

***anti*-Selective Catalytic Asymmetric Nitroaldol Reaction via a Heterobimetallic Heterogeneous Catalyst**

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mshibasa@mol.f.u-tokyo.ac.jp, nkumagai@mol.f.u-tokyo.ac.jp

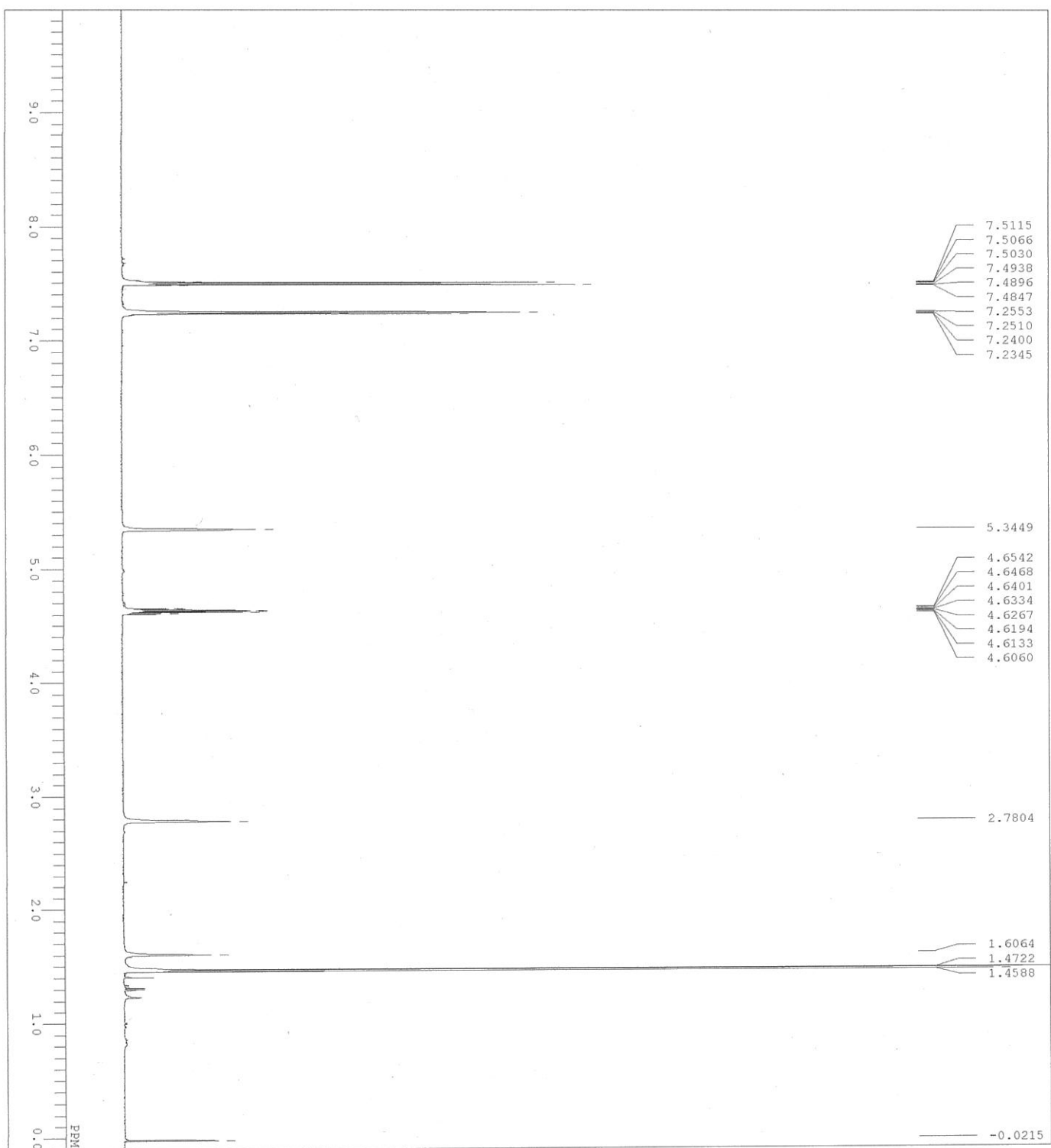
Supporting Information

10. NMR Spectra

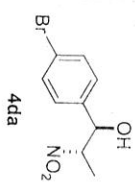
[†] The University of Tokyo.

[‡] Kissei Pharmaceutical Company, Ltd.

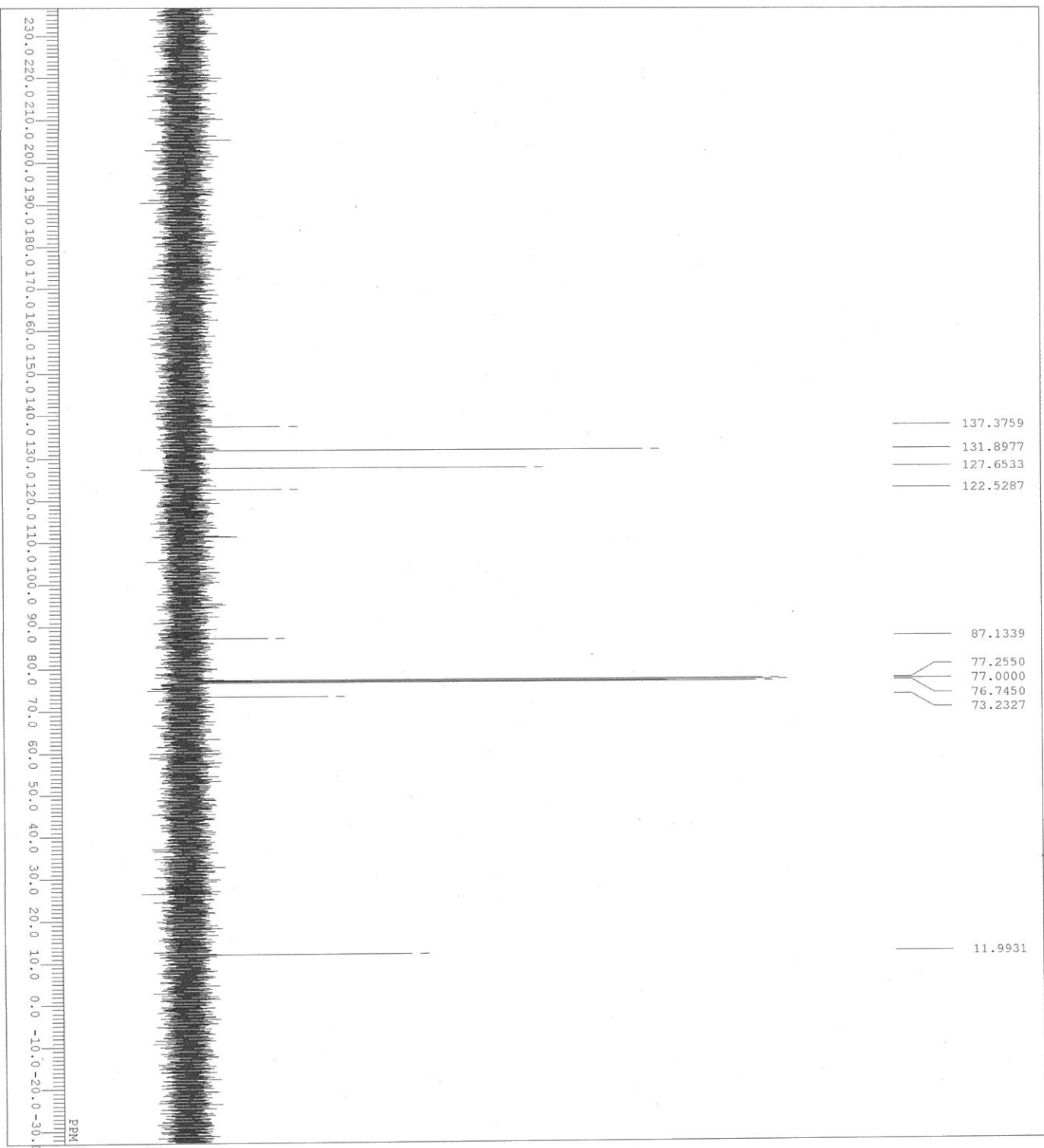
p-Br 1H



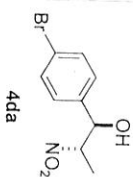
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 OBSET 150.00 KHz
 OBFIN 2150.00 Hz
 POINT 32768
 FREQU 10000.00 Hz
 SCANS 8
 ACQTM 3.2768 sec
 PD 3.7232 sec
 FWI 5.70 usec
 IRNOC 1H
 CTEMP 26.6 C
 SLVNT CDCl3
 EXREF 7.24 ppm
 BF 0.22 Hz
 RGAIN 20

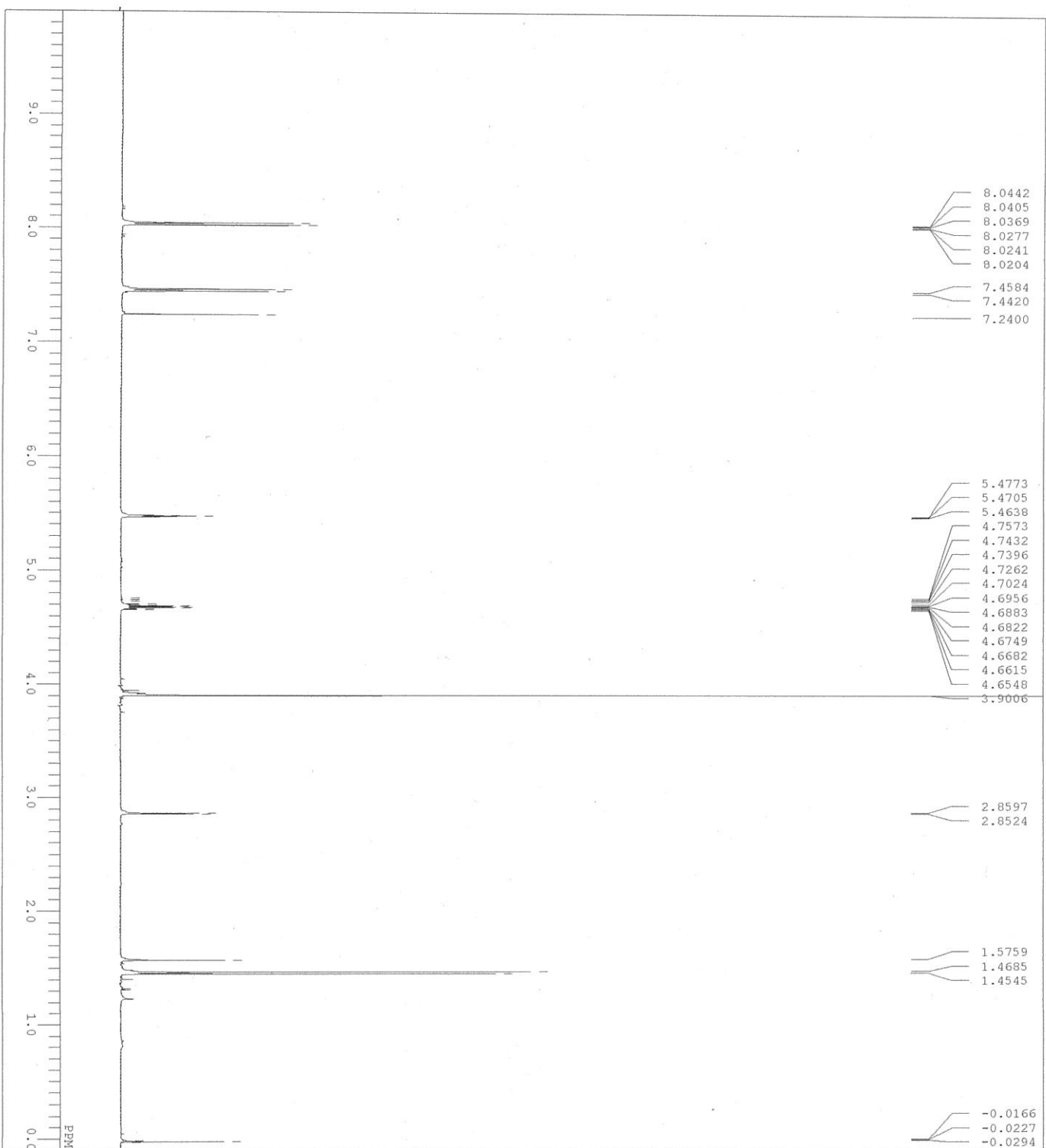


p-br c13

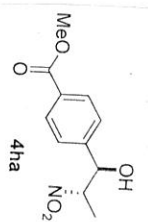


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 EXMOD bcm
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 OBSEI 120.00 KHz
 OBFIN 7958.00 Hz
 POINT 32768
 FREQU 33898.30 Hz
 SCANS 159
 ACQTM 0.9667 sec
 PD 2.0333 sec
 PW1 6.25 usec
 IRNUC 1H
 CTEMP 27.4 c
 SLVMT CDCL3
 EXREF 77.00 ppm
 BF 0.22 Hz
 RGAIN 32



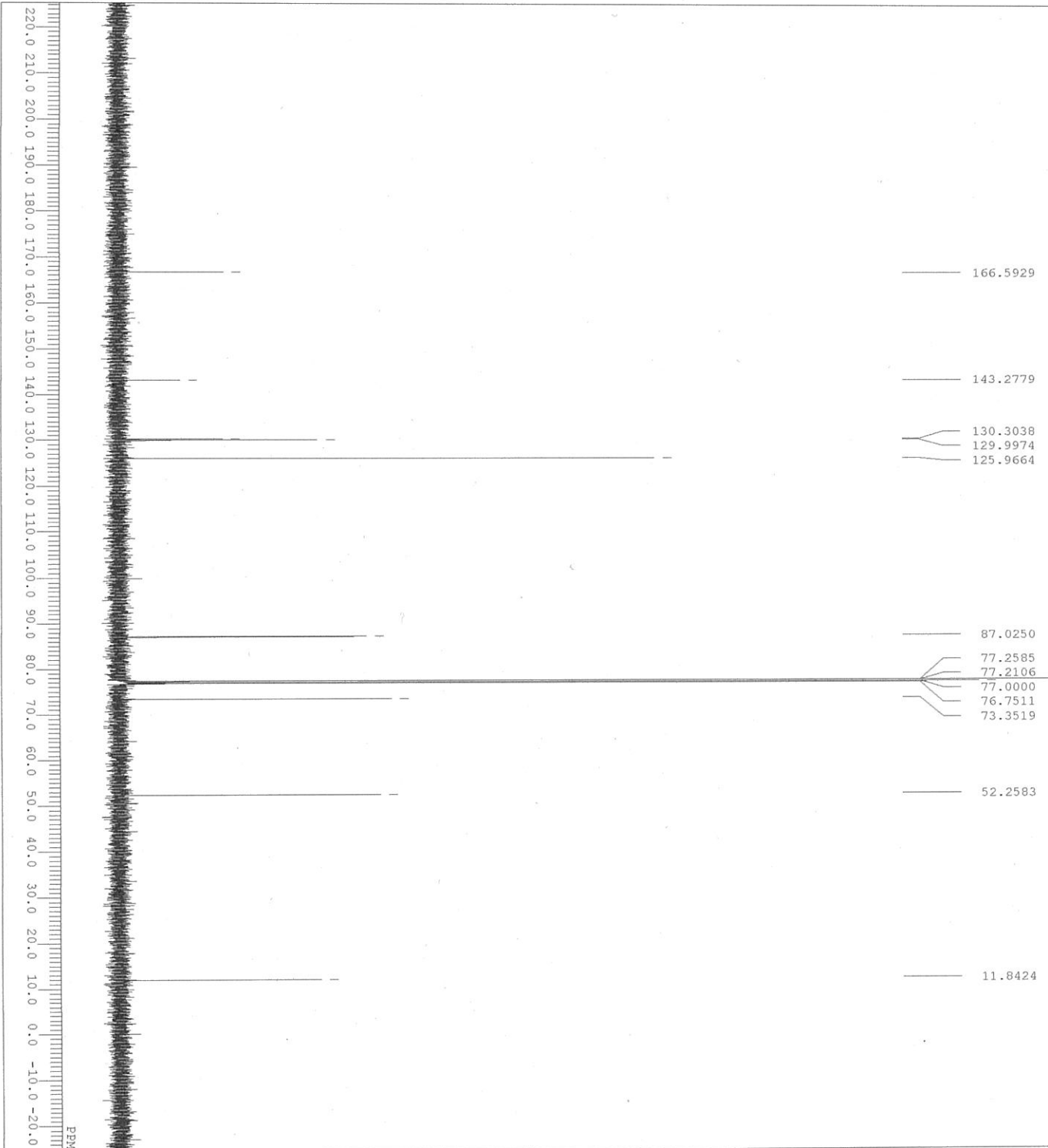


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 OBFIN 2160.00 Hz
 POINT 32768
 FREQUF 10000.00 Hz
 SCANS 7
 ACQTM 3.2768 sec
 PD 3.7232 sec
 PW1 5.70 usec
 IRNUC 1H
 CTEMP 26.8 C
 SLVNT CDCL3
 EXREF 7.24 ppm
 BF 1.00 Hz
 RGAIN 21

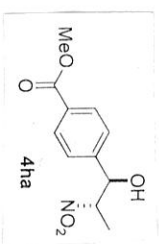


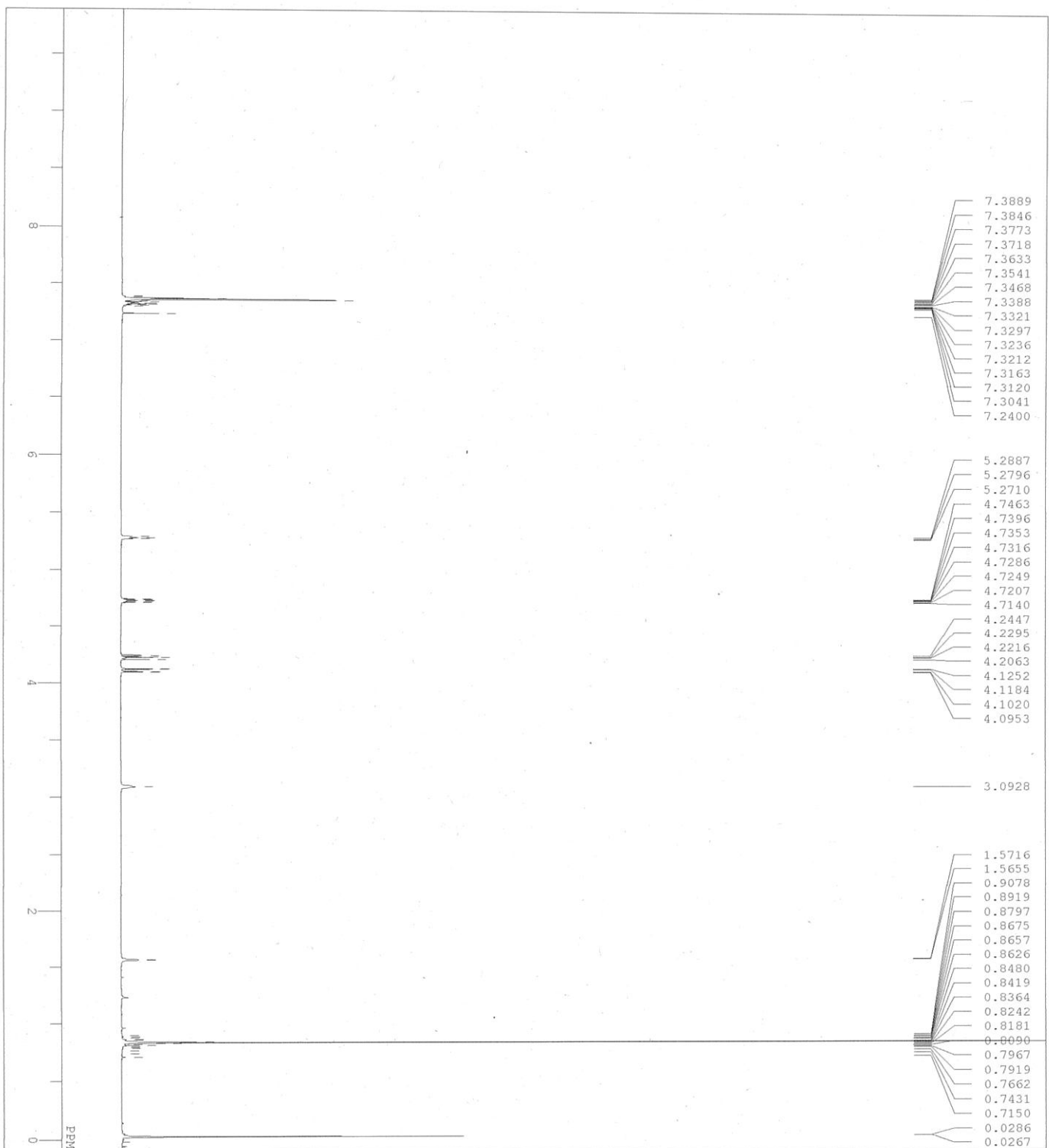
single pulse decoupled gated NOE

D:\MyDocument\1a nojiri\for printing\pco2me c13.als

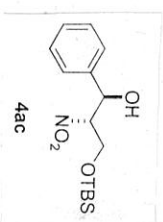


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OBSET 3.45 KHz
OBFIN 6.00 Hz
POINT 26214
FREOU 31249.52 Hz
SCANS 518
ACQTM 0.8389 sec
PD 2.0000 sec
FWI 3.57 usec
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.01 Hz
RGAIN 58

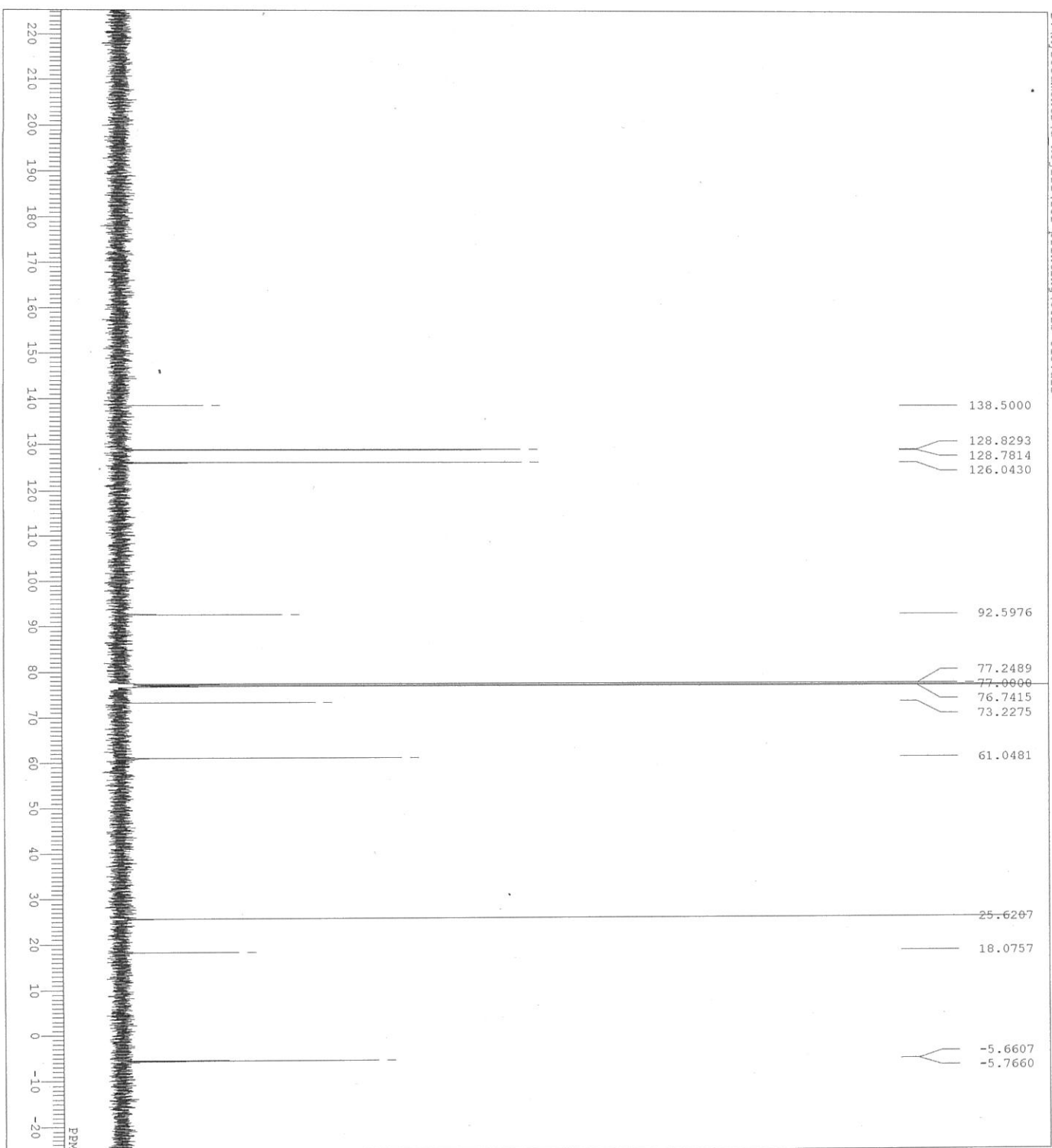




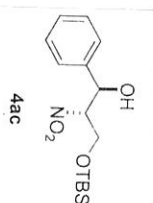
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 EXMOD non
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 OBRIN 2160.00 Hz
 POINT 32768
 FREOU 10000.00 Hz
 SCANS 13
 ACQTM 3.2768 sec
 PD 3.7232 sec
 PUL 5.70 usec
 IRNUC 1H
 CTEMP 26.7 c
 SLVNT CDCl3
 EXREF 7.24 ppm
 BF 1.20 Hz
 RGAIN 18



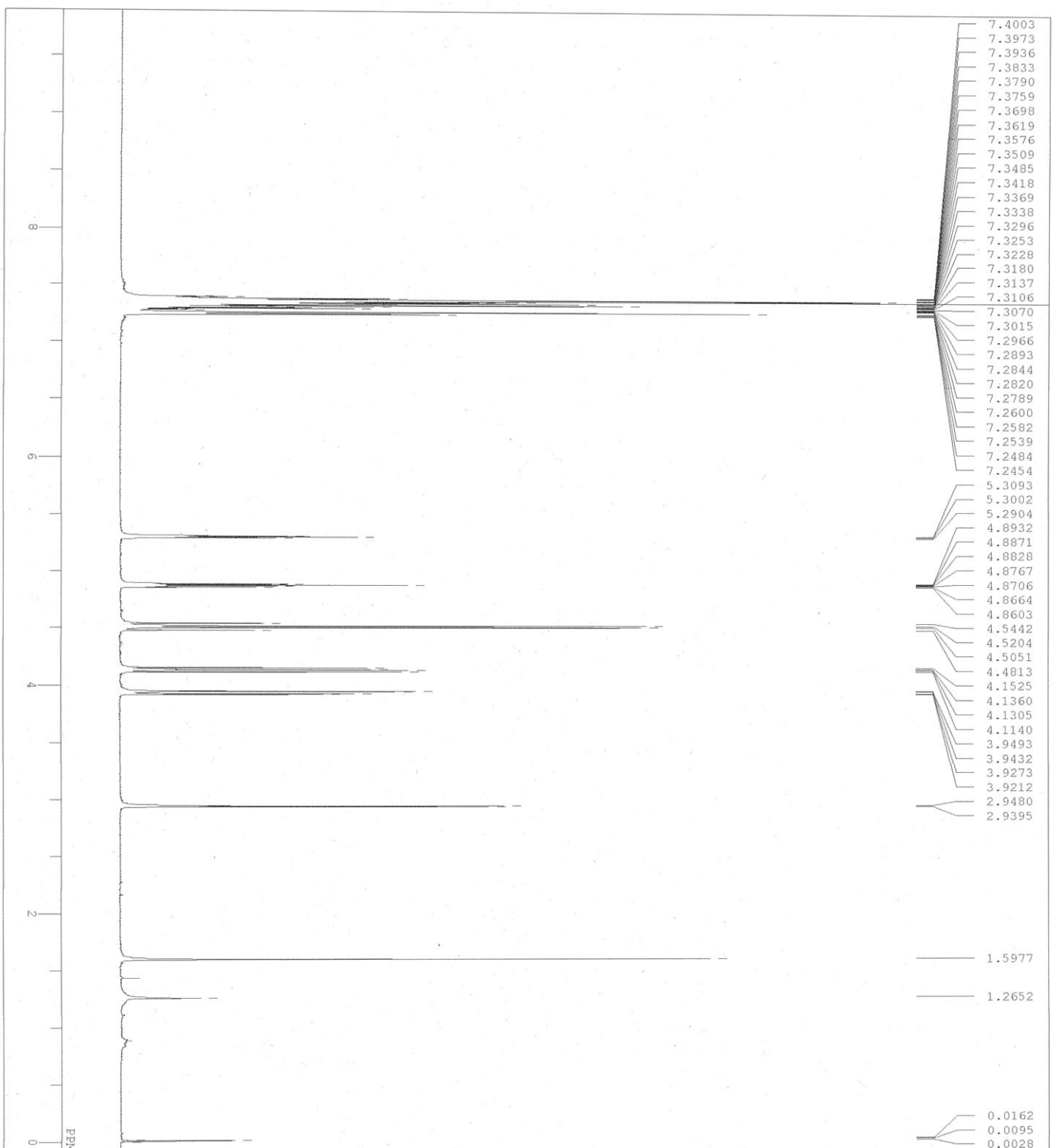
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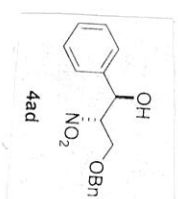
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EXMOD	single	pulse dec
OBFRQ		124.51 MHz
OBFTN		3.45 KHz
POINT		6.00 Hz
FREQU		26214
SCANS		31249.52 Hz
ACQTM		226
PD		0.8389 sec
PM1		2.0000 sec
IRNUC	1H	3.57 usec
CTEMP		21.5 c
SLVNT	CDCl3	
EXREF		77.00 ppm
RF		1.00 Hz
RGAIN		56



1H Line

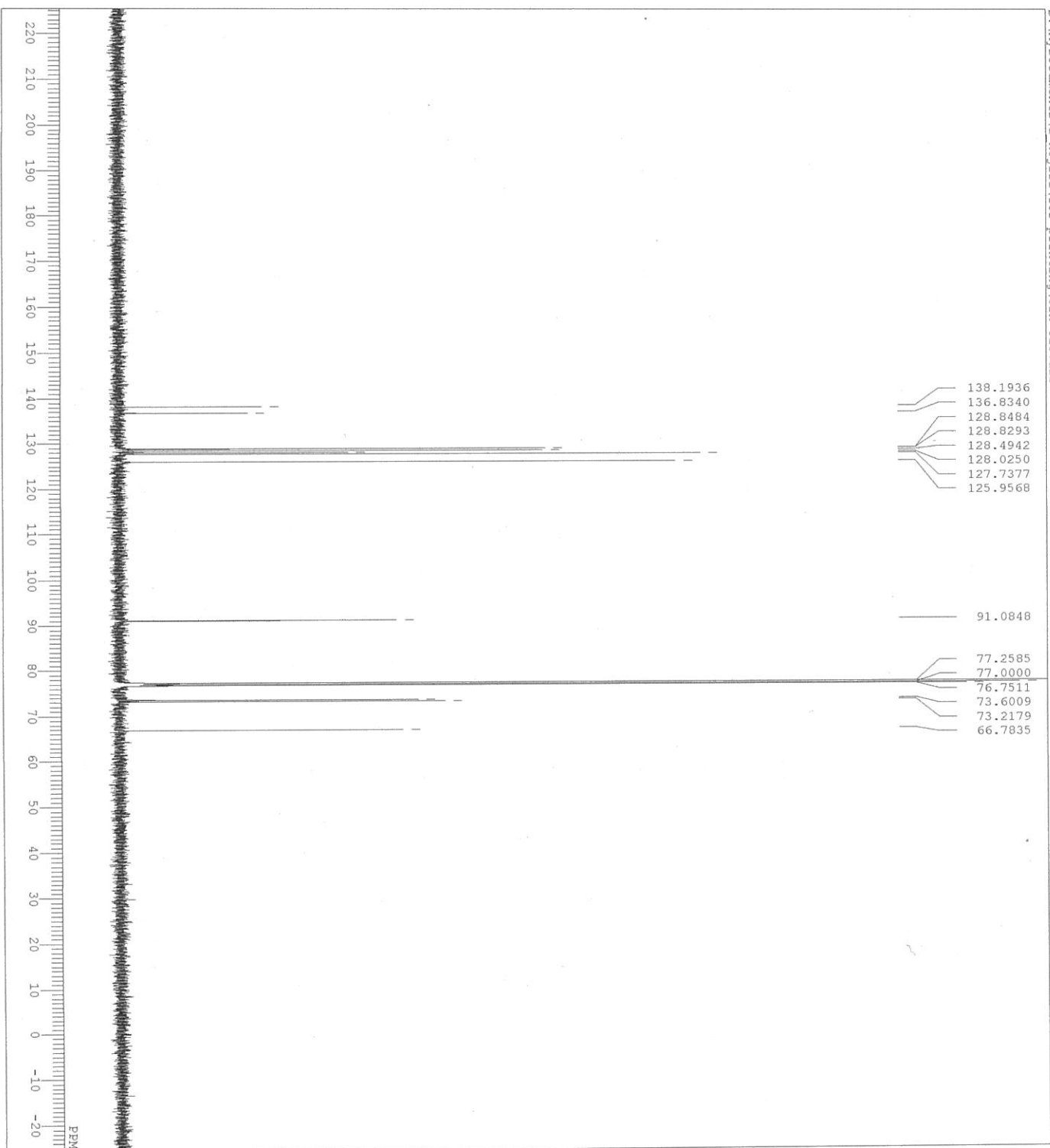


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 EXMOD non
 OFREQ 500.00 MHz
 OSSET 160.00 kHz
 OFBIN 2160.00 Hz
 POINT 32768
 FREQU 10000.00 Hz
 SCANS 17
 ACQTM 3.2768 sec
 PD 3.7232 sec
 PUL 5.70 usec
 IRNUC 1H
 CTMP 26.9 c
 SLYNT CDCL3
 EXREF 7.26 ppm
 BF 1.20 Hz
 RGAIN 19

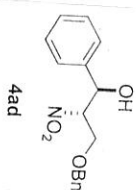


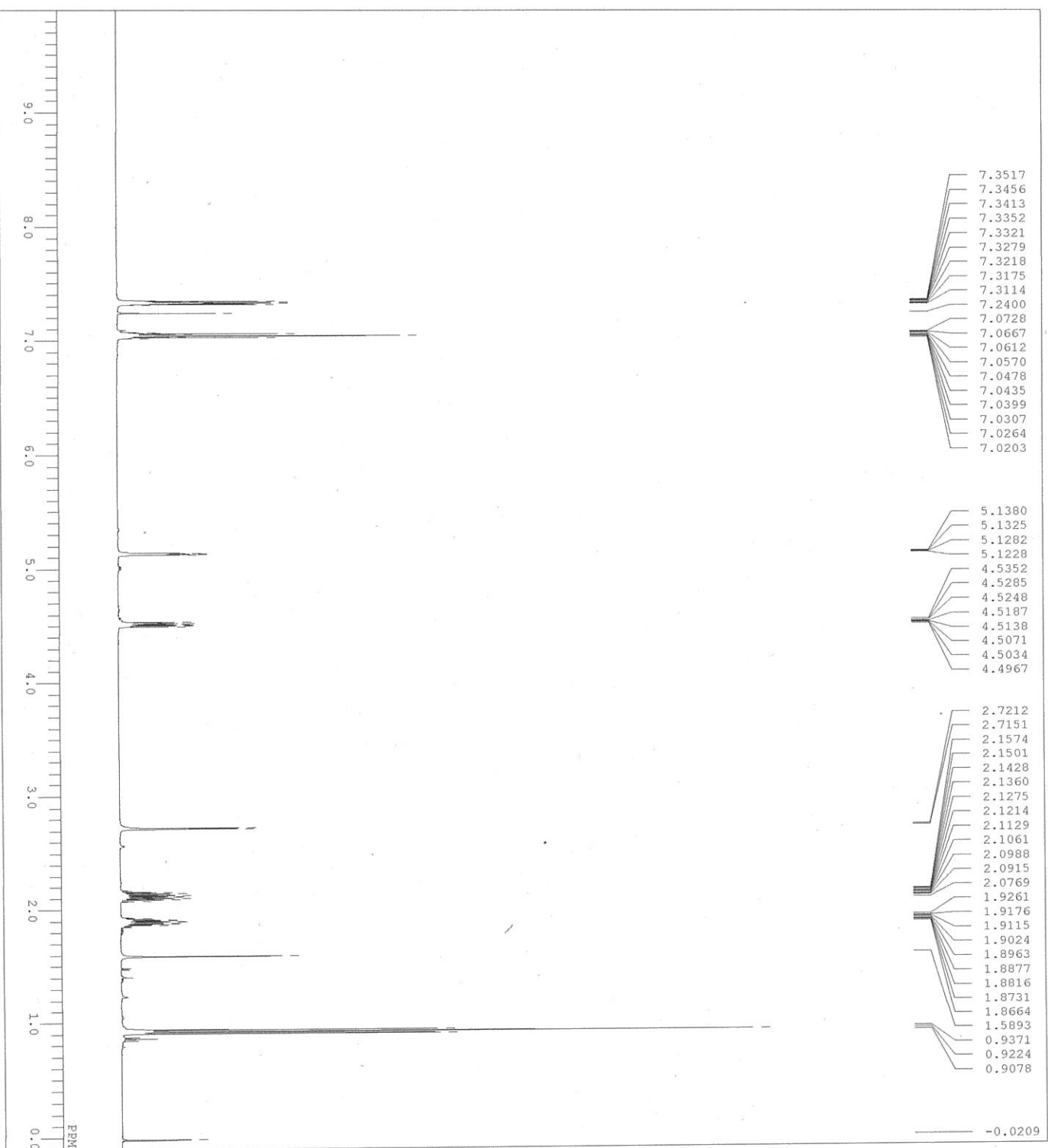
single pulse decoupled gated NOE

D:\MyDocuments\aj nojiri\for printing\Obn C13.als

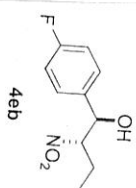


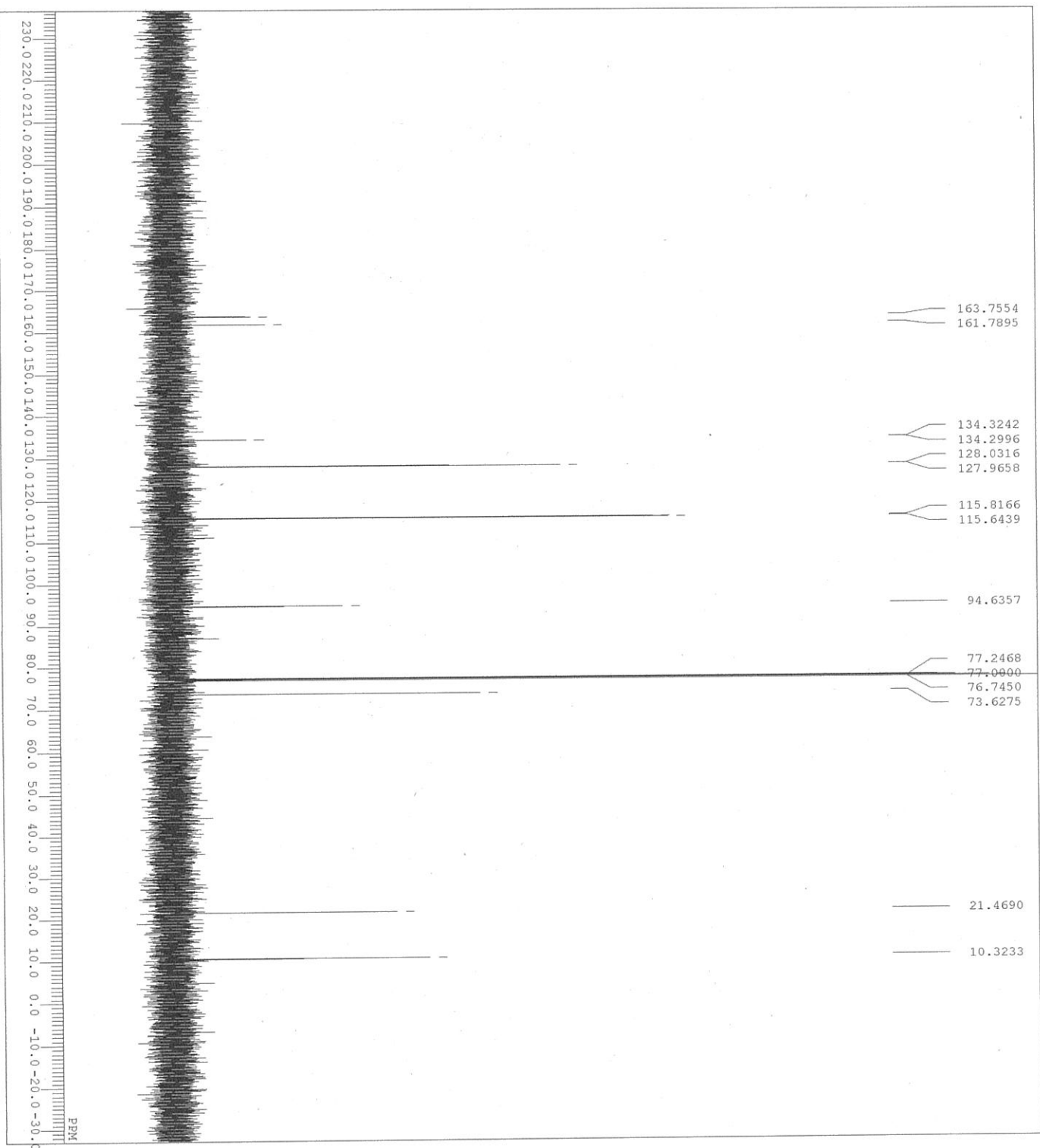
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 OBSET 3.45 kHz
 OBFIN 6.00 Hz
 POINT 26214
 FREQU 31249.52 Hz
 SCANS 480
 ACQTM 0.8389 sec
 PD 2.0000 sec
 FWH 3.57 usec
 IRNUC 1H
 CTEMP 21.3 C
 SLVNT CDCl3
 EXREF 77.00 ppm
 BF 1.00 Hz
 RGAIN 60



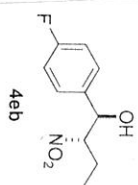


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 OBNIC 1H
 EXMOD non
 OBFREQ 500.00 MHz
 OBSSET 160.00 KHz
 OBSFIN 2160.00 Hz
 POINT 32768
 EREQU 10000.00 Hz
 SCANS 19
 ACQTM 3.2768 sec
 PD 3.7232 sec
 PUL 5.70 usec
 TRNUC 1H
 CTEMP 27.2 c
 SLVNT CDCL3
 EXREF 7.24 ppm
 BF 1.20 Hz
 RGAIN 20



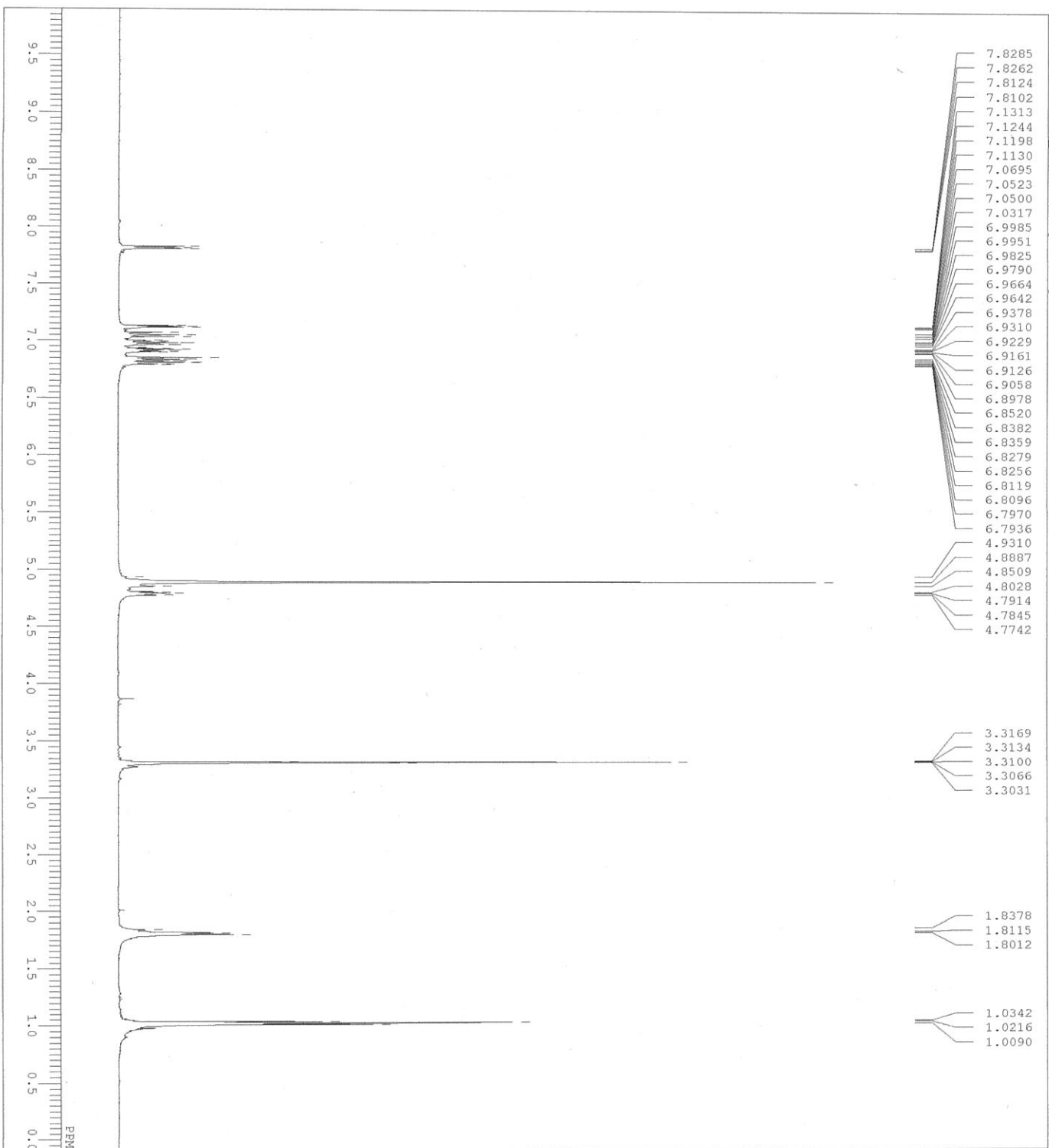


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 EXMOD bcm
 OBSFQ 125.65 MHz
 OBSFQ 120.00 KHz
 OBSFQ 7958.00 Hz
 POINT 32768
 FREQU 33898.30 Hz
 SCANS 267
 ACQTM 0.9667 sec
 PD 2.0333 sec
 PM1 6.25 usec
 IRNUC 1H
 CTEMP 27.7 C
 SLVNT CDCL3
 EXREF 77.00 ppm
 RF 1.20 Hz
 RGAIN 32

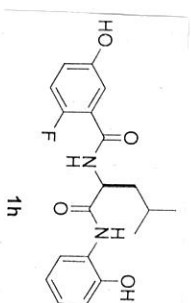


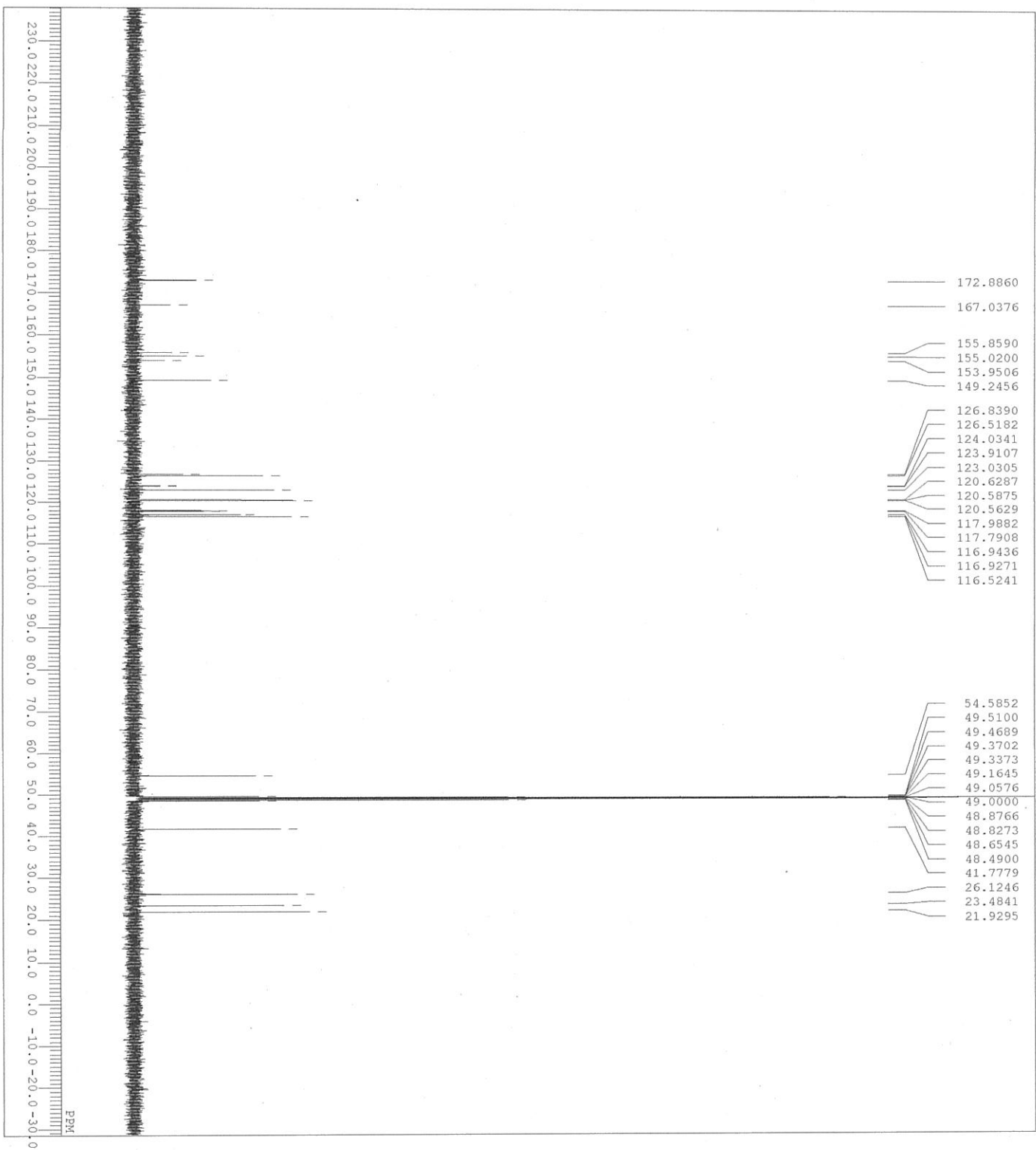
single_pulse

D:\MyDocument\1a nojiri\for printing\6 1H.als

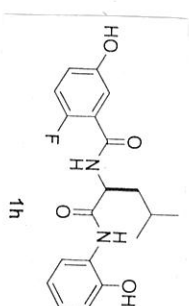


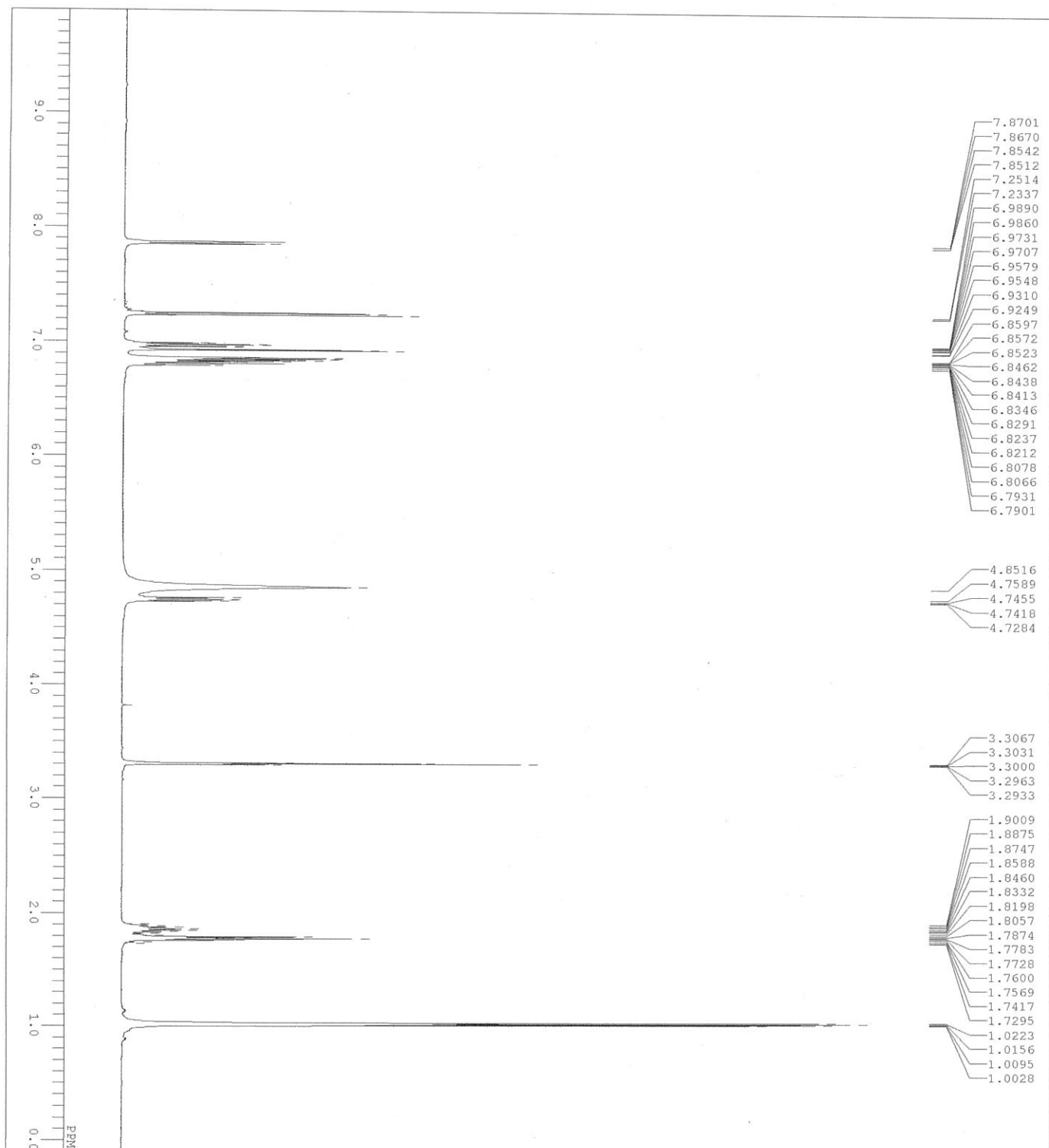
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 EXMOD single_pulse.ex2
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 OBSFT 4.38 KHz
 OBSFT 9.64 Hz
 POINT 13107
 FREQV 7429.31 Hz
 SCANS 8
 ACQTM 1.7642 sec
 PD 5.0000 sec
 PFI 6.20 usec
 IRNUC 1H
 CTEMP 20.4 C
 SLVNT CD3OD
 EXREF 3.31 ppm
 BF 0.01 Hz
 RGAIN 50



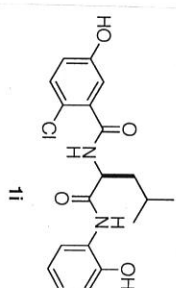


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 DATE Thu Jun 4 15:09:09 2009
 OBNUC 13C
 bcm
 125.65 KHz
 120.00 KHz
 7958.00 Hz
 32768
 33898.30 Hz
 340
 0.9667 sec
 2.0333 sec
 6.25 usec
 27.5 c
 49.00 ppm
 1.00 Hz
 31

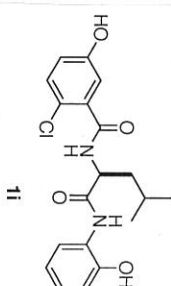




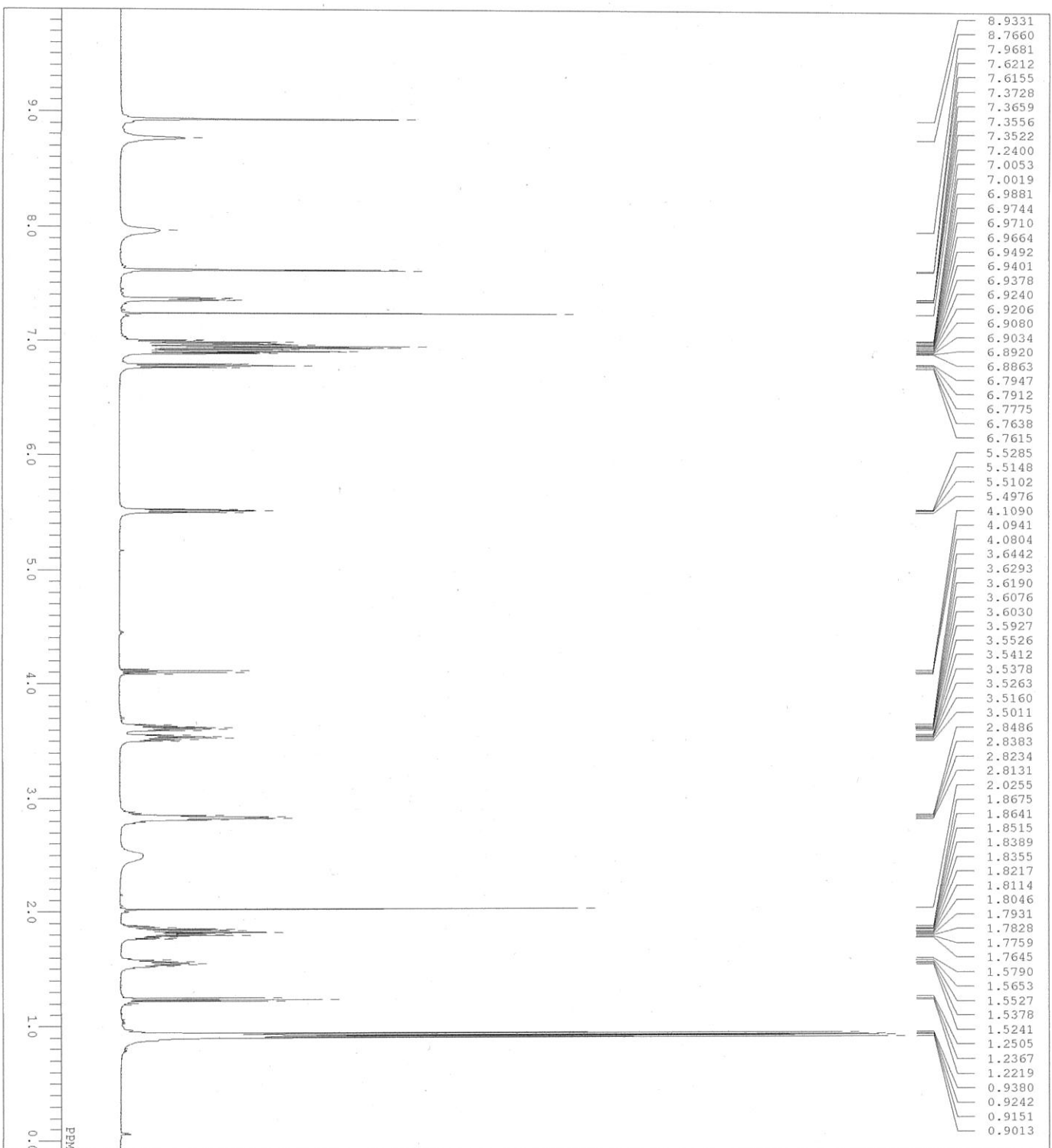
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 COUNT 1H Line
 DATIM Fri Jun 5 10:48:17 2009
 OBNUC 1H
 EXMOD non
 OBFREQ 500.00 MHz
 OBFSET 160.00 KHz
 OBFIN 2160.0 Hz
 POINT 32768
 FRECU 10000.0 Hz
 SCANS 8
 ACQTM 3.277 sec
 PD 3.723 sec
 Fw1 5.7 us
 TRNMC 1H
 CTEMP 26.1 c
 SLVNT CD3OD
 EXREF 3.30 ppm
 BF 0.22 Hz
 RGAIN 19



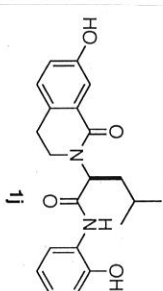
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 ORNUC 13C
 EXMOD bcm
 OBFRQ 125.65 MHz
 OBSET 120.00 KHz
 OBFIN 7958.00 Hz
 POINT 32768
 FREOU 33898.30 Hz
 SCANS 106
 ACQTM 0.9667 sec
 PD 2.0333 sec
 PUL 5.00 usec
 IRNUC 1H
 CTMPC 30.3 c
 SIVNT CD3OD
 EXREF 49.00 ppm
 BF 1.00 Hz
 RGAIN 31



single_pulse



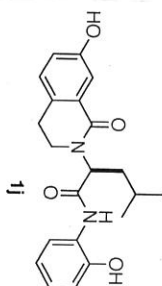
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 EXMOD single_pulse-ex2
 OSFRO 495.13 MHz
 OSFET 4.38 kHz
 OSFET 9.64 Hz
 POINT 13107
 FREOU 7429.31 Hz
 SCANS 8
 ACQTM 1.7642 sec
 PD 5.0000 sec
 PUL 6.20 usec
 INNUC 1H
 CTEMP 24.6 c
 SIVNT CDCL3
 EXREF 7.24 ppm
 BF 1.00 Hz
 RGAIN 42



F:\tn090704lactamligand\tn0907042lactam.als
single pulse decoupled gated NOE

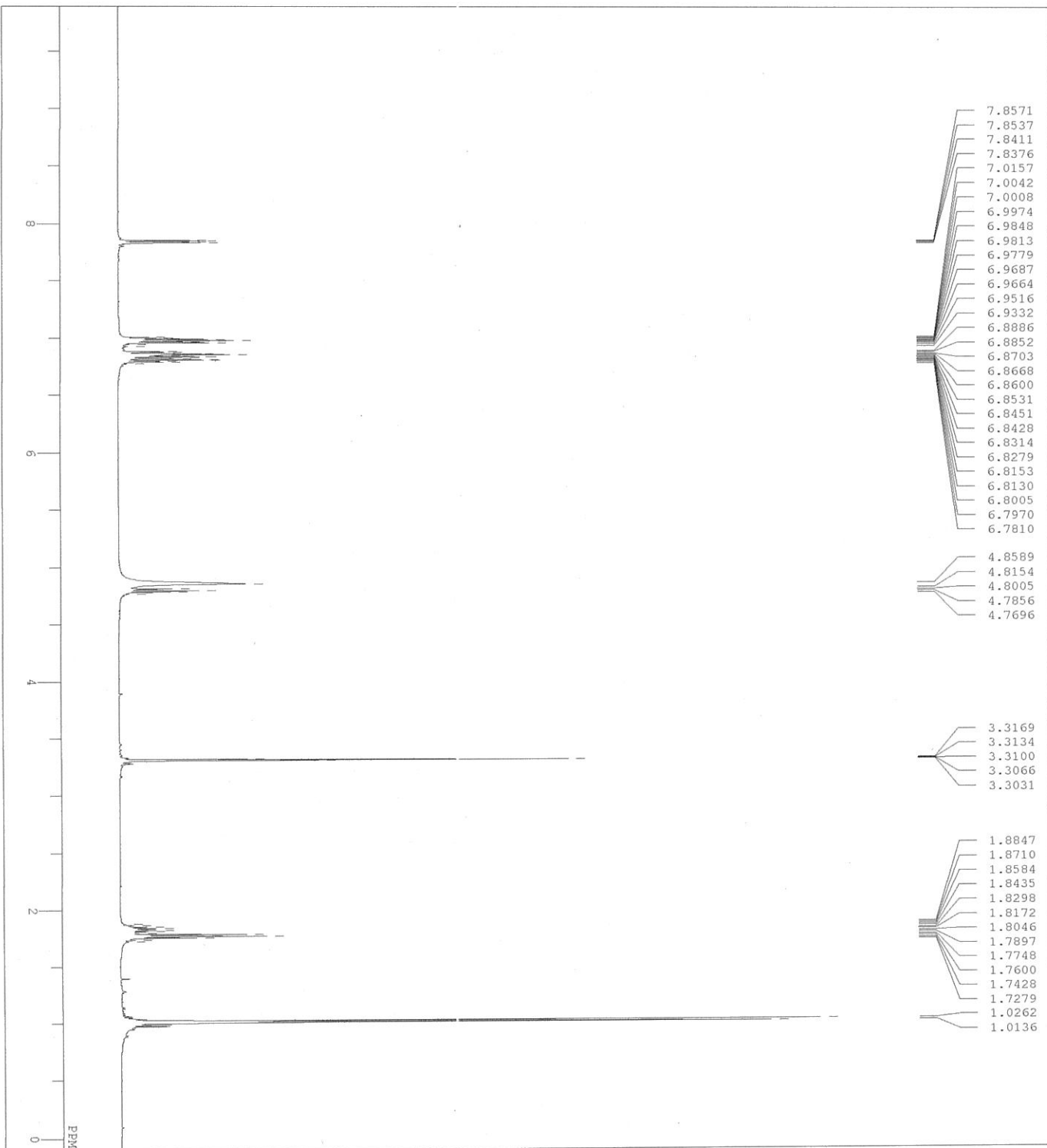


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COMNT single pulse decoupled gated NOE
DATIM 04-07-2009 23:13:36
OBNUC 13C
EXMOD single pulse dec
OBSFQ 124.51 MHz
OBSF 3.45 kHz
OBSF 6.0 Hz
POINT 26214
FREOU 31249.5 Hz
SCANS 1799
ACQTM 0.839 sec
PD 2.000 sec
PWL 3.6 us
IRNUC 1H
CTEMP 25.6 C
SLVNT CDCL3
EKKRF 77.00 ppm
BF 0.22 Hz
RGAIN 56

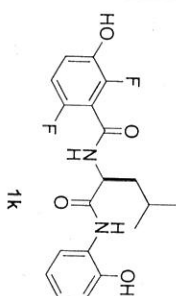


single_pulse

D:\MyDocuments\A nojiri\for printing\reere 7 1H.als

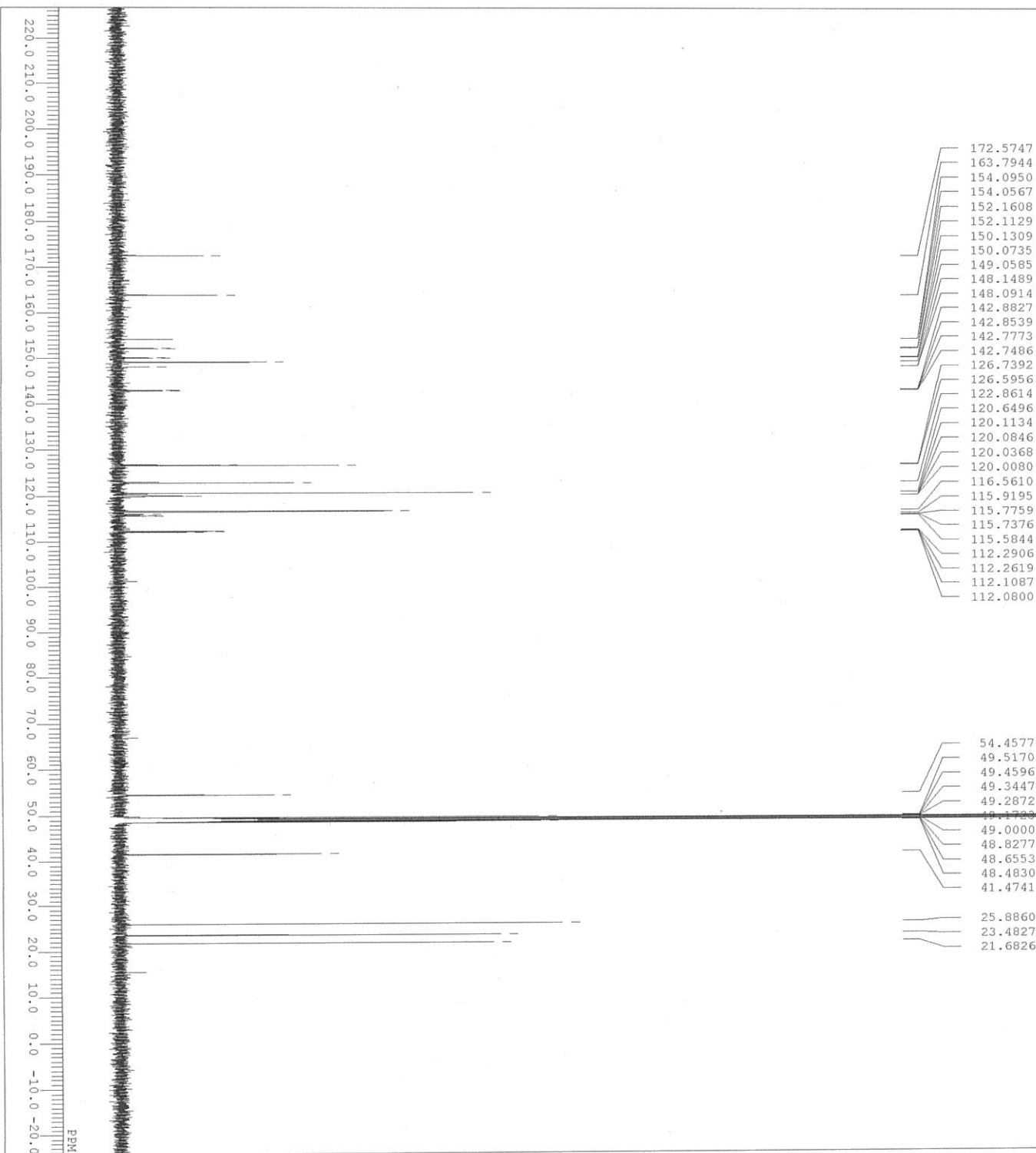


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 EXMOD single_pulse.ex2
 OBFRO 495.13 MHz
 OBFRO 4.38 KHz
 OBFIN 9.64 Hz
 POINT 13107
 FREQU 7429.31 Hz
 SCANS 8
 ACQTM 1.7642 sec
 PD 5.0000 sec
 PUL 6.20 usec
 IRNOC 1H
 CTEMP 24.2 c
 SLVNT CD3OD
 EXREF 3.31 ppm
 BF 0.01 Hz
 RGAIN 50

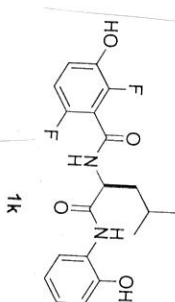


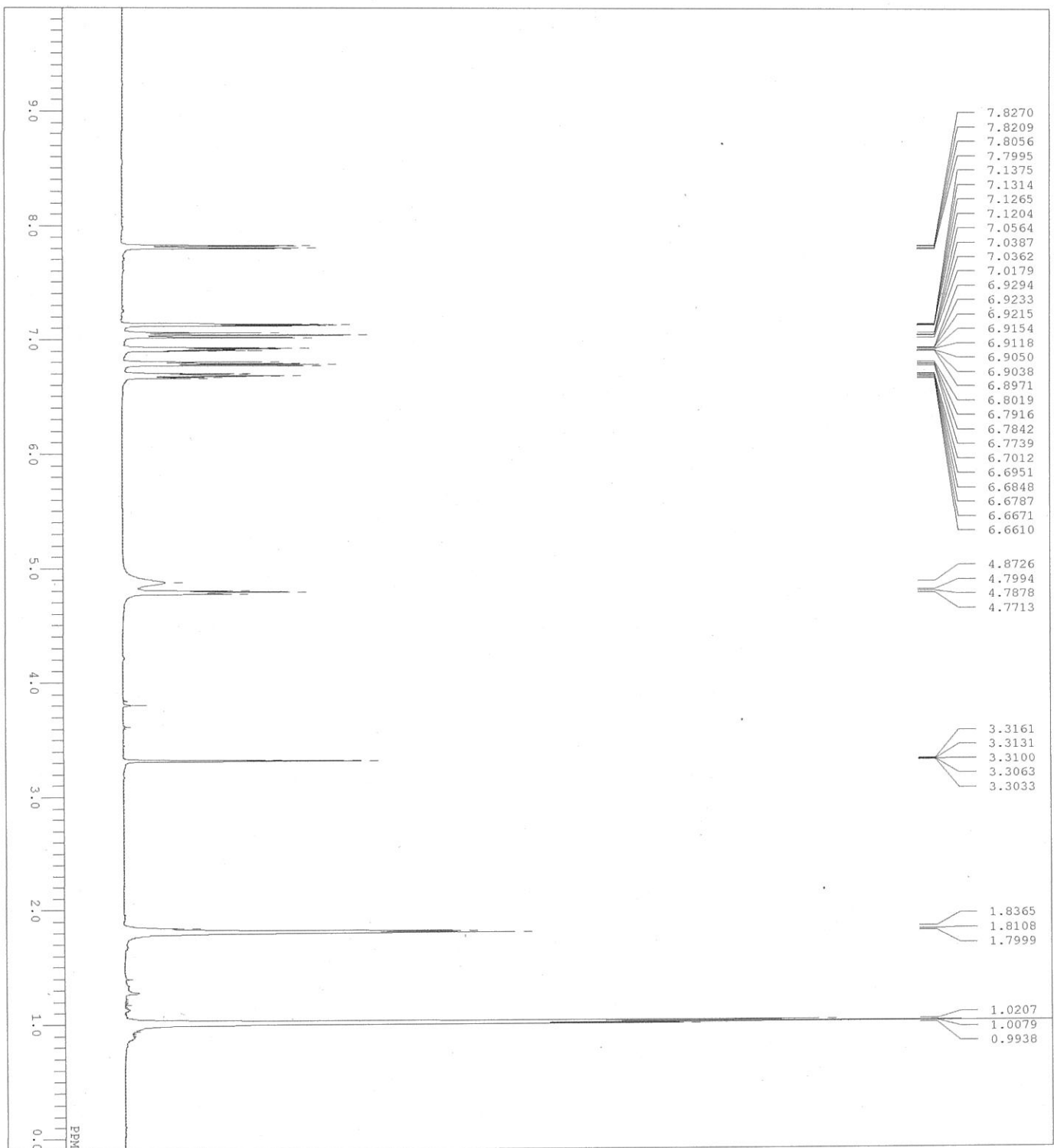
single pulse decoupled gated NOE

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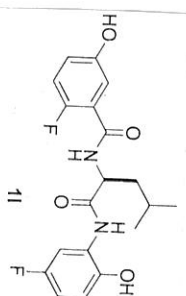


DEFILE re7 C13.als
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 DATIM 09-07-2009 20:26:10
 OBNOC 13C
 EXMOD
 OBFRO 124.51 MHz
 OBFRO 3.45 KHz
 OBFRO 6.00 Hz
 POINT 26214
 FREQU 31249.52 Hz
 SCANS 1811
 ACQTM 0.8389 sec
 PD 2.0000 sec
 PFI 3.57 usec
 IRNUC 1H
 CTEMP 25.6 c
 SIVNT CD3OD
 EXREF 49.00 ppm
 BE 1.00 Hz
 RGAIN 54



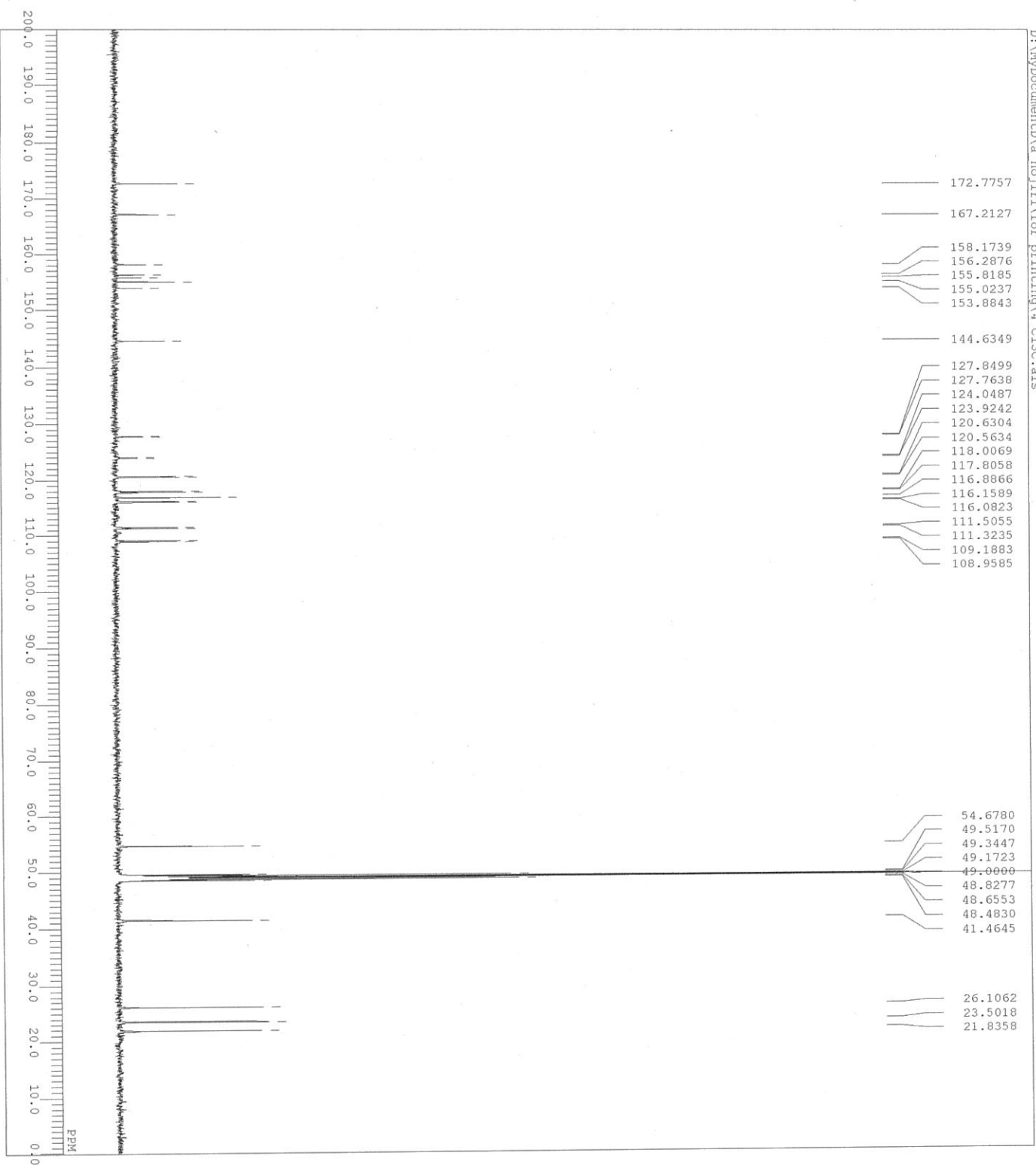


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 EXMOD non
 OBFRO 500.00 MHz
 OBSET 160.00 KHz
 OBFIN 2160.00 Hz
 POINT 32768
 FREOU 10000.00 Hz
 SCANS 8
 ACQTM 3.2768 sec
 PD 3.7232 sec
 PUL 5.70 usec
 IRNUC 1H
 CTEAP 27.0 c
 SIVNT CD3OD
 EXREF 3.31 ppm
 BF 1.00 Hz
 RGAIN 17

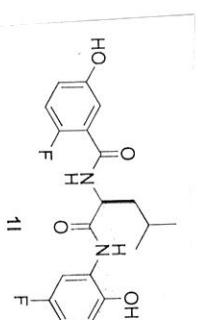


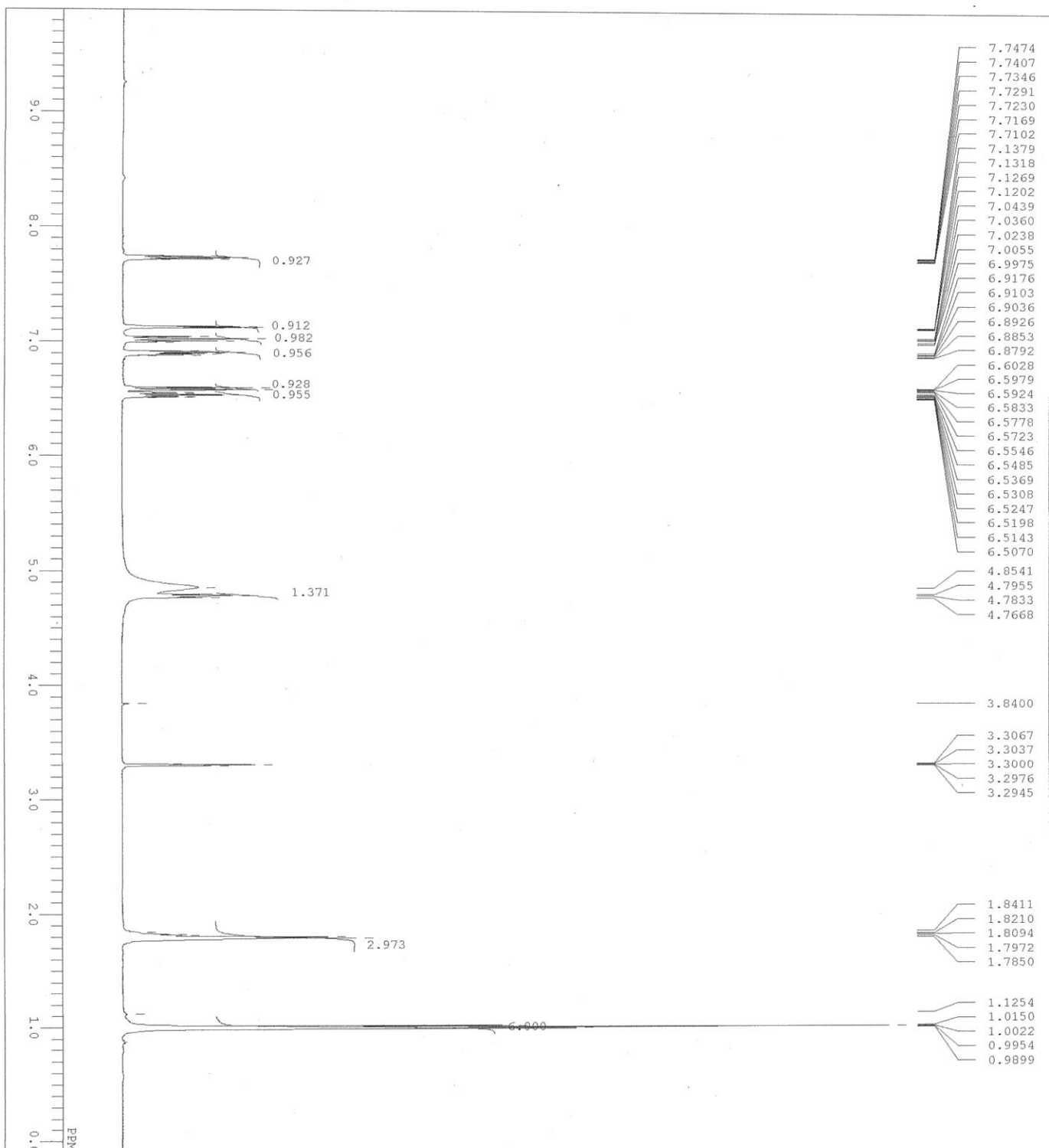
single pulse decoupled gated NOE

D:\MyDocument\4a.noj\1\for printing\4 C13C.als

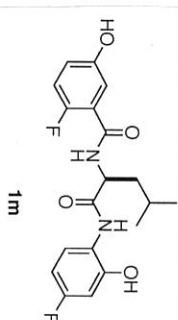


DEFILE 4 C13C.als
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 DATIM 04-06-2009 17:03:31
 ORNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 124.51 MHz
 OBSET 3.45 KHz
 OBFIN 6.00 Hz
 POINT 26214
 FREQU 31249.52 Hz
 SCANS 209
 ACQTM 0.8389 sec
 PD 2.0000 sec
 PUL 3.57 usec
 IRNUC 1H
 CTEMP 21.9 c
 SLYNT CD3OD
 EXREF 49.00 ppm
 BF 3.00 Hz
 RGAIN 60

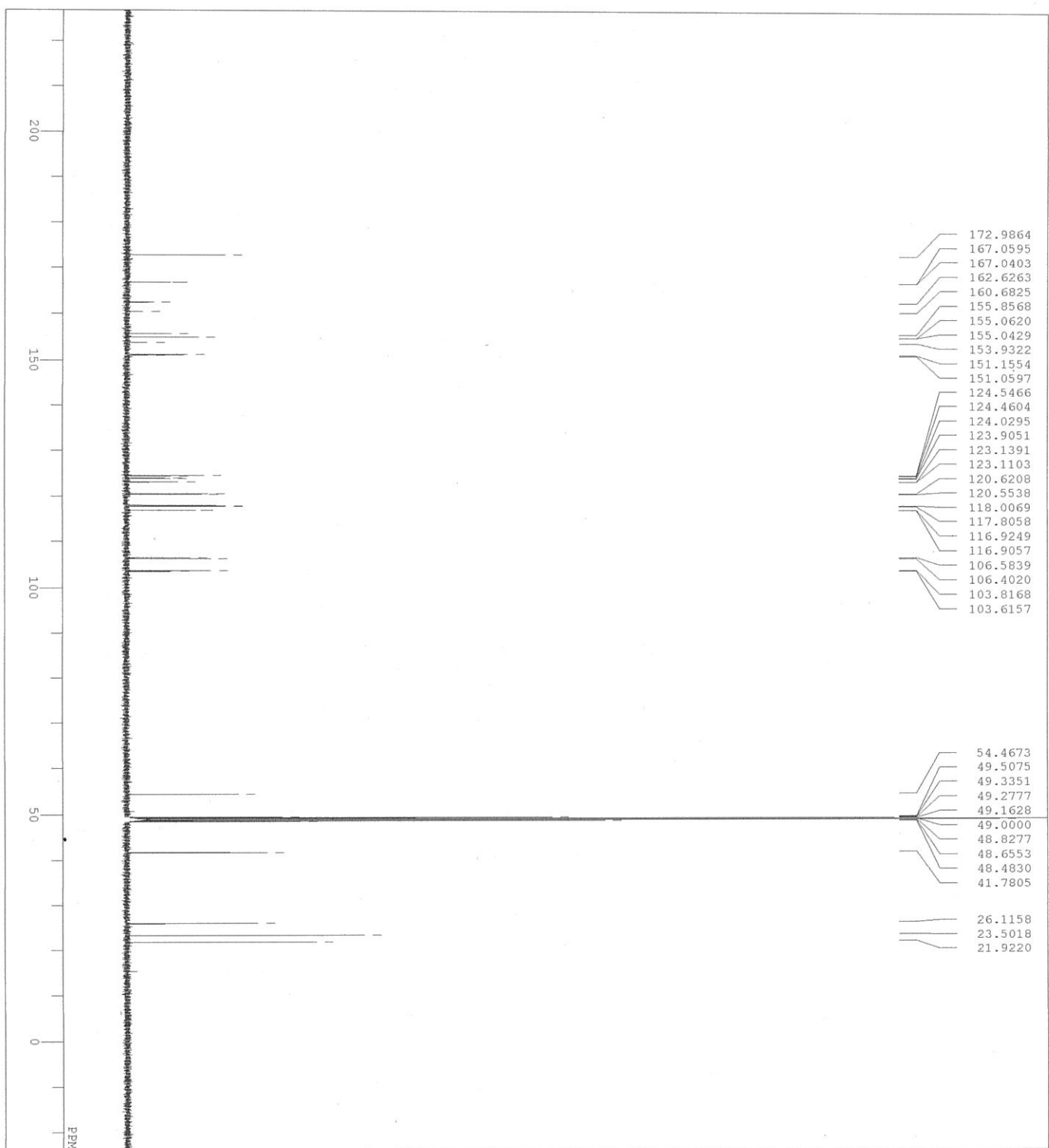




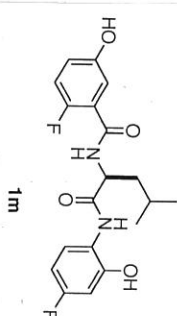
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 DATIM Tue Jul 14 00:00:20 2009
 OENUC 1H
 EXMOD non
 OBERQ 500.00 MHz
 OBERQ 160.00 MHz
 OBERQ 2160.00 Hz
 POINT 32768
 FRECU 10000.00 Hz
 SCANS 9
 ACQTM 3.2168 sec
 PD 3.7232 sec
 PUL 6.50 usec
 IRRUC 1H
 CTEMP 29.6 C
 SLVNT CD3OD
 EXREF 3.30 ppm
 BF 1.00 Hz
 RGAIN 17



single pulse decoupled gated NOE

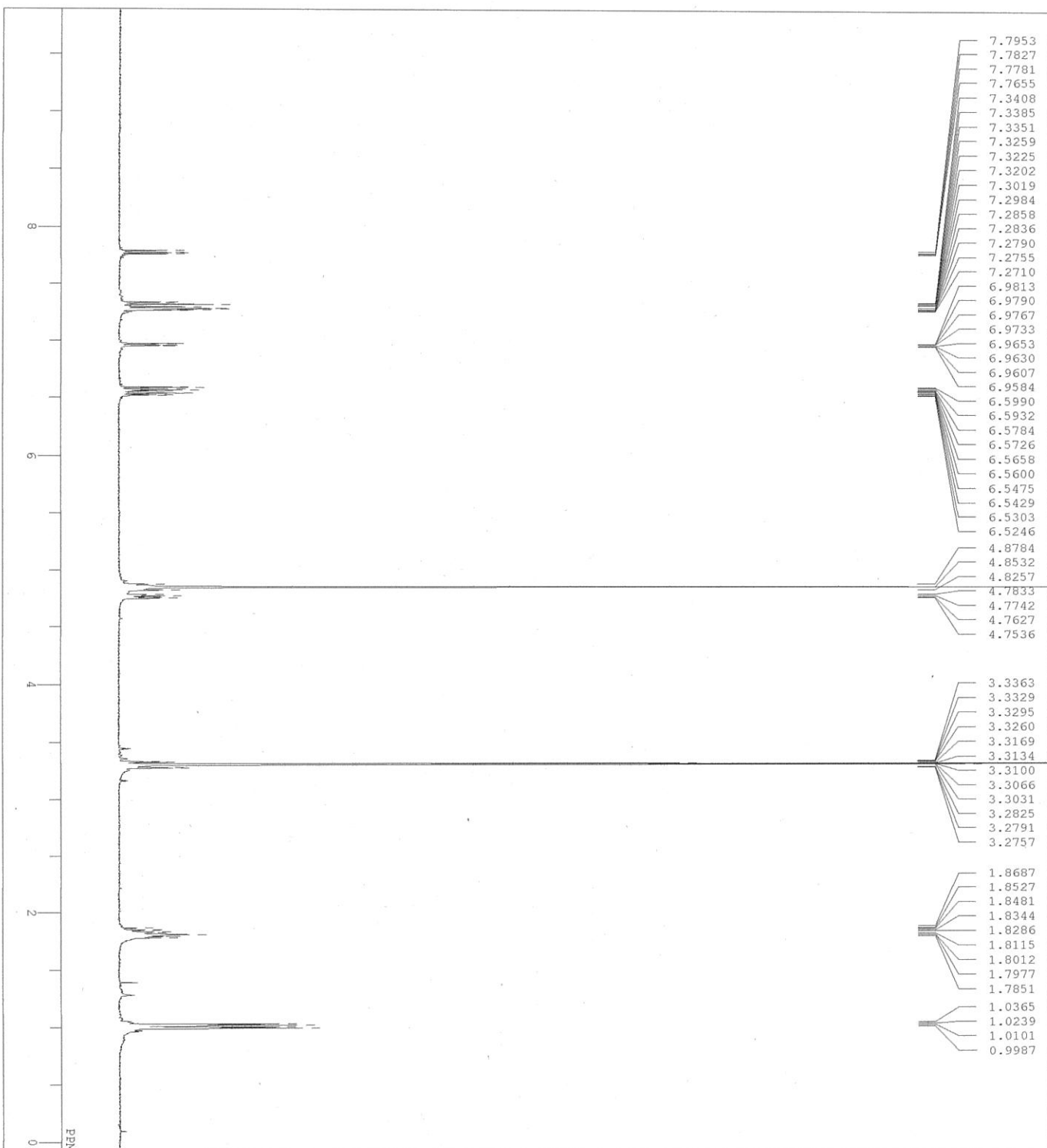


DEFIL tn0012_2.als
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 DATIM 31-01-2009 01:09:42
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 single pulse dec
 EXMOD 124.51 MHz
 OBPRO 3.45 KHz
 OBSET 6.00 Hz
 OBFIN 26214
 POINT 31249.52 Hz
 FREOU 955
 SCANS
 ACQTM 0.8389 sec
 PD 2.0000 sec
 PUL 3.57 usec
 IRNUC 1H
 CTMP 19.9 c
 SLVNT CD3OD
 EXREF 49.00 ppm
 BF 0.50 Hz
 RGAIN 60

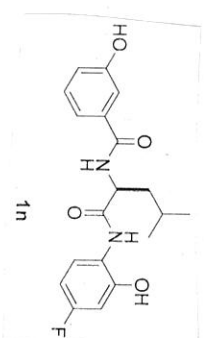


single_pulse

D:\MyDocument\1 a nojiri\for printing\re2 1H.als

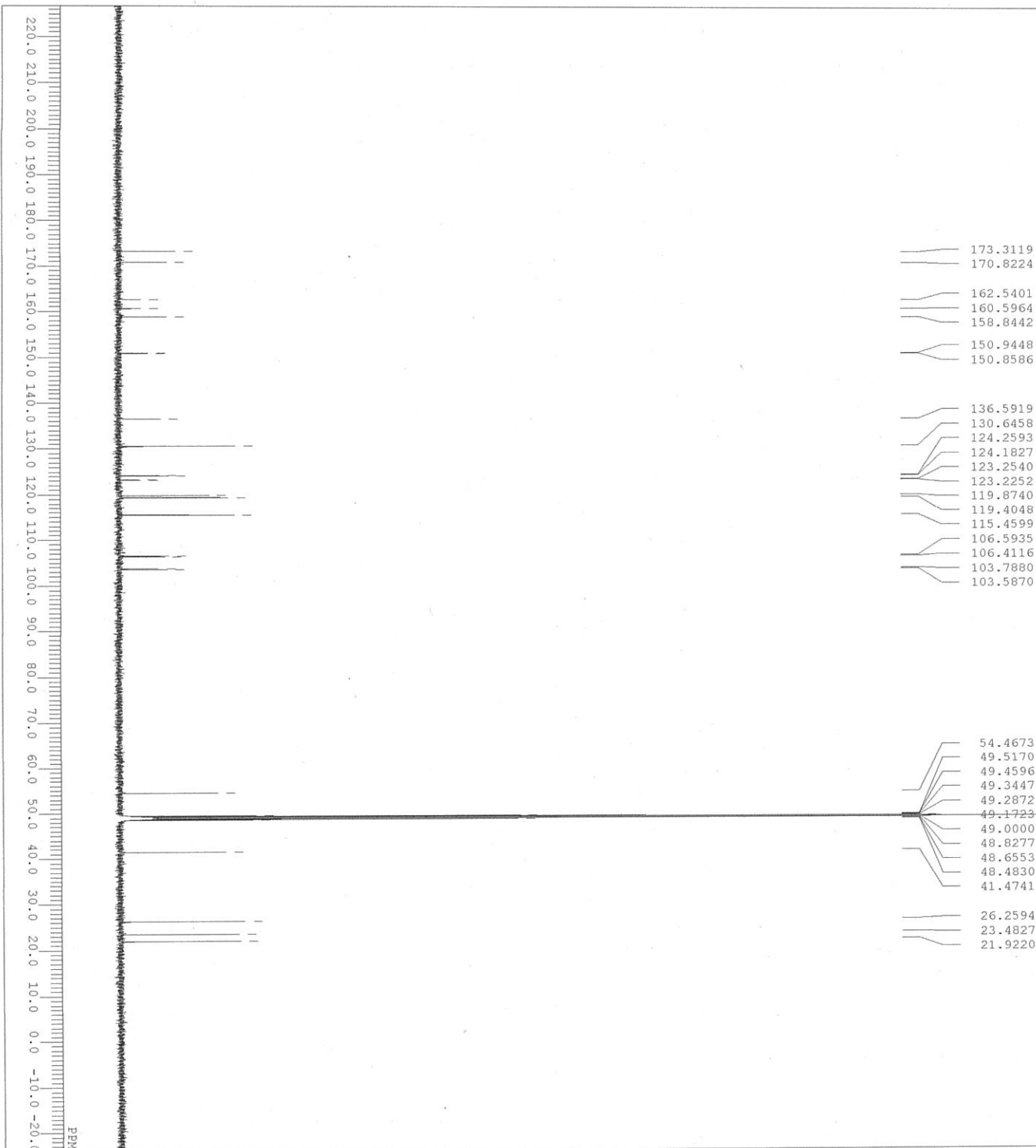


DFIL re2 1H.als
 COMNT single_pulse
 DATIM 10-07-2009 08:47:46
 OBNUC 1H
 EXMOD single_pulse.ex2
 OBFRO 495.13 MHz
 OBSET 4.38 KHz
 OBRIN 9.64 Hz
 POINT 13107
 FREOU 7429.31 Hz
 SCANS 8
 ACQTM 1.7642 sec
 PD 5.0000 sec
 PWL 6.20 usec
 IRNUC 1H
 CTEMF 24.3 C
 SIVNT CD3OD
 EXREF 3.31 ppm
 BE 0.01 Hz
 RGAIN 56

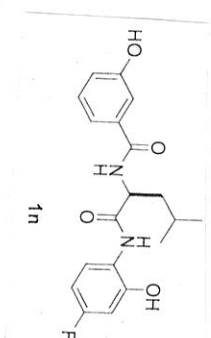


single pulse decoupled gated NOE

D:\MyDocuments\aj\for printing\2 C13.als

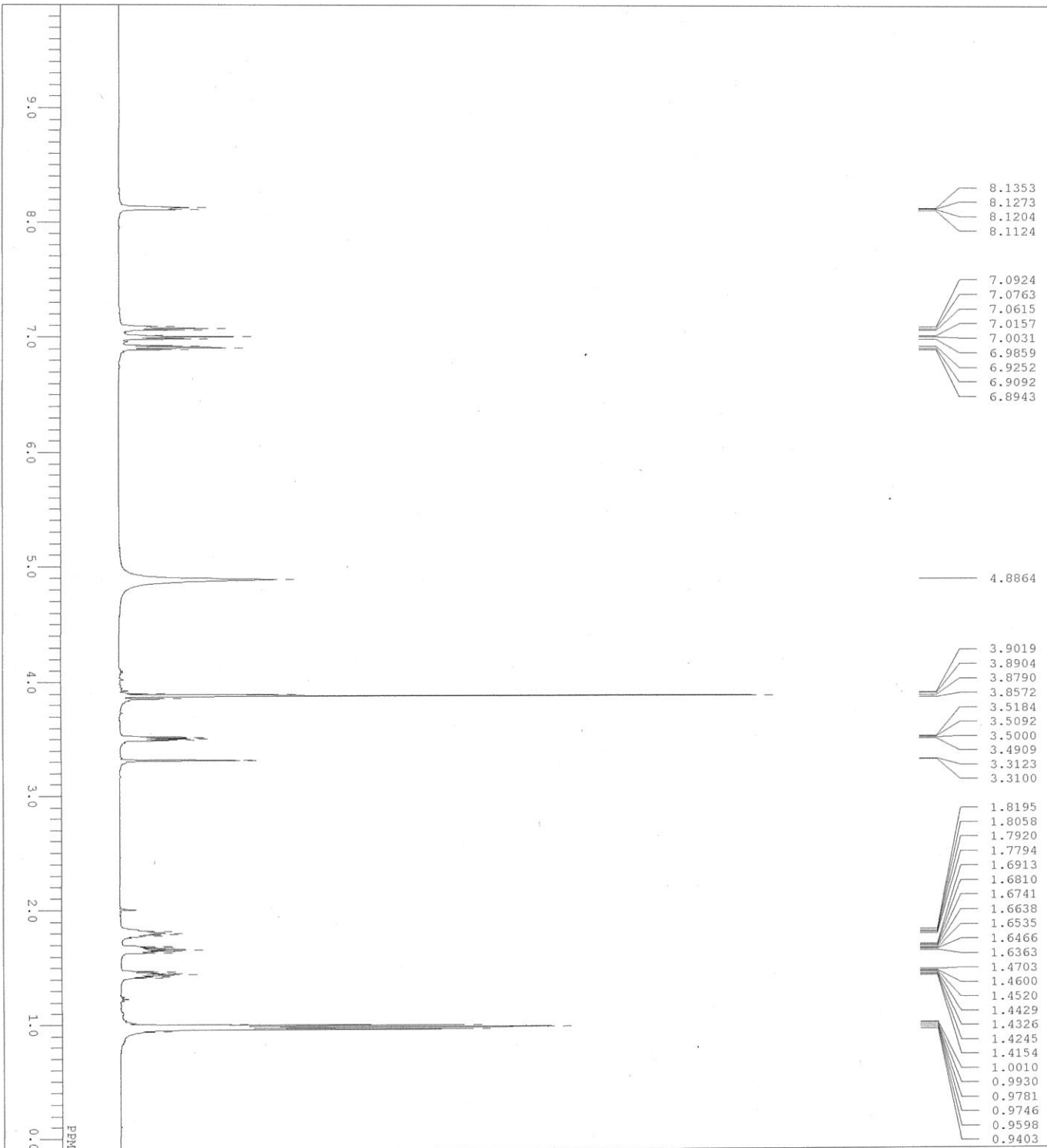


DEFILE 2 C13.als
 COMMT single pulse decoupled gated NOE
 DATIM 05-06-2009 21:23:27
 OBNOC 13C
 EXMOD single_pulse_dec
 OBFRO 124.51 MHz
 OBSET 3.45 KHz
 OBFIN 6.00 Hz
 POINT 26214
 FREQU 31249.52 Hz
 SCANS 175
 ACQTM 0.8389 sec
 PD 2.0000 sec
 PUL 3.57 usec
 IRNOC 1H
 CTEMP 22.1 c
 SLVNT CD3OD
 EXREF 49.00 ppm
 BF 1.00 Hz
 RGAIN 60

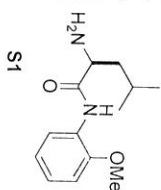


single_pulse

D:\MyDocument\A noiri\for printing\5 1H.als

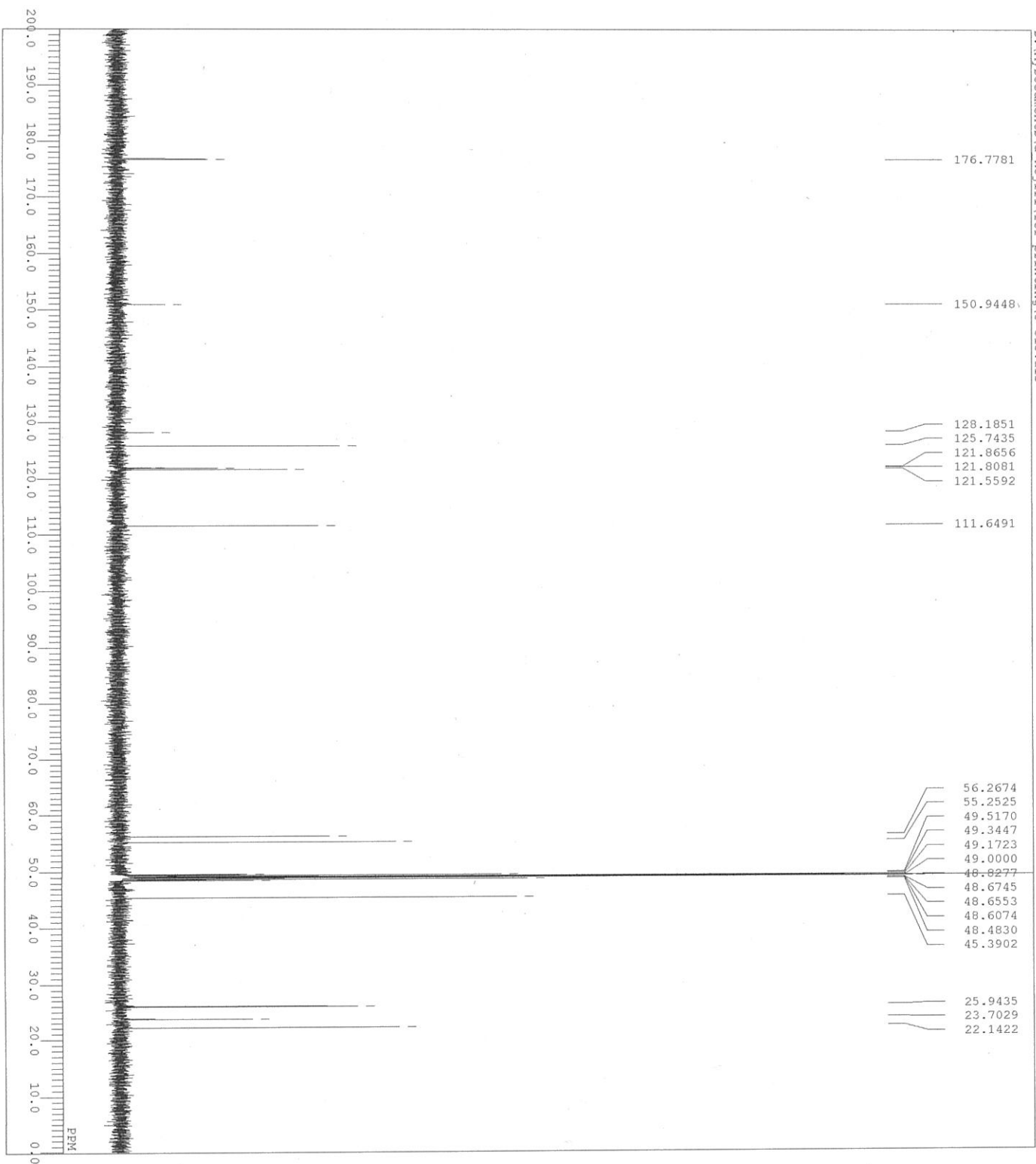


DEFILE 5 1H.als
 COMNT single_pulse
 DATIM 05-06-2009 10:47:08
 OBNOC 1H
 EXMOD single_pulse-ex2
 OBFREQ 495.13 MHz
 OBFREQ 4.38 KHz
 OBFREQ 9.64 Hz
 POINT 13107
 FREQ0 7429.31 Hz
 SCANS 8
 ACQTM 1.7642 sec
 PD 5.0000 sec
 PUL 6.20 usec
 IRNUC 1H
 CTEMP 20.4 c
 SLVNT CD3OD
 EXREF 3.31 ppm
 BF 0.10 Hz
 RGAIN 40

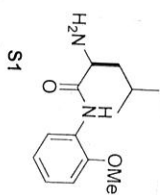


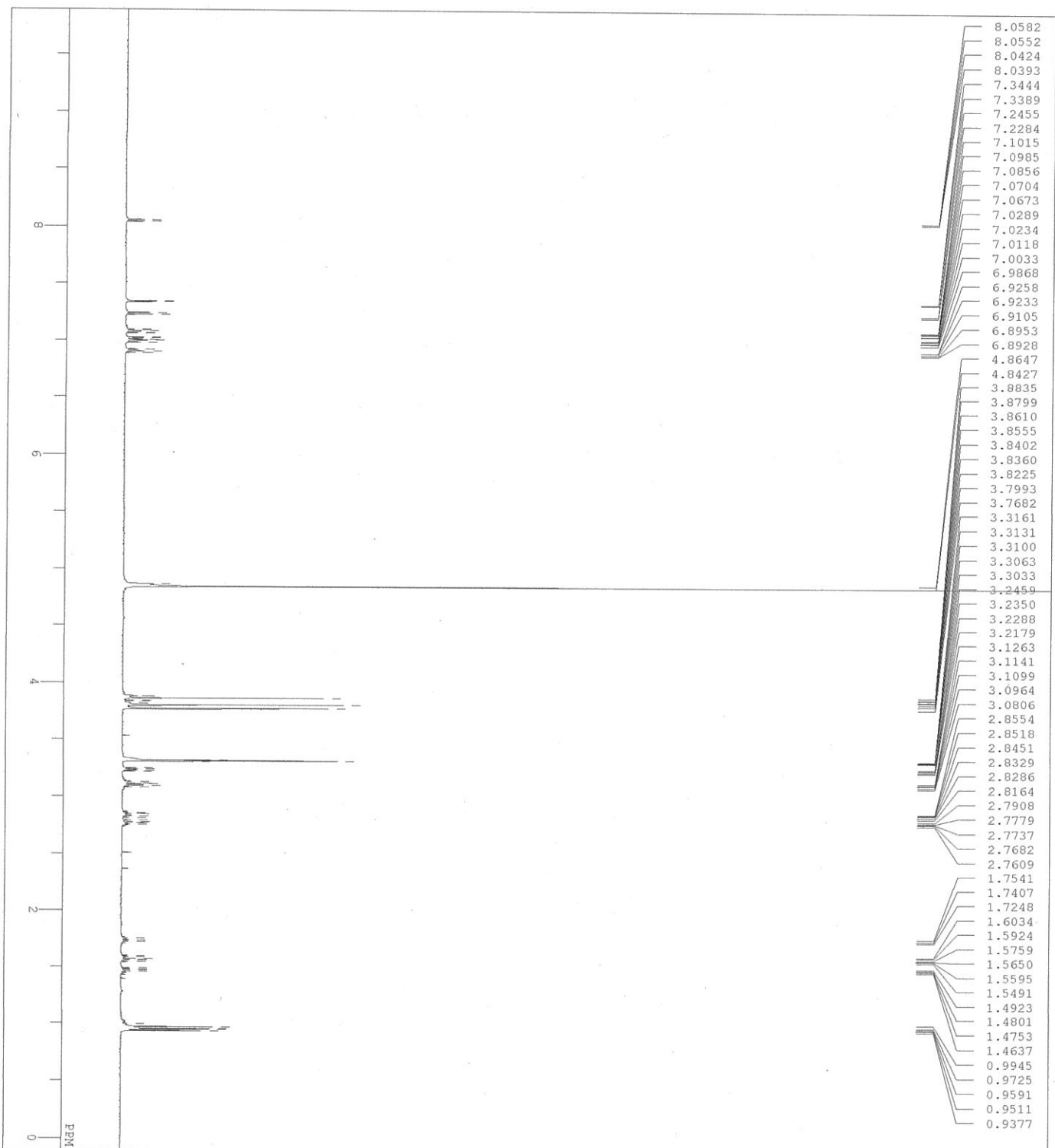
single pulse decoupled gated NOE

D:\MyDocumentDVA\for printing\5 C13.als

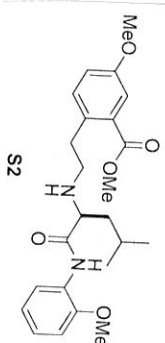


DFIL 5 C13.als
 COMNT single pulse decoupled gated NOE
 DATIM 05-06-2009 10:59:18
 OBNUC 13C
 EXMOC single pulse dec
 OBRPO 124.51 MHz
 OBRPO 3.45 KHz
 OBRPO 6.00 Hz
 OFIN 26214
 FREQ 31249.52 Hz
 SCANS 235
 ACQTM 0.8389 sec
 PD 2.0000 sec
 PM 3.57 usec
 IRNUC 1H
 CTMPC 22.1 c
 CD3OD 49.00 ppm
 SLVNT 0.10 Hz
 EXREF 58
 BE
 RGAIN

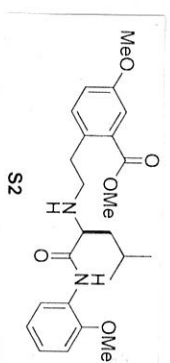
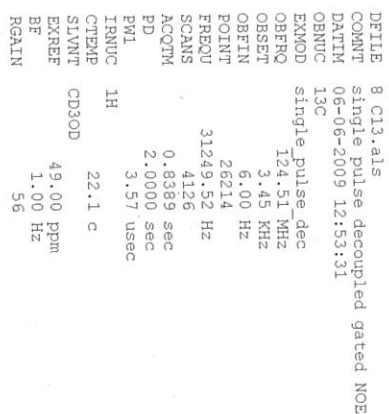




FILE AN090605c.nm.fid
 COUNT 81 H
 DATEIN Fri Jun 5 18:03:40 2009
 OBNUC 1H
 EXMOD non
 OBFRQ 500.00 MHz
 OBSET 160.00 KHz
 OBFTN 2160.00 Hz
 POINT 32768
 FREQ 10000.00 Hz
 SCANS 16
 ACQTM 3.2768 sec
 PD 3.7232 sec
 FWH 5.70 usec
 IRNUC 1H
 CTEMP 26.8 c
 SLVNT CD3OD
 EXREF 3.31 ppm
 BF 1.00 Hz
 RGAIN 20

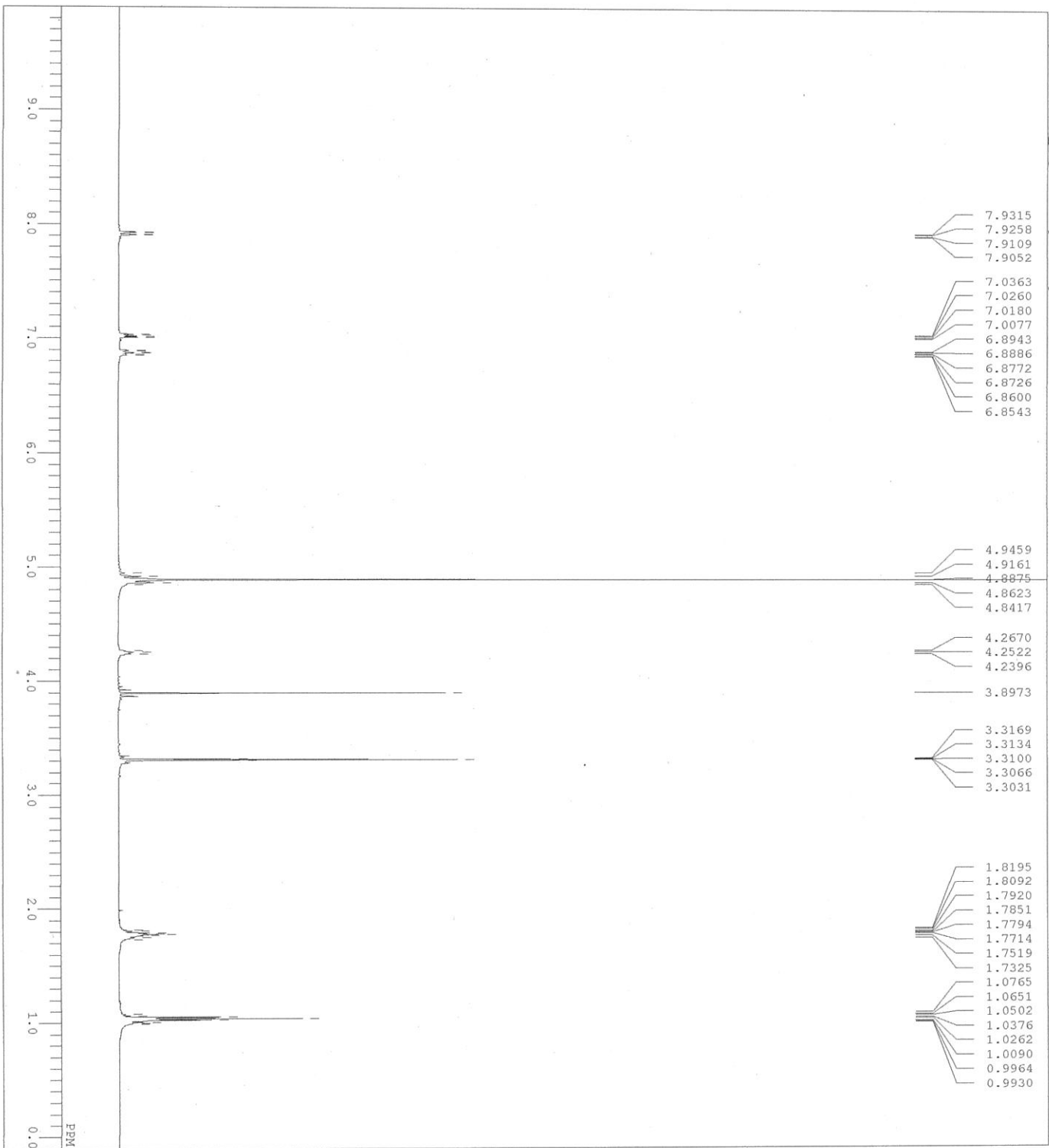


D:\MyDocument\anojiri\for printing\8 Cl3.als

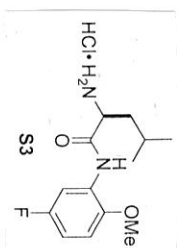


single_pulse

D:\MyDocument\A nojiri\for printing\3_1H.als



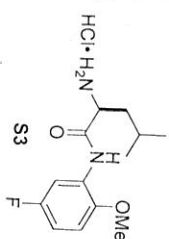
DEFILE 3 1H.als
 COMENT single_pulse
 DATIM 05-06-2009 10:20:32
 ORNUC 1H
 EXMOD single_pulse.ex2
 OBEFO 495.13 MHz
 OBEFO 4.38 KHz
 OBEIN 9.64 Hz
 POINT 13107
 FREOU 7429.31 Hz
 SCANS 8
 ACQTM 1.7642 sec
 PD 5.0000 sec
 PUL 6.20 usec
 IRNUC 1H
 CTEMP 20.6 c
 SIVNT CD3OD
 EXREF 3.31 ppm
 BF 0.10 Hz
 RGAIN 50

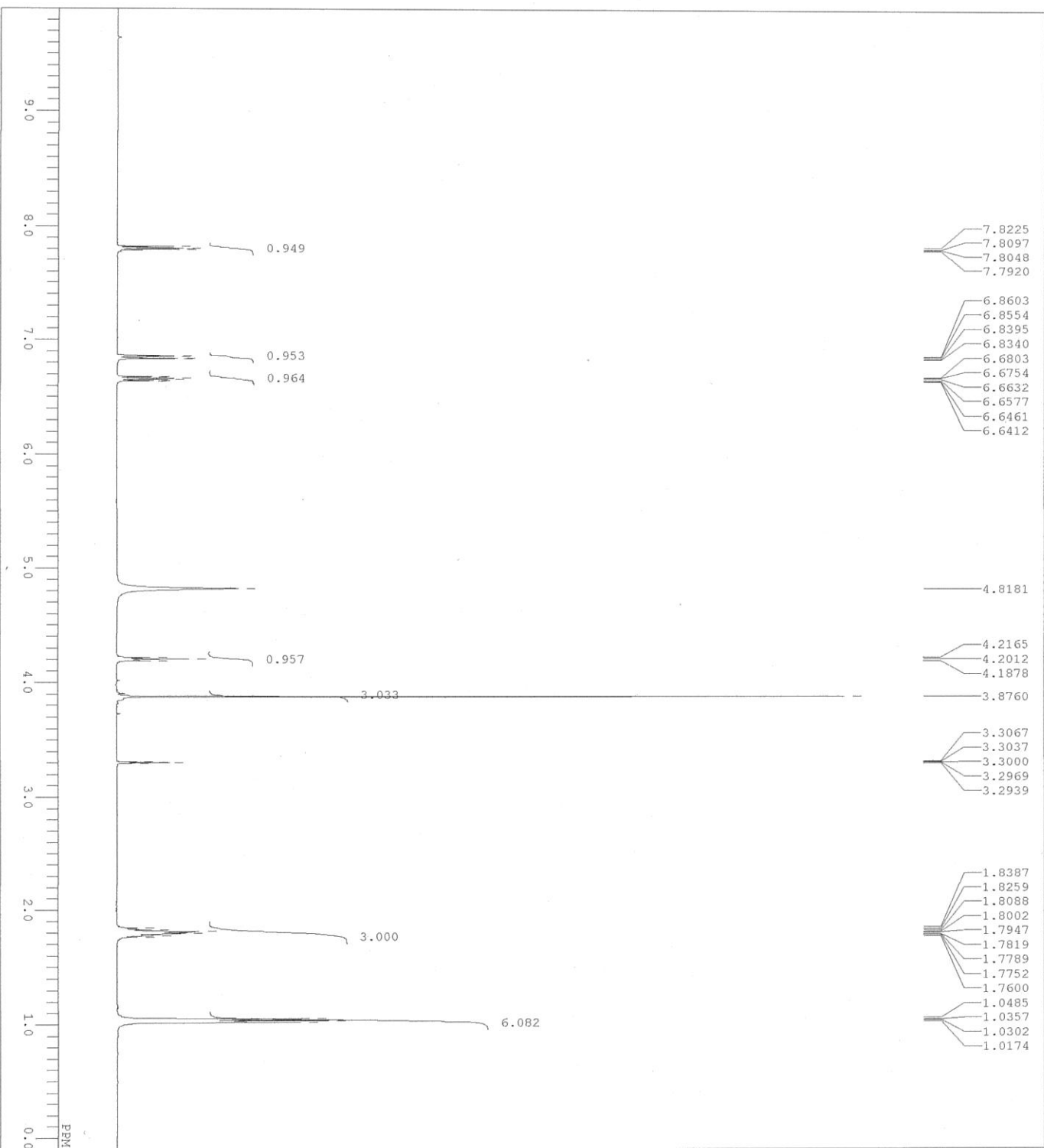


single pulse decoupled gated NOE

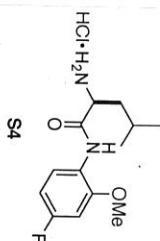


FILE 3 C13 e-als
 COMNT single pulse decoupled gated NOE
 DATIM 06-06-2009 09:26:25
 OBNUC 13C
 EXMOD single-pulse-dec
 OBFRQ 124.51 MHz
 OBSET 3.45 KHz
 OBSIN 6.00 Hz
 POINT 26214
 FREQU 31249.52 Hz
 SCANS 14980
 ACQTM 0.8389 sec
 PD 2.0000 sec
 PUL 3.57 usec
 IRNUC 1H
 CTEMP 22.2 c
 CD3OD
 EXREF 49.00 ppm
 BF 1.00 Hz
 RGAIN 56

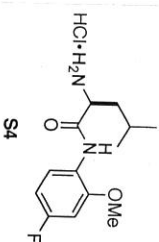
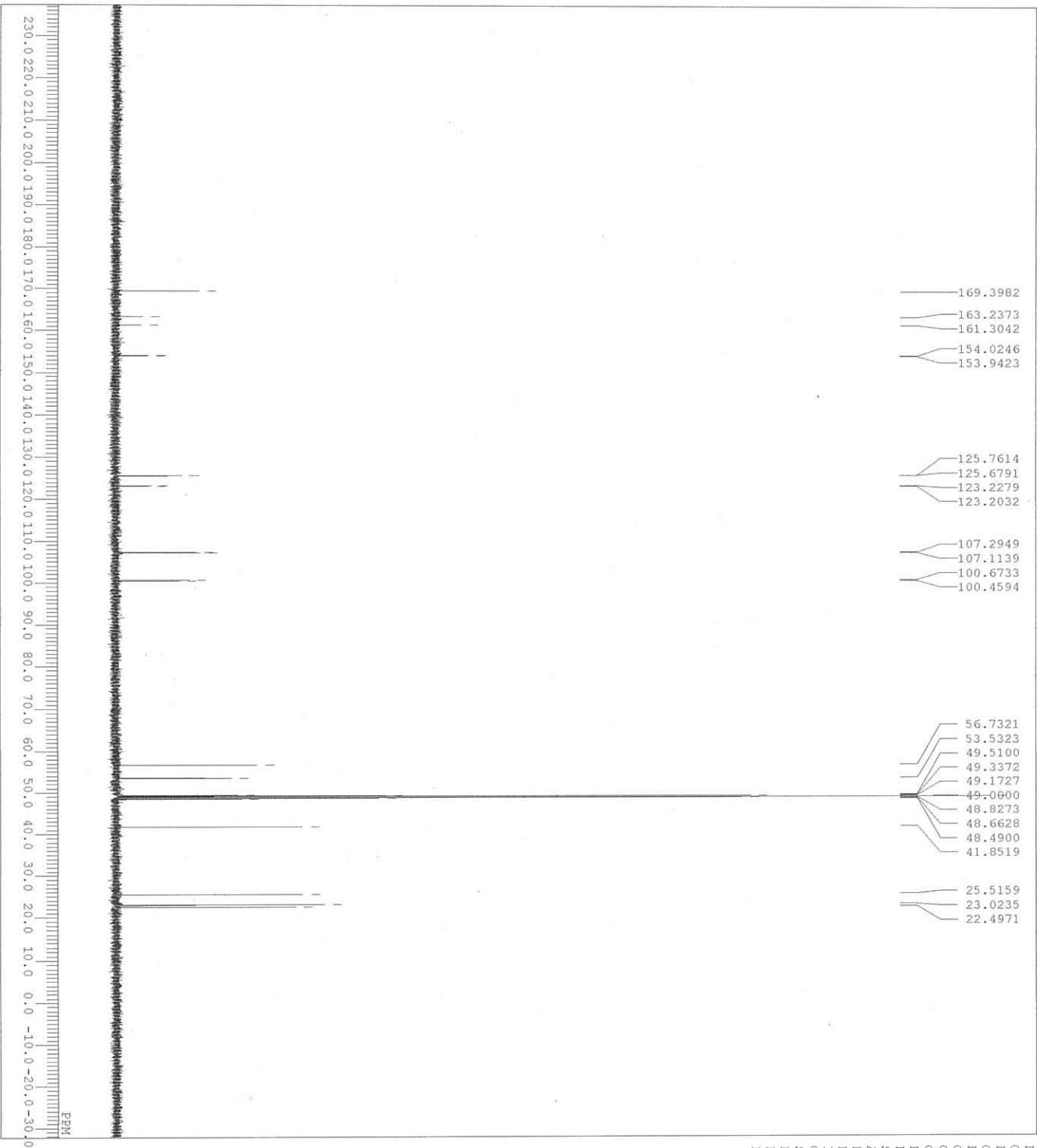




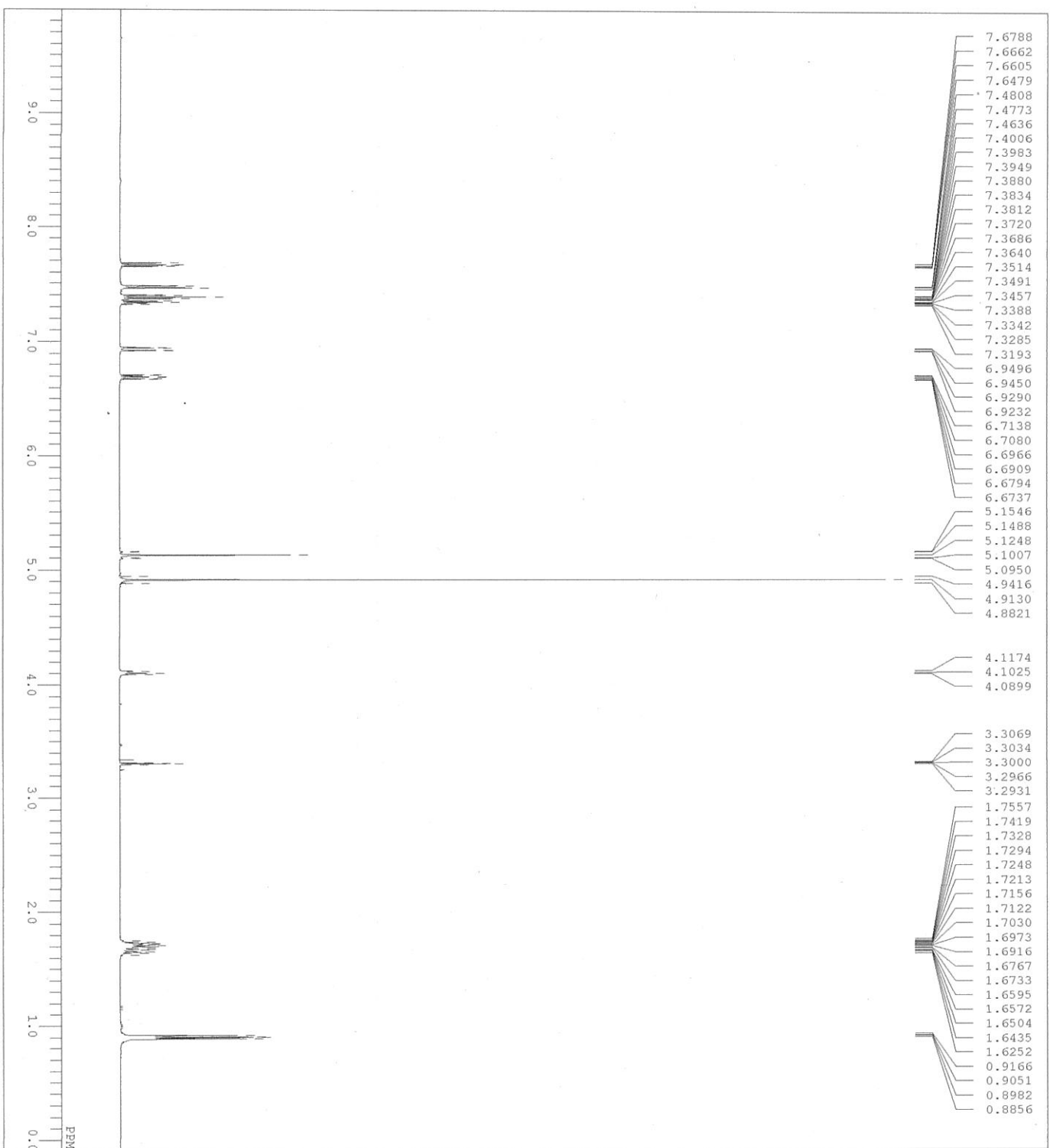
DEFILE D:\MyDocument\NITABARU\tn090710\ntmnd\tn090710\ntmnd1
 COMNT 1H line
 DATIM Fri Jul 10 18:06:53 2009
 OENUC 1H
 EXMOD non
 OBFRO 500.00 MHz
 OBSET 160.00 KHz
 OBFIN 2160.0 Hz
 POINT 32768
 FREOU 10000.0 Hz
 SCANS 29
 ACQTM 3.277 sec
 PD 3.723 sec
 PUL 6.5 us
 IRNUC 1H
 CTMP 29.7 c
 SLVNT CD3OD
 EXREF 3.30 ppm
 BF 0.22 Hz
 RGAIN 14



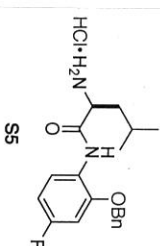
DEFILE D:\MyDocument\NITABARU\tn090710intmd\tn090710intmd1
 COUNT 1H Line 125.65 MHz
 DATIM Fri Jul 10 17:57:09 2009
 OBNUC 13C
 EXMOD bcm
 OBFREQ 120.00 kHz
 OBFINT 7958.0 Hz
 POINT 32768
 FREOU 33898.3 Hz
 SCANS 205
 ACQTM 0.967 sec
 PD 2.033 sec
 PM 5.0 us
 IRNUC 1H
 CTEMP 30.5 C
 SLVNT CD3OD
 EREF 49.00 ppm
 BF 0.22 Hz
 RGAIN 32

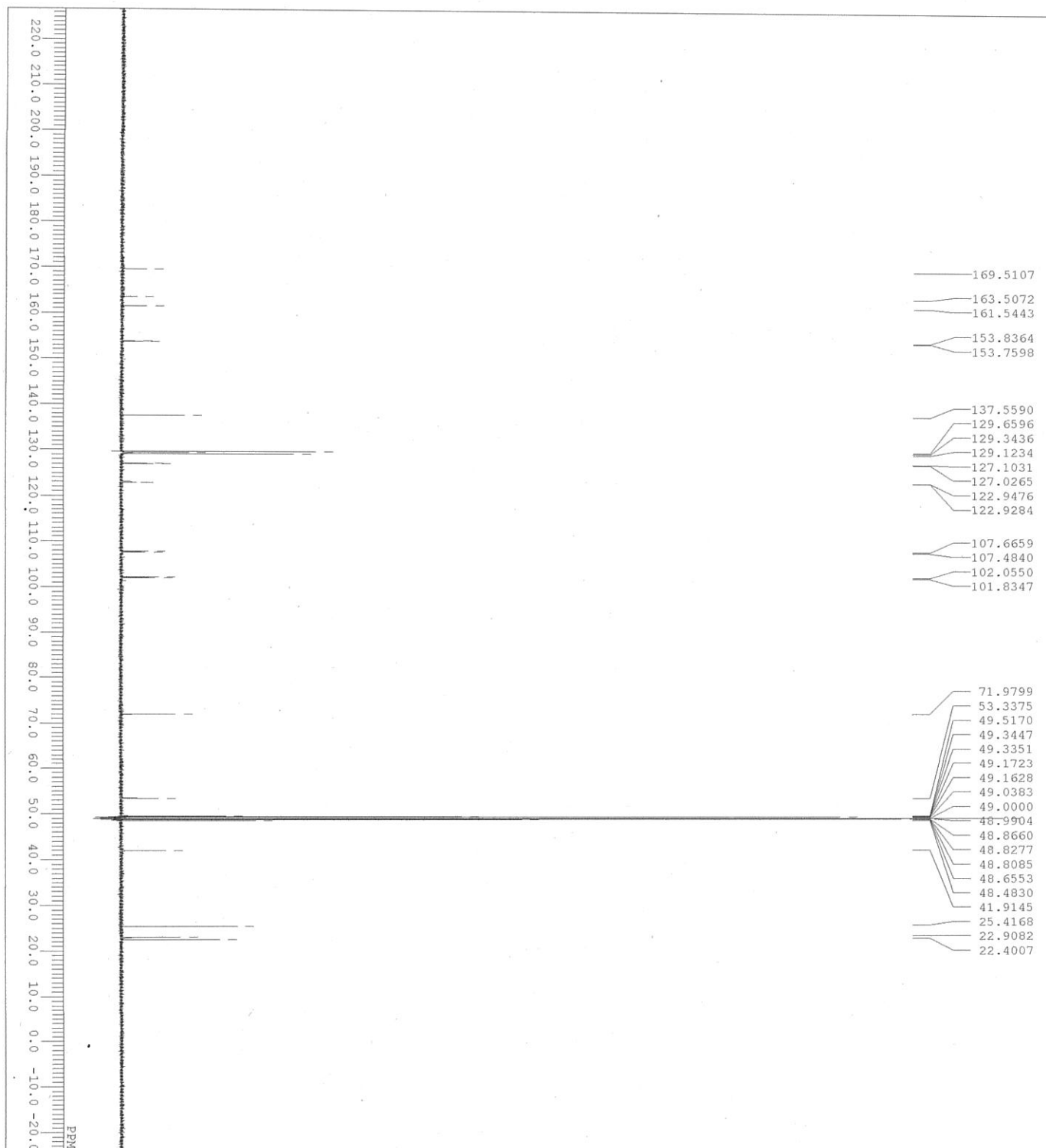


single_pulse



DEFILE tn0010.1
 COMNT single_pulse
 DATIM 30-01-2009 05:26:26
 OBNUC 1H
 EXMOD single_pulse.ex2
 OBFRO 495.13 MHz
 OBSRT 4.38 kHz
 OBSIN 9.64 Hz
 POINT 16384
 FREOU 9286.78 Hz
 SCANS 8
 ACQTM 1.7642 sec
 PD 5.0000 sec
 PUL 6.20 usec
 IRNUC 1H
 CTMPC 18.1 c
 SLVNT CD3OD
 EXREF 3.30 ppm
 BF 1.00 Hz
 RGAIN 36





DFILE F:\tn0010\tn0010_2.als
 COMNT single pulse decoupled gated NOE
 DATIM 30-01-2009 05:47:12
 OBNUC 13C
 EXMOD single pulse dec
 OBERQ 124.51 MHz
 OBERF 3.45 kHz
 OBERIN 6.0 Hz
 POINT 26214
 FREQU 31249.5 Hz
 SCANS 295
 ACQTM 0.839 sec
 PD 2.000 sec
 PUL 3.6 us
 IRNUC 1H
 CTEMP 19.3 C
 SLVNT CD3OD
 EXREF 49.00 ppm
 BF 0.22 Hz
 RGAIN 58

