

Supplementary Materials

Total Synthesis and Antimicrobial Evaluation of 23-Demethyleushearilide and Extensive Antimicrobial Evaluation of All Synthetic Stereoisomers of (16*Z*,20*E*)-Eushearilide and (16*E*,20*E*)-Eushearilide

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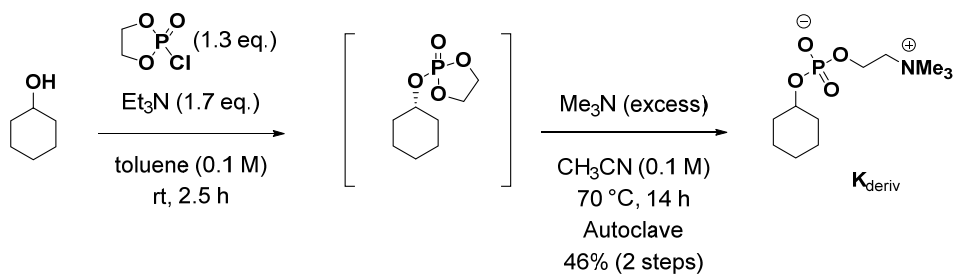
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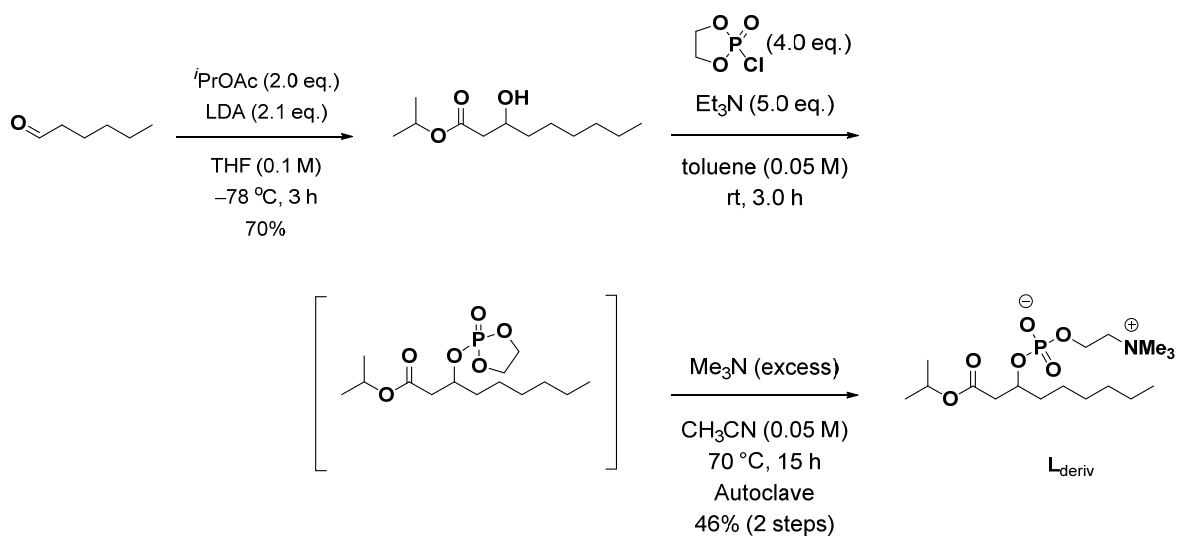
1. Preparation of Phosphates **K**_{deriv} and **L**_{deriv}: S2
2. Copies of ¹H and ¹³C NMR spectra: S3 to S24

1. Preparation of Phosphates **K_{deriv}** and **L_{deriv}**

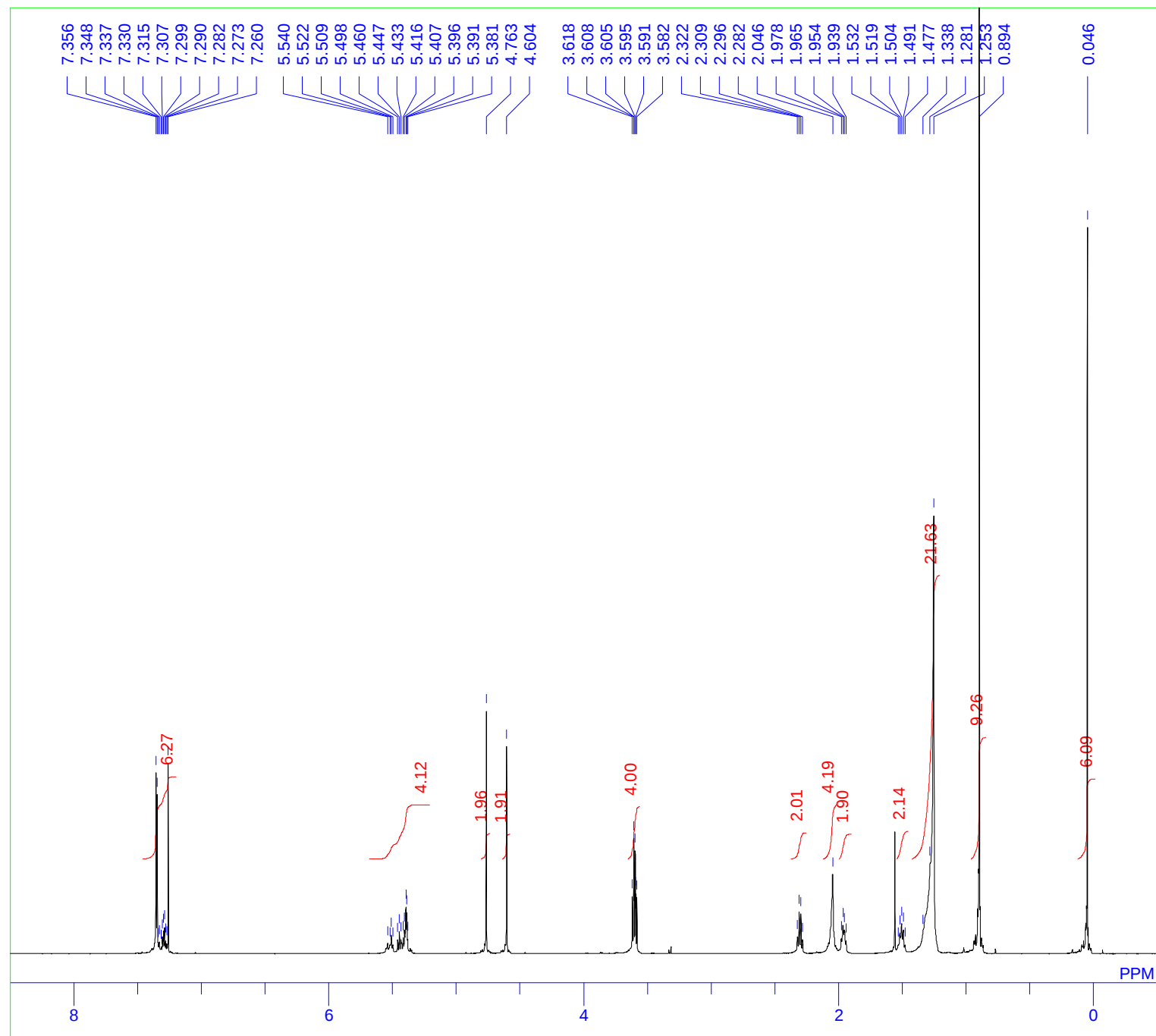
Phosphate **K_{deriv}** was prepared as shown below.



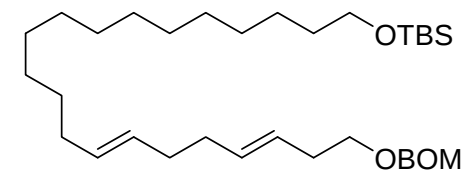
Phosphate **L_{deriv}** was prepared as shown below.



single_pulse

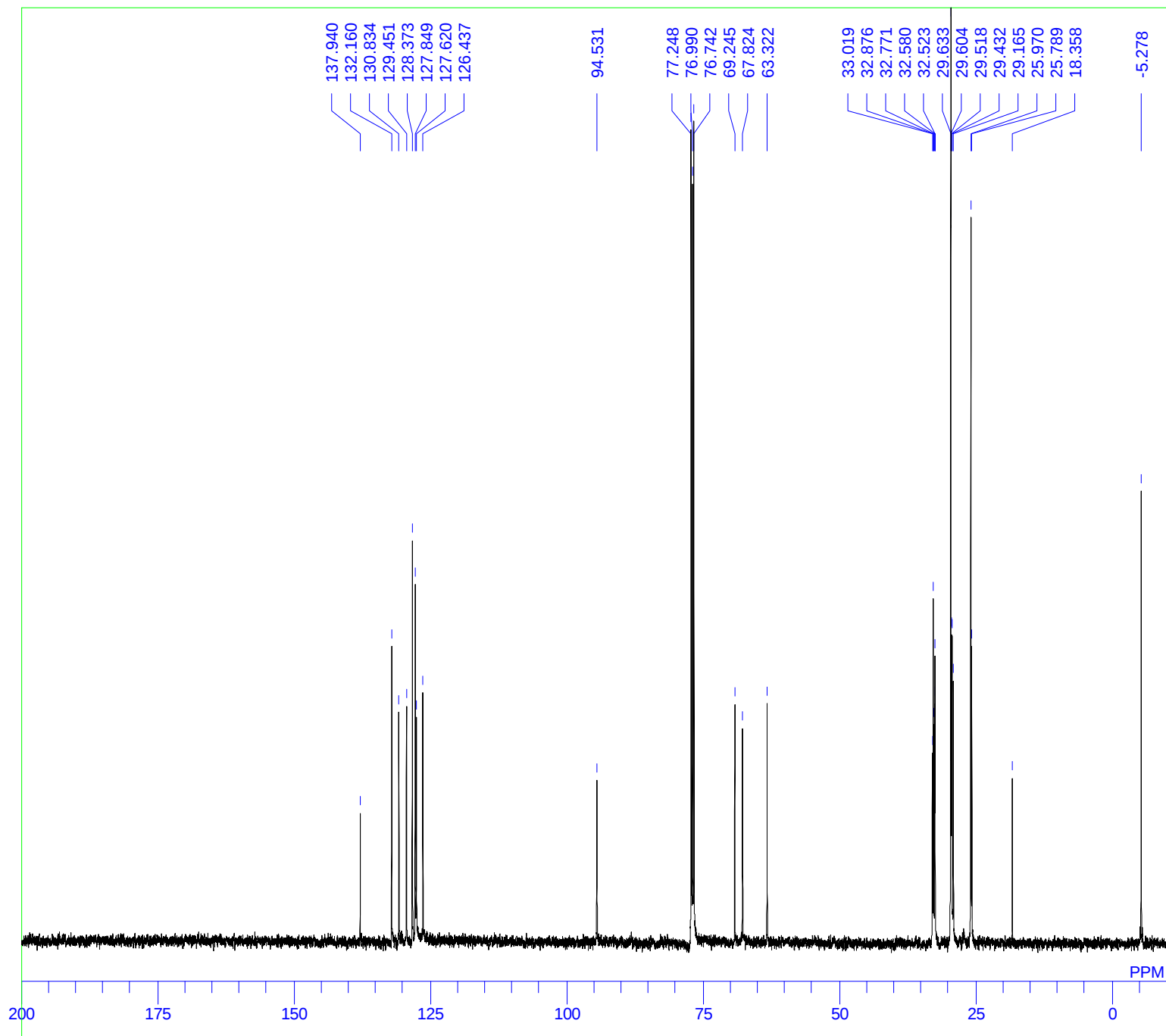


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 EXMOD proton.jpg
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 OBFIN 6.01 Hz
 POINT 16384
 FREQU 9384.38 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 5.0000 sec
 PW1 3.52 usec
 IRNUC 1H
 CTEMP 23.0 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 42

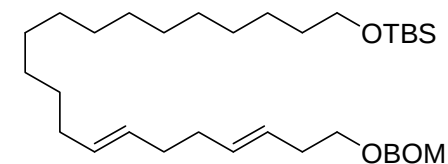


(3E,7E)-21-*tert*-Butyldimethylsilyloxy-1-benzoyloxymethoxyhenicos-3,7-diene (**9**)

single pulse decoupled gated NOE

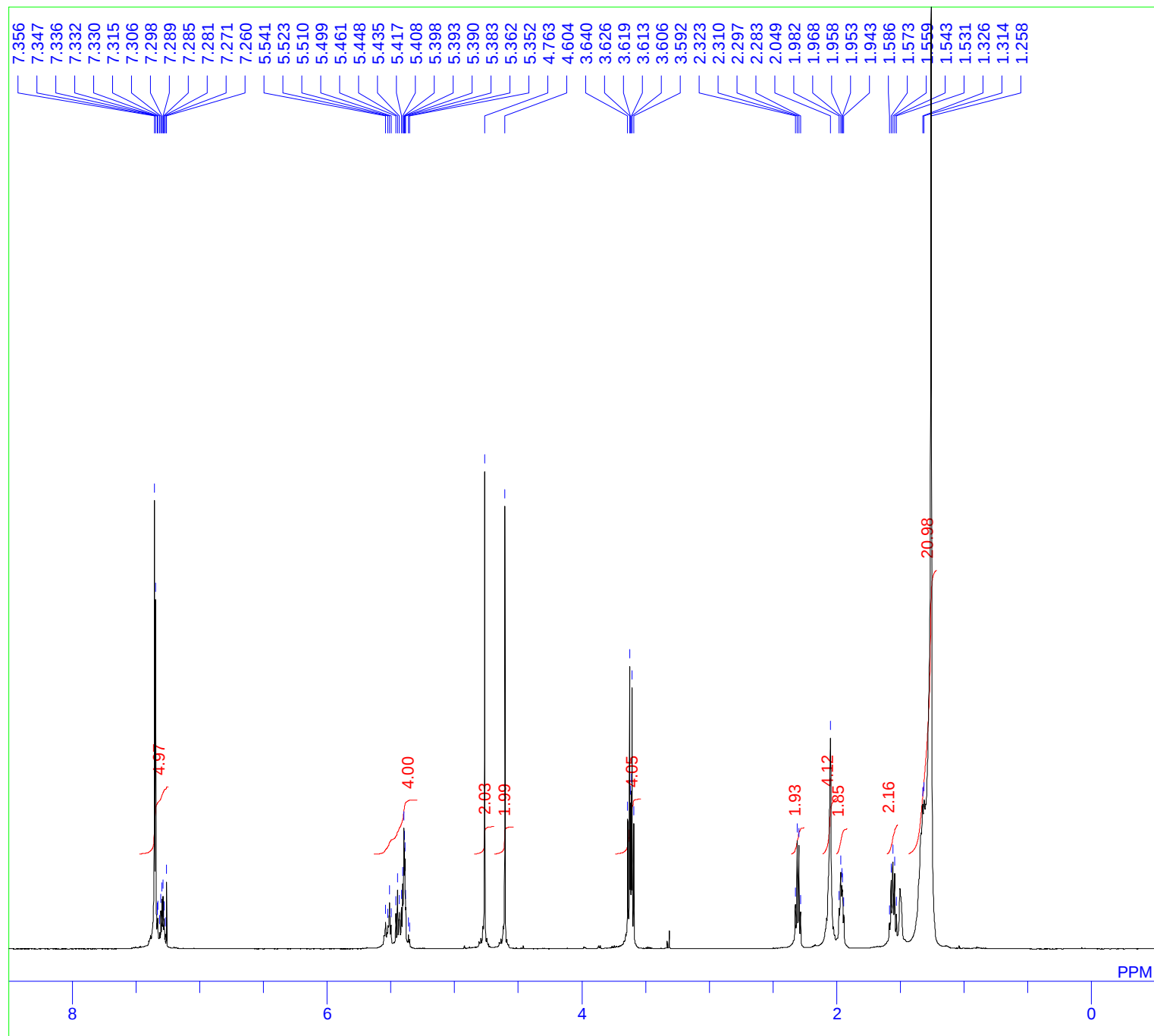


DFILE IT-3-014-E_Carbon-1-2.als
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 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 39308.18 Hz
 SCANS 532
 ACQTM 0.8336 sec
 PD 2.0000 sec
 PW1 3.74 usec
 IRNUC 1H
 CTEMP 23.1 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.12 Hz
 RGAIN 24

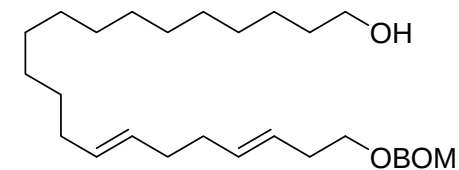


(3E,7E)-21-*tert*-Butyldimethylsilyloxy-1-benzyloxymethoxyhenicosa-3,7-diene (**9**)

single_pulse

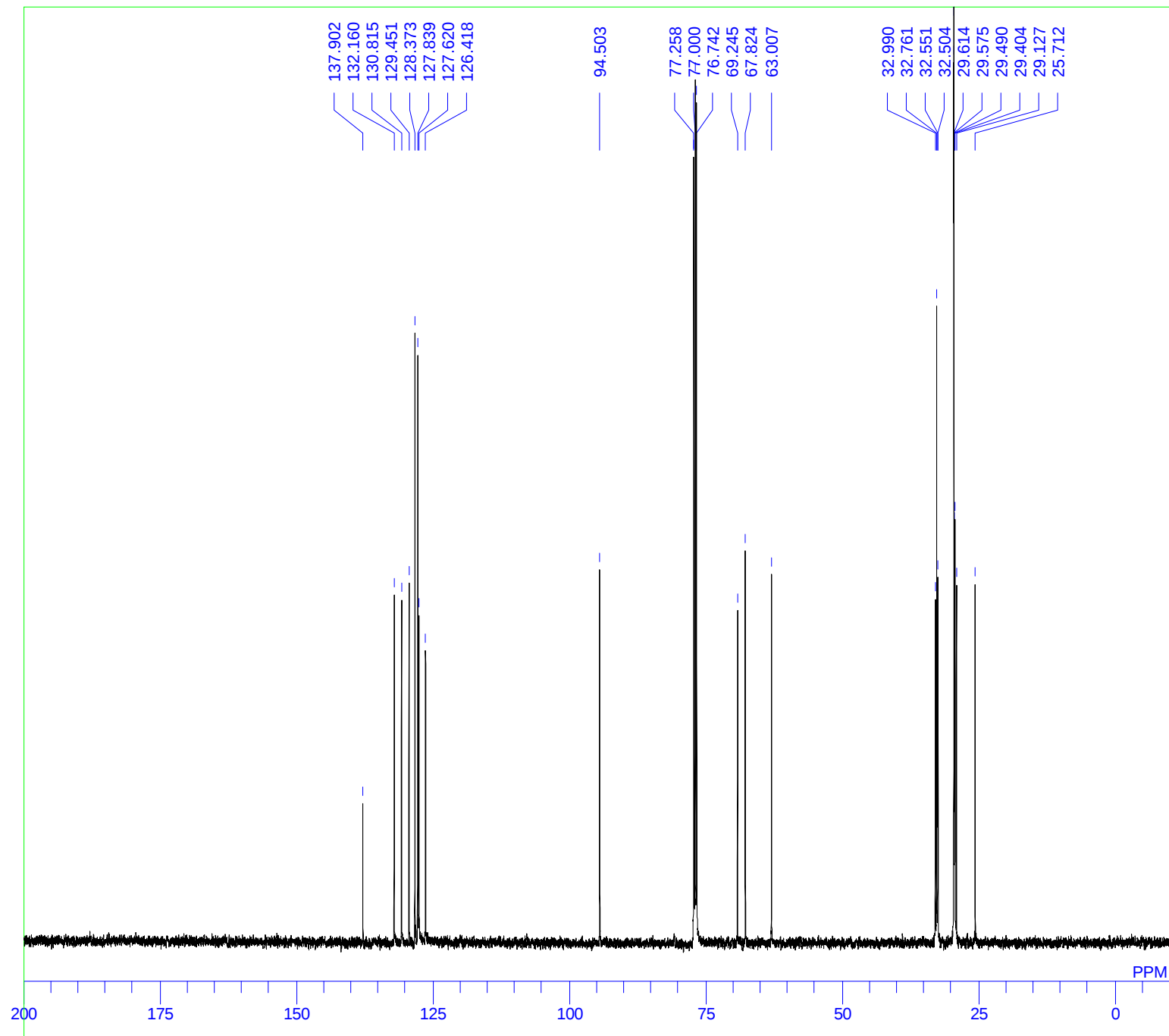


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 OBNUC 1H
 EXMOD proton.jpg
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16384
 FREQU 9384.38 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 5.0000 sec
 PW1 3.52 usec
 IRNUC 1H
 CTEMP 23.1 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.25 Hz
 RGAIN 22

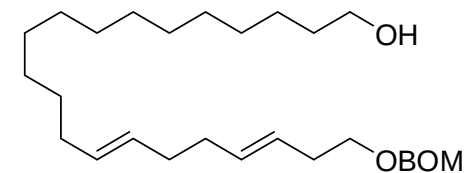


(14*E*,18*E*)-21-Benzyloxymethoxyhenicosa-14,18-dien-1-ol (**10**)

single pulse decoupled gated NOE

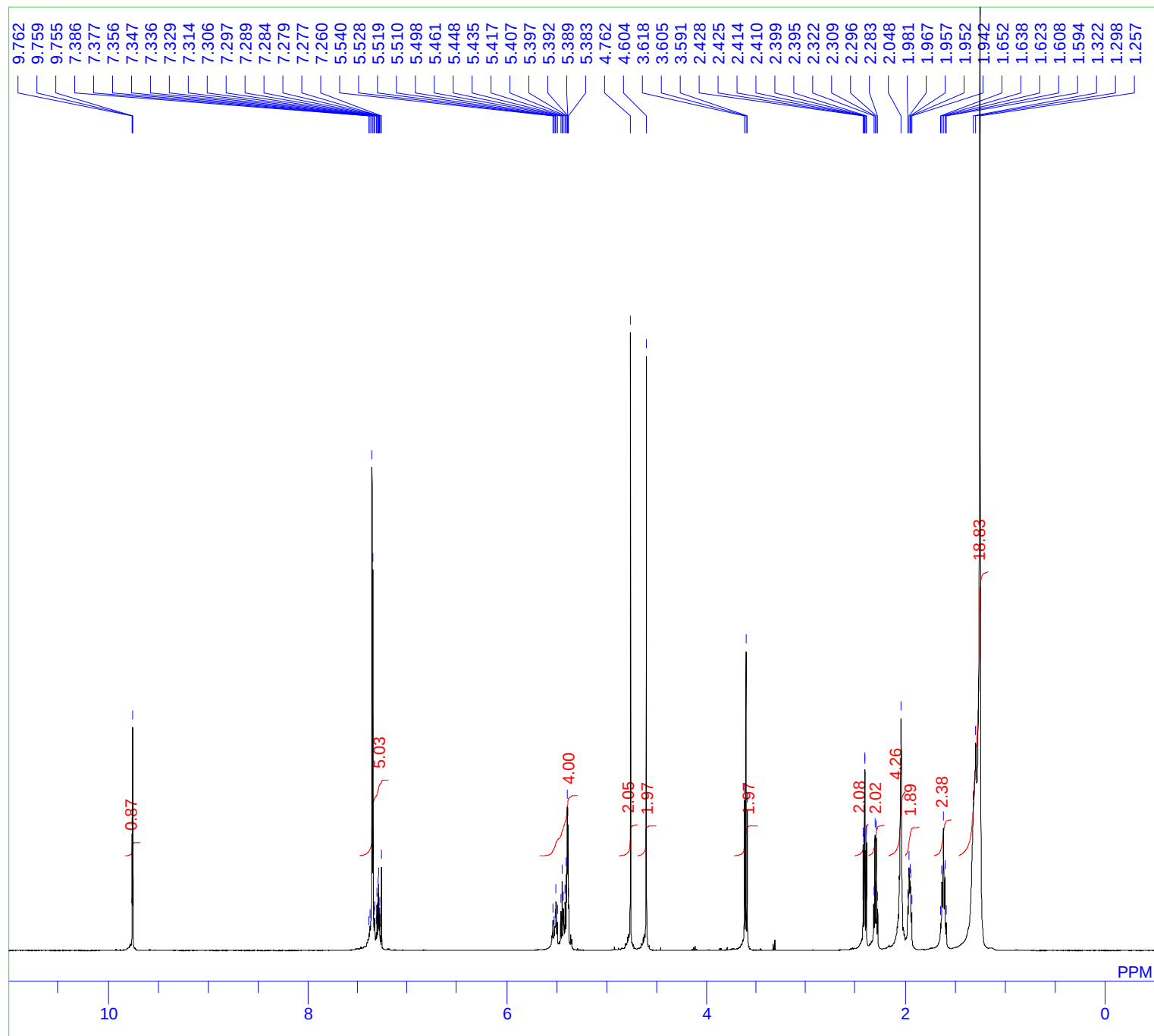


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 POINT 32768
 FREQU 39308.18 Hz
 SCANS 530
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 PD 2.0000 sec
 PW1 3.74 usec
 IRNUC 1H
 CTEMP 23.1 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.25 Hz
 RGAIN 24

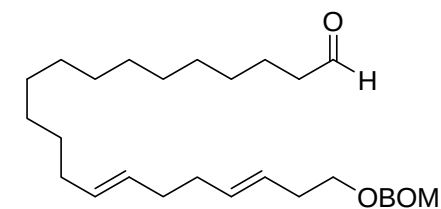


(14*E*,18*E*)-21-Benzyloxymethoxyhenicosa-14,18-dien-1-ol (10)

single_pulse

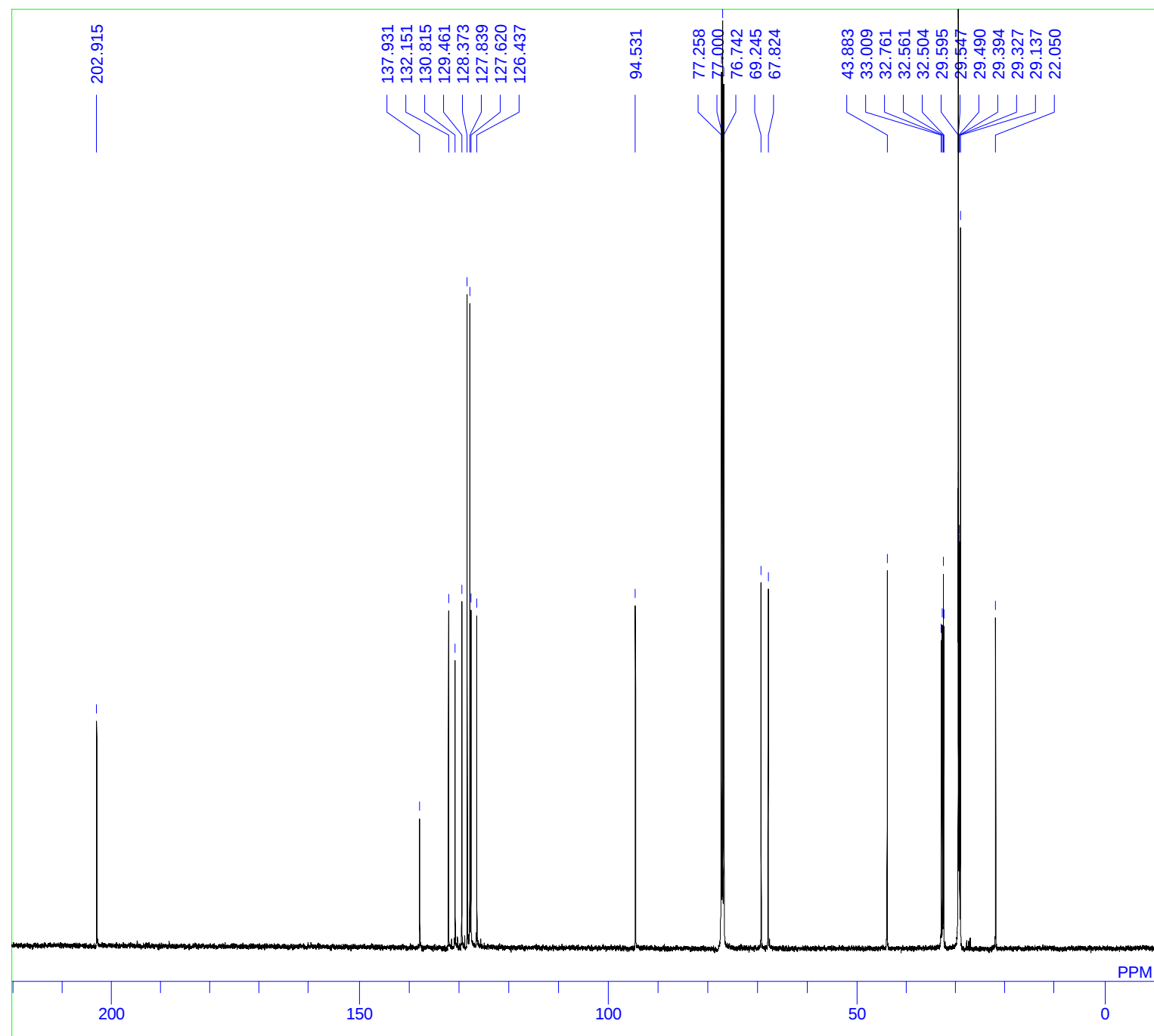


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 EXMOD proton.jxp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16384
 FREQU 9384.38 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 5.0000 sec
 PW1 3.52 usec
 IRNUC 1H
 CTEMP 23.4 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.25 Hz
 RGAIN 24

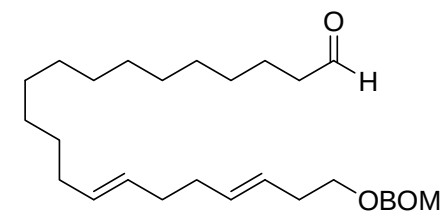


(14*E*,18*E*)-21-Benzyloxymethoxyhenicosa-14,18-dienal (**6**)

single pulse decoupled gated NOE

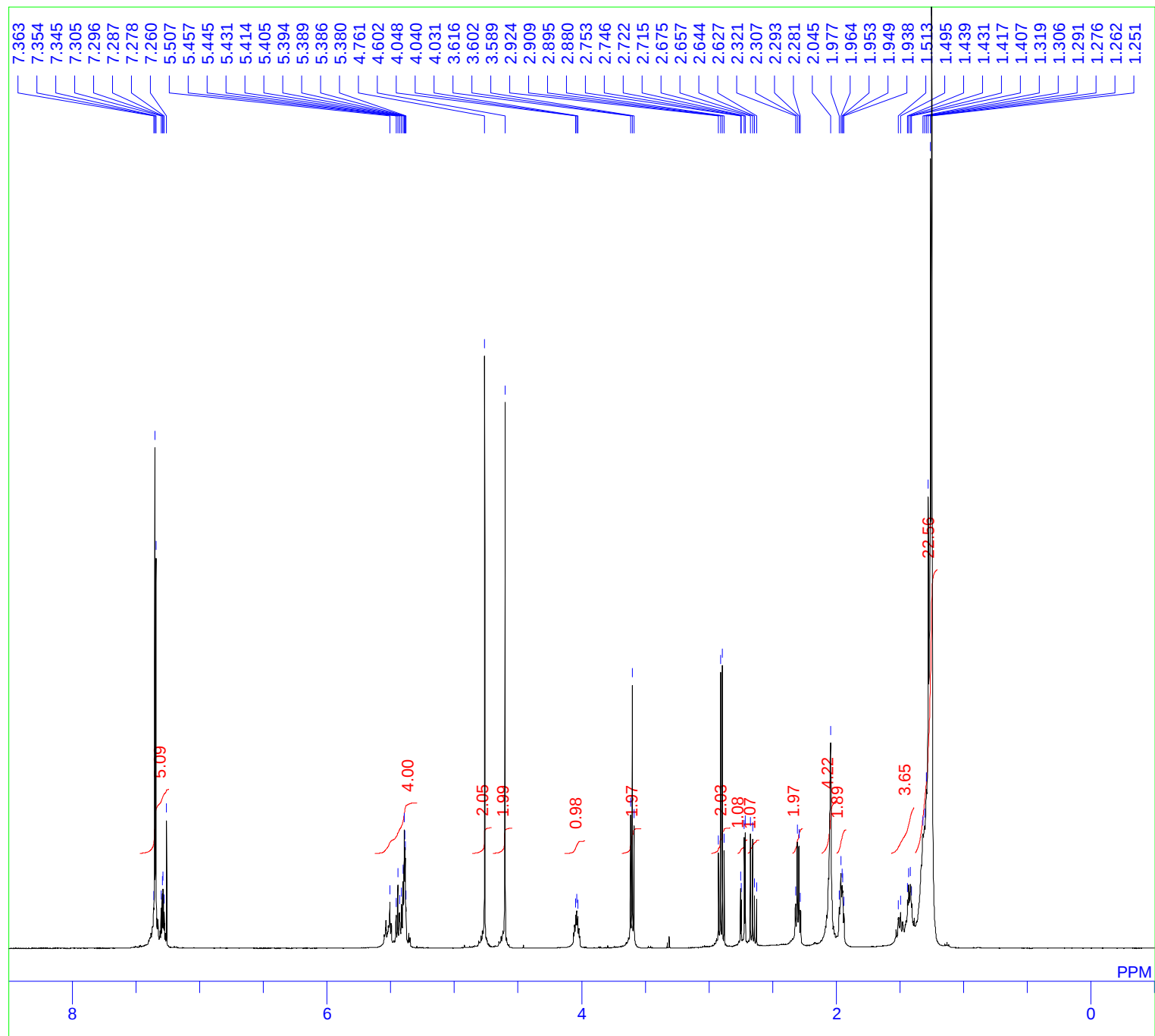


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 EXMOD carbon.jxp
 OBFRQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 39308.18 Hz
 SCANS 4000
 ACQTM 0.8336 sec
 PD 2.0000 sec
 PW1 3.74 usec
 IRNUC 1H
 CTEMP 23.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.25 Hz
 RGAIN 24

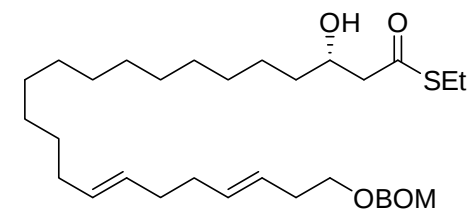


(14*E*,18*E*)-21-Benzyloxymethoxyhenicosa-14,18-dienal (6)

single_pulse

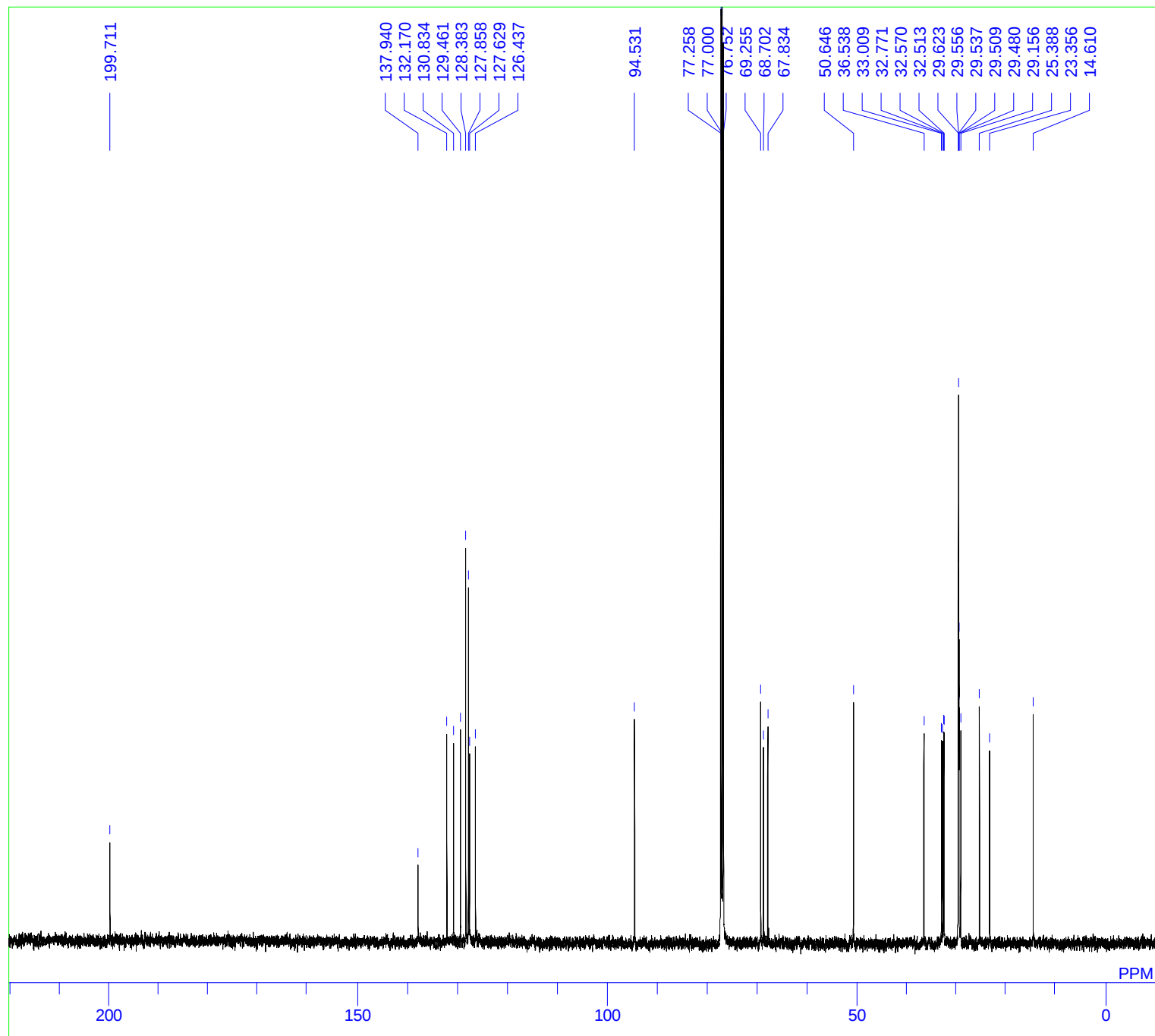


DFILE IT-3-035_Proton-1-2.als
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 DATIM 2016-09-08 14:43:40
 OBNUC 1H
 EXMOD proton.jpg
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16384
 FREQU 9384.38 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 5.0000 sec
 PW1 3.52 usec
 IRNUC 1H
 CTEMP 23.4 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.25 Hz
 RGAIN 26

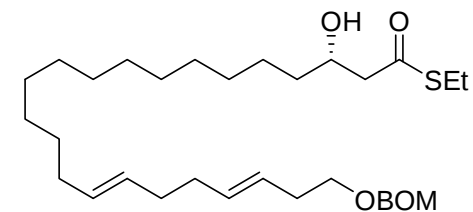


(3S,16E,20E)-Ethyl 23-benzyloxymethoxy-3-hydroxytricos-16,20-dienethioate (11)

single pulse decoupled gated NOE

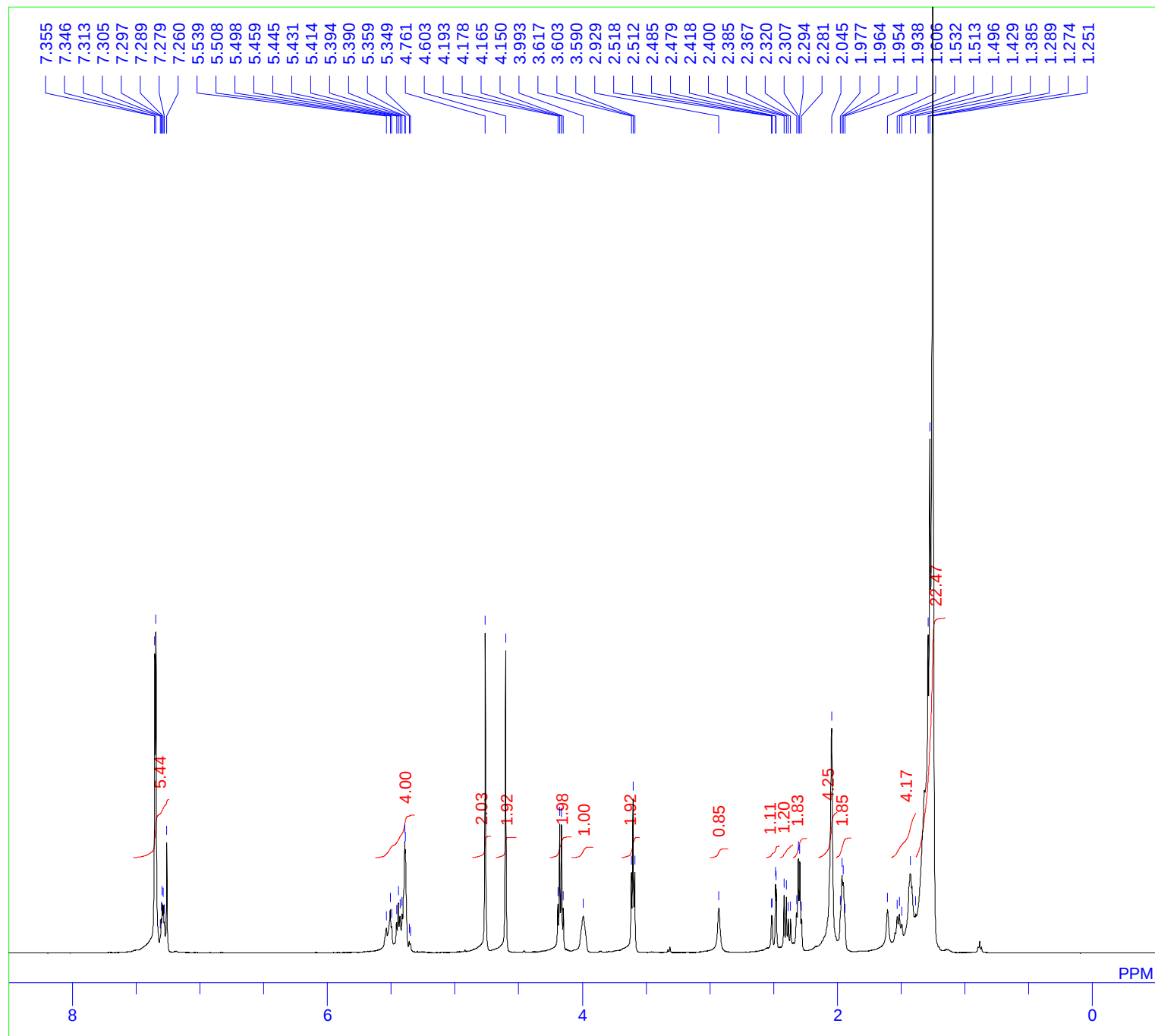


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 EXMOD carbon.jxp
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 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 39308.18 Hz
 SCANS 535
 ACQTM 0.8336 sec
 PD 2.0000 sec
 PW1 3.74 usec
 IRNUC 1H
 CTEMP 23.4 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.25 Hz
 RGAIN 24

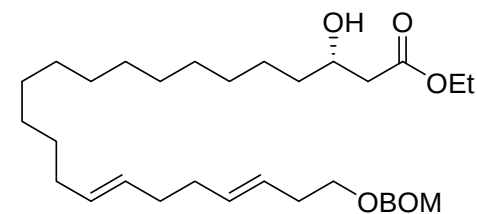


(3*S*,16*E*,20*E*)-Ethyl 23-benzyloxymethoxy-3-hydroxytricos-16,20-dienethioate (**11**)

single_pulse

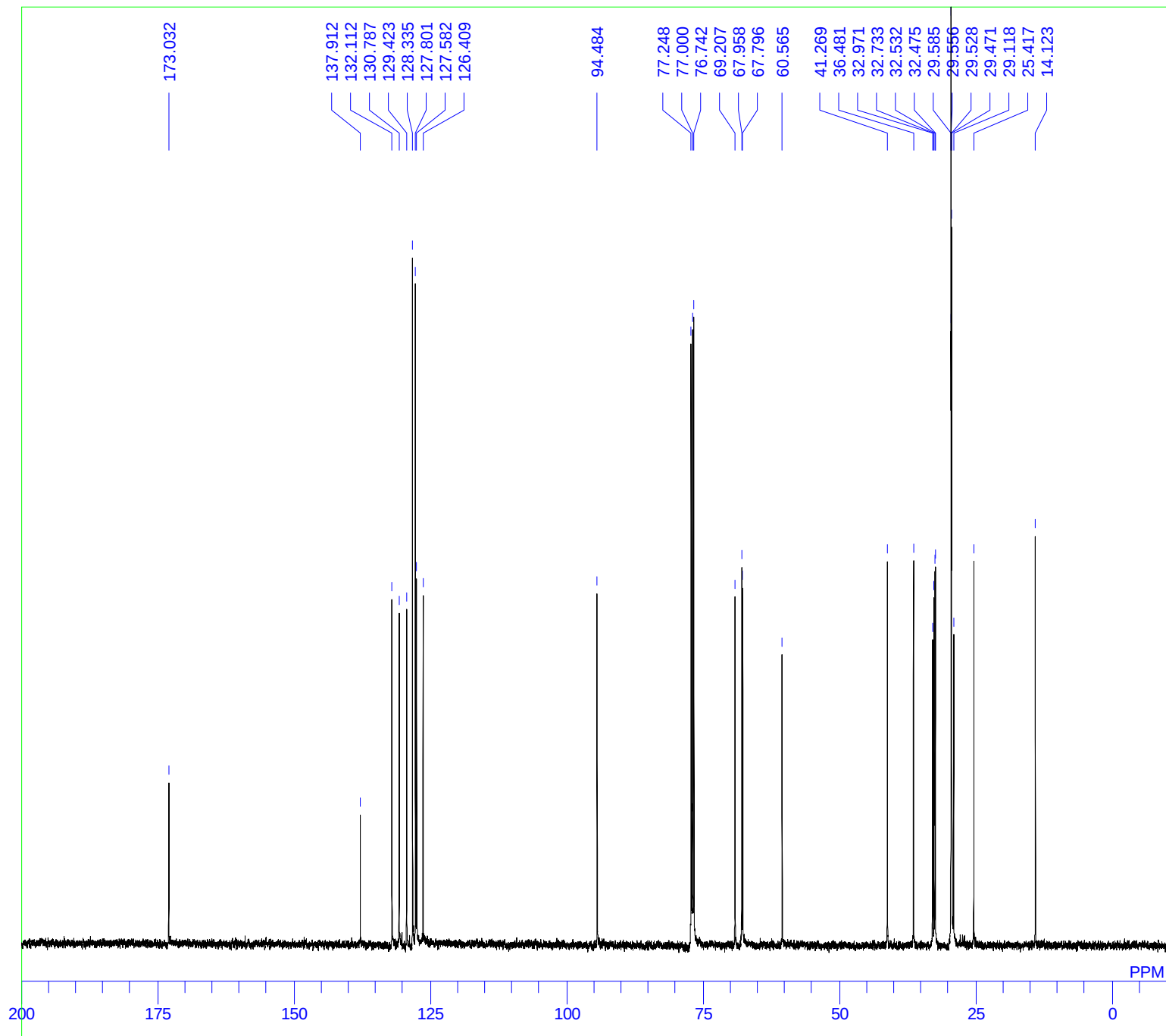


DFILE IT-3-040_Proton-1-2.als
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 DATIM 2016-09-13 11:06:38
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 EXMOD proton.jxp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16384
 FREQU 9384.38 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 5.0000 sec
 PW1 3.52 usec
 IRNUC 1H
 CTEMP 23.2 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.25 Hz
 RGAIN 30

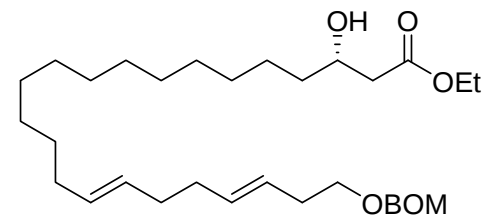


(3*S*,16*E*,20*E*)-Ethyl 23-benzyloxymethoxy-3-hydroxytricos-16,20-dienoate (**12**)

single pulse decoupled gated NOE

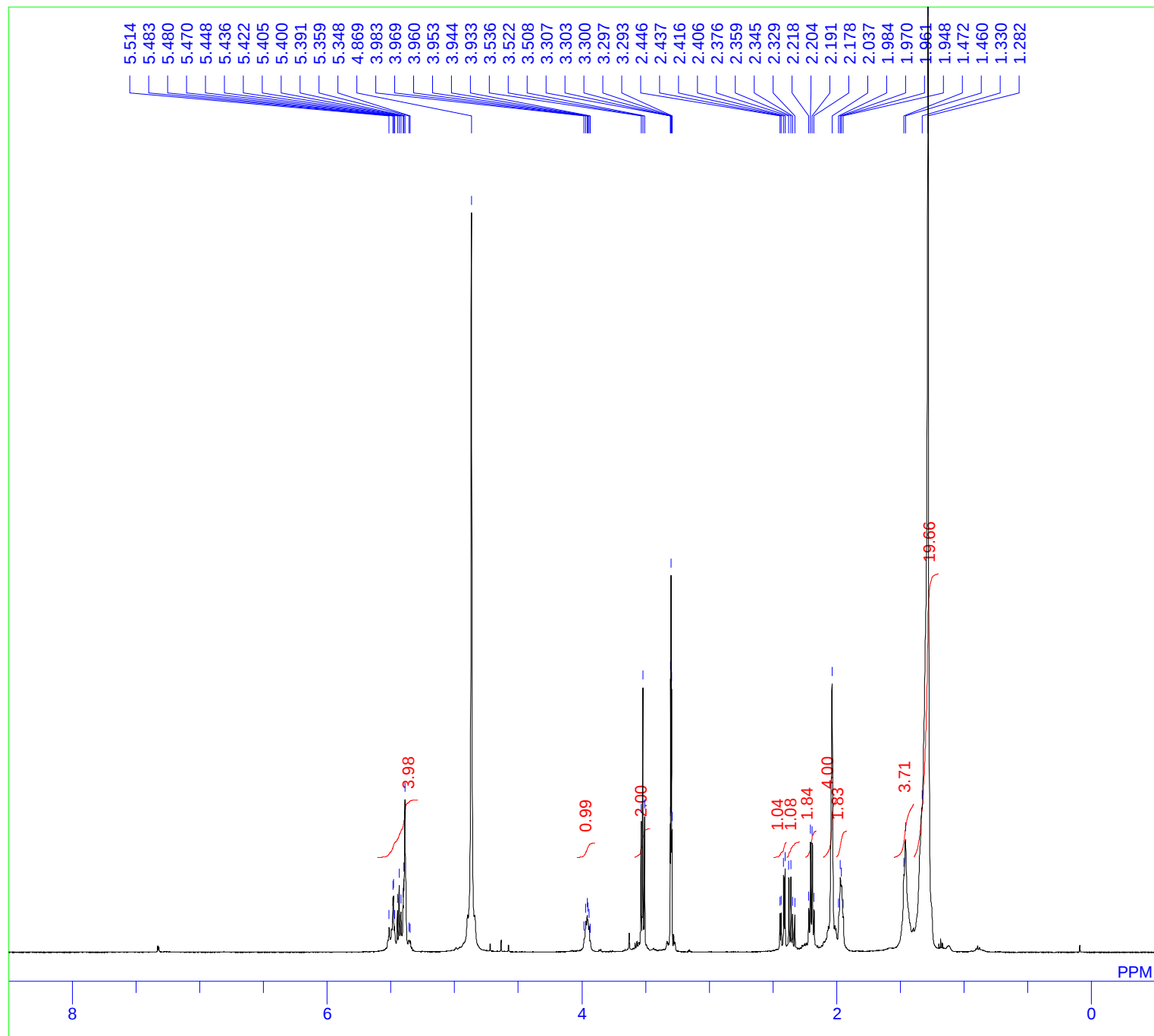


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 EXMOD carbon.jxp
 OBFRQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 39308.18 Hz
 SCANS 542
 ACQTM 0.8336 sec
 PD 2.0000 sec
 PW1 3.74 usec
 IRNUC 1H
 CTEMP 24.5 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.25 Hz
 RGAIN 26

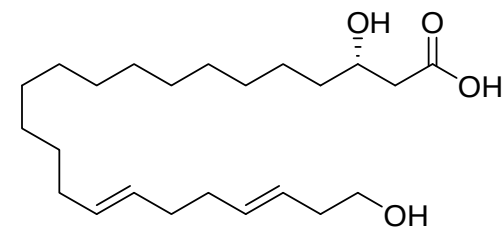


(3*S*,16*E*,20*E*)-Ethyl 23-benzyloxymethoxy-3-hydroxytricos-16,20-dienoate (**12**)

single_pulse

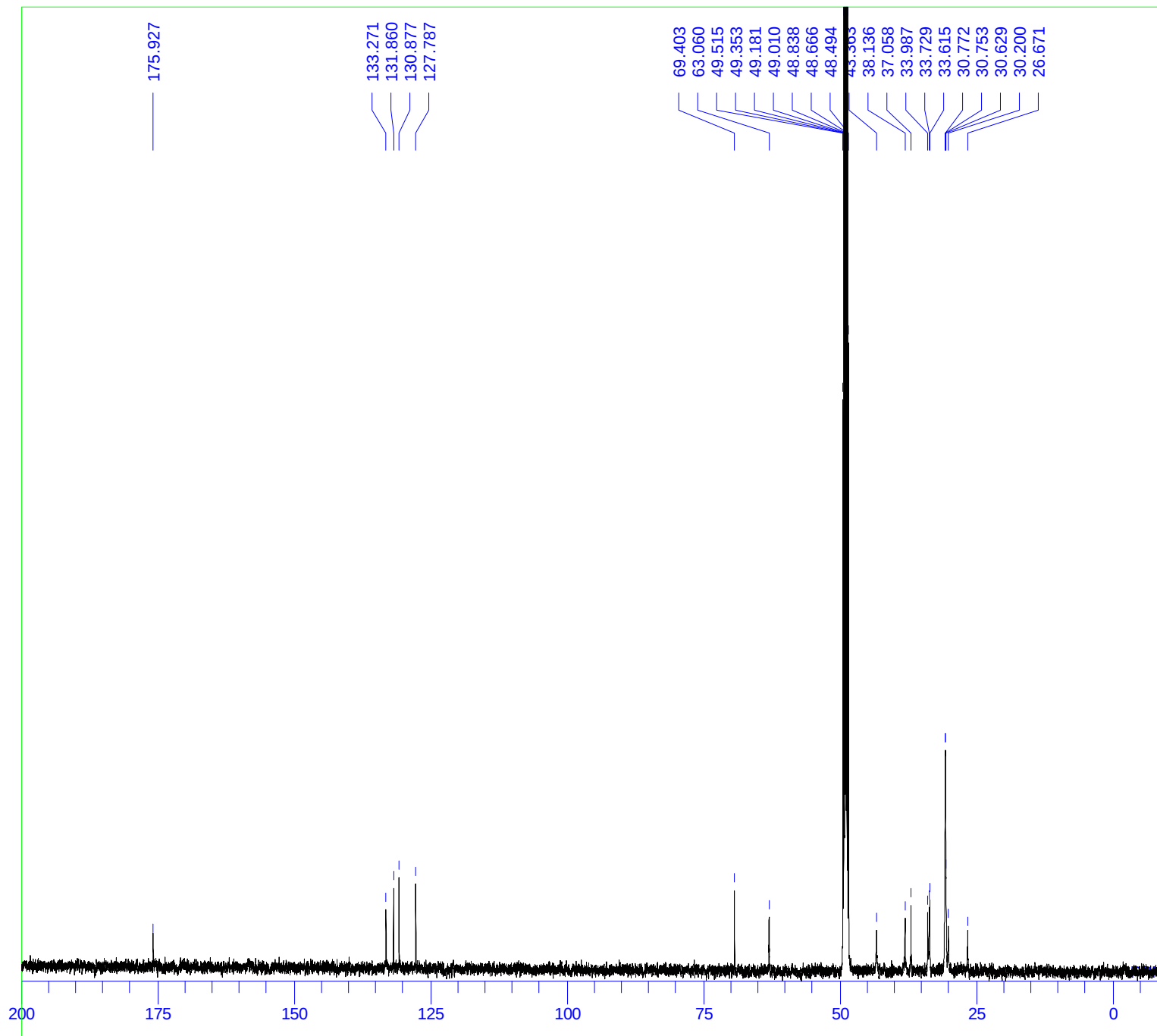


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 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16384
 FREQU 9384.38 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 5.0000 sec
 PW1 3.52 usec
 IRNUC 1H
 CTEMP 23.3 c
 SLVNT CD3OD
 EXREF 3.30 ppm
 BF 0.25 Hz
 RGAIN 42

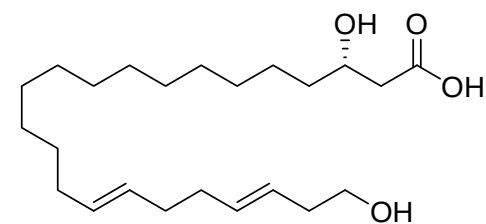


(3S,16E,20E)-3,23-Dihydroxytricos-16,20-dienoic acid (**5**)

single pulse decoupled gated NOE

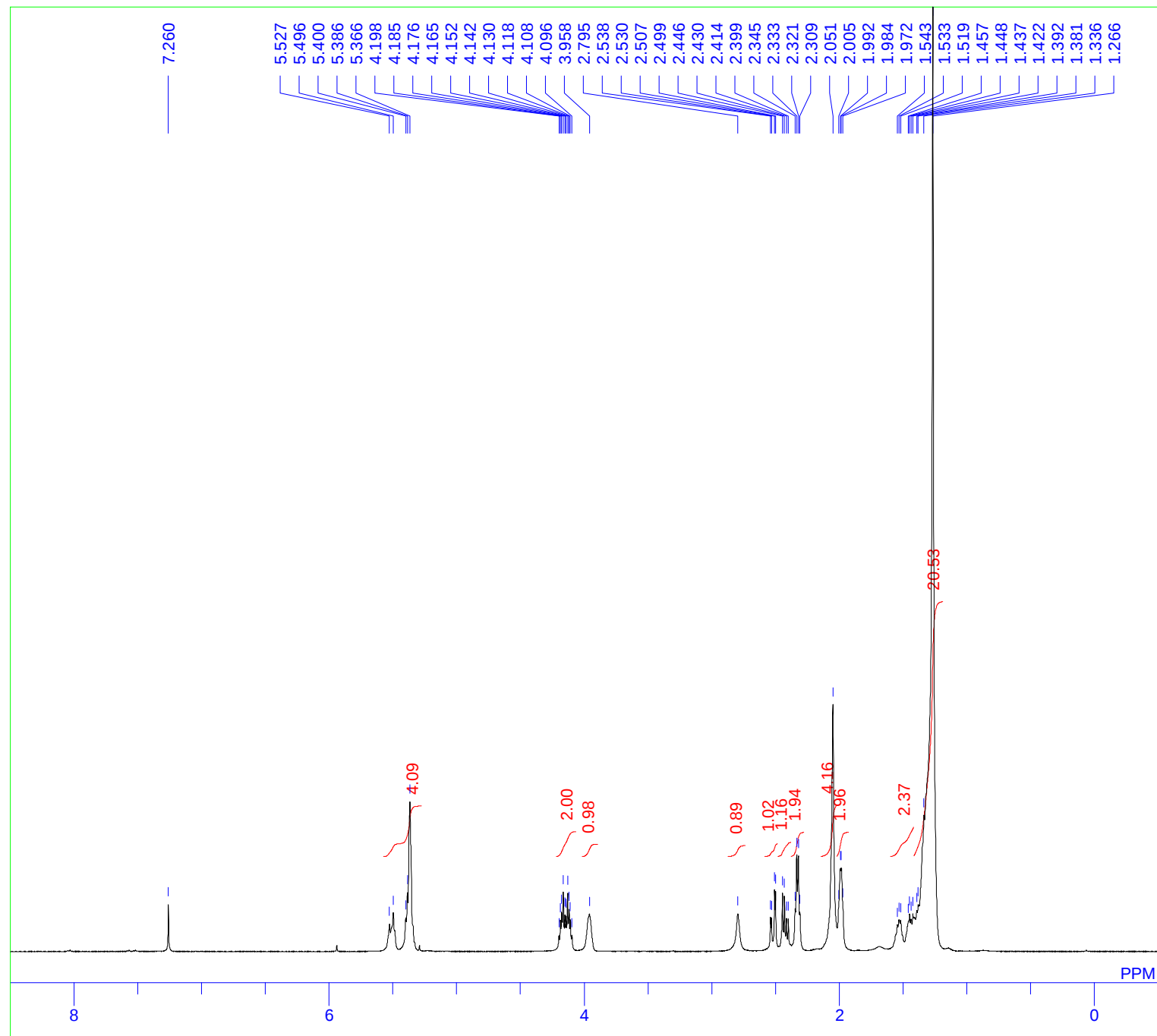


DFILE IT-3-062_Carbon-1-2.als
 COMNT single pulse decoupled gated NOE
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 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 39308.18 Hz
 SCANS 742
 ACQTM 0.8336 sec
 PD 2.0000 sec
 PW1 3.74 usec
 IRNUC 1H
 CTEMP 23.2 c
 SLVNT CD3OD
 EXREF 49.00 ppm
 BF 0.25 Hz
 RGAIN 22

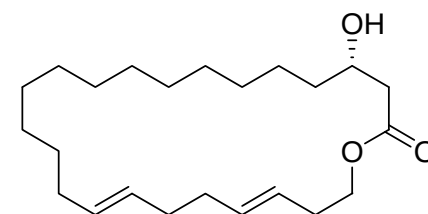


(3*S*,16*E*,20*E*)-3,23-Dihydroxytricos-16,20-dienoic acid (**5**)

single_pulse

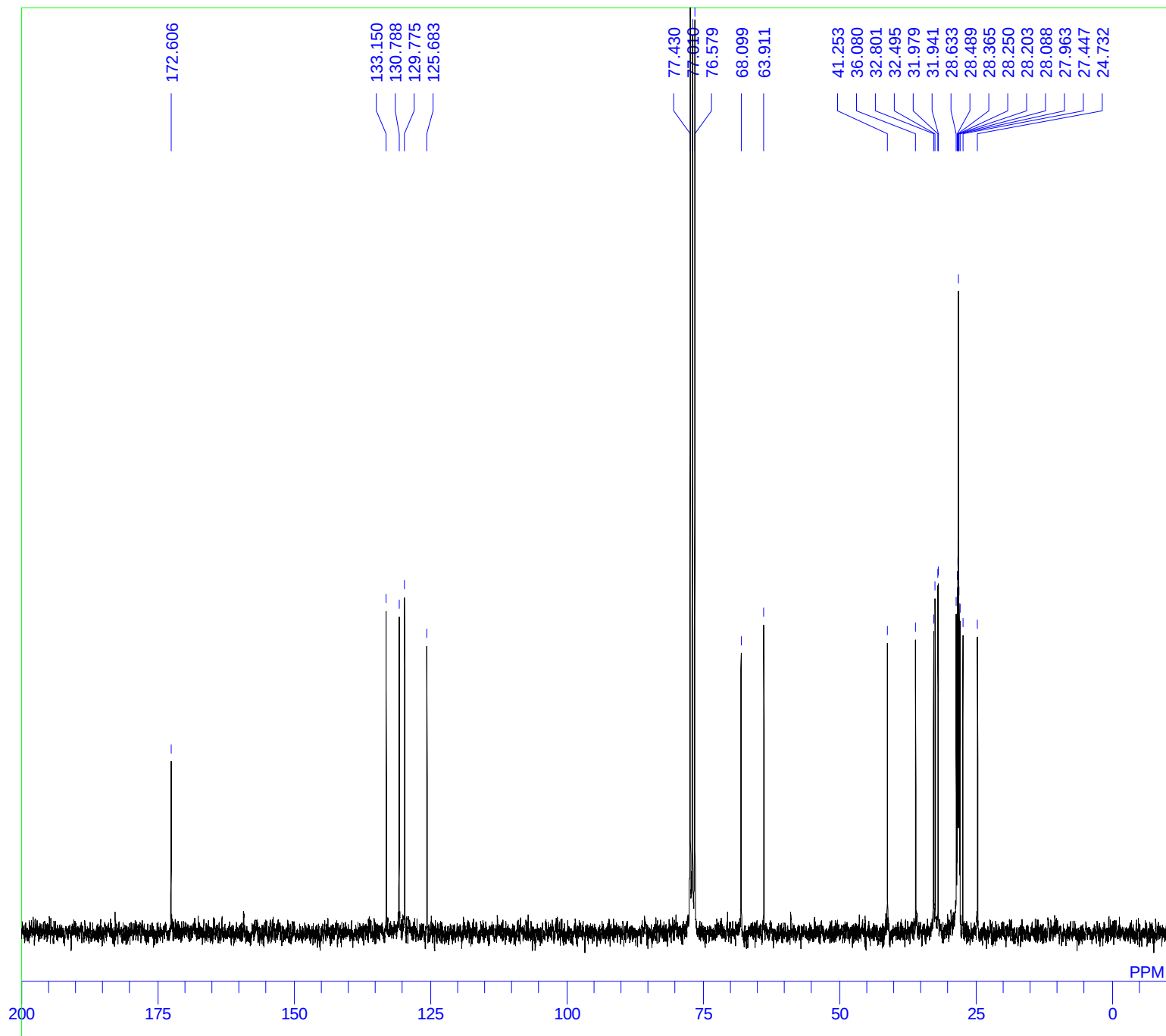


DFILE IT-3-064_Proton-1-2.als
 COMNT single_pulse
 DATIM 2016-10-01 14:25:22
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16384
 FREQU 9384.38 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 5.0000 sec
 PW1 3.52 usec
 IRNUC 1H
 CTEMP 23.0 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.25 Hz
 RGAIN 46

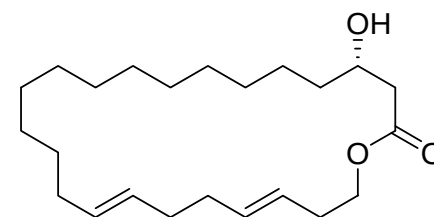


(3*S*,16*E*,20*E*)-3-Hydroxytricosa-16,20-dienolide (**4**)

single pulse decoupled gated NOE

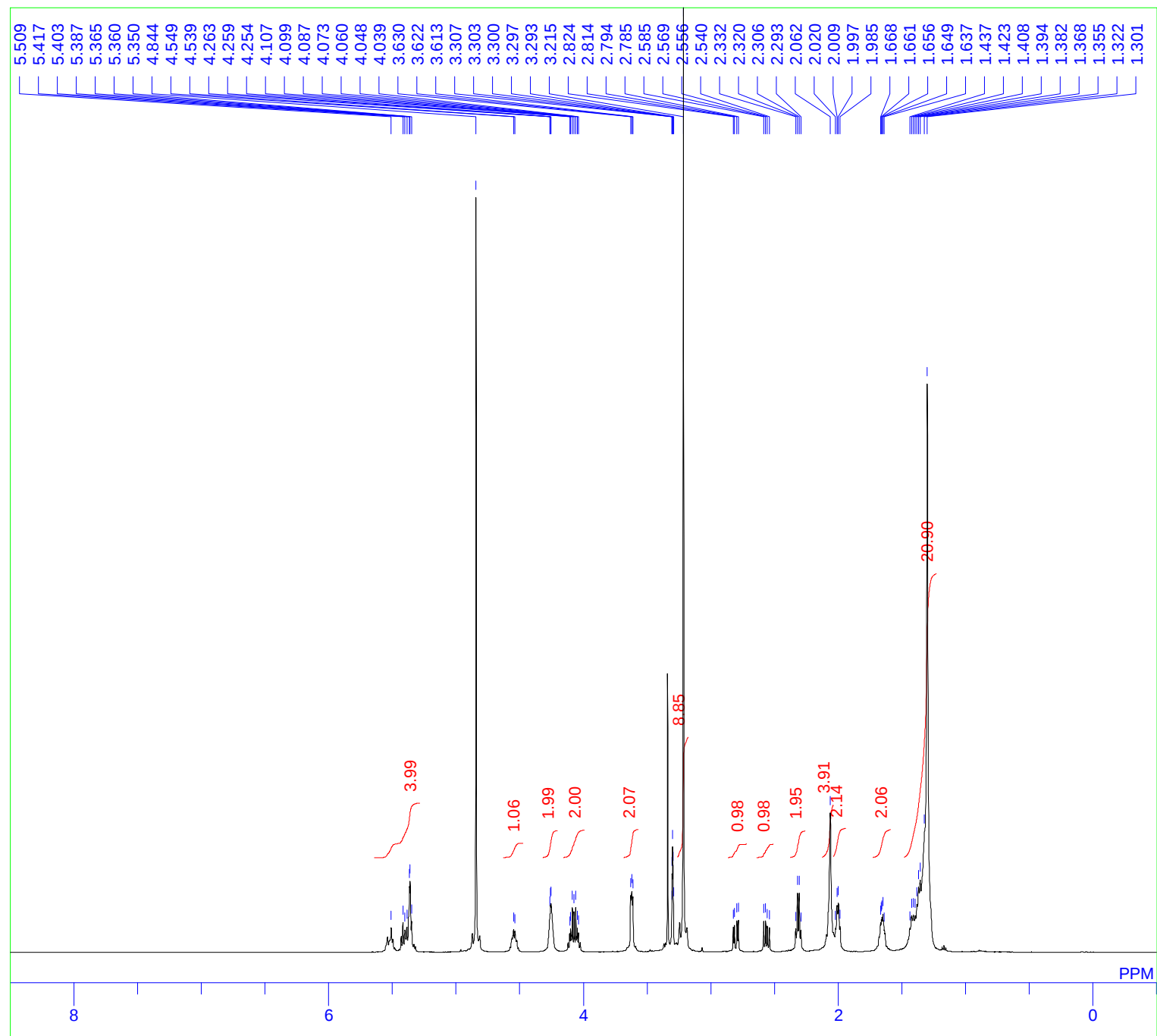


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 OBNUC 13C
 EXMOD carbon.jxp
 OBFRQ 75.57 MHz
 OBSET 5.79 KHz
 OBFIN 1.08 Hz
 POINT 32768
 FREQU 23674.24 Hz
 SCANS 406
 ACQTM 1.3841 sec
 PD 2.0000 sec
 PW1 3.27 usec
 IRNUC 1H
 CTEMP 22.8 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 0.25 Hz
 RGAIN 50

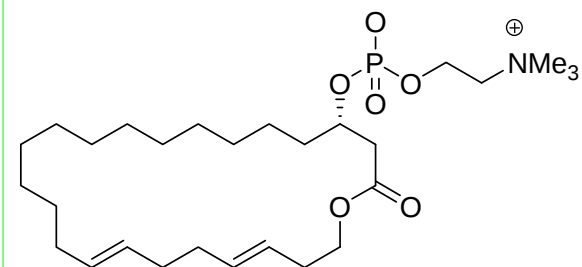


(3*S*,16*E*,20*E*)-3-Hydroxytricosal-16,20-dienolide (**4**)

single_pulse

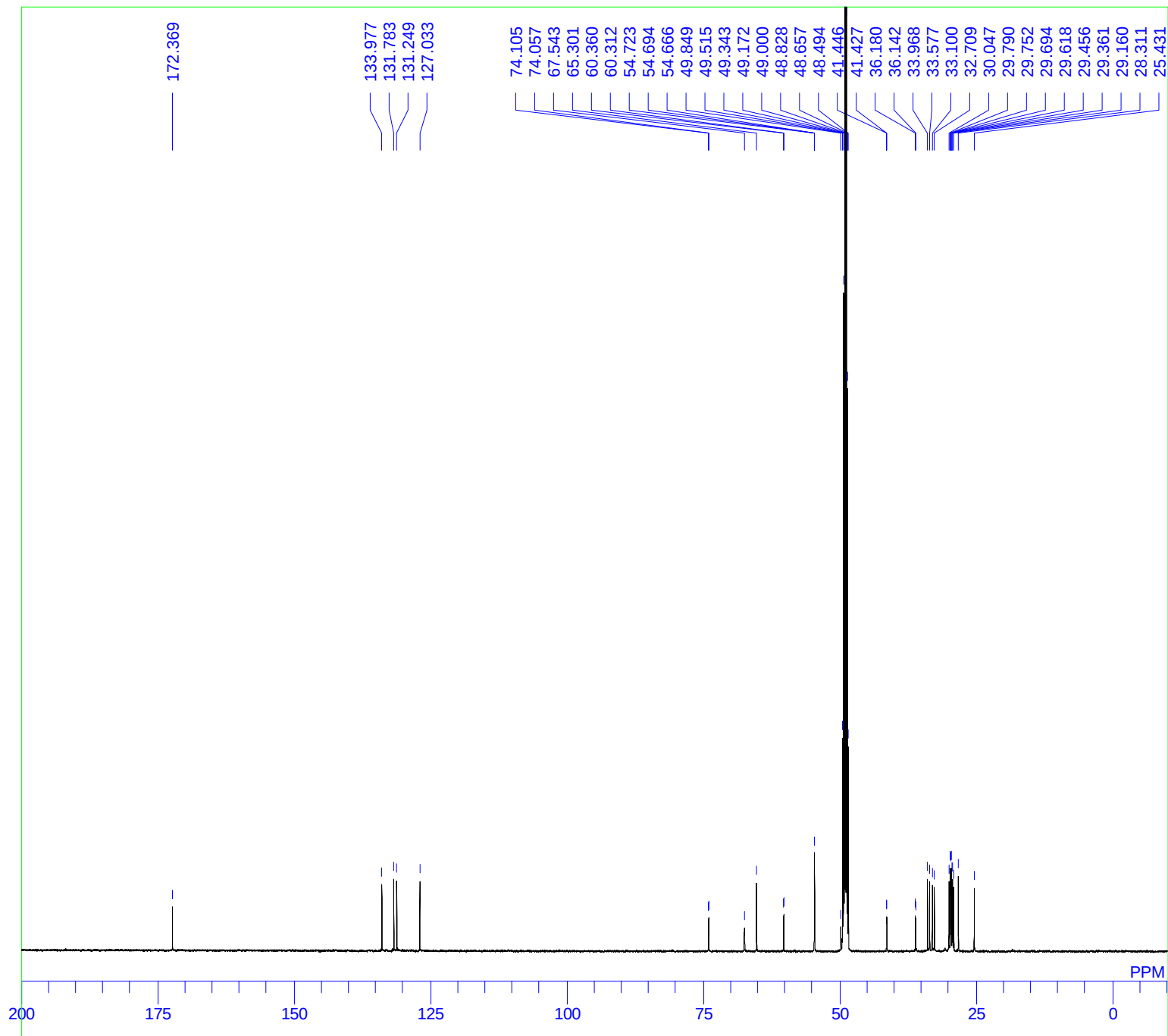


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 EXMOD proton.jpg
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16384
 FREQU 9384.38 Hz
 SCANS 16
 ACQTM 1.7459 sec
 PD 5.0000 sec
 PW1 3.52 usec
 IRNUC 1H
 CTEMP 25.0 c
 SLVNT CD3OD
 EXREF 3.30 ppm
 BF 0.25 Hz
 RGAIN 30



23-Demethyleushearilide (3)

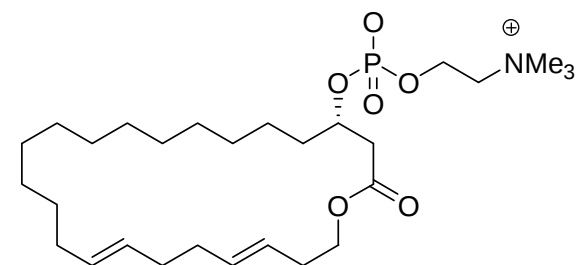
single pulse decoupled gated NOE



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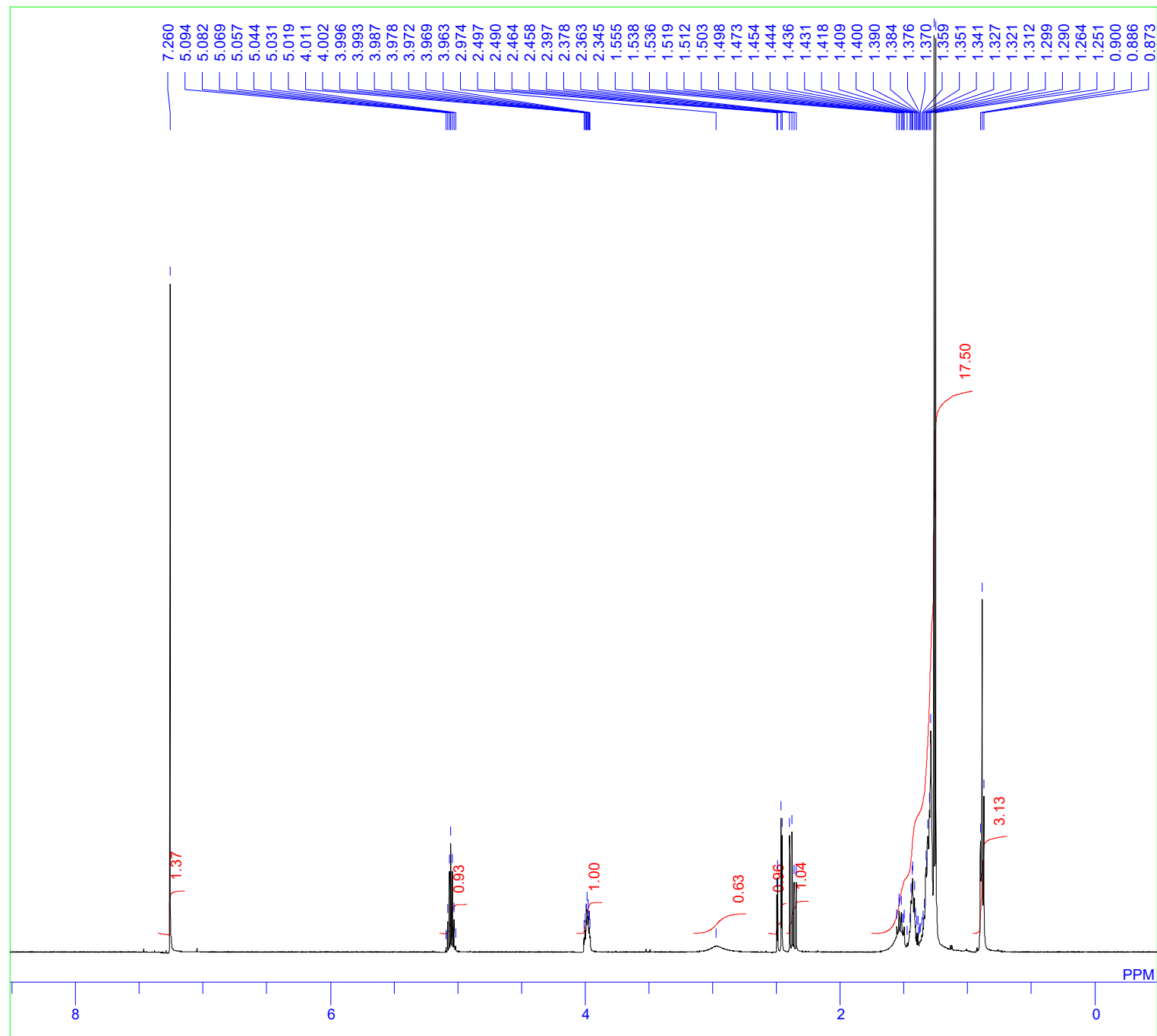
23:43:17 FILE          IT-3-066_Carbon-1-2.als
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OBNUC        13C
EXMOD        carbon.jxp
OBFREQ       125.77 MHz
OBSET        7.87 KHz
OBFIN        4.21 Hz
POINT        32768
FREQU        39308.18 Hz
SCANS        6014
ACQTM        0.8336 sec
PD           2.0000 sec
PW1          3.74 usec
IRNUC        1H
CTEMP        25.0 c
SLVNT        CD3OD
EXREF        49.00 ppm
BF           0.25 Hz
RGAIN        26

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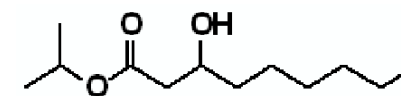


23-Demethyleushearilide (3)

single_pulse

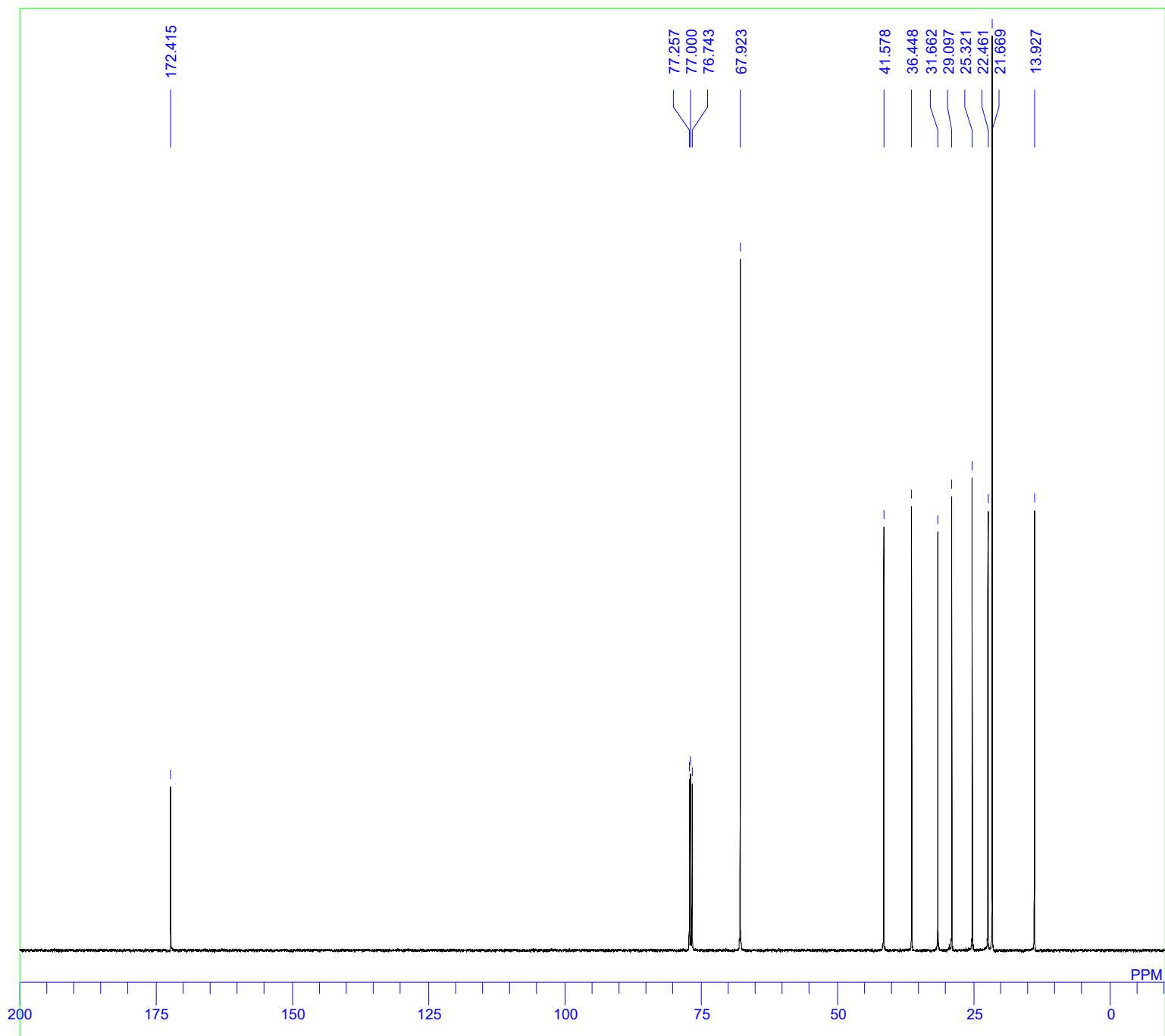


DFILE MA-1-139-Aldol__Proton-1-4.als
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EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13120
FREQU 7507.51 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 3.84 usec
IRNUC 1H
CTEMP 24.1 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 44

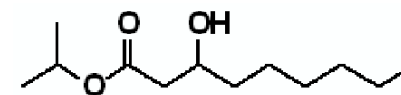


Isopropyl 3-hydroxynonananoate

single pulse decoupled gated NOE

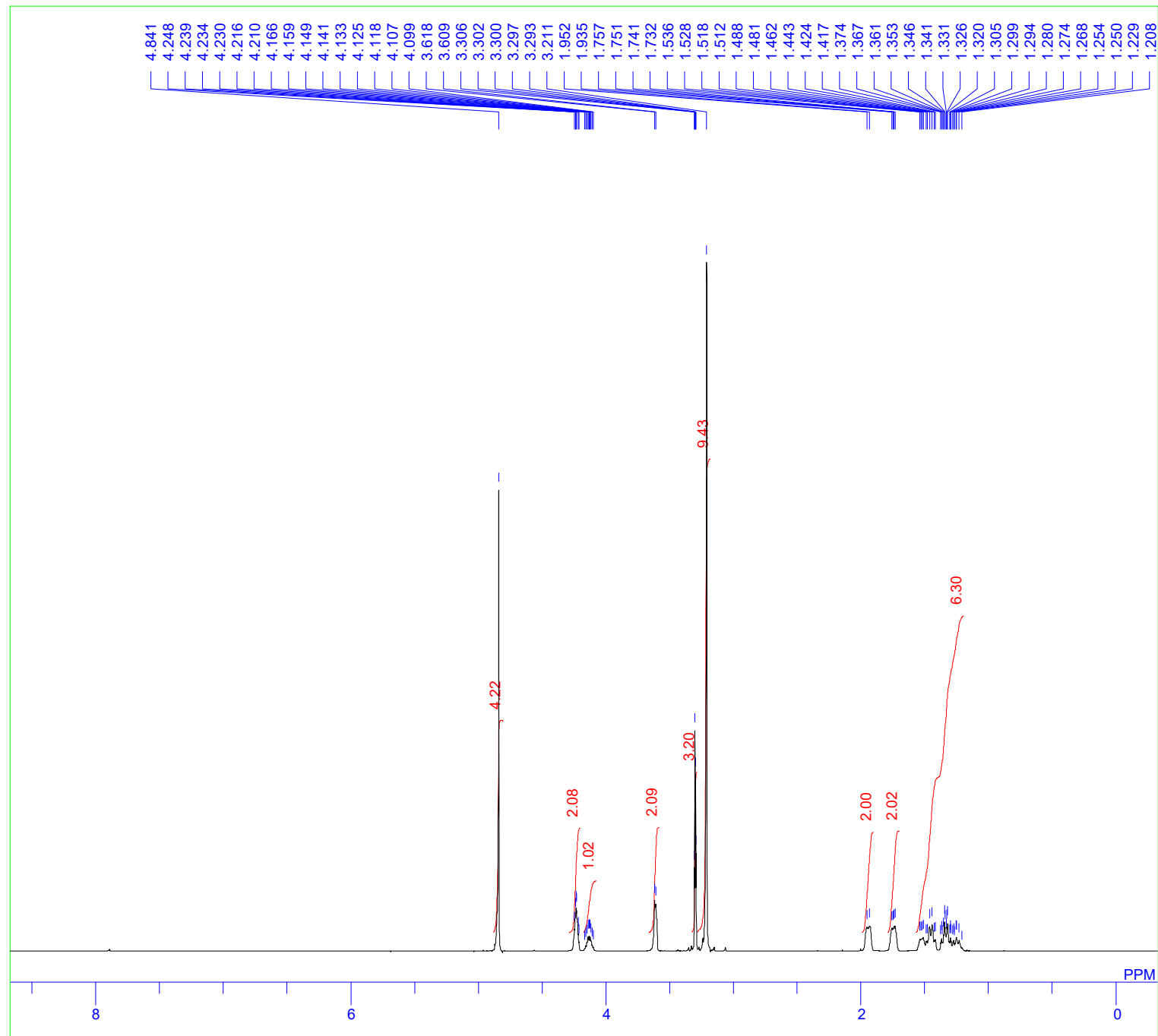


DFILE ts-1-034-TM_Carbon-2-3.als
COMNT single pulse decoupled gated NOE
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OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26224
FREQU 31446.54 Hz
SCANS 512
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.58 usec
IRNUC 1H
CTEMP 24.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 18

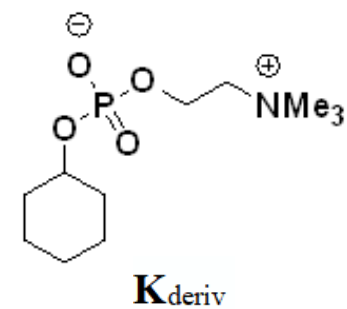


Isopropyl 3-hydroxynonanoate

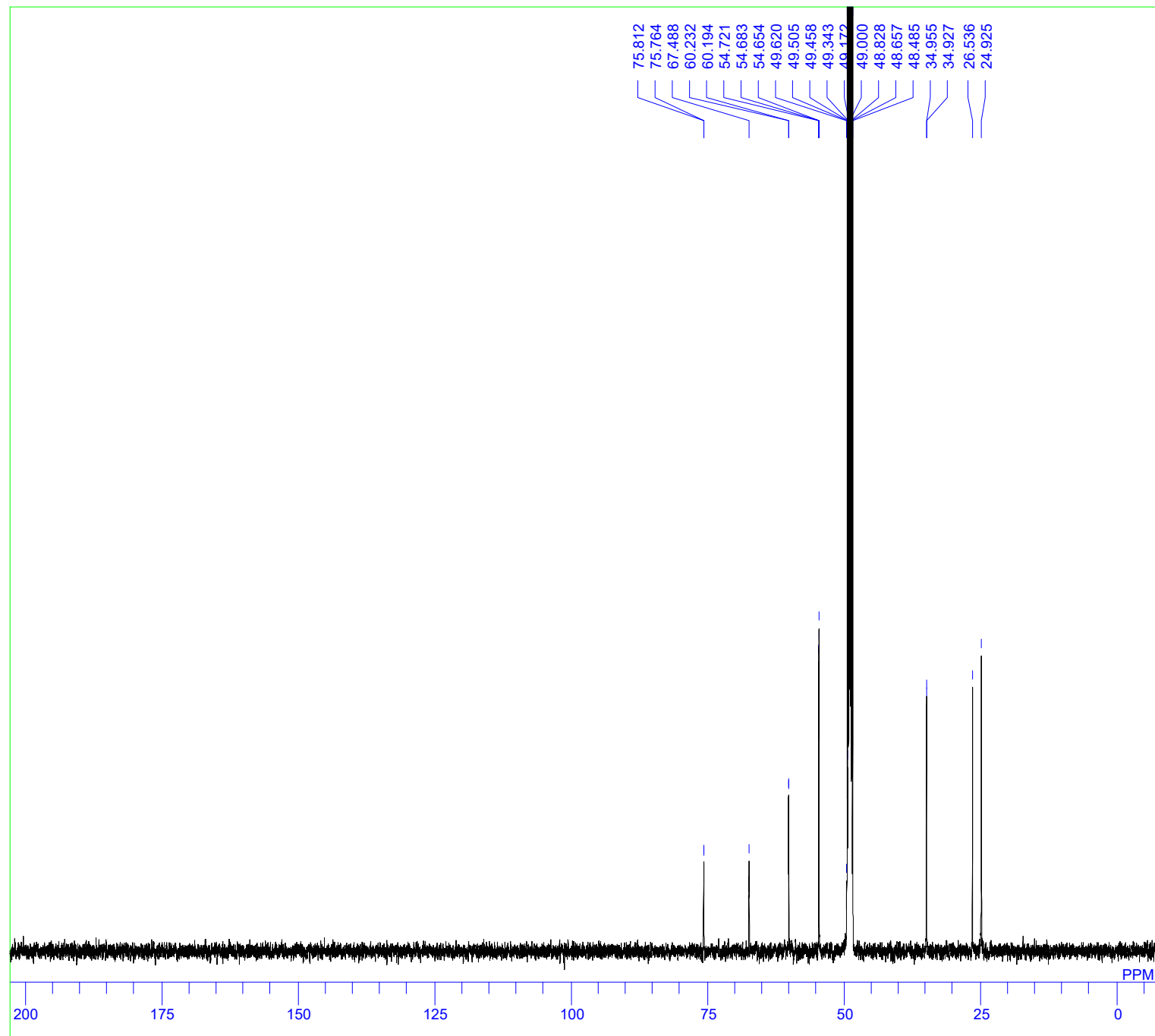
single_pulse



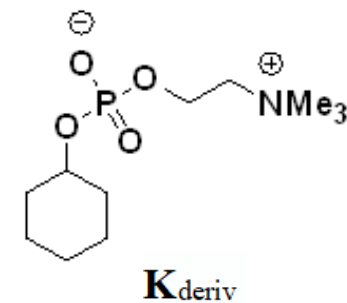
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 COMNT single_pulse
 DATIM 2017-08-01 18:59:30
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16384
 FREQU 9384.38 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 5.0000 sec
 PW1 3.45 usec
 IRNUC 1H
 CTEMP 24.6 c
 SLVNT CD3OD
 EXREF 3.30 ppm
 BF 0.12 Hz
 RGAIN 38



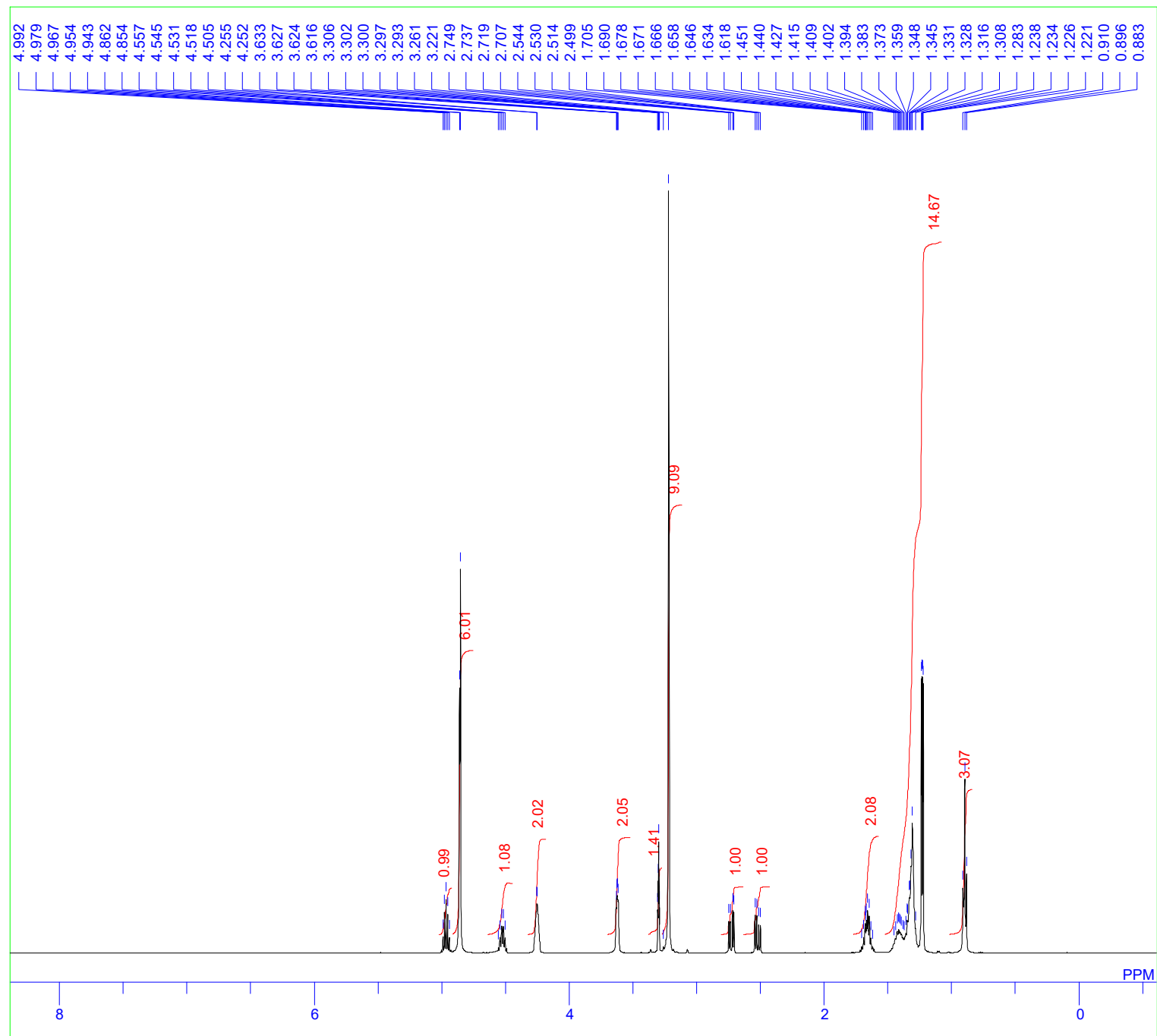
single pulse decoupled gated NOE



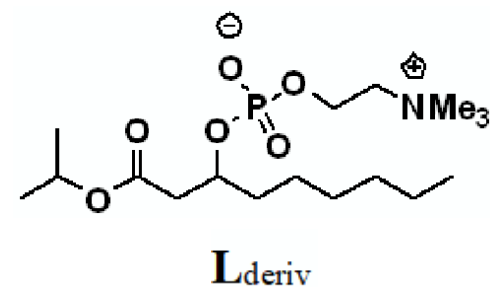
DFILE MA-1-137-P1_Carbon-1-6.als
COMNT single pulse decoupled gated NOE
DATIM 2019-05-31 19:10:29
OBNUC ¹³C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32780
FREQU 39308.18 Hz
SCANS 2048
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.87 usec
IRNUC ¹H
CTEMP 24.1 c
SLVNT CD3OD
EXREF 49.00 ppm
BF 0.12 Hz
RGAIN 32



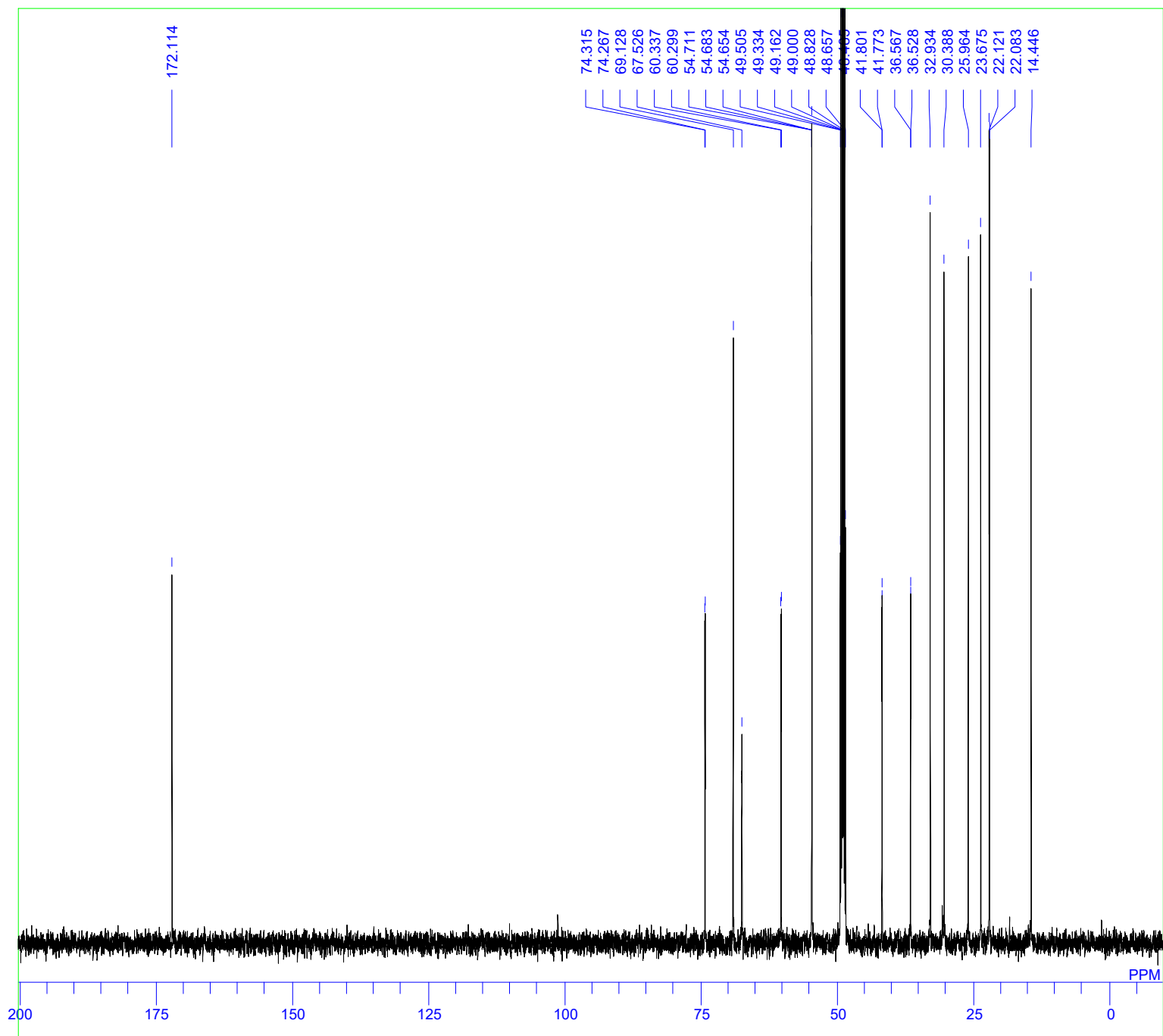
single_pulse



DFILE YN-24-TM-4_Proton-1-4.als
 COMNT single_pulse
 DATIM 2017-07-22 10:08:14
 OBNUC 1H
 EXMOD proton.jxp
 OBFRQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 13120
 FREQU 7507.51 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 5.0000 sec
 PW1 3.45 usec
 IRNUC 1H
 CTEMP 23.7 c
 SLVNT CD3OD
 EXREF 3.30 ppm
 BF 0.12 Hz
 RGAIN 28



single pulse decoupled gated NOE



DFILE ts-1-036-TM_Carbon-2-4.als
COMNT single pulse decoupled gated NOE
DATIM 2015-09-14 13:39:01
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32780
FREQU 39308.18 Hz
SCANS 363
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.58 usec
IRNUC 1H
CTEMP 24.3 c
SLVNT CD3OD
EXREF 49.00 ppm
BF 0.12 Hz
RGAIN 22

