

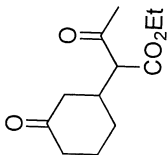
**Efficient organocatalytic Michael addition reaction of
 β -ketoesters under high pressure**

Maya Moritaka, Keiji Nakano, Yoshiyasu Ichikawa, and Hiyoshizo Kotsuki*

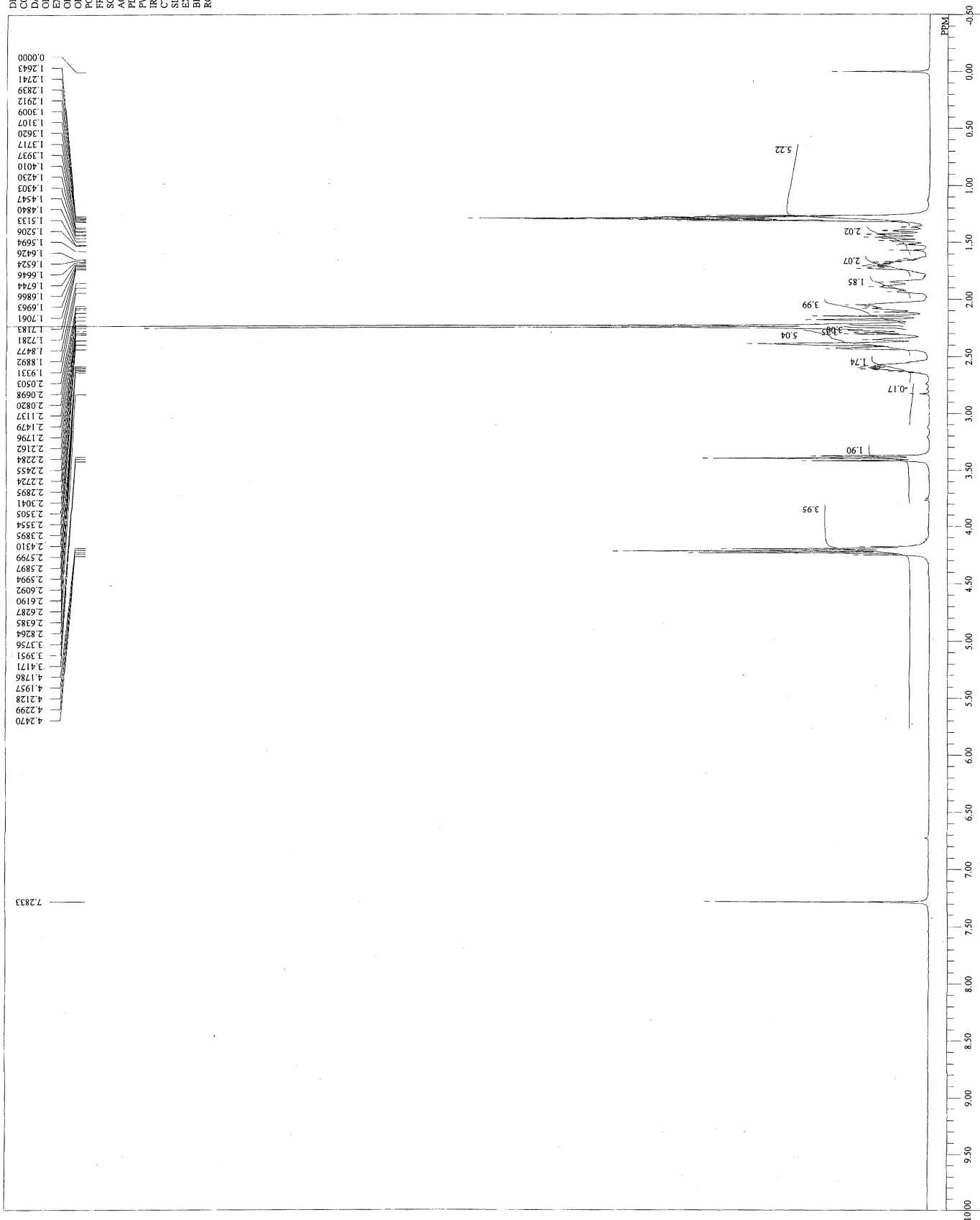
*Laboratory of Natural Products Chemistry,
Faculty of Science, Kochi University,
Akebono-cho, Kochi 780-8520, Japan
kotsuki@kochi-u.ac.jp*

**¹H & ¹³C NMR Data
(24 Pages)**

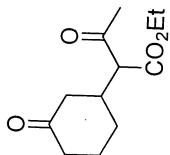
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 OBFRQ: 134300.00 Hz
 POINT: 8192
 FREQU: 7993.60 Hz
 SCANS: 8
 ACQTM: 1.0248 sec
 PD: 5.9752 sec
 PW1: 5.75 usec
 IRNUC: 1H
 CTEMP: 17.1 c
 N1PRG: CDCCL3
 N1VER: 0.00 ppm
 EXREF: 0.12 Hz
 BR: 17
 RGAIN: 17



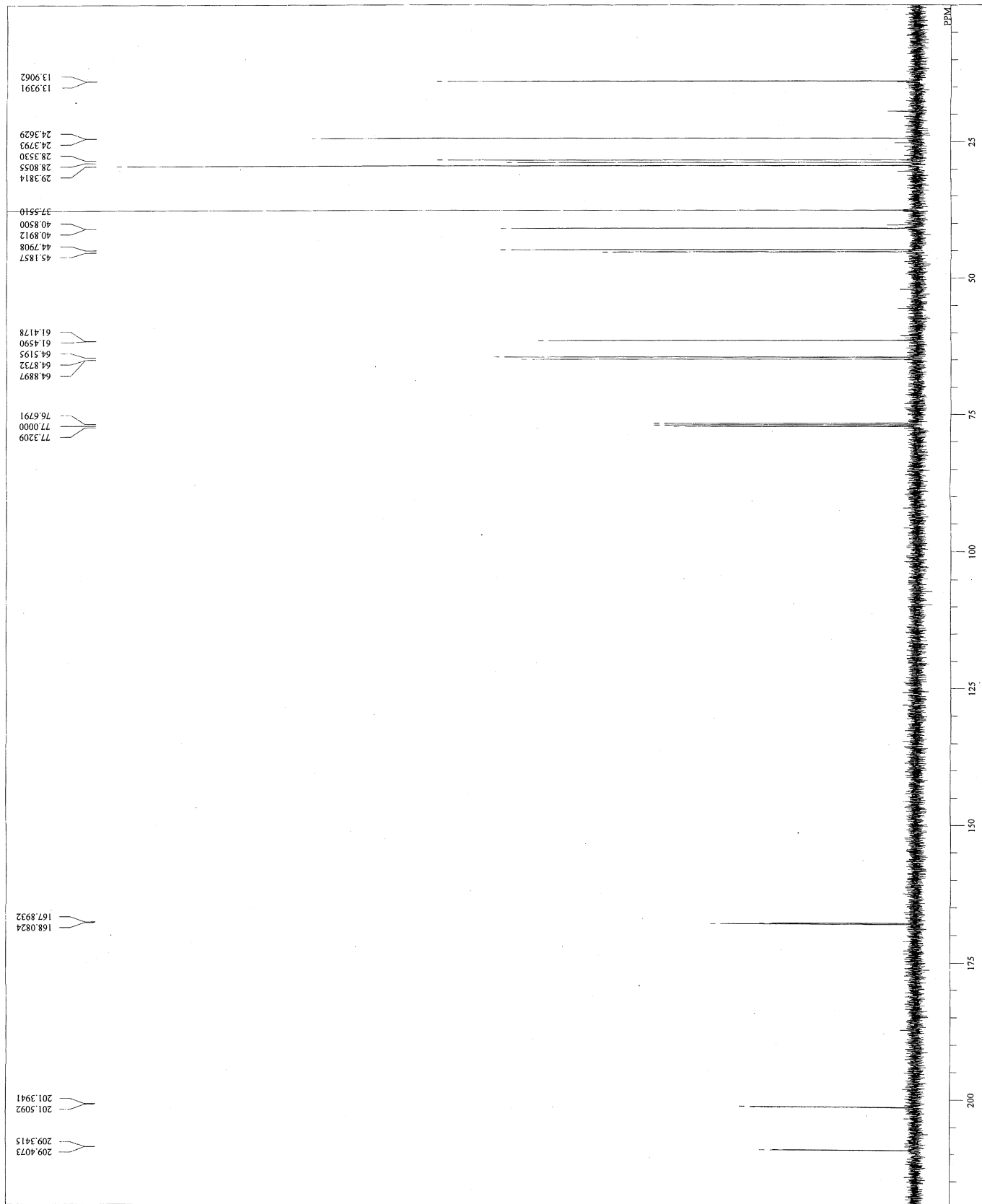
3a
¹H NMR
 (400 MHz, CDCl₃)



MYY-508-13cals
 Sat Apr 09 17:51:54 2011
 13C
 bom
 100.40 MHz
 135500.00 Hz
 32768
 27100.27 Hz
 128
 1.2091 sec
 1.7909 sec
 5.75 usec
 1H
 20.5 c
 CDCl₃
 77.00 ppm
 0.12 Hz
 26
 RGAIN



¹³C NMR
 (100 MHz, CDCl₃)



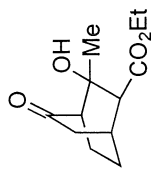
single_pulse

DFILE
COMNT
DATIM
OBNUC
EXMOD
OBFREQ
OBSFET
OBSFIN
POINT
FREQU
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

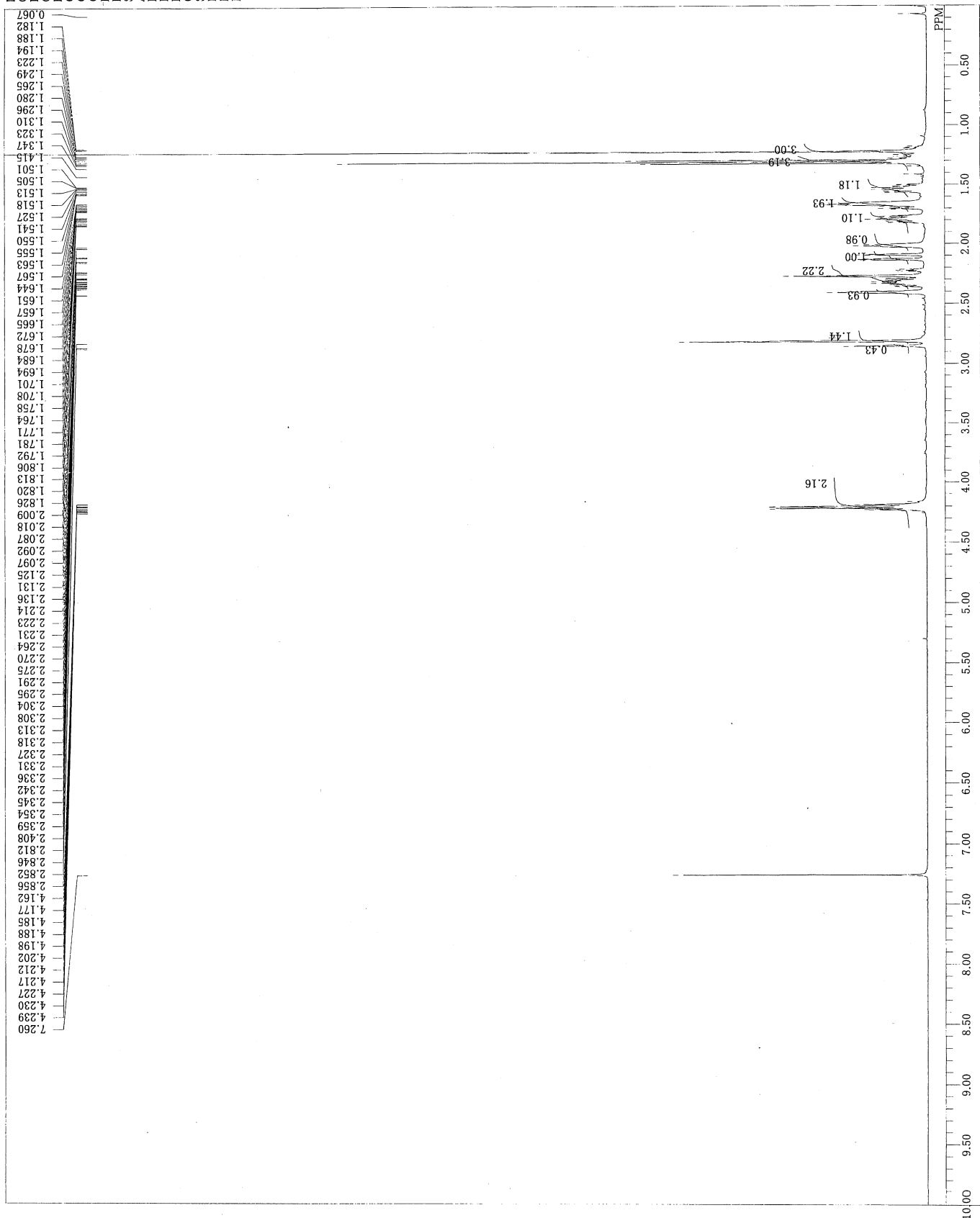
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1.182
1.188
1.194
1.203
1.235
1.249
1.263
1.280
1.296
1.310
1.323
1.347
1.415
1.501
1.505
1.513
1.518
1.527
1.541
1.550
1.555
1.563
1.567
1.644
1.651
1.657
1.665
1.672
1.678
1.684
1.694
1.701
1.708
1.758
1.764
1.771
1.781
1.792
1.806
1.813
1.820
1.826
2.009
2.018
2.087
2.092
2.097
2.125
2.131
2.136
2.214
2.223
2.231
2.264
2.270
2.275
2.291
2.295
2.304
2.308
2.313
2.318
2.327
2.331
2.336
2.342
2.345
2.354
2.359
2.408
2.812
2.846
2.852
2.856
4.162
4.177
4.185
4.188
4.198
4.202
4.212
4.217
4.227
4.230
4.239
7.260

MY-861bypro.proton-2-1.jdf
single_pulse
2012-03-10 11:14:17
1H

proton:kp
500.16 MHz
2.41 KHz
6.01 Hz
16400
9384.38 Hz
8
1.7459 sec
5.0000 sec
4.68 usec
1H 16.8 c
CDCl3
12.51 ppm
1.00 Hz
44

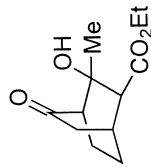


¹H NMR
(500 MHz, CDCl₃)



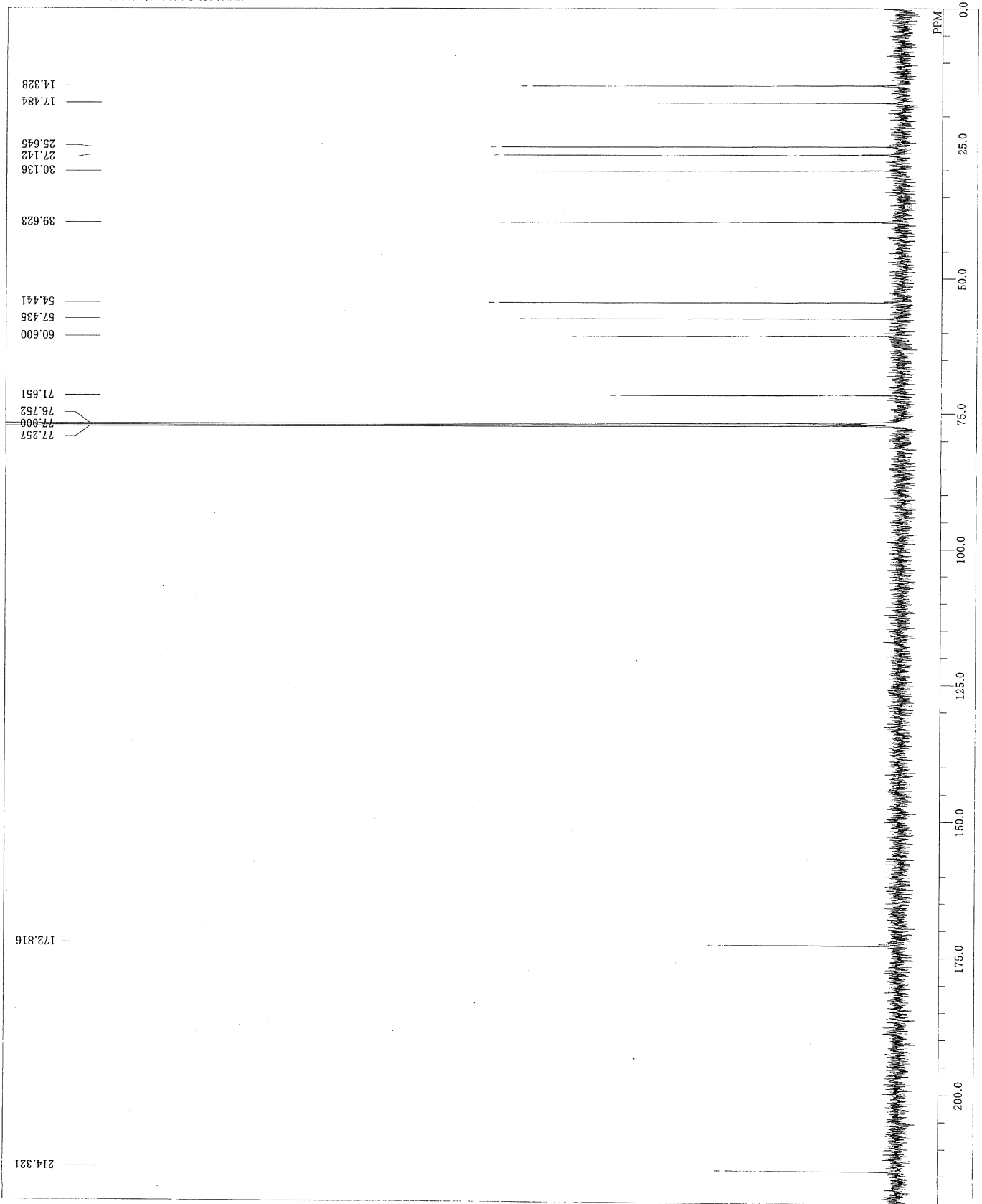
MYY-861hypro_Carbon_copy3-1-jk
 single pulse decoupled gated NOE
 2012-03-05 18:43:08
 13C
 carbon_1yp

125.77 MHz
 7.87 KHz
 4.21 Hz
 32780
 39308 18 Hz
 65
 0.0000 sec
 2.0000 sec
 2.72 usec
 1H 17.2 c
 CDCl3 77.00 ppm
 1.00 Hz
 54



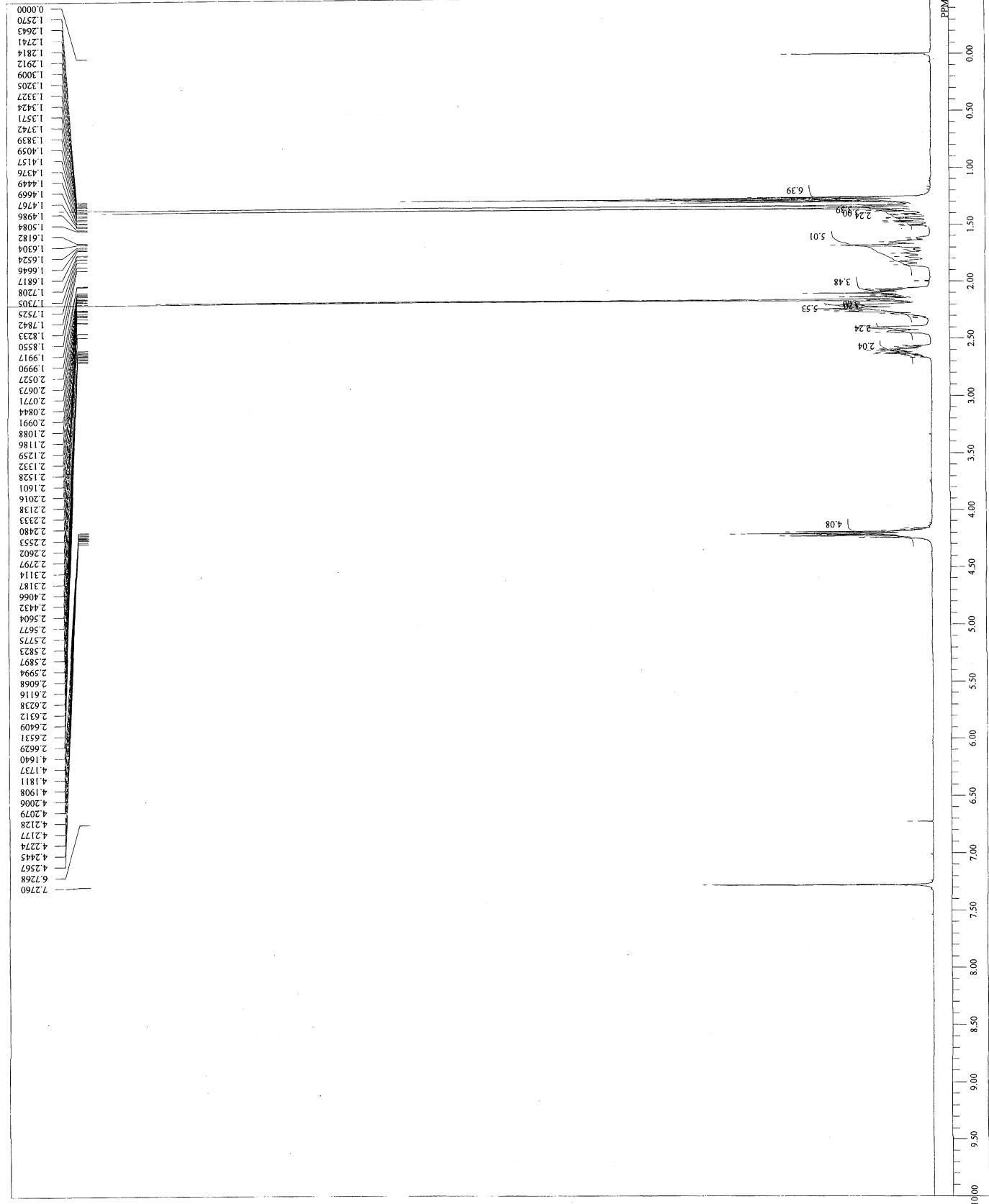
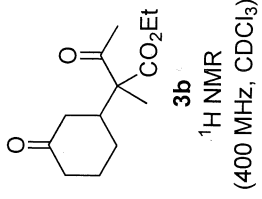
¹³C NMR
 (125.8 MHz, CDCl₃)

DFILE
 COMNT
 DATIM
 DBNJC
 EXMOD
 OBFRQ
 OBSET
 OBFTN
 POINT
 FREQU
 SCANS
 ACQTM
 PD
 PW1
 IRNUC
 CTMP
 SLVNT
 EXREF
 BF
 RGAIN

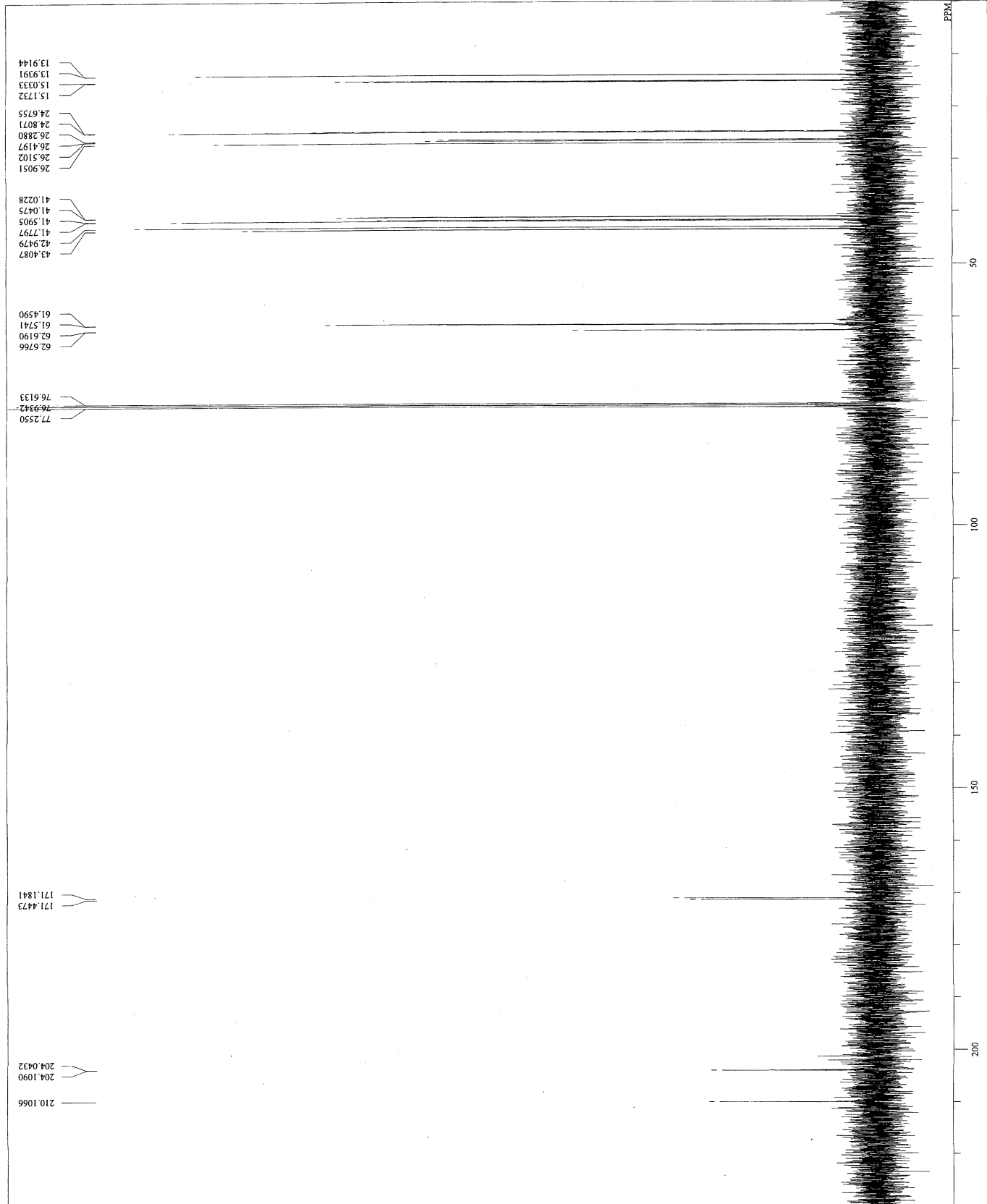
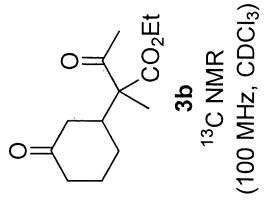


MYY-555 fr-13-18.als
 Mon Feb 14 23:30:38 2011
 1H
 100 399.65 MHz
 134300.00 Hz
 8192
 7993.60 Hz
 8
 1.0248 sec
 5.9752 sec
 5.75 usec
 1H
 16.1 c
 CDCl3
 0.00 ppm
 0.12 Hz
 19
 RGAIN

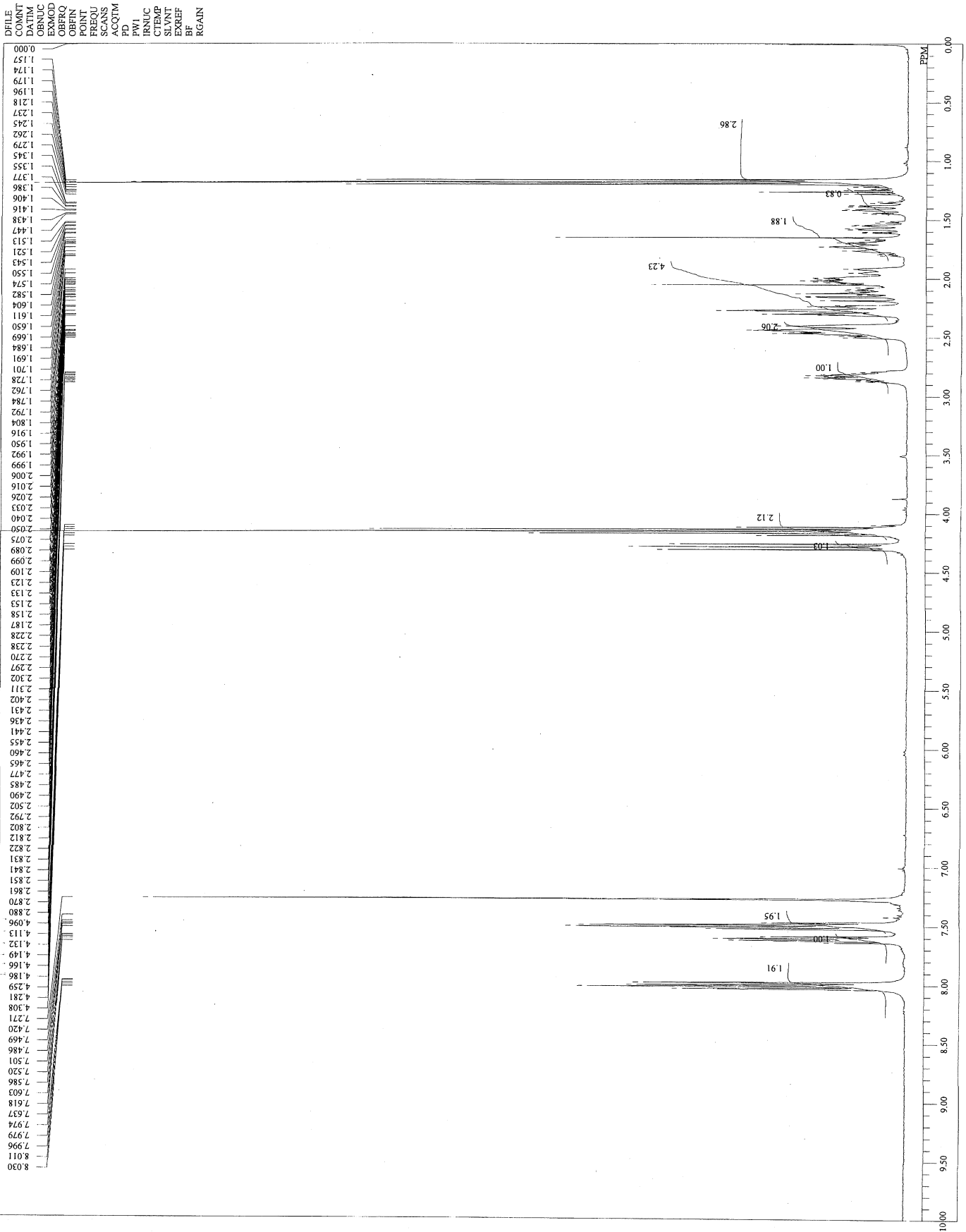
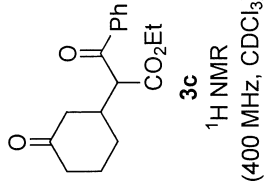
DPFILE
 COMINT
 DATIM
 EXAMID
 EXAMOD
 OBREIN
 POINT
 FREOU
 SCANS
 ACQTM
 PD
 PW1
 IRNUC
 CTEMP
 SLVNT
 EXREF
 BF
 RGAIN



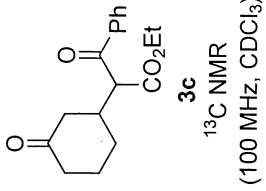
MYY-555 13C.als
 Thu Apr 21 22:26:47 2011
 13C
 bom
 100.40 MHz
 135500.00 Hz
 32768
 27100.27 Hz
 128
 1.2091 sec
 1.9989 sec
 5.75 msec
 1H
 20.0 c
 CDCl3
 77.00 ppm
 0.12 Hz
 28
 RGAIN



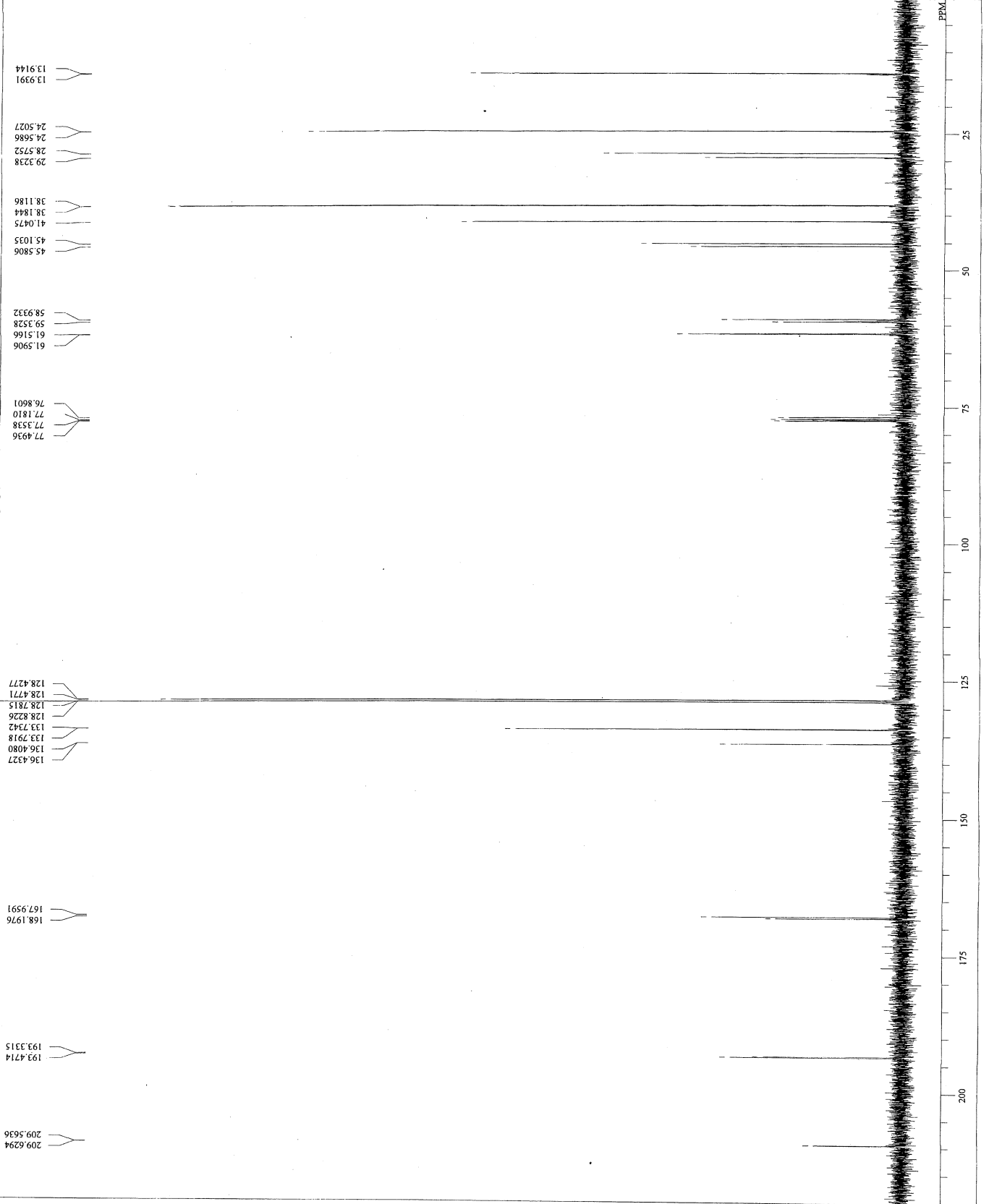
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 1H
 non
 399.65 MHz
 134300.00 Hz
 8192
 7993.60 Hz
 8
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 59752 sec
 5.75 usec
 1H
 TRANUC
 15.9 c
 CDCl3
 0.00 ppm
 0.12 Hz
 20
 RGAIN



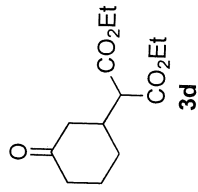
MYY-548-13c.als
 Sat Apr 09 17:38:38 2011
 13C
 hsm
 100.40 MHz
 135300.00 Hz
 32768
 27116.27 Hz
 65
 1.2091 sec
 1.7909 sec
 5.75 usec
 PD
 PW1 20.8 c
 IRNUC 1H
 CTEMP 77.00 ppm
 CDCL3
 SLVNT
 EXREF 0.12 Hz
 BF 28
 RGAIN



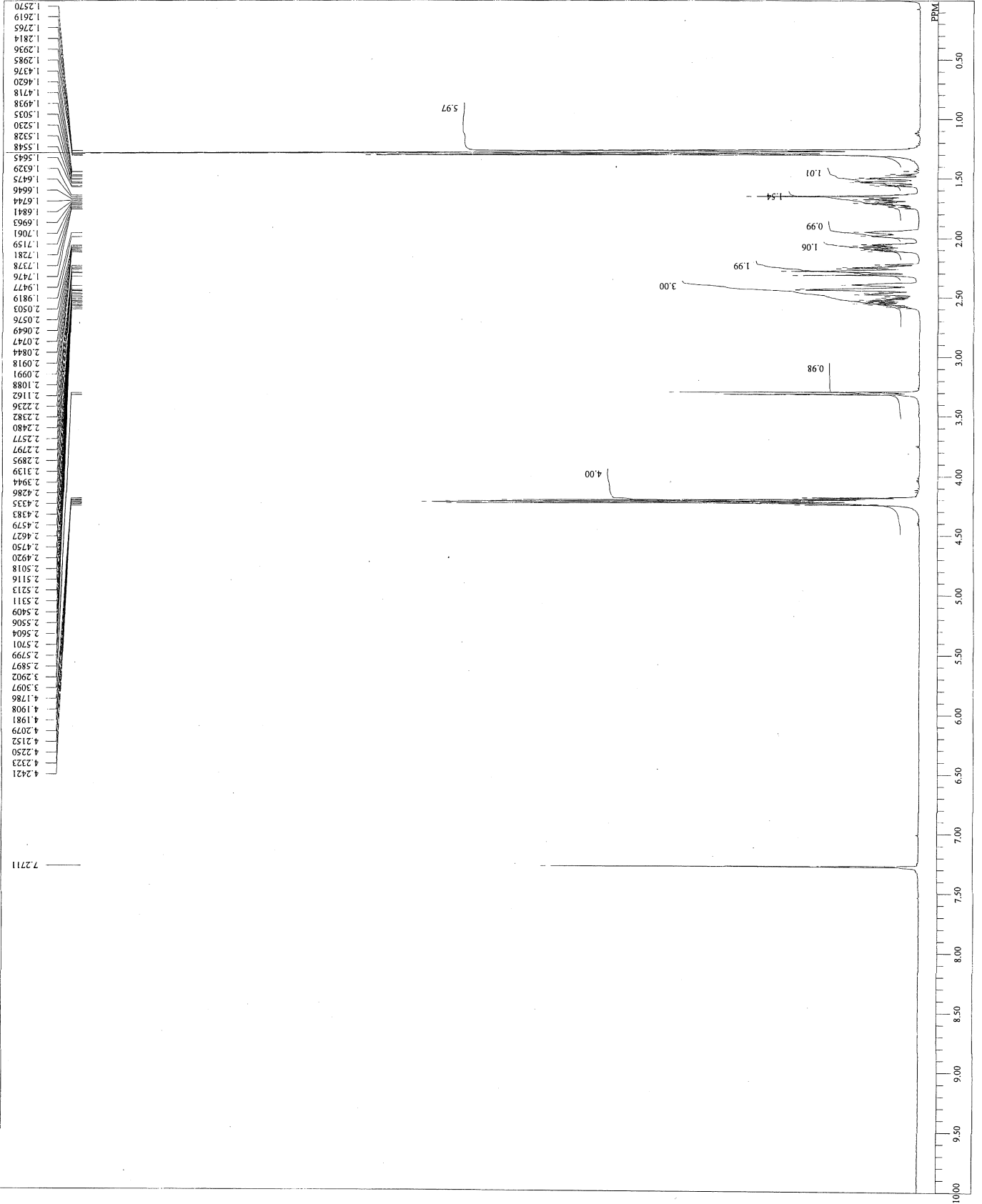
DFILE
 COMPT
 DATIM
 OBNUC
 EXMOD
 OBPRQ
 OBFIN
 POINT
 FOCUS
 SCANS
 ACQTM
 PD
 PW1
 IRNUC
 CTEMP
 SLVNT
 EXREF
 BF
 RGAIN



MYX-500 pure.als
 Thu Feb 17 19:11:23 2011
 1H
 399.65 MHz
 134300.00 Hz
 8192
 7993.60 Hz
 8
 1.0248 sec
 5.9752 sec
 5.75 usec
 1H
 18.9 c
 CDCL3
 0.00 ppm
 0.12 Hz
 18
 RGAIN

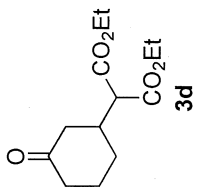


¹H NMR
 (400 MHz, CDCl₃)

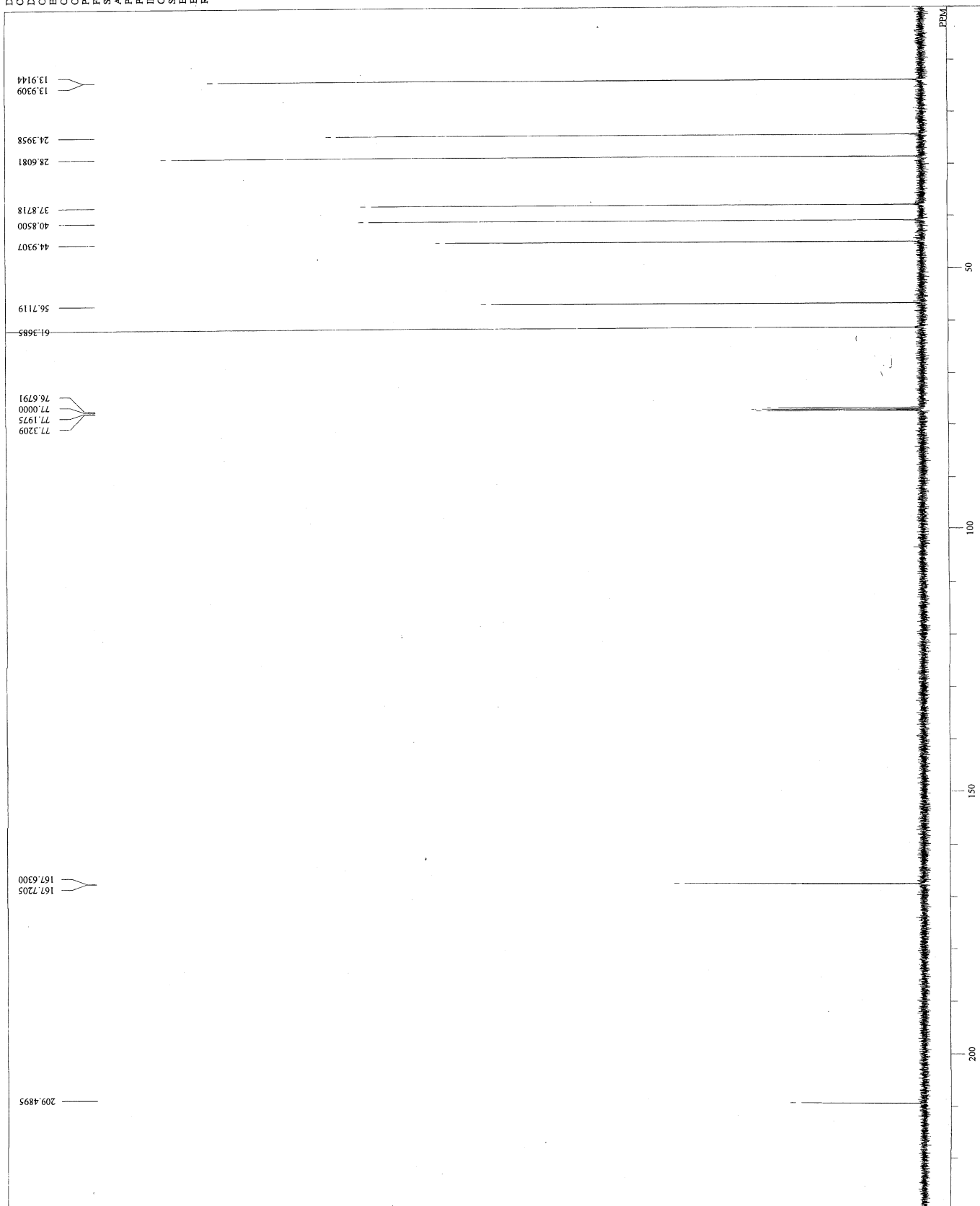


DFILE
COMNT
DATIM
OBNUC
EXMCD
OBFRO
OBFEN
POINT
FREQU
SCANS
ACQTM
PD
PW1
IRNUC
CTEMP
SOLVENT
EXREF
RF
RGAIN

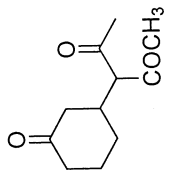
MY-560 13C.als
Thu Apr 21 22:14:57 2011
13C
ben
100.40 MHz
135500.00 Hz
32768
27100.27 Hz
128
1.2091 sec
1.7909 sec
5.75 usec
IH 20.1 c
CDCl₃
77.00 ppm
0.12 Hz
28



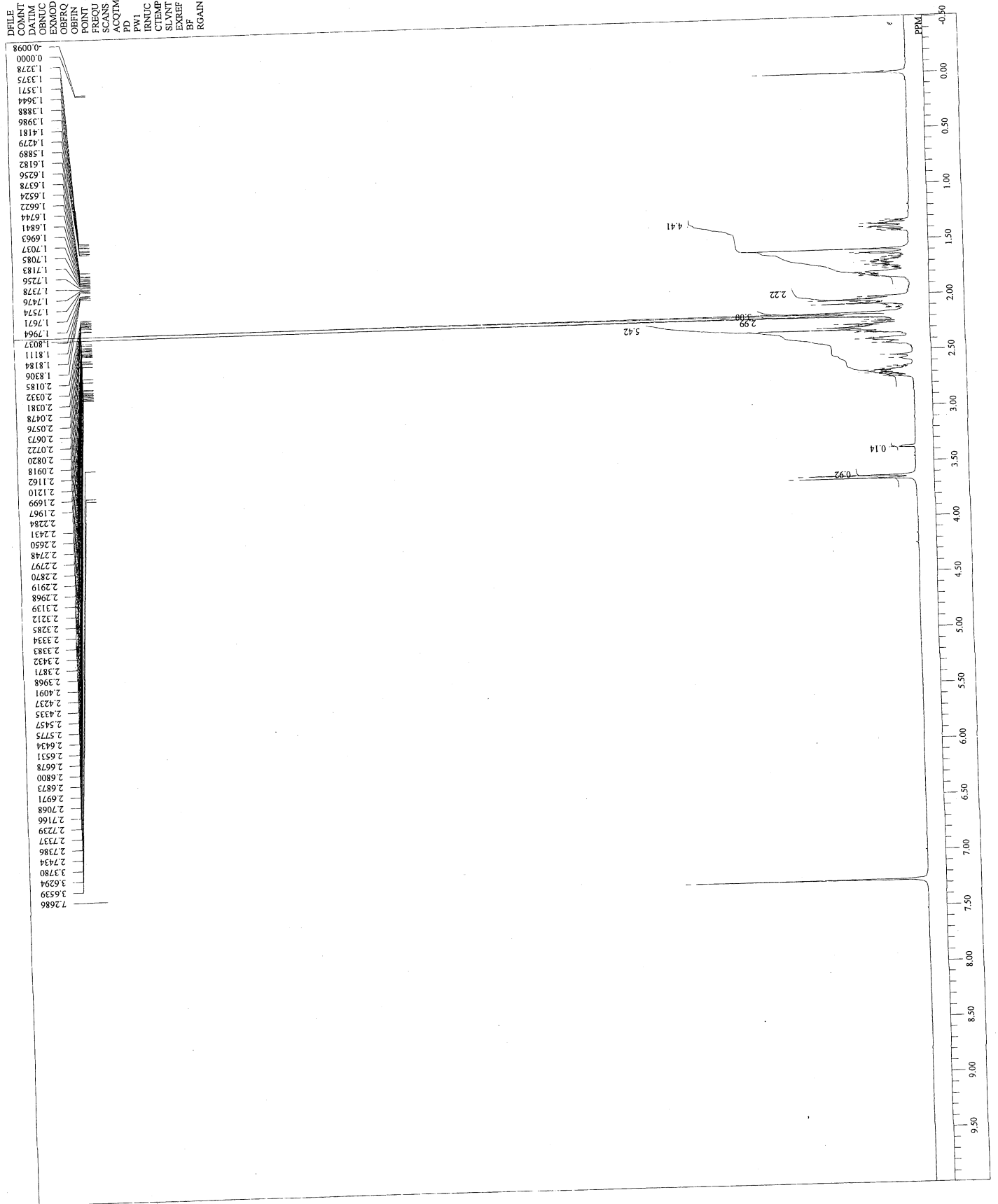
¹³C NMR
(100 MHz, CDCl₃)



MYY-558 pure.als
 Sat Feb 19 23:39:18 2011
 1H
 nu 99.65 MHz
 EXMOD 134300.00 Hz
 OBFRC 8192
 OBFIT 7993.60 Hz
 FREQU 8
 SCANS 1.0248 sec
 ACQTM 5.9752 sec
 PD 5.75 usec
 PW1 17.9 c
 IRNUC CDCL3
 CTEMP 0.00 ppm
 SLVNT EXREF 0.12 Hz
 BF 20
 RGAIN 20

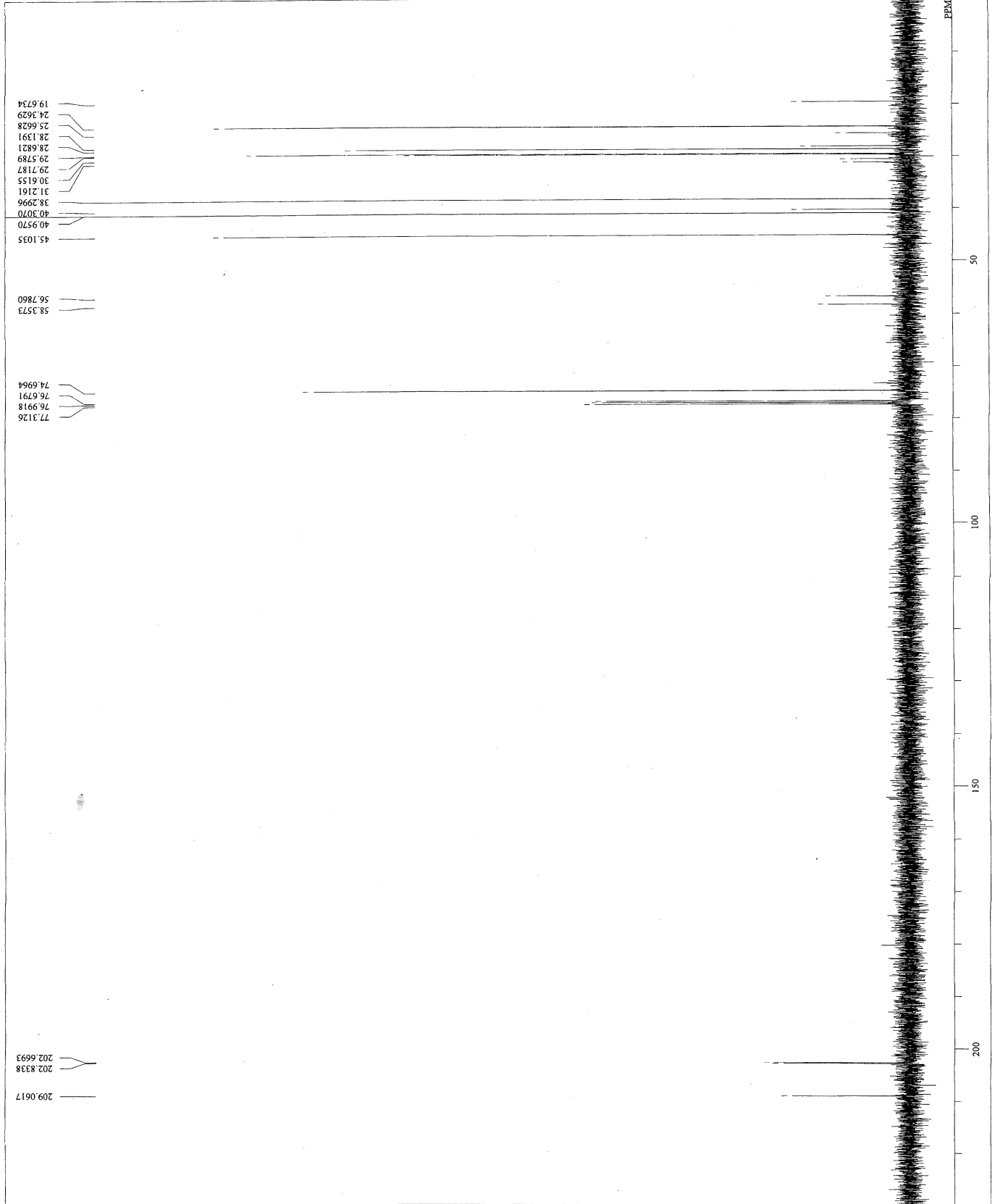
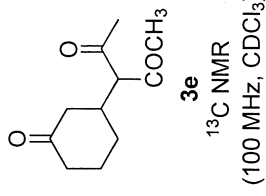


¹H NMR
 (400 MHz, CDCl₃)

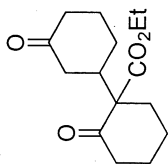


MYY-558 13C-2.ms
 Wed Apr 20 16:46:56 2011
 13C
 100.40 MHz
 135500.00 Hz
 32768
 27100.27 Hz
 64
 1.2091 sec
 1.7909 sec
 5.75 usec
 20.4 c
 CDCl3
 77.00 ppm
 0.3 Hz
 29

DFILF
 COMPT
 DATUM
 OBINIC
 EXMOD
 OBFRQ
 OBFIN
 POINT
 FREQU
 SCANS
 ACQTM
 PD
 PW1
 IRNUC
 CTMP
 SLVNT
 EXREF
 RGAIN

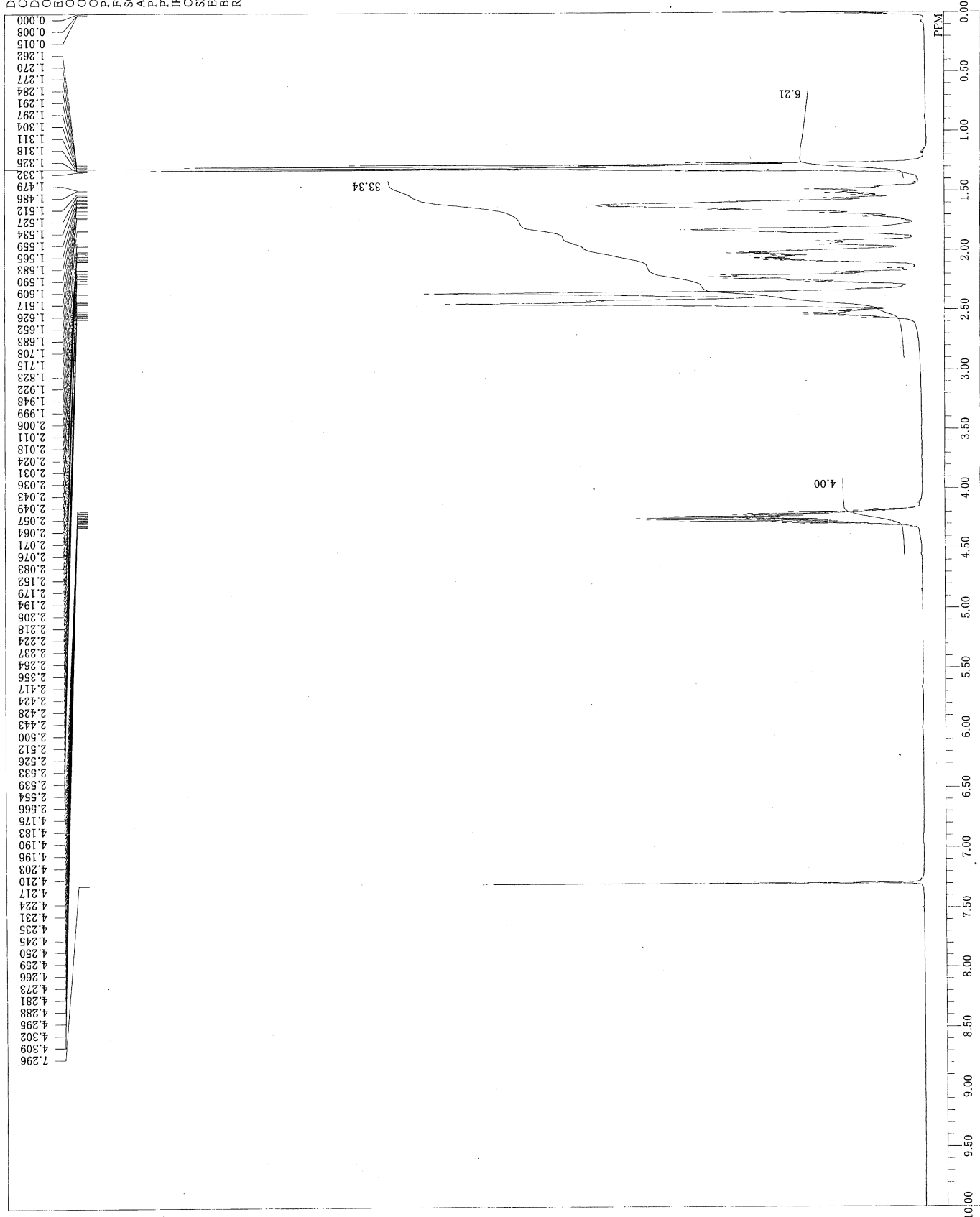


MYY-870pure_proton-1-1.jdf
 single_pulse
 2012-03-13 16:44:55
 1H
 proton.kxp
 500.16 MHz
 2.41 KHz
 6.01 Hz
 16400
 9384.38 Hz
 8
 1.7459 sec
 5.0000 sec
 4.68 usec
 1H
 17.0 c
 CDCL3
 0.00 ppm
 1.00 Hz
 30



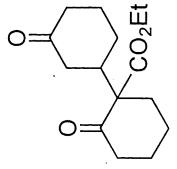
3f
¹H NMR
 (500 MHz, CDCl₃)

DFILE
 COMINT
 DATIM
 OBNUC
 EXMOD
 OBRFQ
 OBSEI
 OBFIN
 POINT
 FREQU
 SCANS
 ACQTM
 PD
 PW1
 IRNUC
 CTEMP
 SLVNT
 EXREF
 BF
 RGAIN



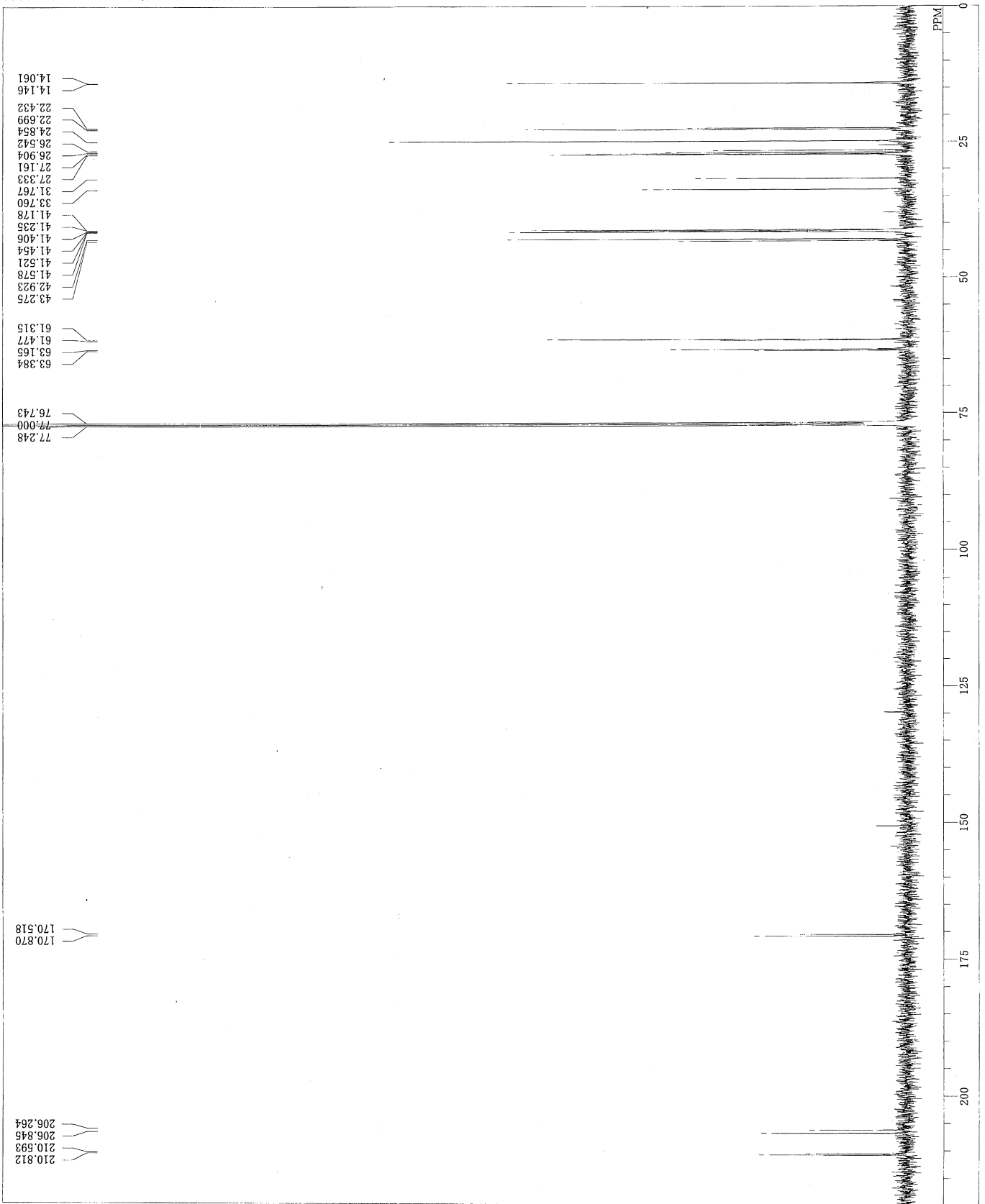
MYY-870_Carbon_copy2-1-1.jdf
 single pulse decoupled gated NOE
 2012-03-13 16:53:17
 13C
 carbon.jp

125.77 MHz
 7.87 KHz
 4.21 Hz
 32780
 39308.18 Hz
 129
 0.0000 sec
 2.0000 sec
 2.72 usec
 1H
 17.3 c
 CDCl₃
 77.00 ppm
 1.00 Hz
 54

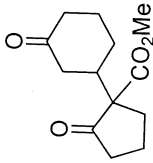


3f
¹³C NMR
 (125.8 MHz, CDCl₃)

DFILE
 COMNT
 DATIM
 OBNUC
 EXMOD
 OBFREQ
 OBSET
 OBFIN
 POINT
 FREQU
 SCANS
 ACQTM
 PD
 PW1
 IRNUC
 CTEMP
 SLVNT
 EXREF
 BF
 RGAIN

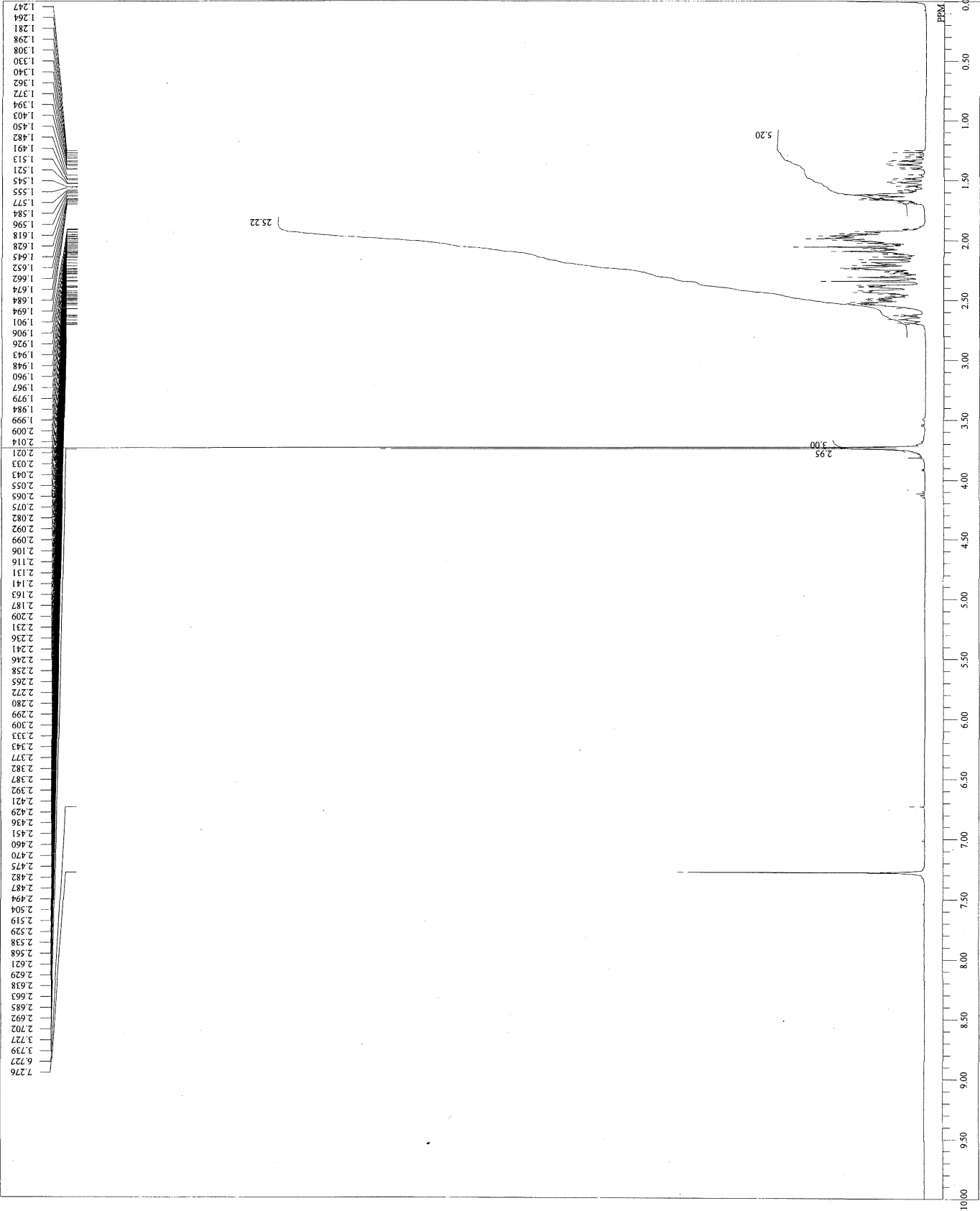


MYY-492 pure.als
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 non
 399.65 MHz
 134300.00 Hz
 8192
 7993.60 Hz
 1.0248 sec
 5.9753 sec
 5.75 usec
 1H
 15.2 e
 CDCL3
 0.00 ppm
 0.12 Hz
 17

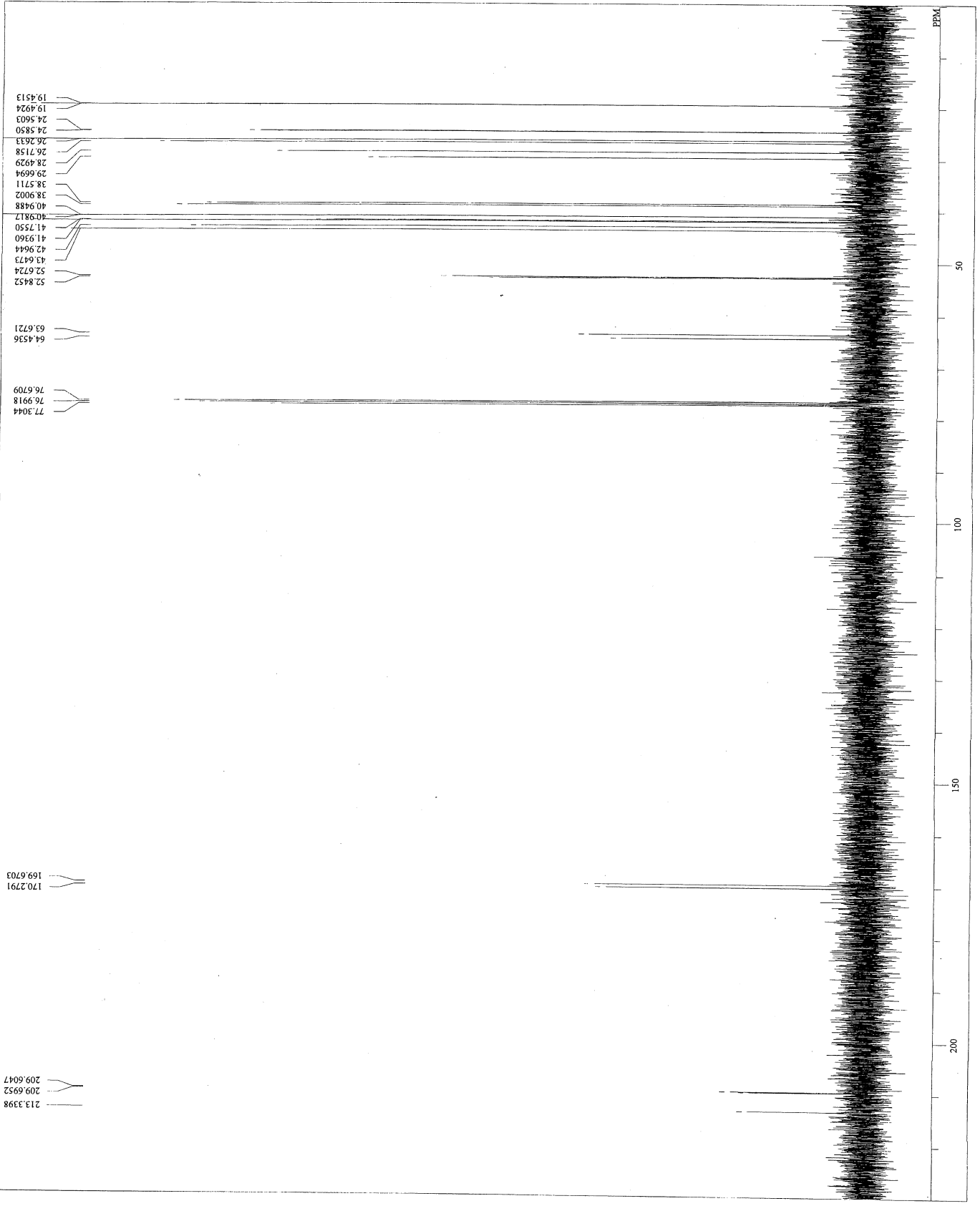
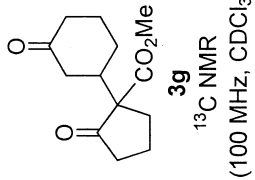


3g
¹H NMR
 (400 MHz, CDCl₃)

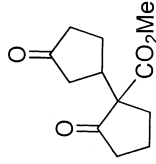
DPFILE
 COMANT
 DATUM
 OBNUC
 EXMOD
 OBFREQ
 FREQQU
 POINT
 ACQUIS
 PCQTM
 PD
 PW1
 IRNUC
 CTMP
 SLVNT
 EXREF
 BF
 RGAIN



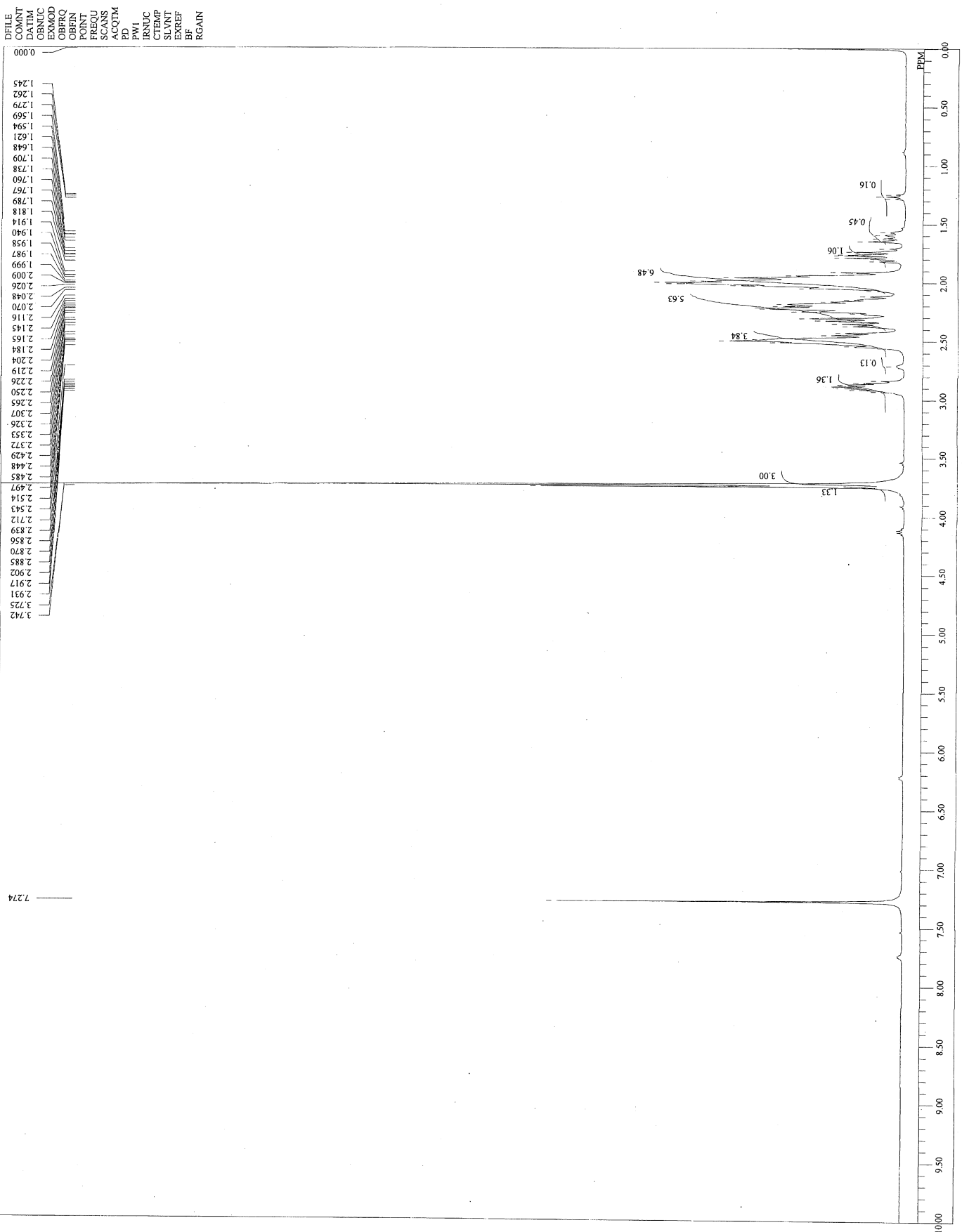
MYY-503 13C.als
 Wed Apr 20 16:32:09 2011
 13C
 bom
 EXMOD
 DATIM
 OBNUC
 OBPRQ
 OBNVT
 POINT
 FREQU
 SCANS
 ACQTM
 PD
 PW1
 IRNUC
 CTEMP
 SLVNT
 EXREF
 BF
 RGAIN



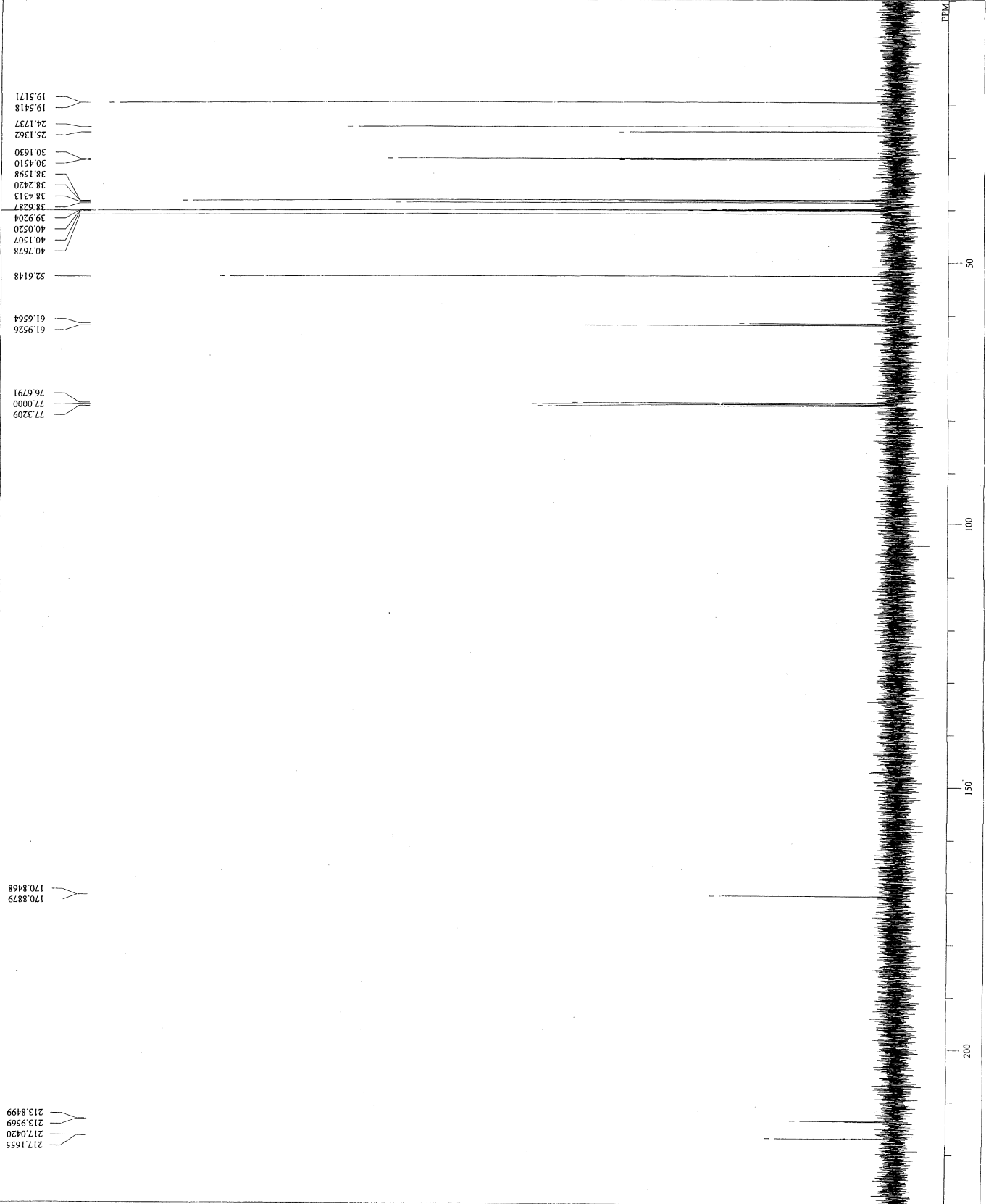
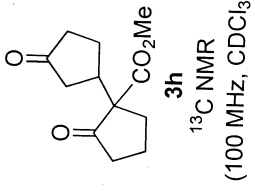
MYY-504-pure.als
 Tue Jan 18 09:23:47 2011
 1H
 130.00 MHz
 130.00 Hz
 8192
 7993.60 Hz
 8
 1.0248 sec
 5.9752 sec
 5.75 usec
 20.4 c
 CDCl3
 0.00 ppm
 1.00 Hz
 20



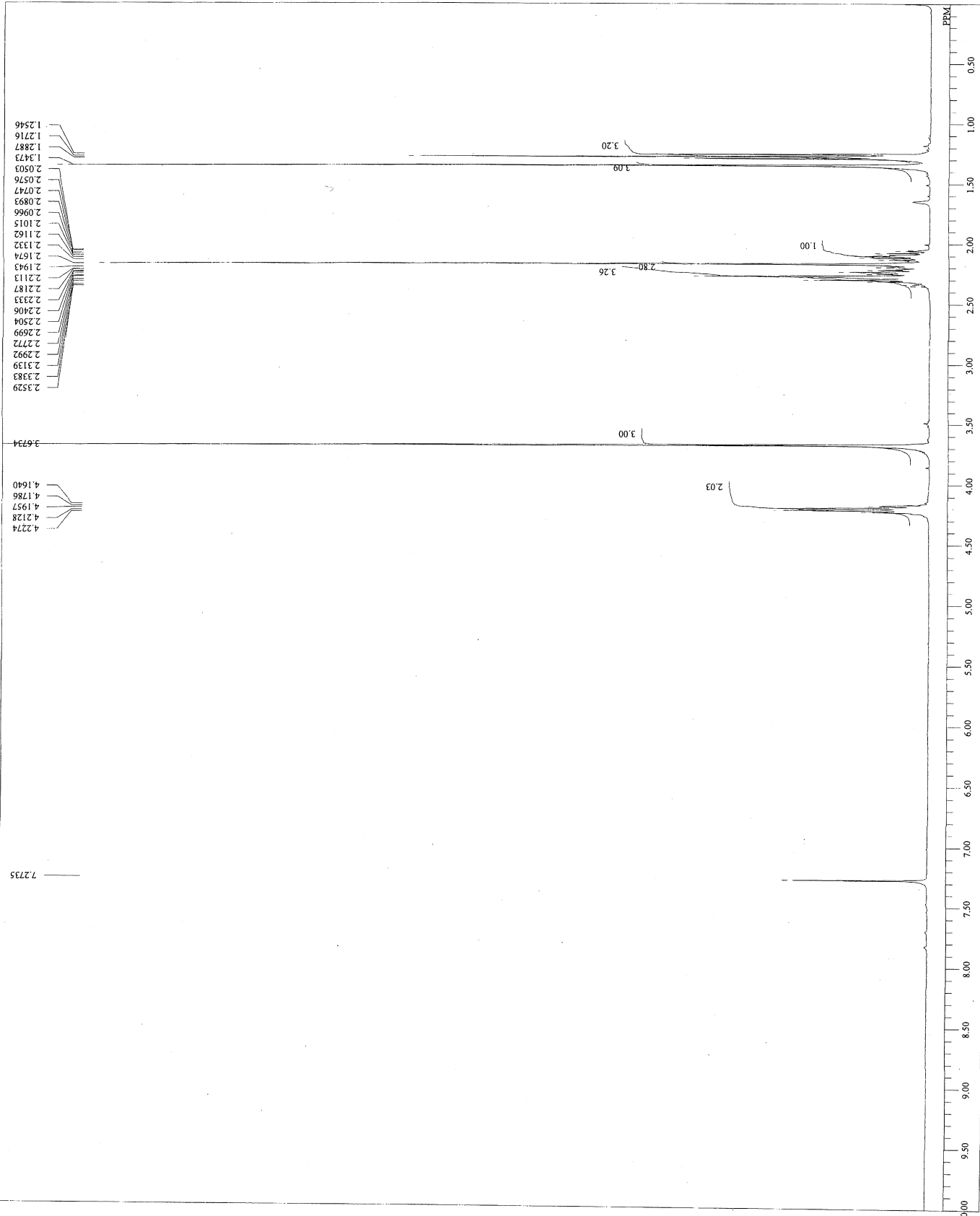
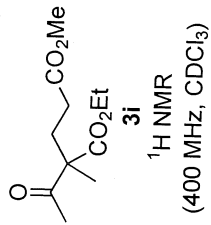
¹H NMR
 (400 MHz, CDCl₃)



MYY-504 13C-2.als
 Wed Apr 20 16:19:56 2011
 13C
 100.40 MHz
 135500.00 Hz
 32768
 27100.27 Hz
 64
 1.2091 sec
 1.7909 sec
 5.75 usec
 1H
 20.6 c
 77.00 ppm
 CDCL3
 0.12 Hz
 29
 RGAIN

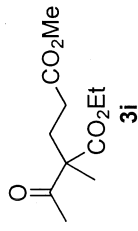


MTT-514-purcals
 DATE: Fri, Jan 21 14:50:50 2011
 COMPT: 1H
 DATUM: 399.65 MHz
 OBNUC: 134300.00 Hz
 EXMOD: non
 OBFRO: 8192
 POINT: 7993.60 Hz
 OBFEN: 8
 FREQU: 1.0248 sec
 PD: 5.9752 sec
 SCANS: 5.75 usec
 ACQTM: 17.5 c
 PW1: 1H
 IRNUC: CDCL3
 CTEMP: 0.00 ppm
 SLVNT: 0.12 Hz
 EXREF: 19
 RGAIN:



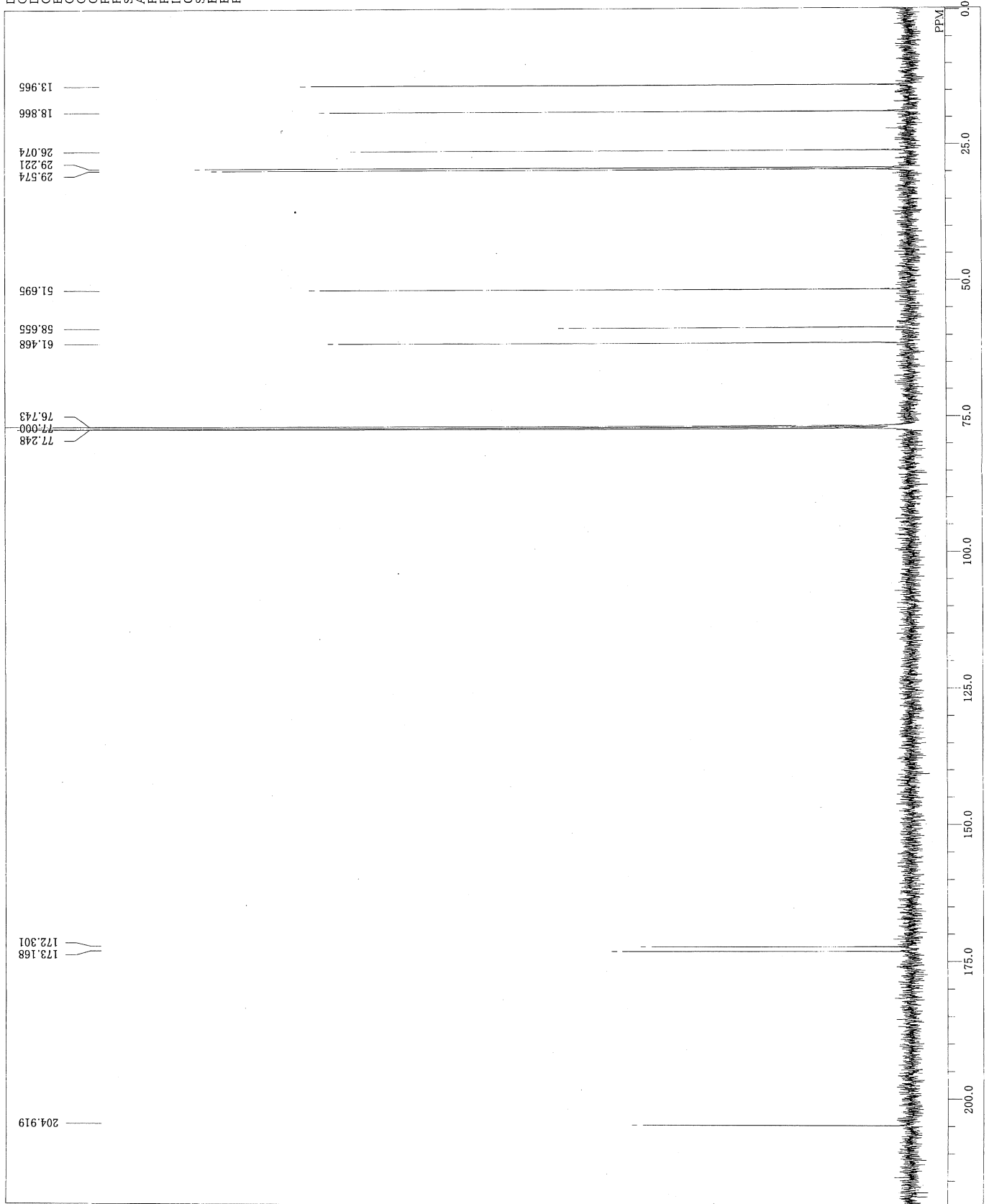
MXY-476_Carbon_copy3-1-1.tif
 single pulse decoupled gated NOE
 2012-02-21 00:02:26
 13C
 carbon.fxp

125.77 MHz
 7.87 KHz
 4.21 Hz
 32780
 39308.18 Hz
 65
 0.0000 sec
 2.0000 sec
 2.72 usec
 1H 17.3 c
 CDCL3 77.00 ppm
 EXREF 0.12 Hz
 BF 60
 RGAIN 60



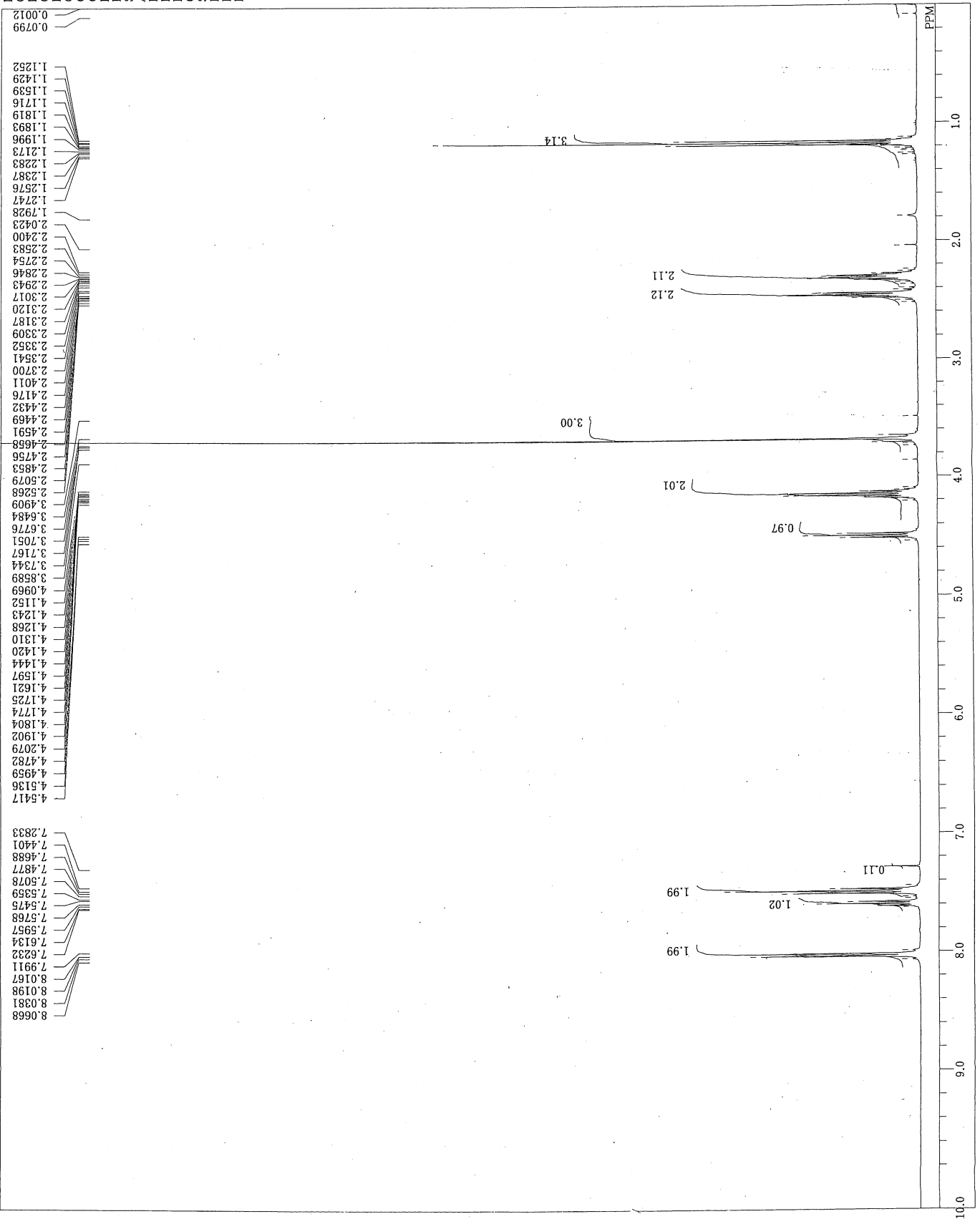
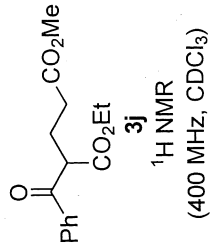
¹³C NMR
 (125.8 MHz, CDCl₃)

DEFL
 CONV
 DATM
 DENUC
 EXMOD
 OBRQ
 OBSF
 OBFIN
 POINT
 FREQU
 SCANS
 ACQTM
 PD
 PW1
 IRNUC
 CTEMP
 SLVNT
 EXREF
 BF
 RGAIN



DFILE
 COMMENT
 DATIM
 OBNUC
 EXMOD
 OBSFRQ
 OBSFT
 ORFN
 POINT
 FREQ
 SCANS
 ACQTM
 PD
 PW1
 IRNUC
 CTMP
 SLVNT
 EXREF
 BF
 RGAIN

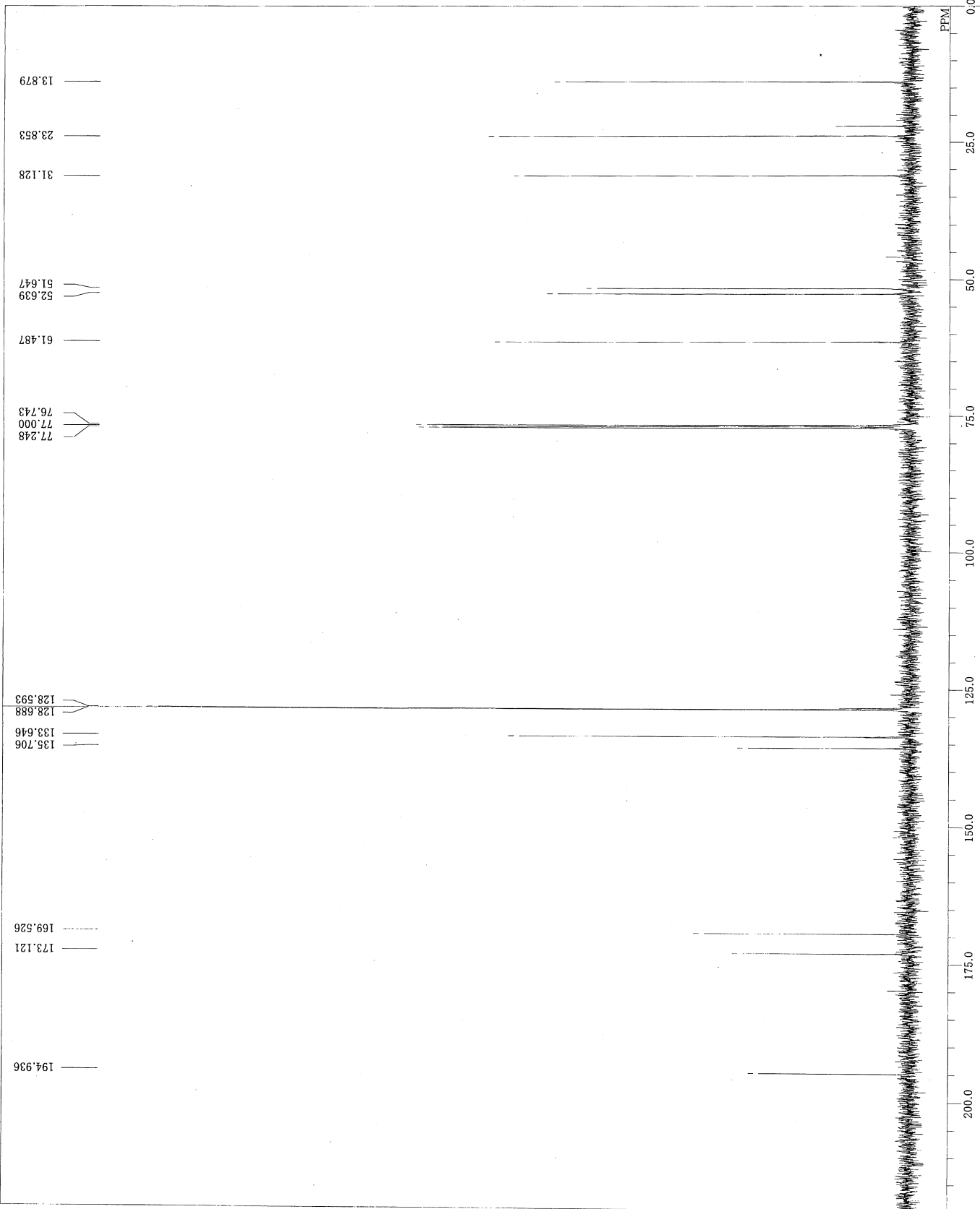
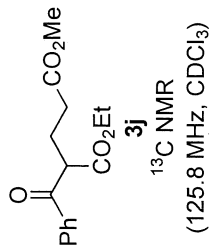
KMK-458up.nmdata
 KMK-458 up
 Mon Feb 9 21:05:34 2009
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 399.65 MHz
 130.00 KHz
 4300.00 Hz
 32768
 7993.60 Hz
 4
 4.0993 sec
 2.9007 sec
 7.00 usec
 1H
 22.2 c
 CDCL3
 0.00 ppm
 0.12 Hz
 16



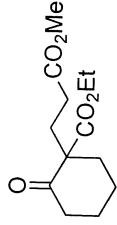
MY-547_Carbon_copy1-1-1.fif
single pulse decoupled gated NOE
2012-02-20 23:51:27

DFILE
COMINT
DATIM
DNRUC
EXMOD
OBNUC
OBFRC
OBSFT
OBSFT
POINT
PREQU
SCANS
ACQTM
PD
PWI
IRNUC
CTEMP
SLVNT
EXREF
BF
RGAIN

carbon.kp
195.77 MHz
7.87 KHz
4.21 Hz
32780
39306.18 Hz
33
0.0000 sec
2.0000 sec
2.72 usec
1H 17.0 c
CDCL3
77.00 ppm
0.12 Hz
60

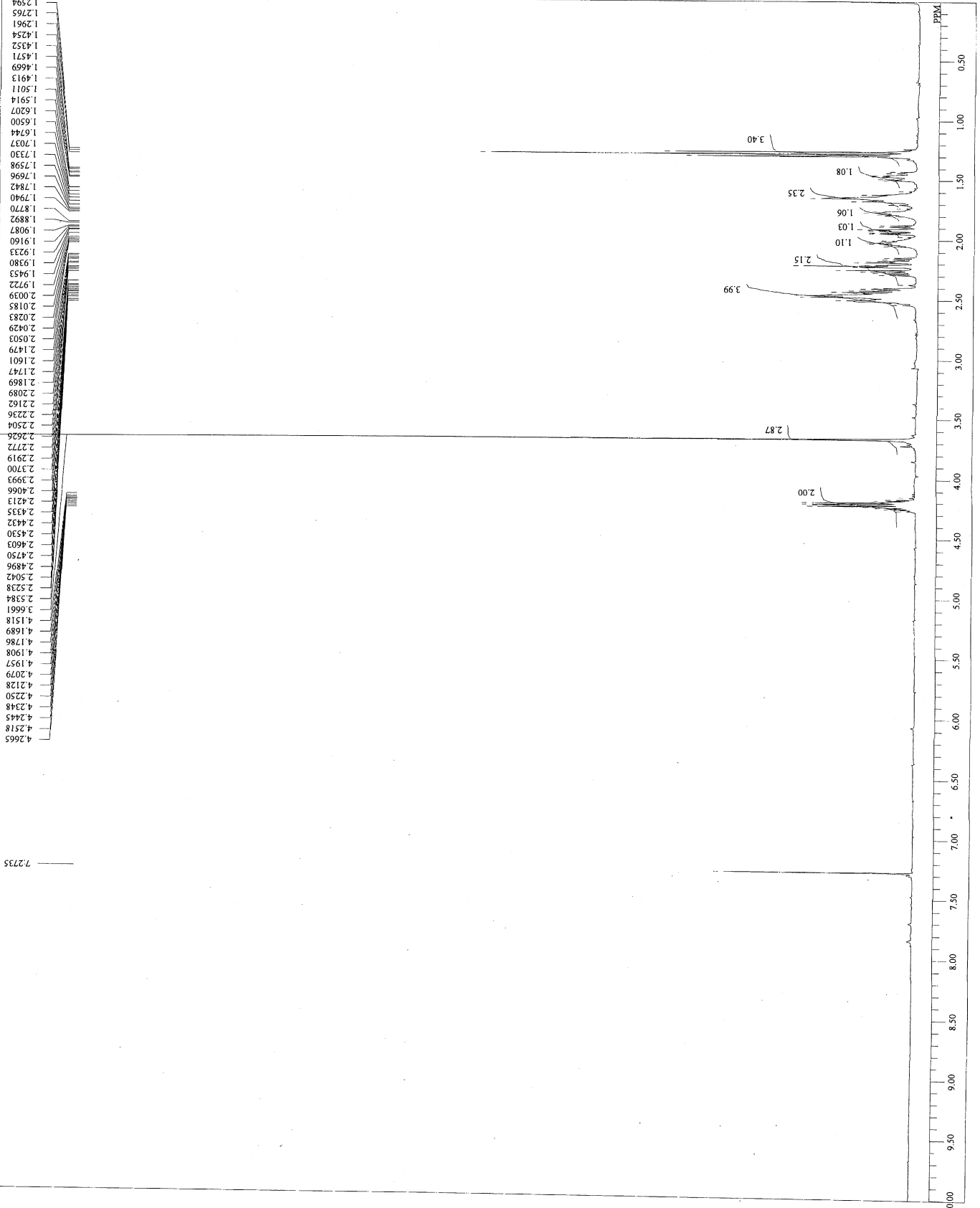


MYY-489 pureals
 Fri Dec 10 15:44:10 2010
 1H
 NON
 399.65 MHz
 134300.00 Hz
 8192
 7993.60 Hz
 32
 1.0248 sec
 5.9752 sec
 5.00 usec
 19.4 c
 CDCl₃
 0.00 ppm
 0.12 Hz
 19
 RGAIN



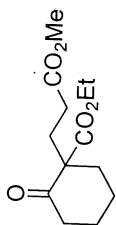
3k
¹H NMR
 (400 MHz, CDCl₃)

DFILE
 COMNT
 DATIM
 OBNUC
 EXMOD
 OBFRO
 POINT
 FREQU
 SCANS
 ACQTIM
 PD
 PULP
 CTEMP
 SILVNT
 EXREF
 BF
 RGAIN



MYY-489 Carbon copy3-1-1.tif
 single pulse decoupled gated NOE
 2012-02-20 23:40:27
 13C

carbon.jpg
 125.77 MHz
 7.87 kHz
 4.21 Hz
 32780
 39308.18 Hz
 76
 0.0000 sec
 2.0000 sec
 2.72 usec
 1H 16.9 c
 CDCL3
 77.00 ppm
 1.00 Hz
 58



3k
¹³C NMR
 (125.8 MHz, CDCl₃)

DFILE
 COMNT
 DATIM
 OBNUC
 EXMOD
 OBFERQ
 OBSEI
 OBSEI
 POINT
 FREQU
 SCANS
 ACQTM
 PD
 PWT
 IRNUC
 CTEMP
 SLVNT
 EXREF
 BF
 RGAIN

