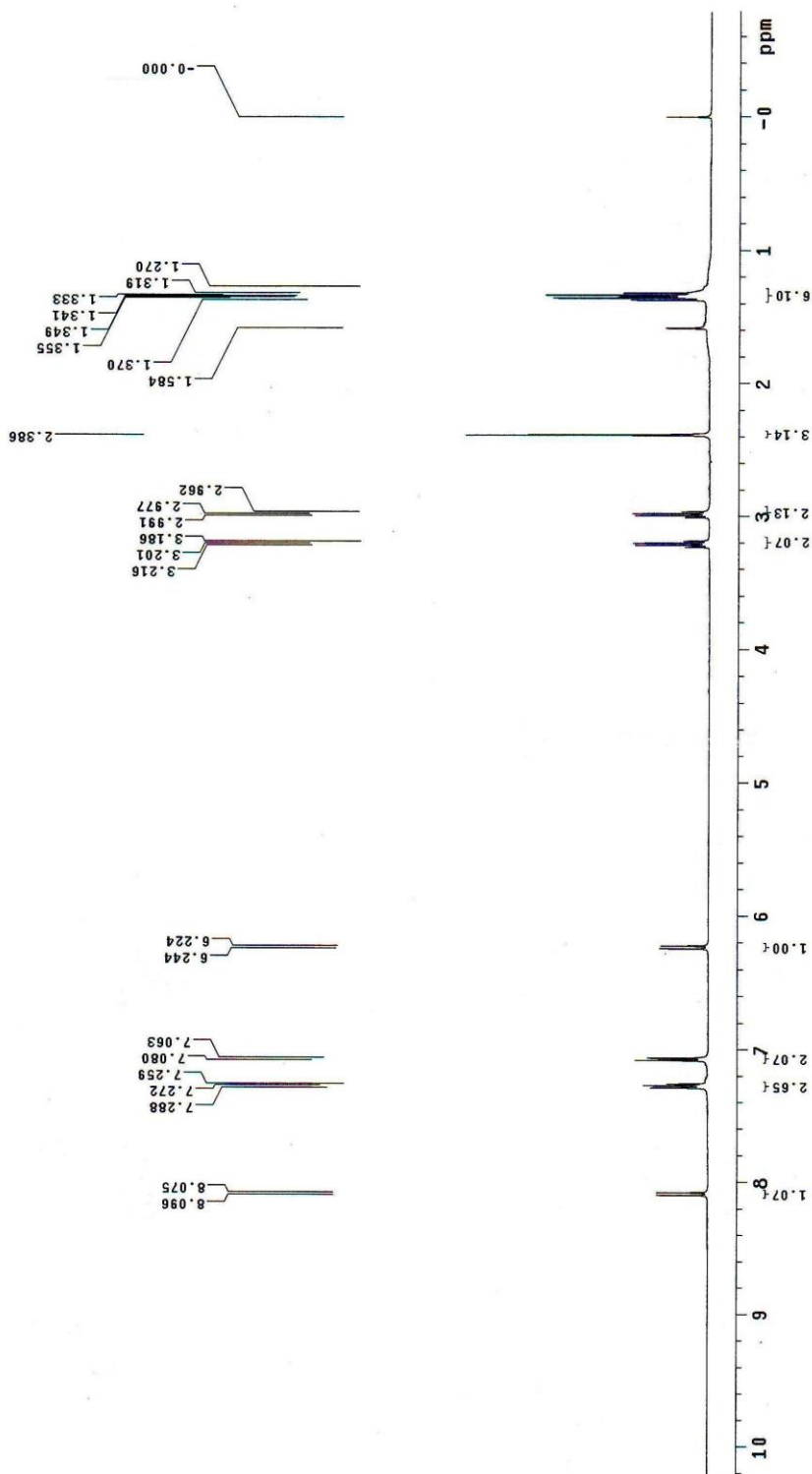
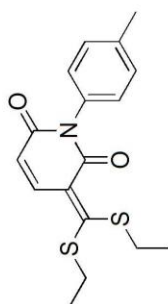
32 repetitions

OBSERVE H1, 499.8025928 MHz

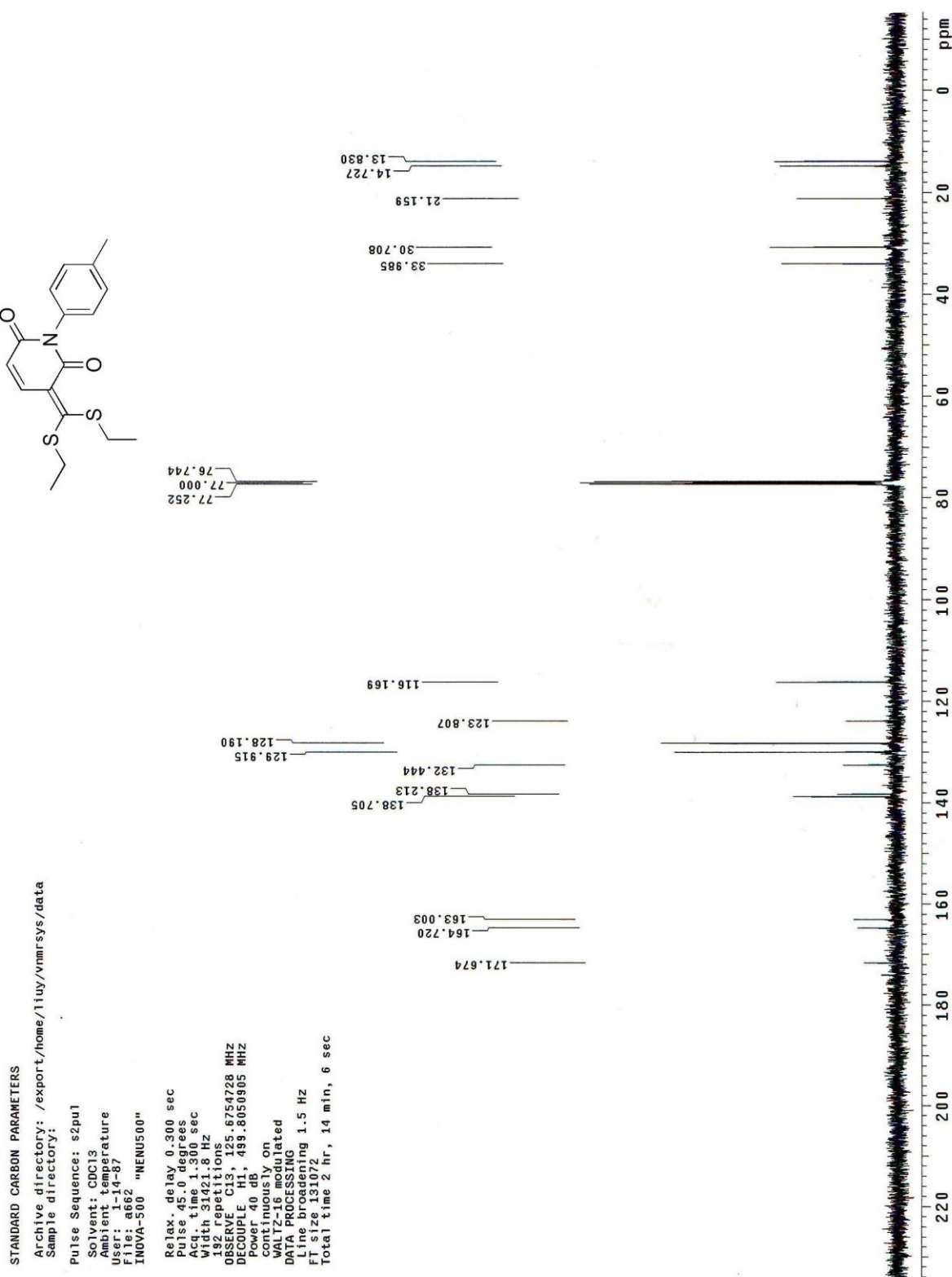
DATA PROCESSING

File: f712-65536

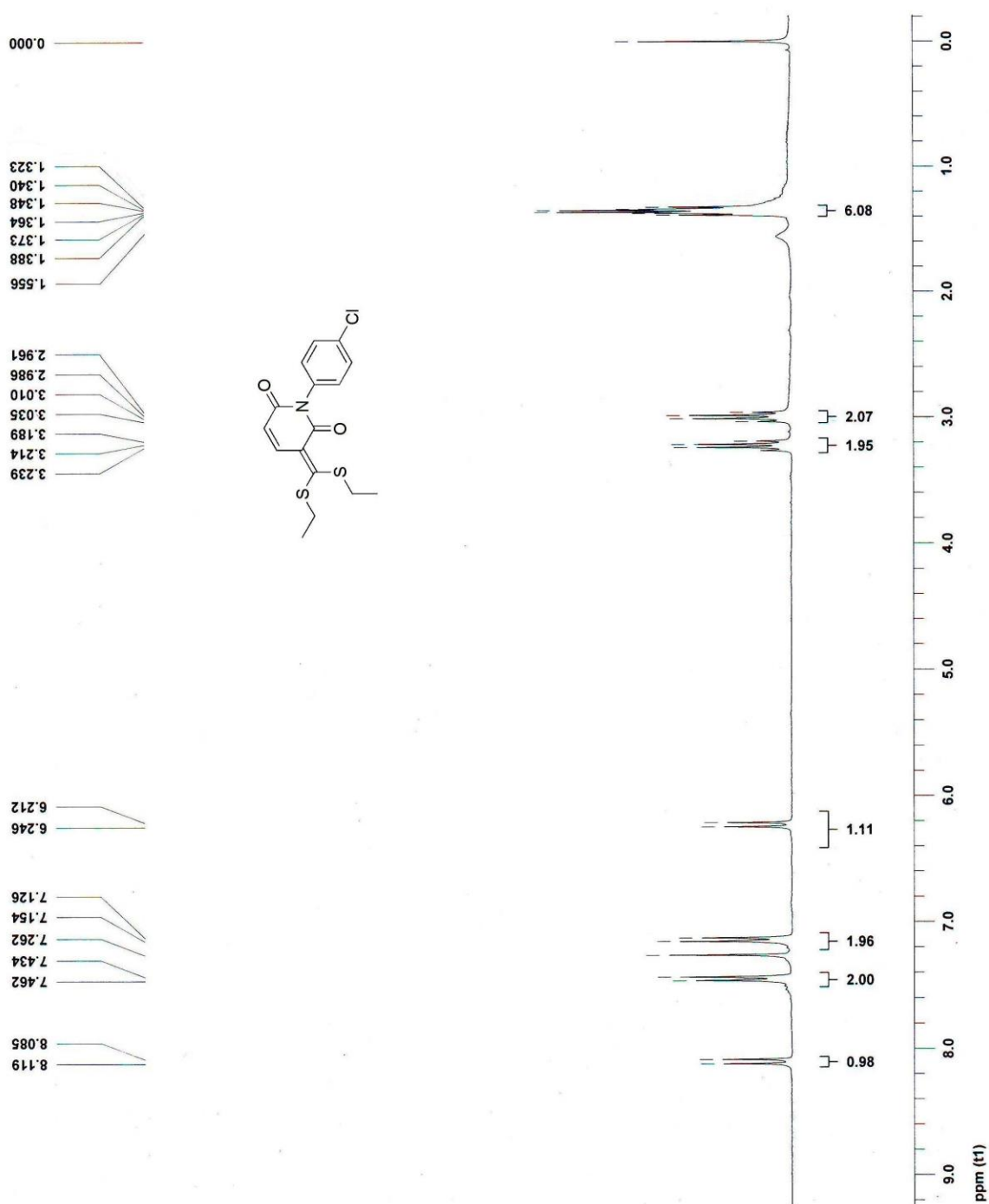
Total time 1 min, 32 sec



2h



Date: 24 May 2010
 Document's Title: Cl
 Spectrum Title: None
 Frequency (MHz): (f1) 300.132
 Original Points Count: (f1) 16384
 Actual Points Count: (f1) 32768
 Acquisition Time (sec): (f1) 2.7329
 Spectral Width (ppm): (f1) 19.975
 Pulse Program: ZG
 Temperature: 673.16
 Number of Scans: 5
 Acq. Date: Fri Jan 22 04:42:25 PM



GFJ-3

Current Data Parameters
 NAME GFJ
 EXPNO 1
 PROCNO 1

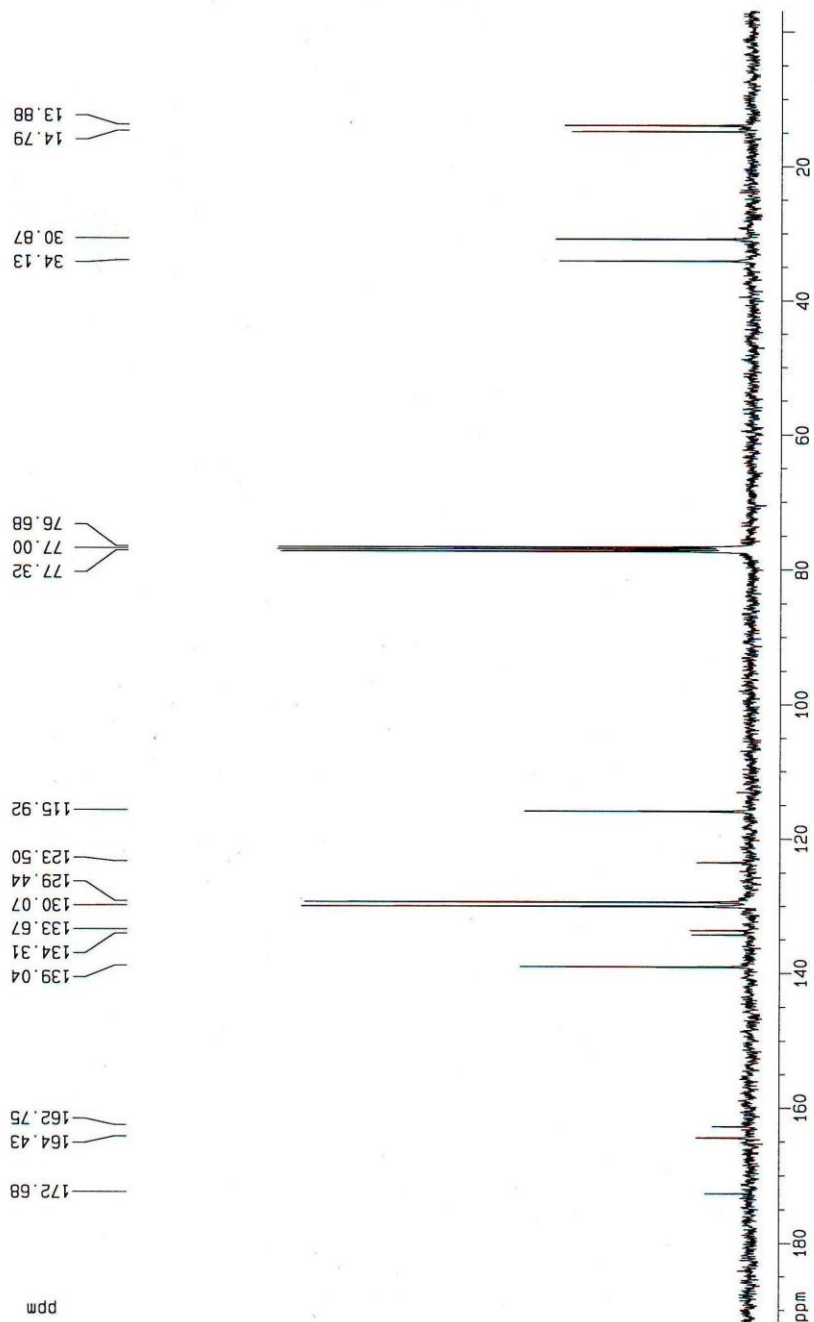
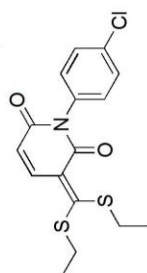
F2 - Acquisition Parameters
 Date_ 20100330
 Time 19.56
 INSTRUM av400
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg
 TD 16384
 SOLVENT COC13
 NS 506
 DS 0
 SWH 25062.656 Hz
 FIDRES 1.529703 Hz
 AQ 0.3269108 sec
 RG 8192
 DM 19.950 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.69999999 sec
 d11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 ¹³C
 P1 2.80 usec
 PL1 -2.00 dB
 SF01 100.5785294 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 ¹H
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 18.50 dB
 SF02 399.9515998 MHz

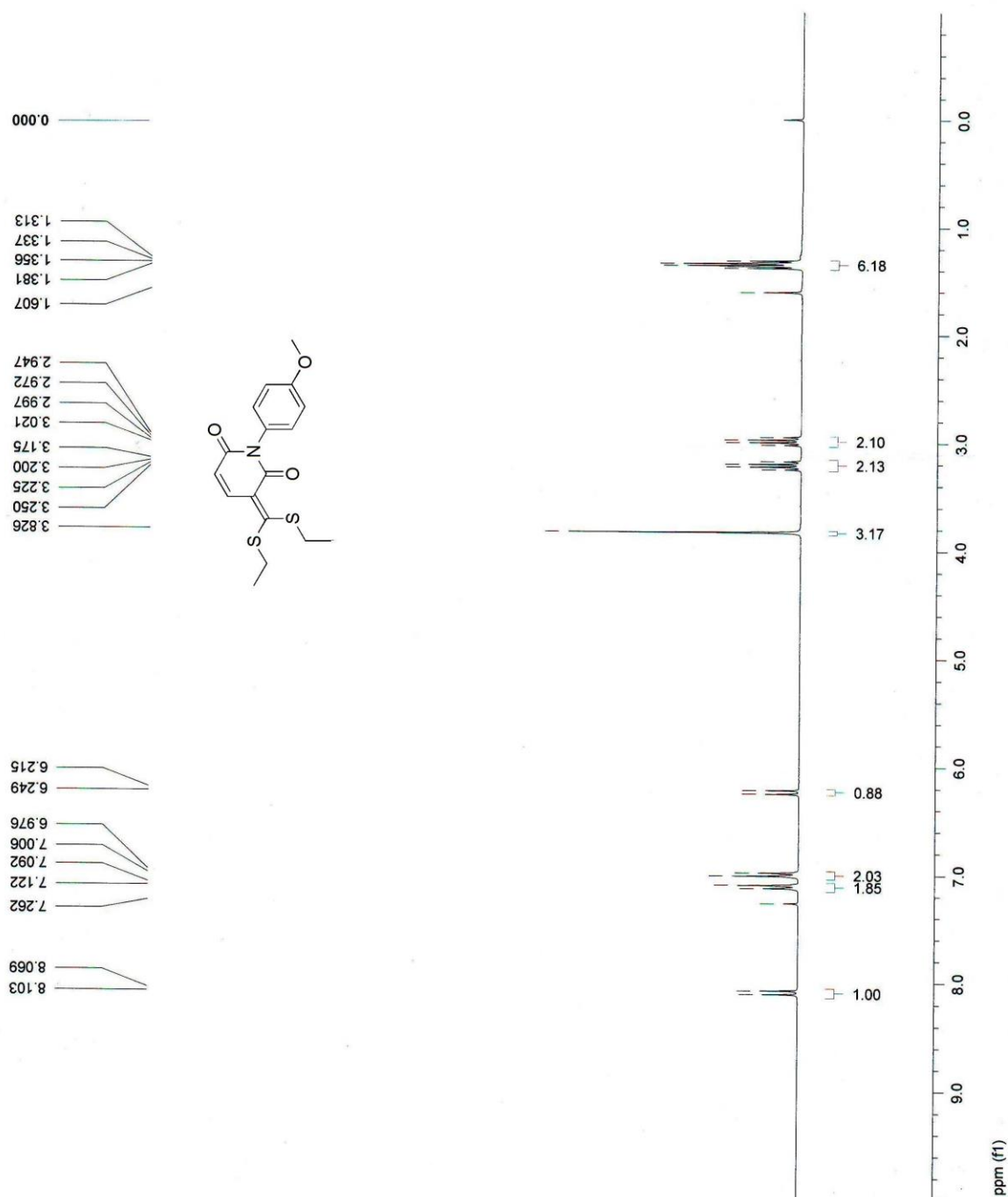
F2 - Processing parameters
 SI 65536
 SF 100.5675137 MHz
 MDW EM
 SSB 0
 LB 4.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 22.00 cm
 CY 8.00 cm
 F1P 191.716 ppm
 F1 19280.40 Hz
 F2P -3.128 ppm
 F2 -314.61 Hz
 PPMCM 8.85655 ppm/cm
 HZCM 890.68231 Hz/cm

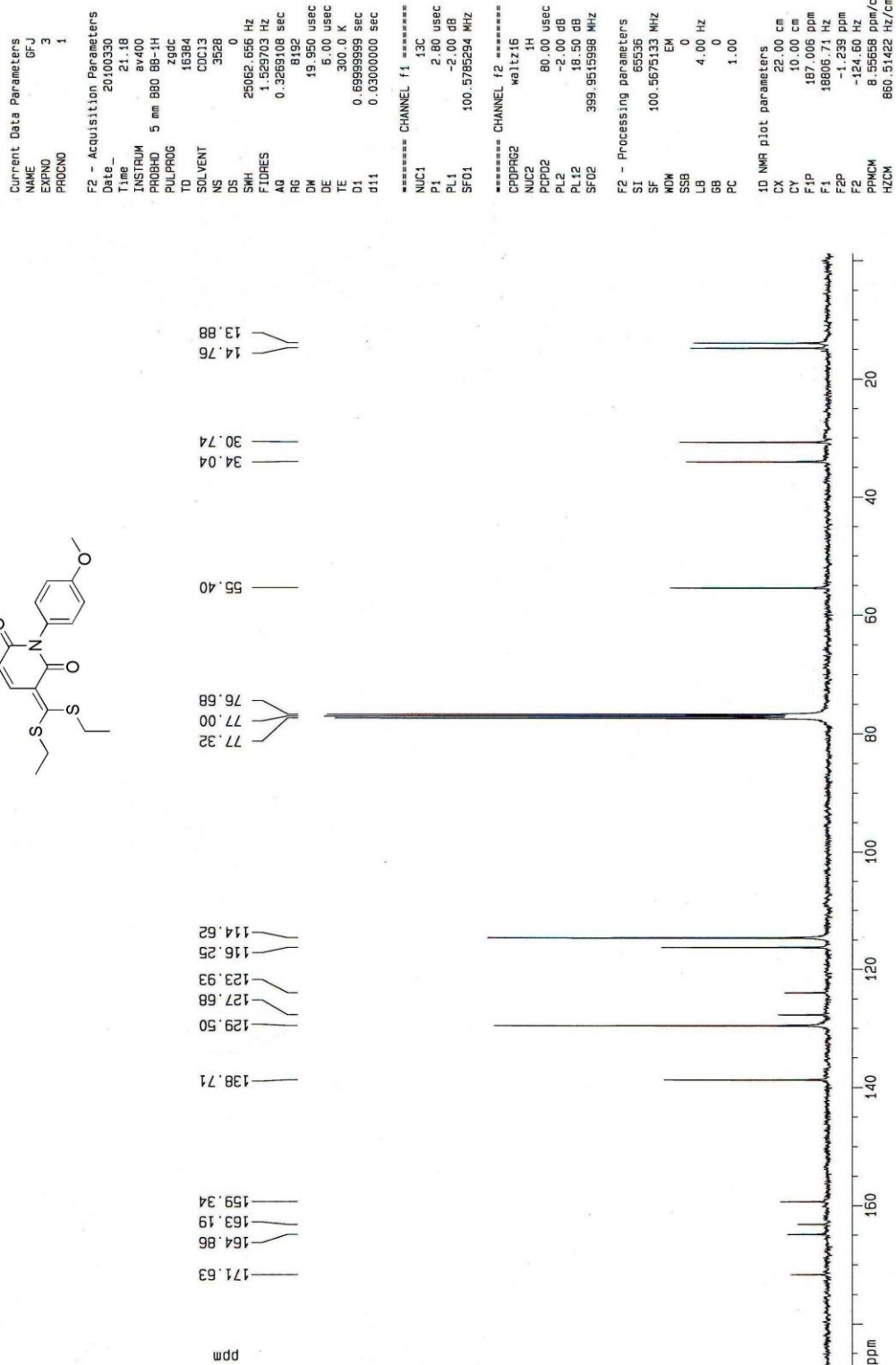
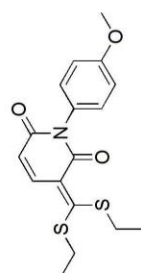


2j

Date: 11 Jul 2009
Document's Title: fid
Spectrum Title: None
Frequency (MHz): (f1) 300.132
Original Points Count: (f1) 16384
Actual Points Count: (f1) 32768
Acquisition Time (sec): (f1) 2.7329
Spectral Width (ppm): (f1) 19.975
Pulse Program: ZG
Temperature: 673.16
Number of Scans: 5
Acq. Date: Fri Jul 10 05:51:38 PM

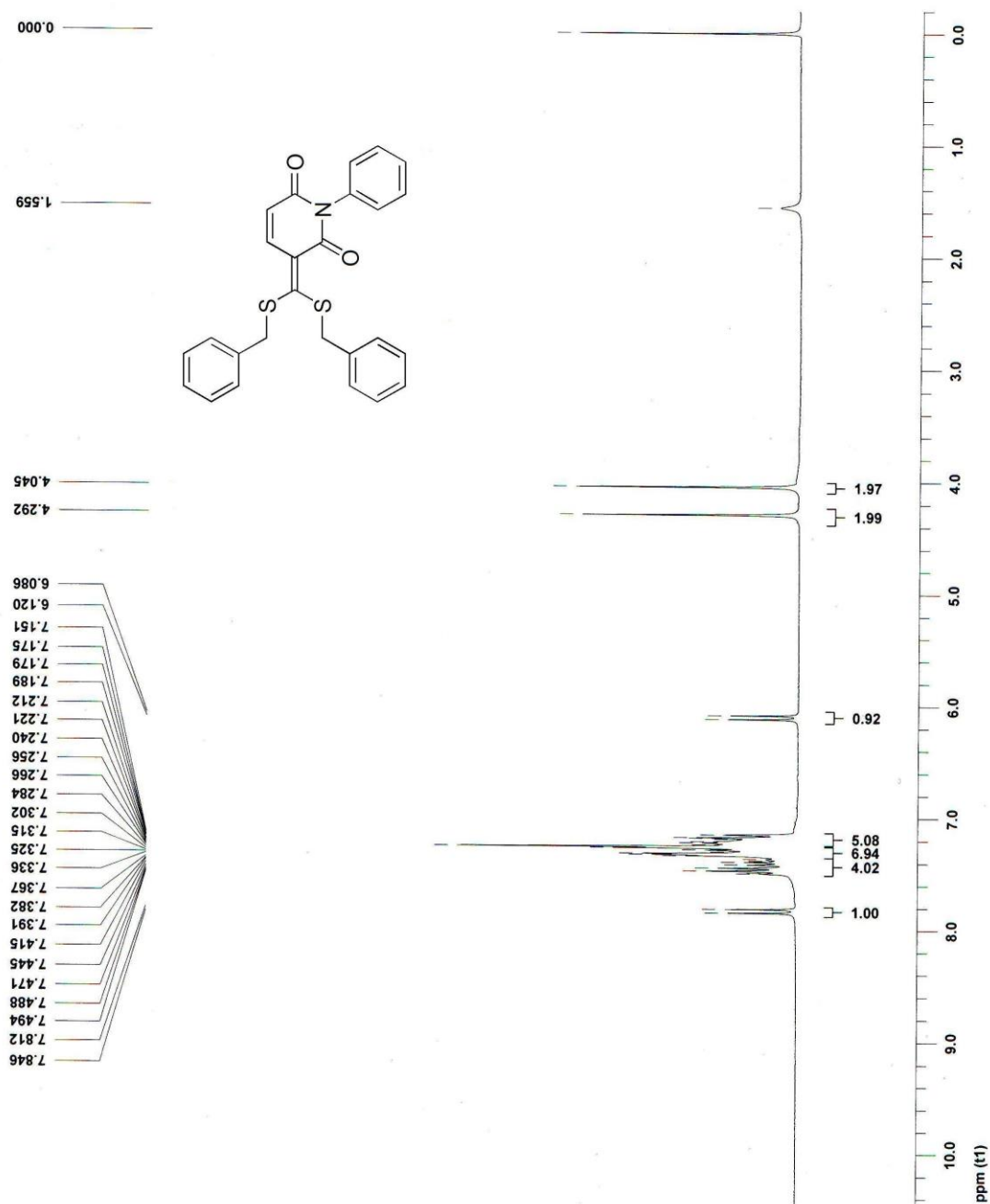


gfj-1



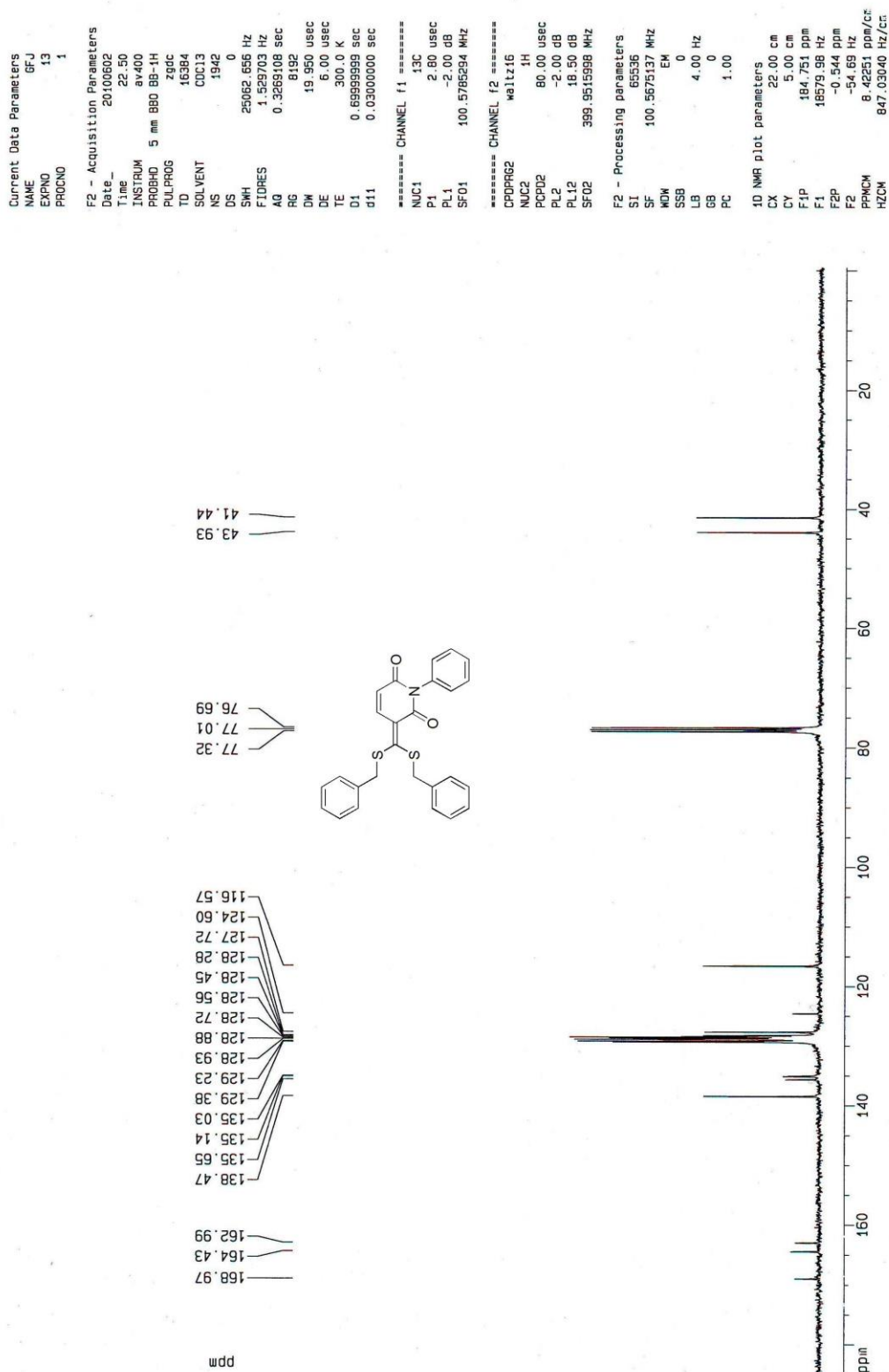
2k

Date: 30 May 2010
 Document's Title: fid9
 Spectrum Title: None
 Frequency (MHz): (f1) 300.132
 Original Points Count: (f1) 16384
 Actual Points Count: (f1) 32768
 Acquisition Time (sec): (f1) 2.7329
 Spectral Width (ppm): (f1) 19.975
 Pulse Program: ZG
 Temperature: 673.16
 Number of Scans: 5
 Acq. Date: Fri May 28 05:00:32 PM

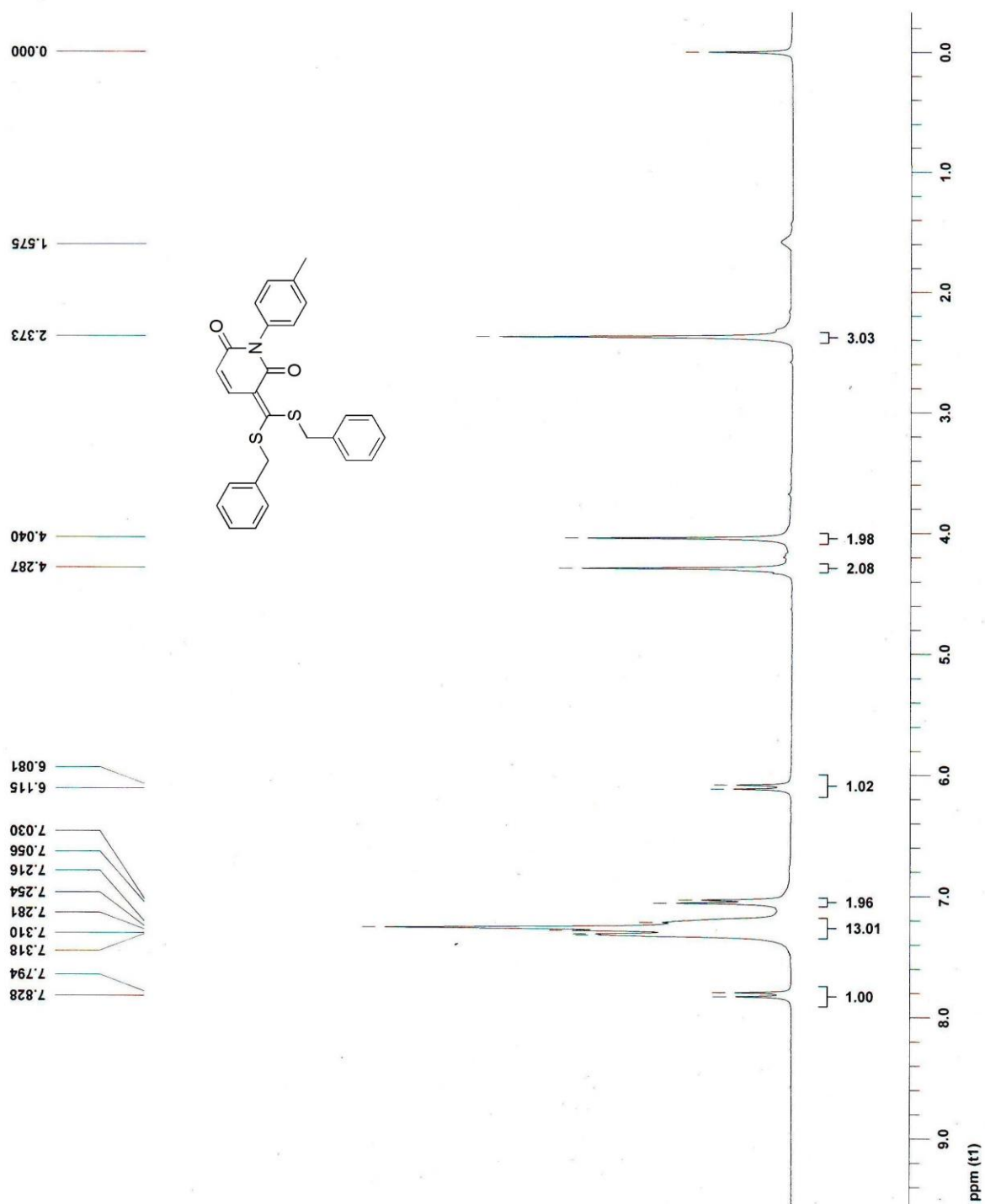


2k

GFJ-1



Date: 24 May 2010
 Document's Title: fid1
 Spectrum Title: None
 Frequency (MHz): (f1) 300.132
 Original Points Count: (f1) 16384
 Actual Points Count: (f1) 32768
 Acquisition Time (sec): (f1) 2.7329
 Spectral Width (ppm): (f1) 19.975
 Pulse Program: ZG
 Temperature: 673.16
 Number of Scans: 11
 Acq. Date: Fri May 14 04:01:46 PM



GFJ-3

Current Data Parameters
 NAME GFJ
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20100531
 Time 20.51
 INSTRUM av400
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 16384
 SOLVENT CDCl3
 NS 2208
 DS 0
 SWH 25062.656 Hz
 FIDRES 1.523703 Hz
 AQ 0.3269108 sec
 RG 8192
 DW 19.950 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.69999999 sec
 d11 0.03000000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 2.80 usec
 PL1 -2.00 dB
 SF01 100.5785294 MHz

===== CHANNEL f2 =====
 CPOPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.00 dB
 PL12 18.50 dB
 SF02 399.9515998 MHz

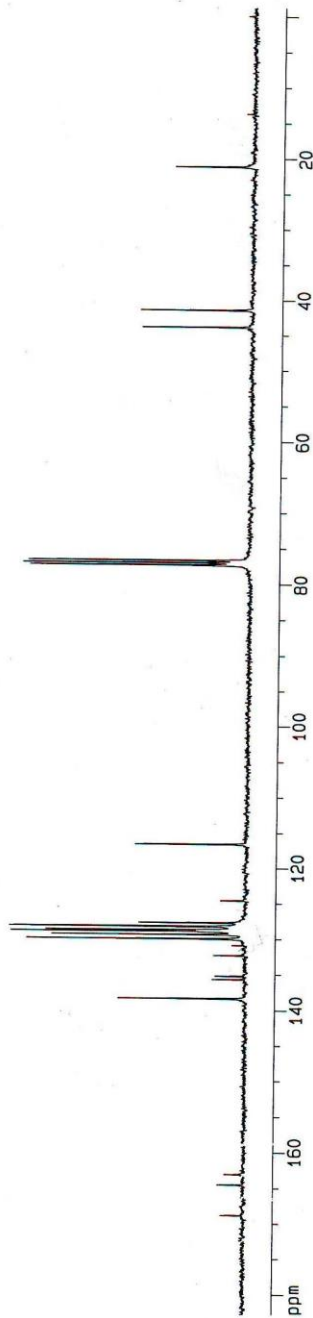
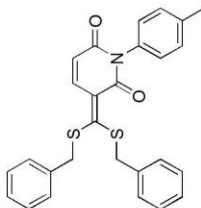
F2 - Processing parameters
 SI 65536
 SF 100.5675145 MHz
 MDW EM
 SSB 0
 LB 4.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 22.00 cm
 CY 4.00 cm
 F1P 182.807 ppm
 F1 18384.43 Hz
 F2P -1.197 ppm
 F2 -120.38 Hz
 PPMCM 8.36381 ppm/cm
 HZCM 841.12781 Hz/cm

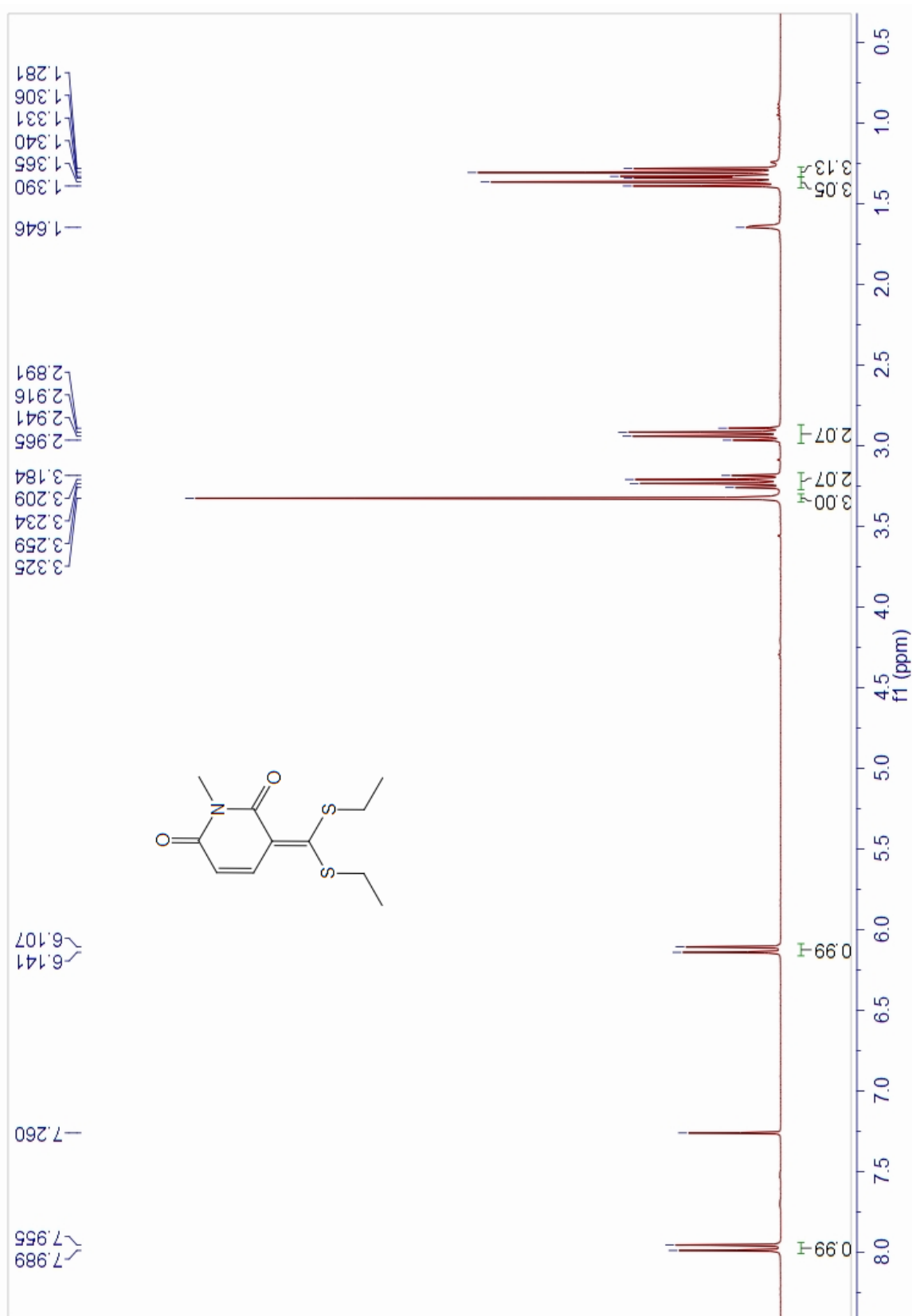
21.19

41.44
43.8776.68
77.00
77.32
 116.61
 124.59
 127.69
 128.19
 128.25
 128.70
 128.87
 128.91
 129.35
 129.97
 132.30
 135.20
 135.66
 138.36
163.05
164.54
168.81

ppm



2m



2m

