Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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Electronic supplementary information (ESI)

Highly Stereoselective Directed Reactions and an Efficient Synthesis of Azafuranose from a Chiral Aziridine

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Table of Contents

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Std proton

File: xp

Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
Operator: LWK
VNMRS-400 "Varian-NMR"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.049 sec
 Width 6410.3 Hz
 8 repetitions
OBSERVE H1, 399.7193723 MHz
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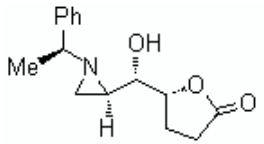
FT size 65536
Total time 0 min, 30 sec



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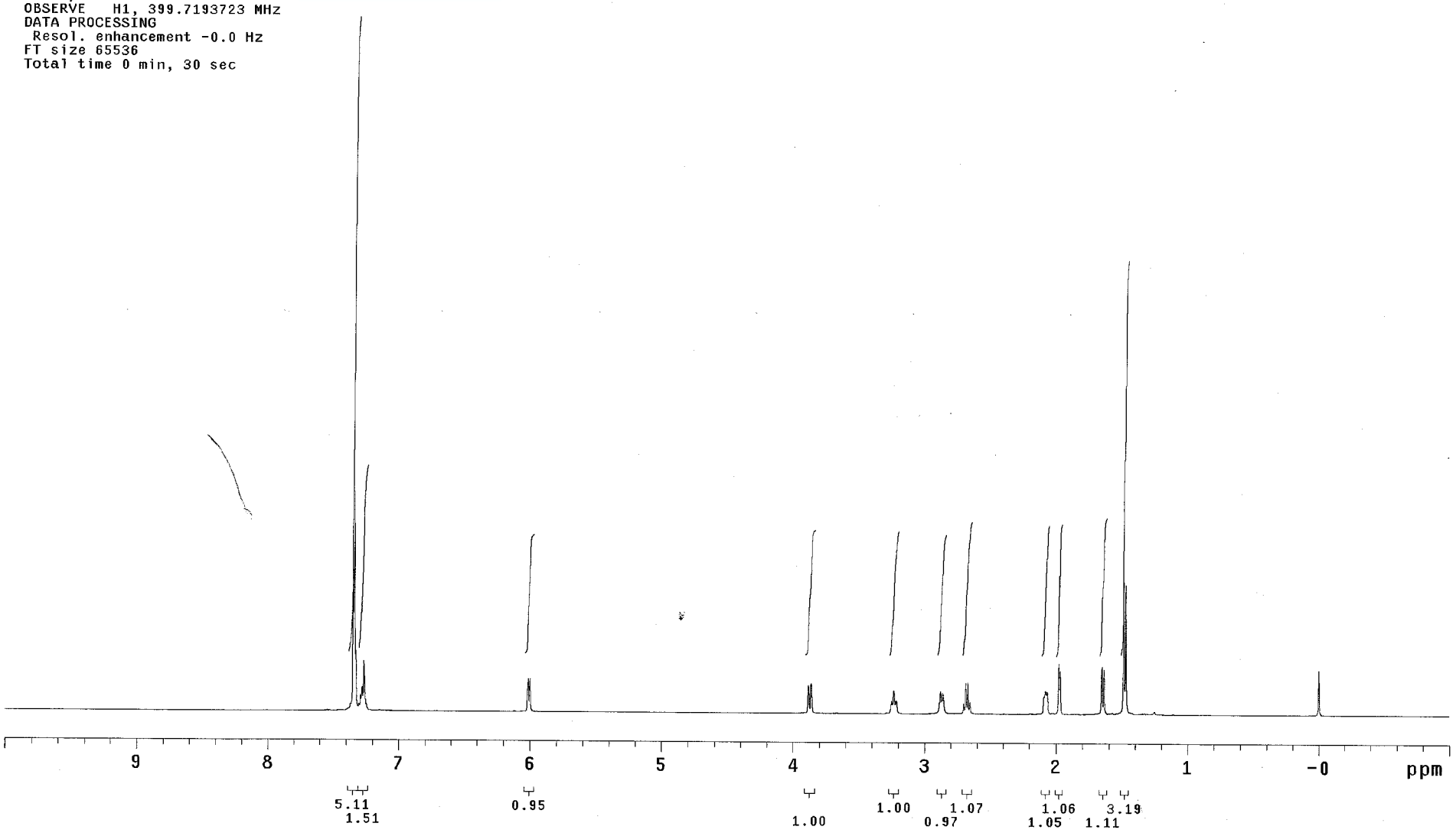

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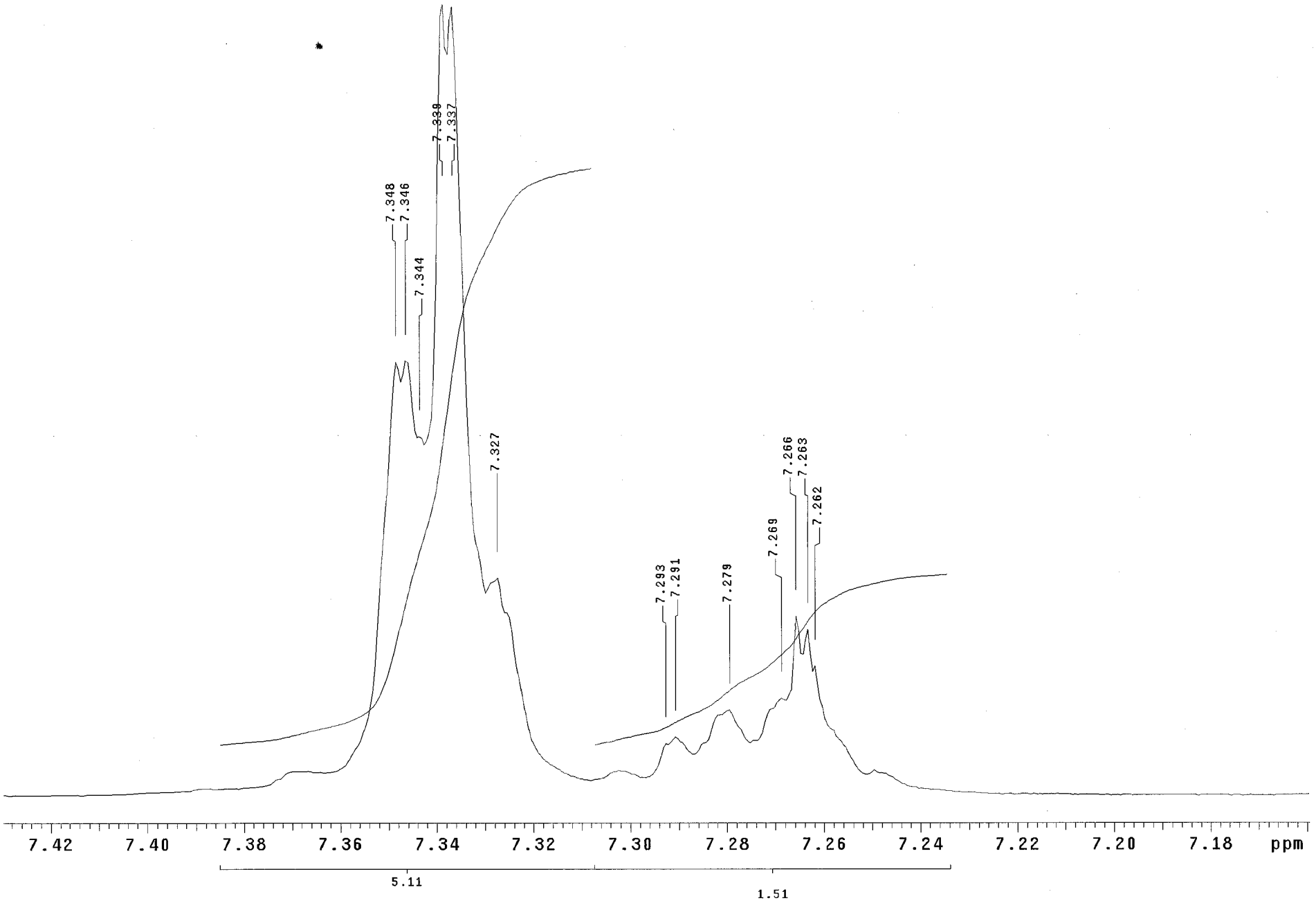

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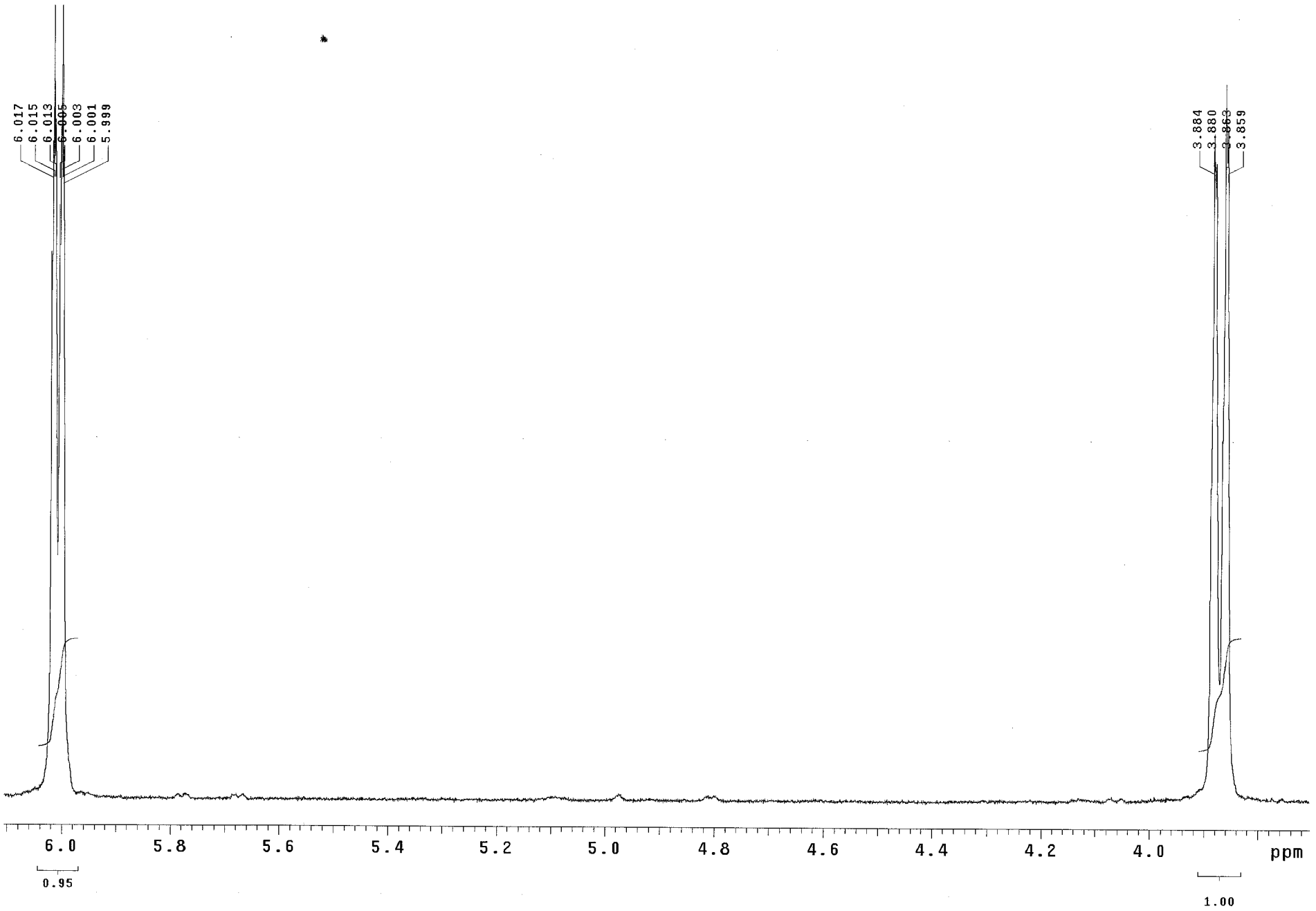

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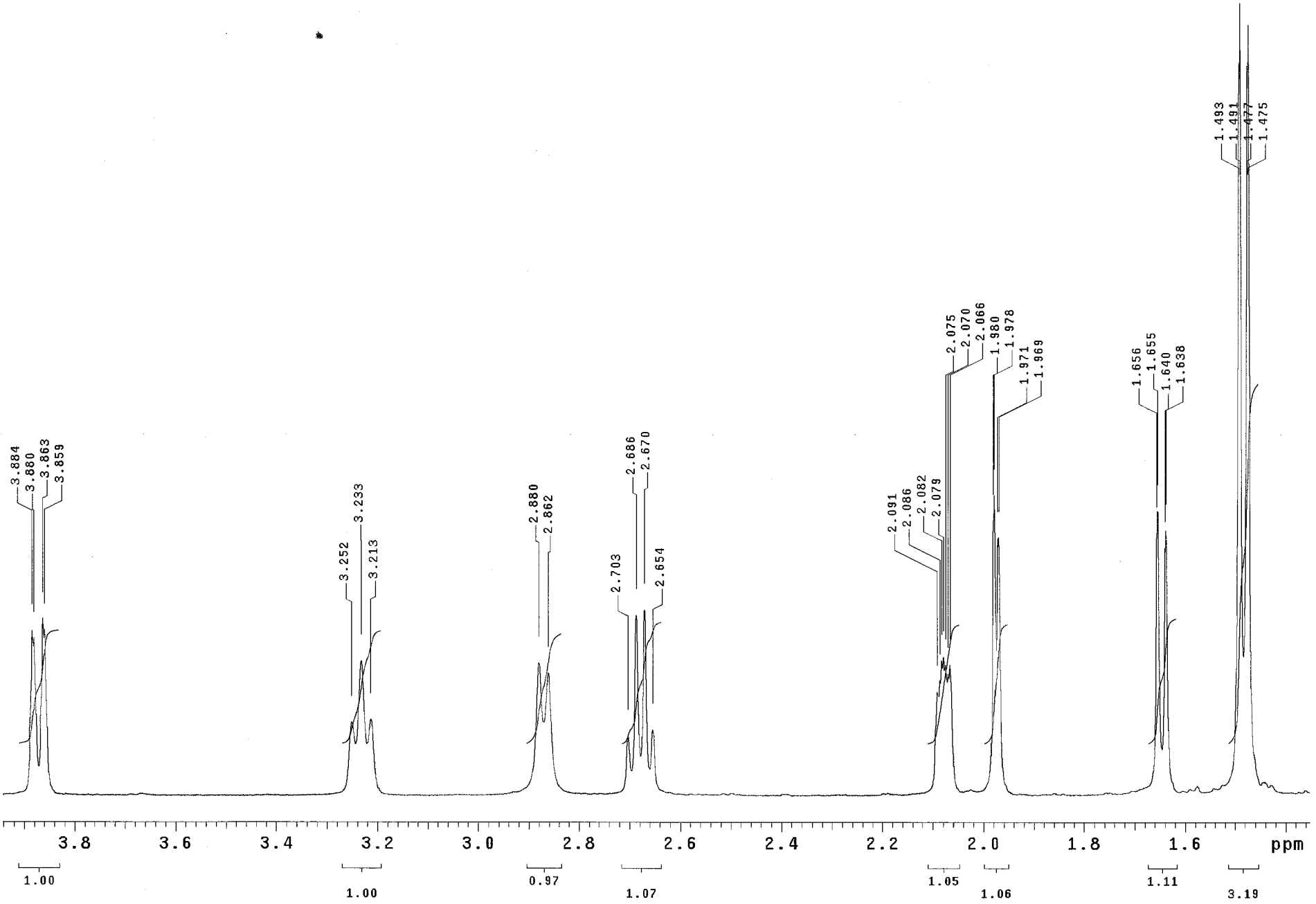

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Std carbon

File: xp

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: LWK
VNMRS-400 "Varian-NMR"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
360 repetitions
OBSERVE C13, 100.5150857 MHz
DECOUPLE H1, 399.7435210 MHz
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continuously on
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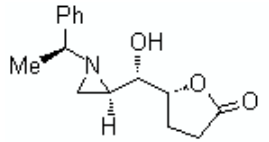
Total time 38 min, 21 sec



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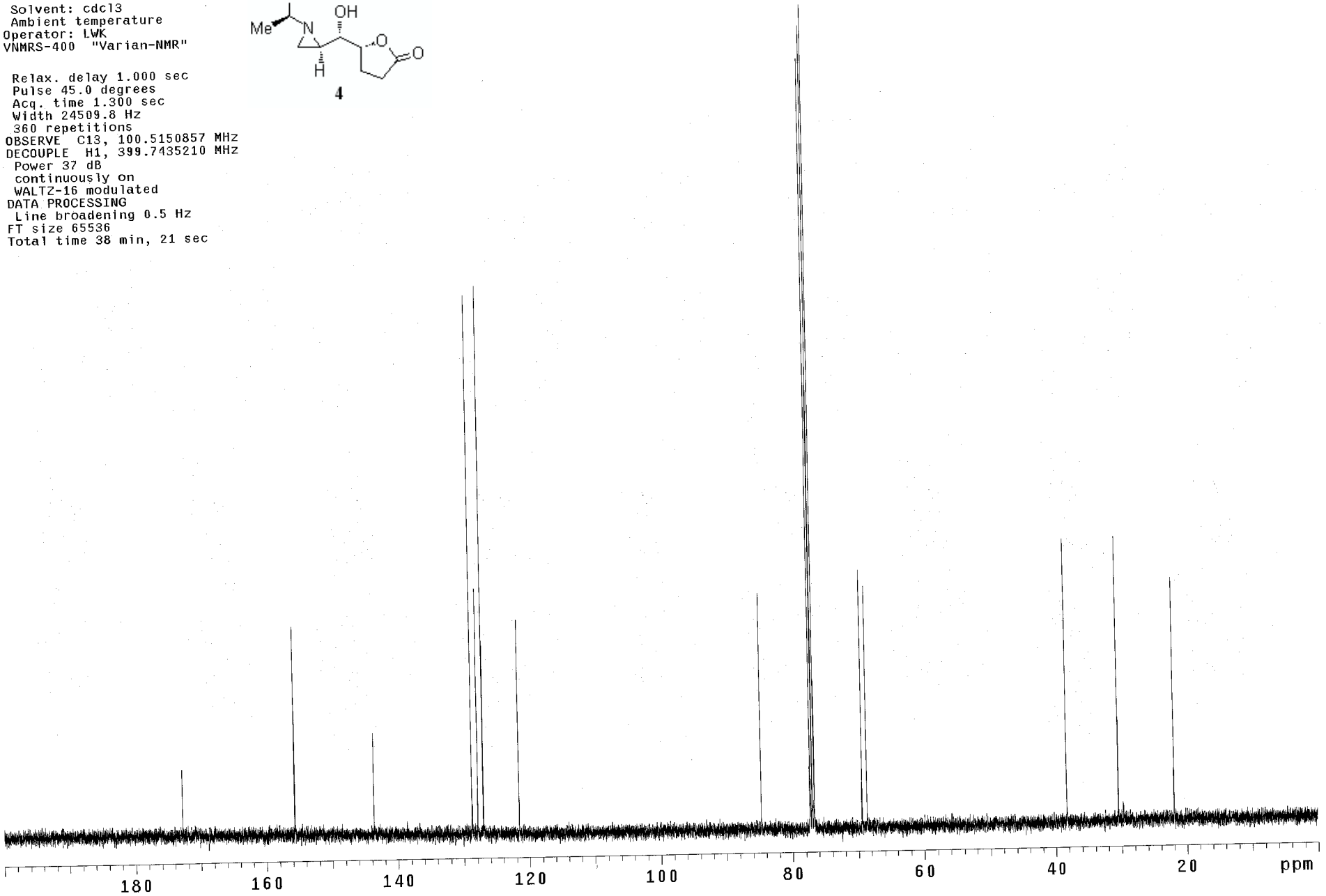

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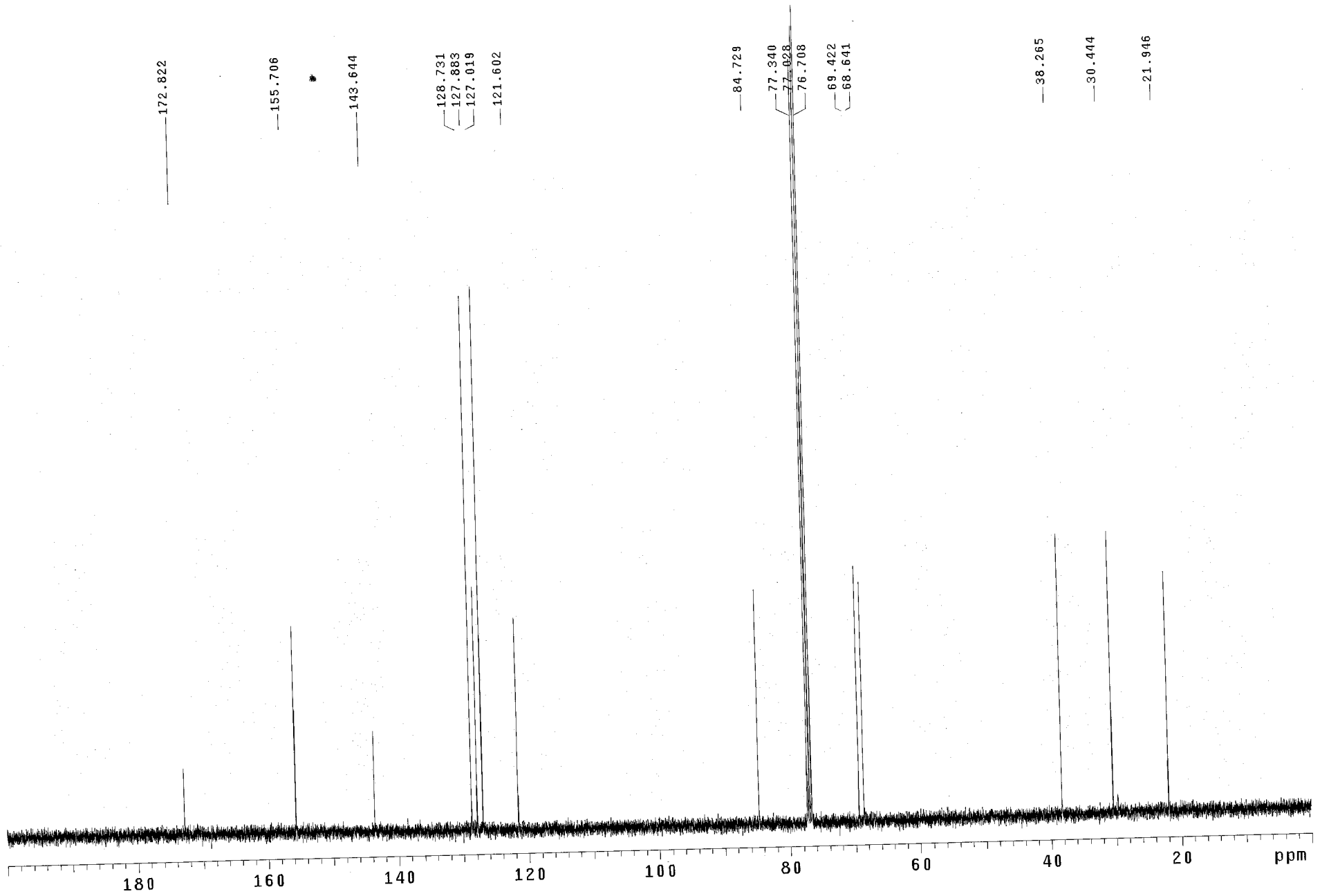

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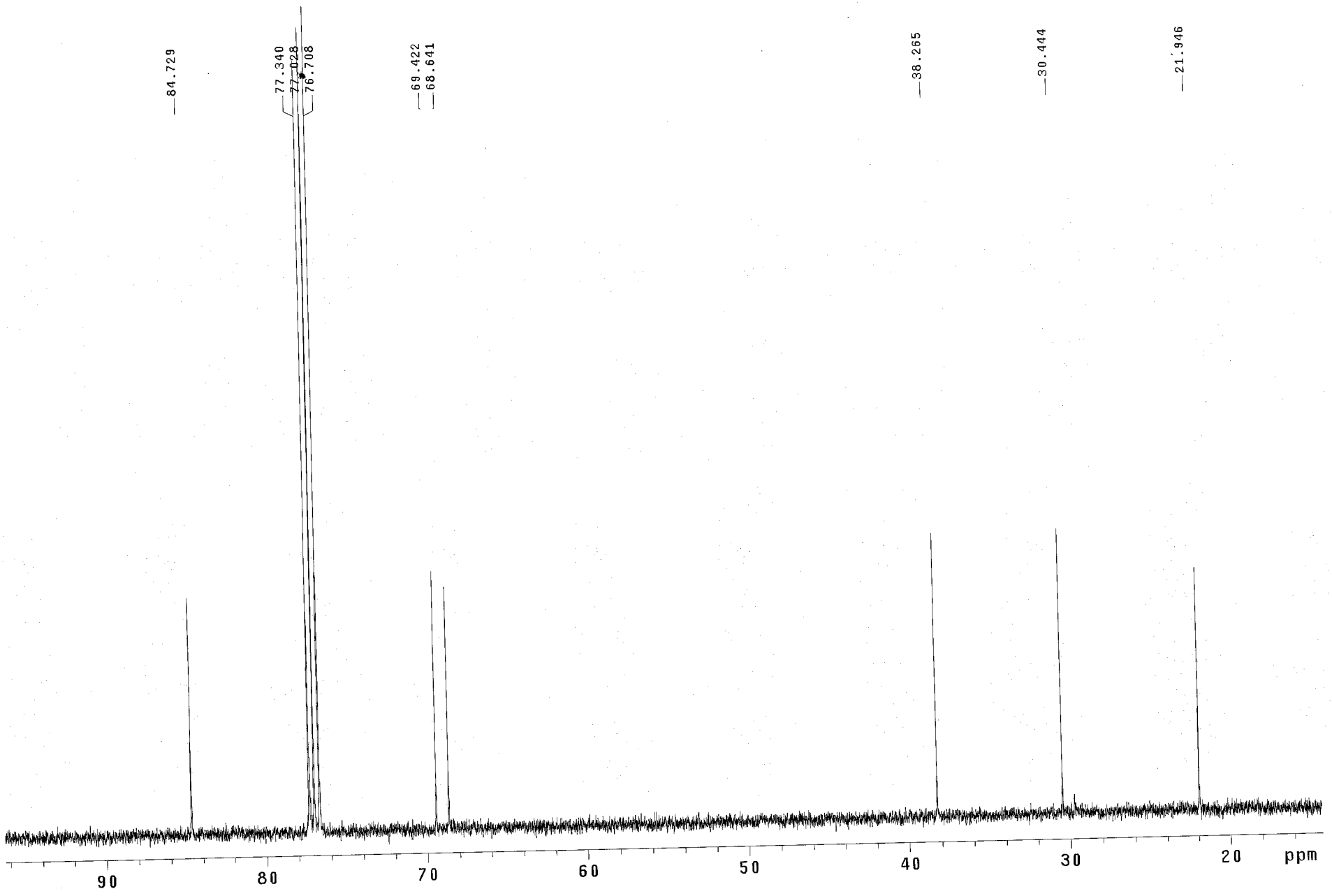

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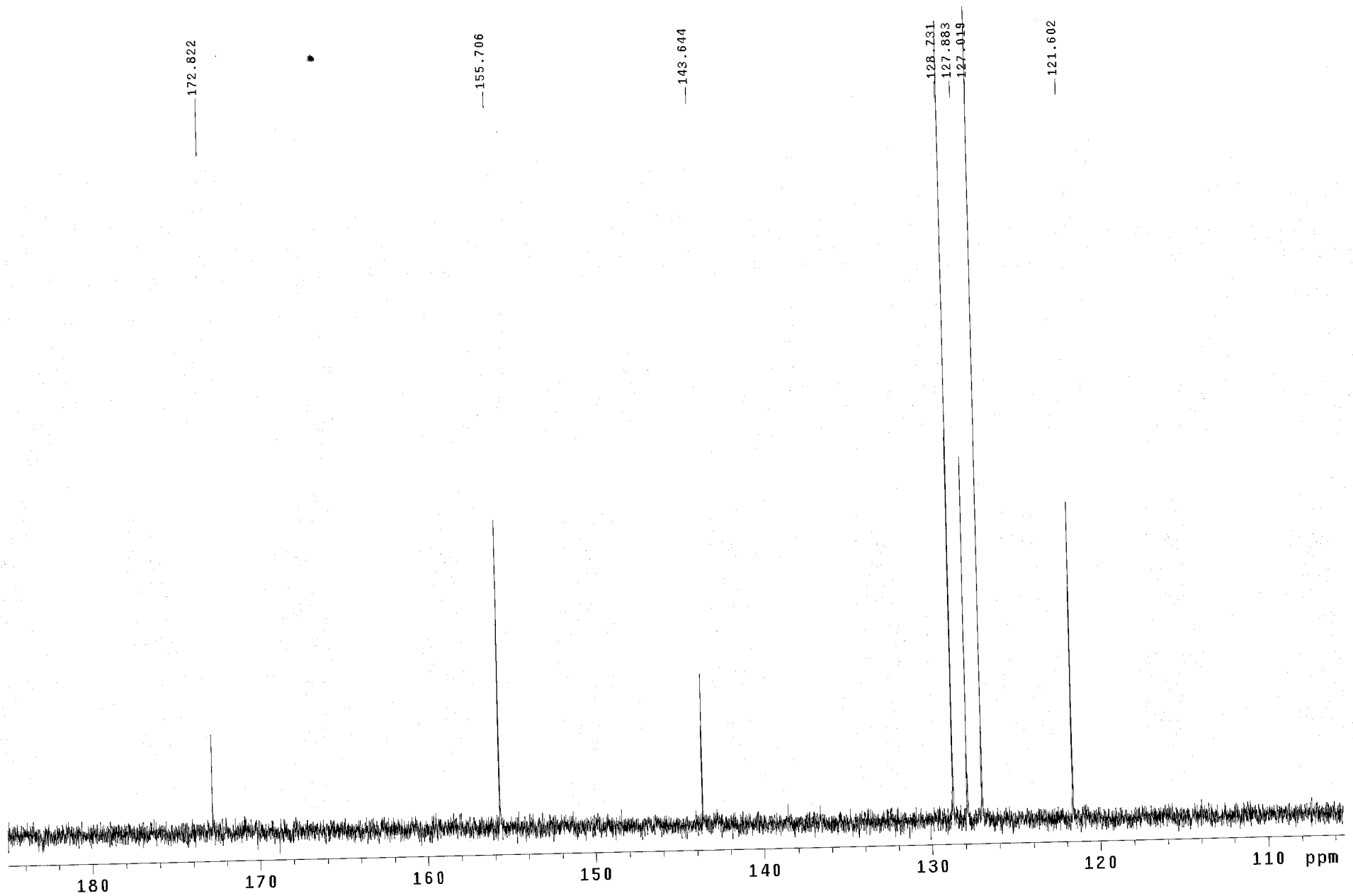

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Std proton

File: xp

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: LWK
VNMRS-400 "Varian-NMR"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
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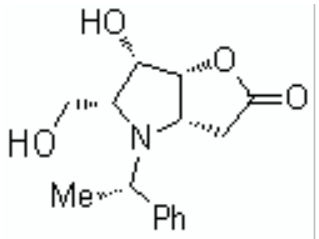

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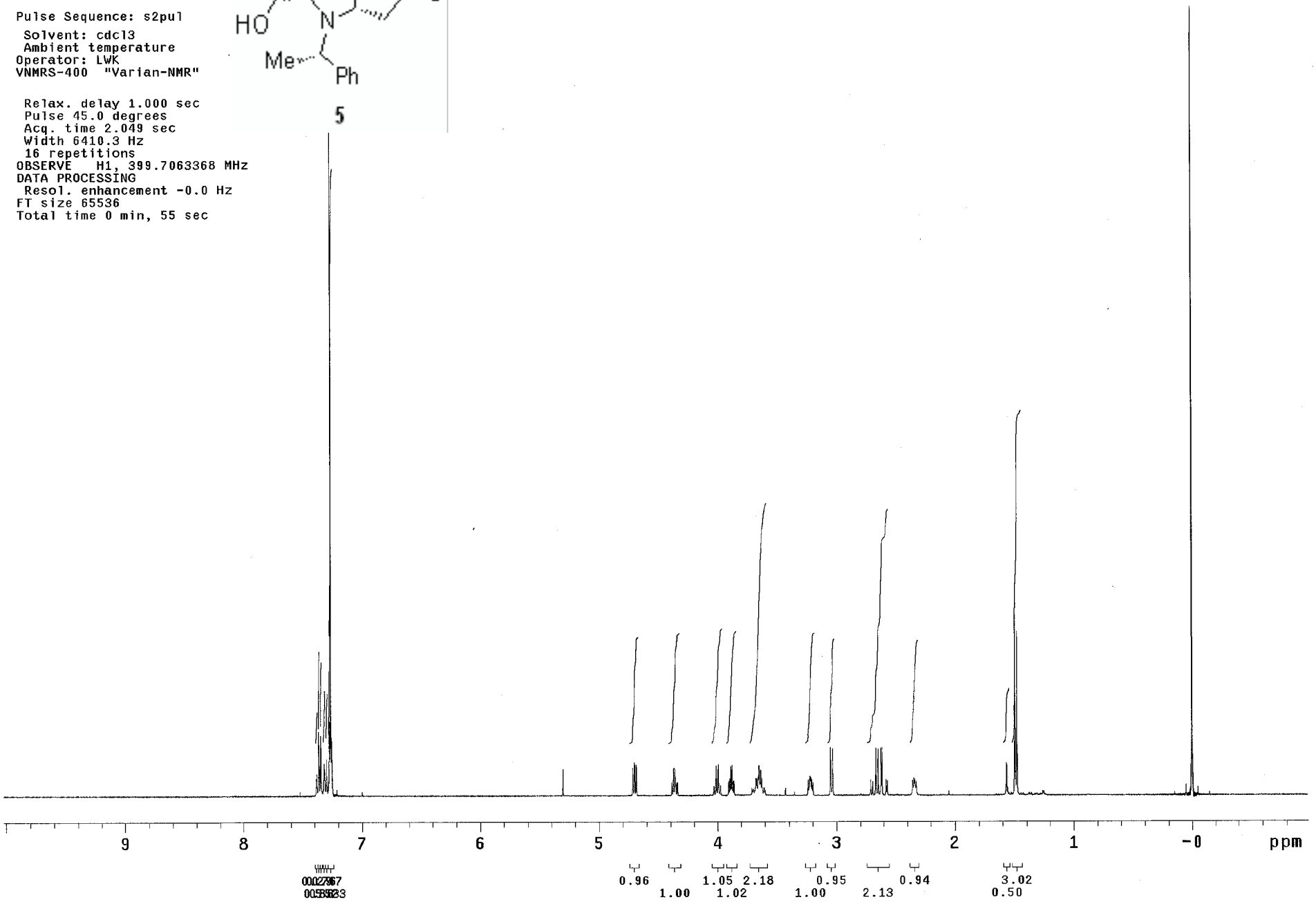

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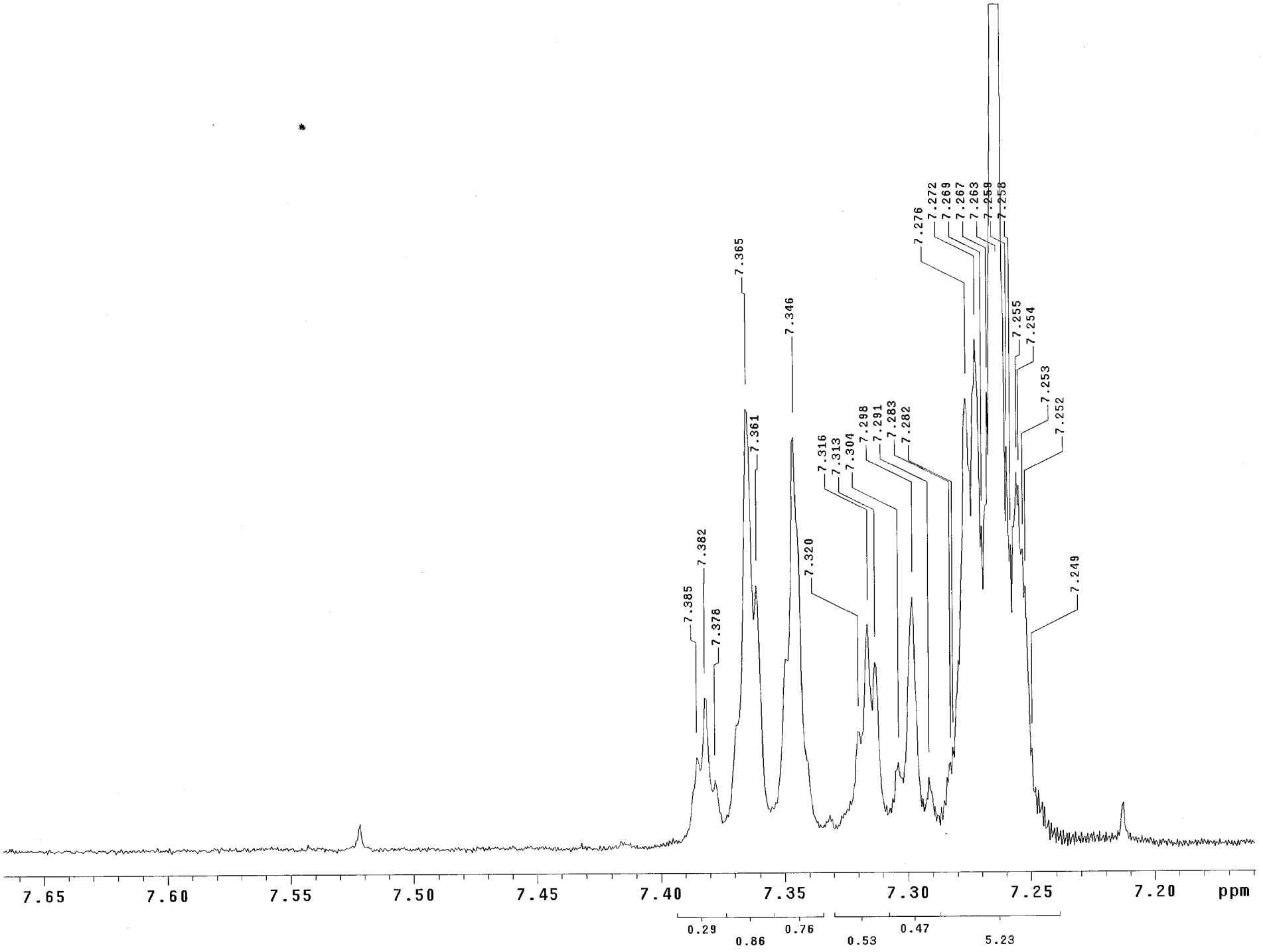

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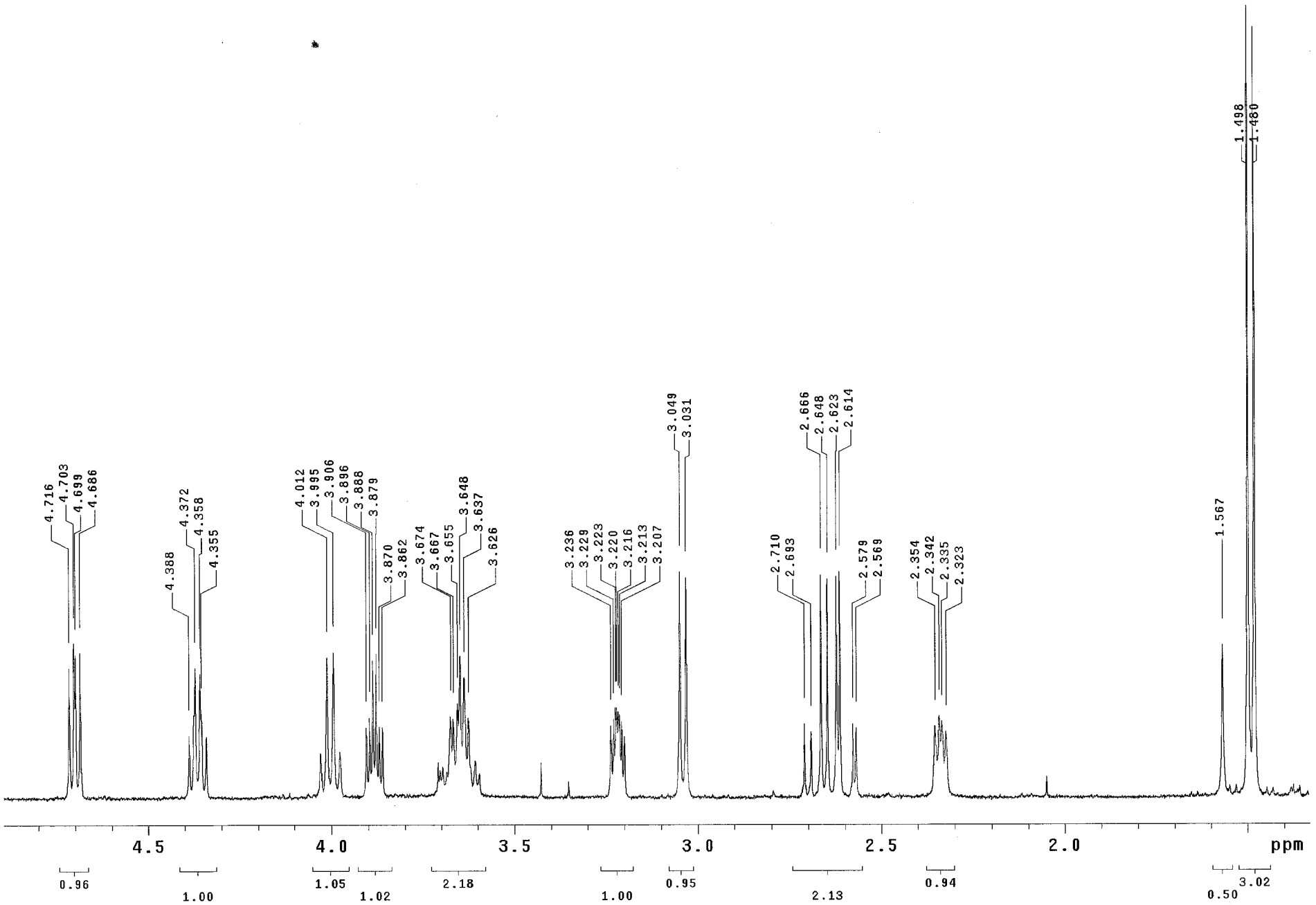

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Std carbon

File: xp

Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
Operator: LWK
VNMRS-400 "Varian-NMR"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24509.8 Hz
 192 repetitions
OBSERVE C13, 100.5039471 MHz
DECOUPLE H1, 399.6992231 MHz
 Power 37 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
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Total time 1 hr, 16 min, 41 sec

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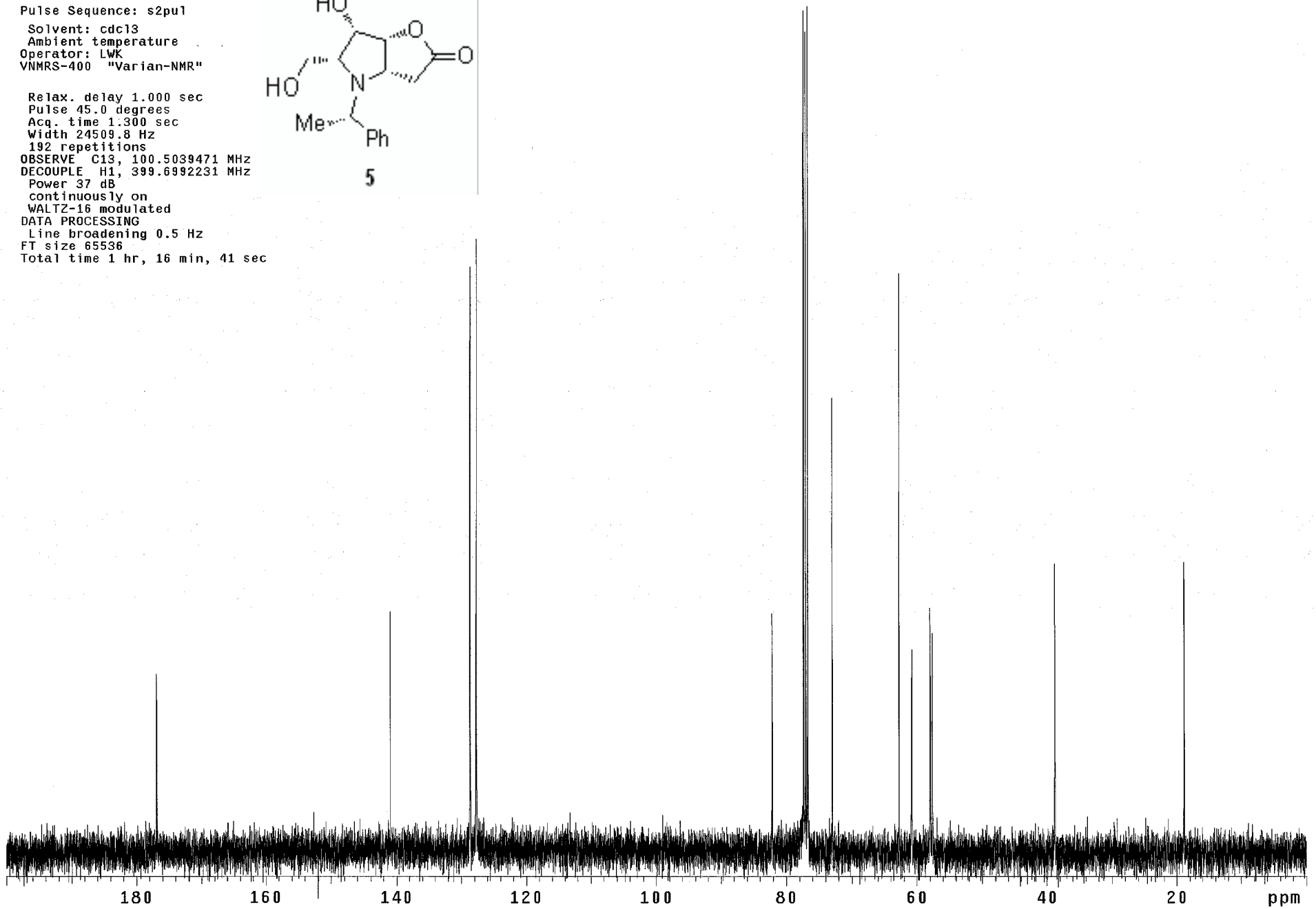

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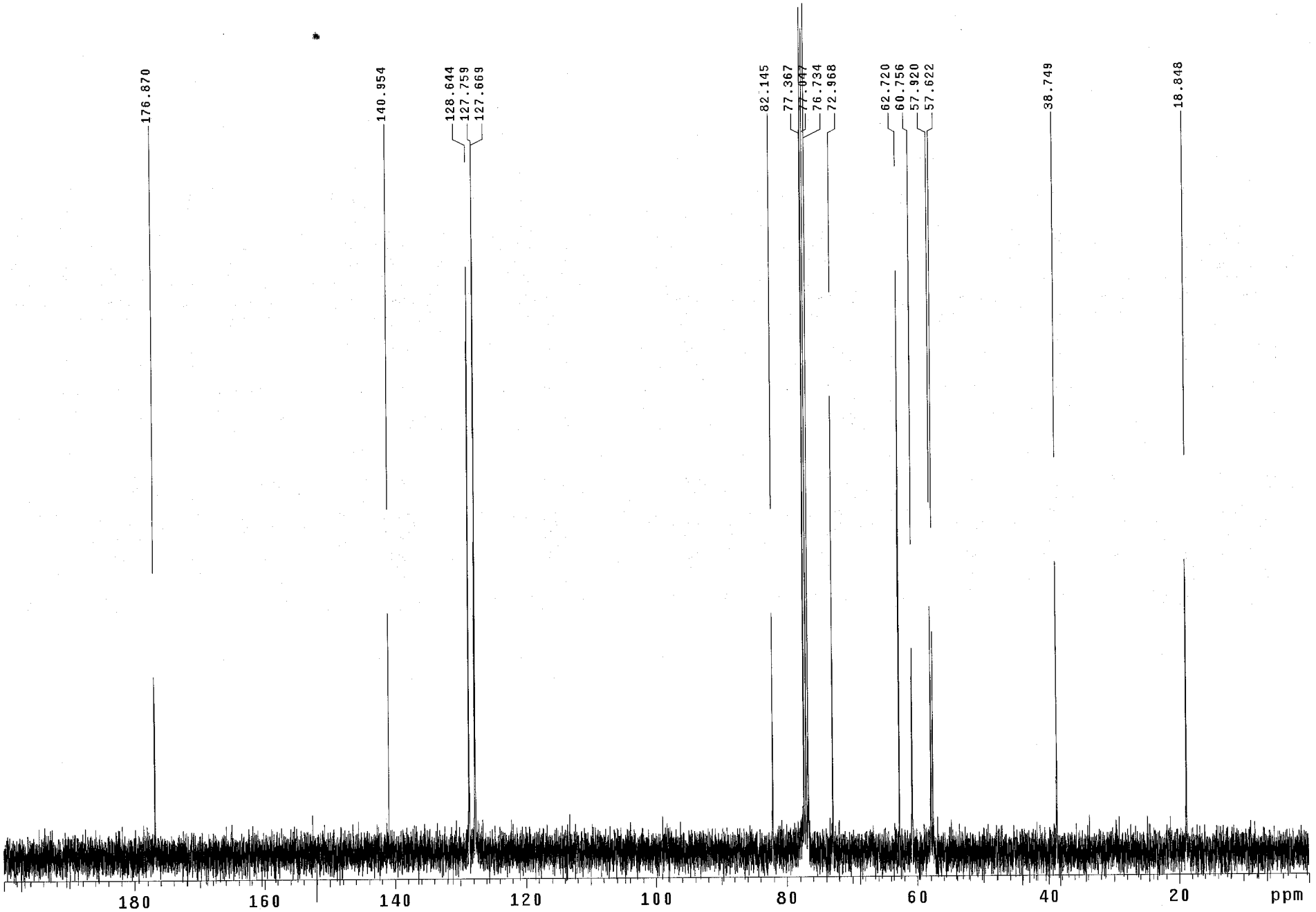

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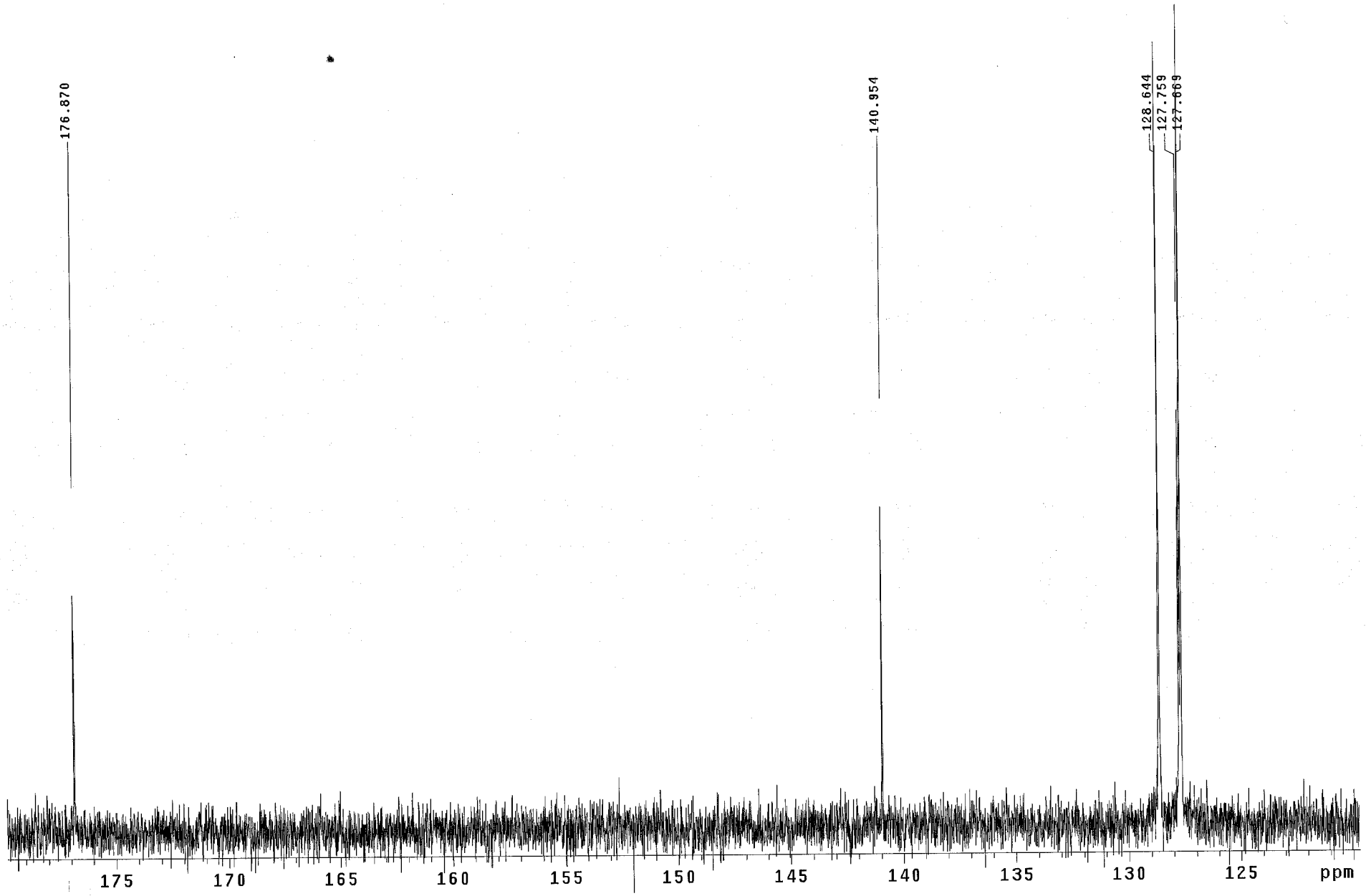

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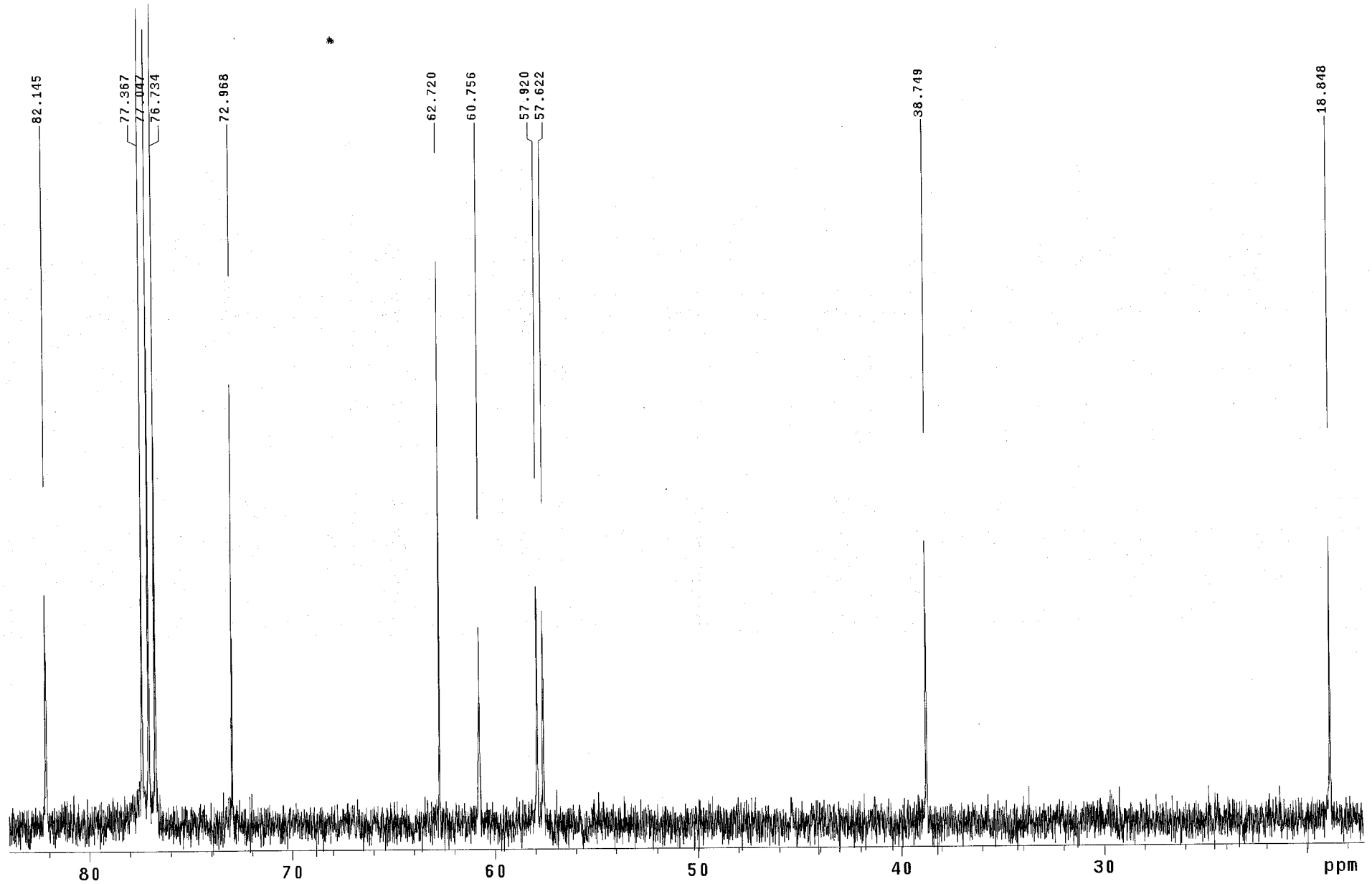

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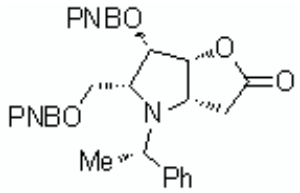
Std proton

File: xp

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
Operator: LWK
VNMRS-400 "Varian-NMR"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
16 repetitions
OBSERVE H1, 399.7063376 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 55 sec



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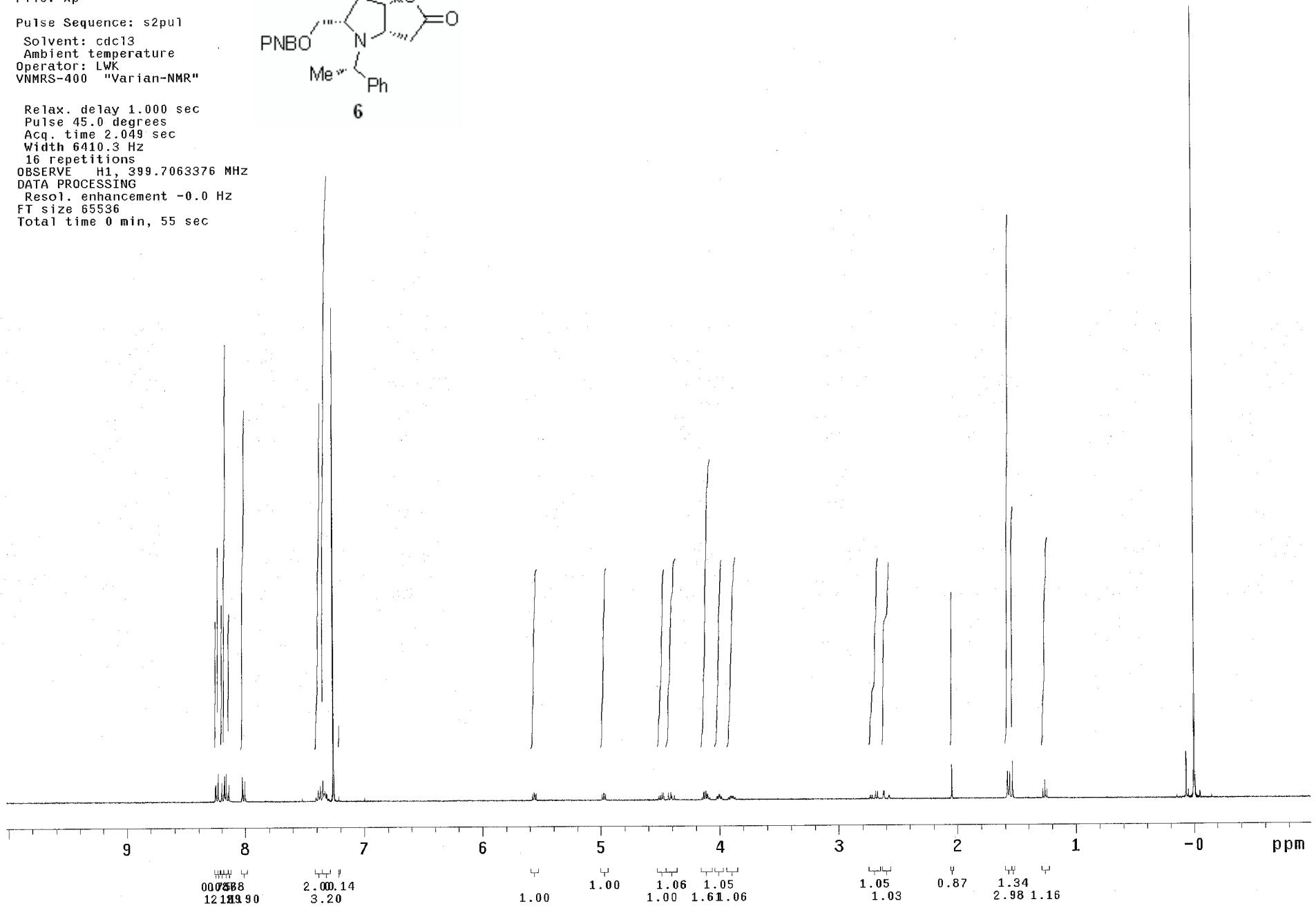

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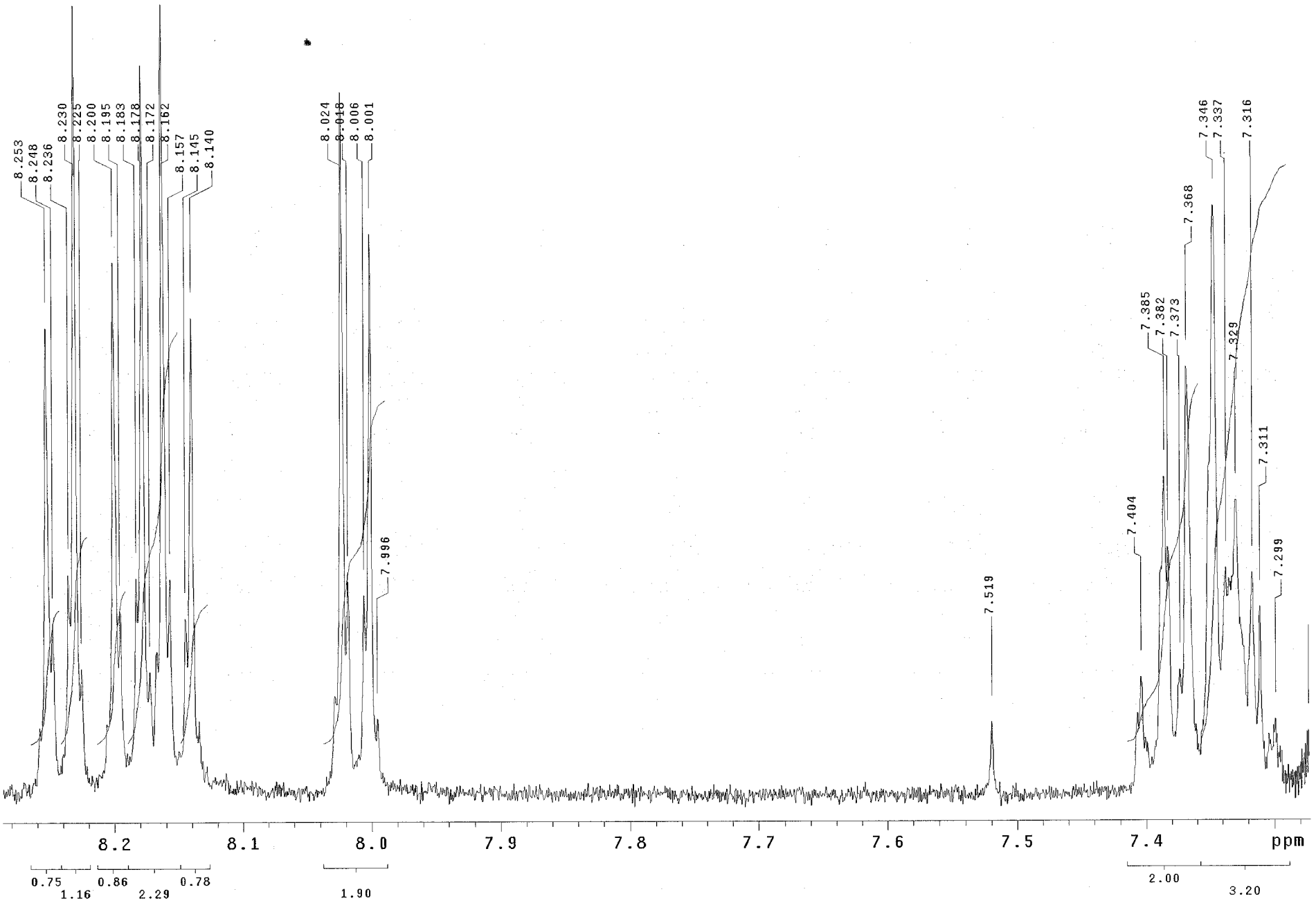

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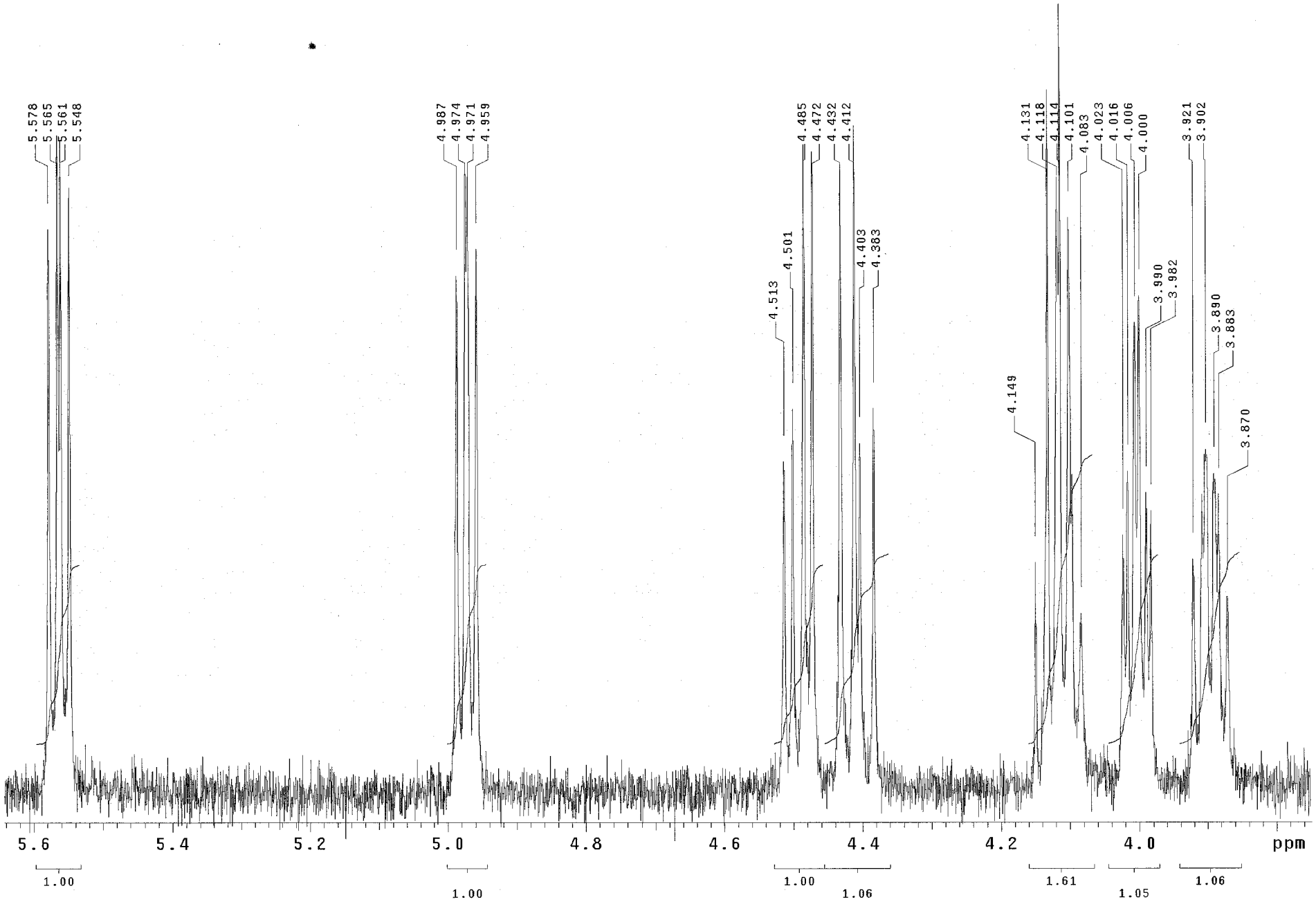

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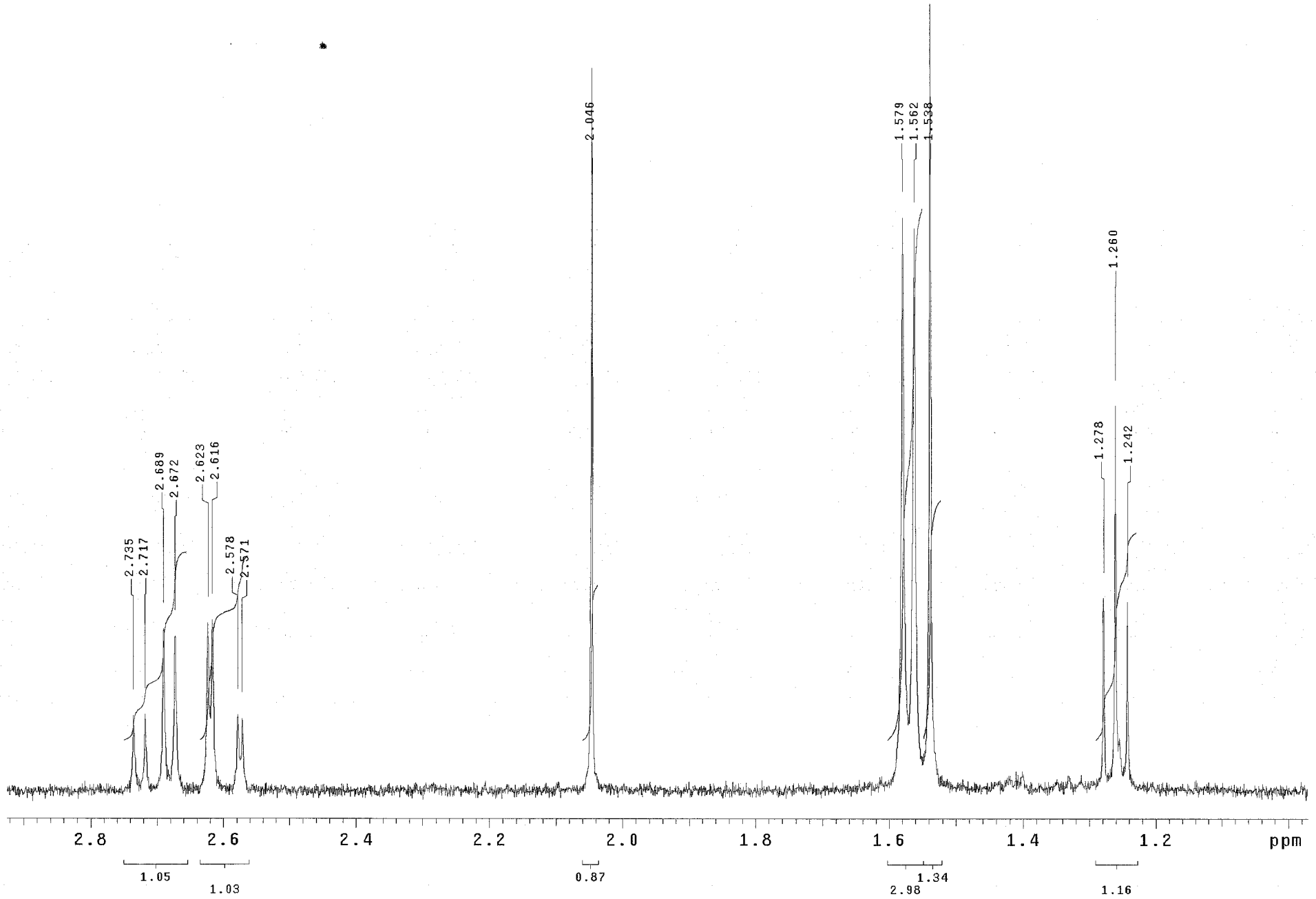

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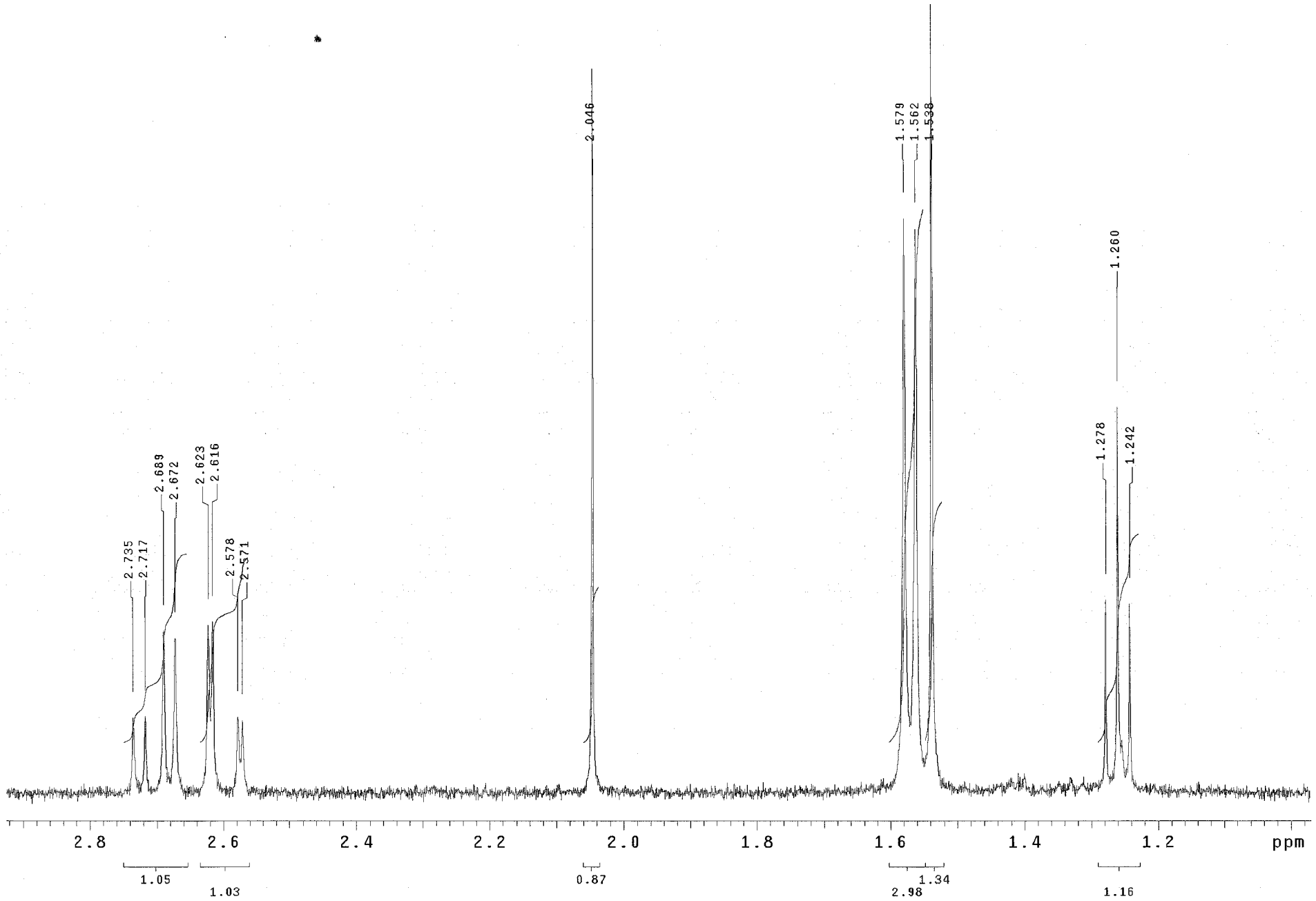

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Std carbon

File: xp

Pulse Sequence: s2pul
 Solvent: cdcl3
 Temp. 25.0 C / 298.1 K
Operator: LWK
VNMRS-400 "Varian-NMR"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24509.8 Hz
 480 repetitions
OBSERVE C13, 100.4988692 MHz
DECOUPLE H1, 399.6790285 MHz
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 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 16 min, 41 sec

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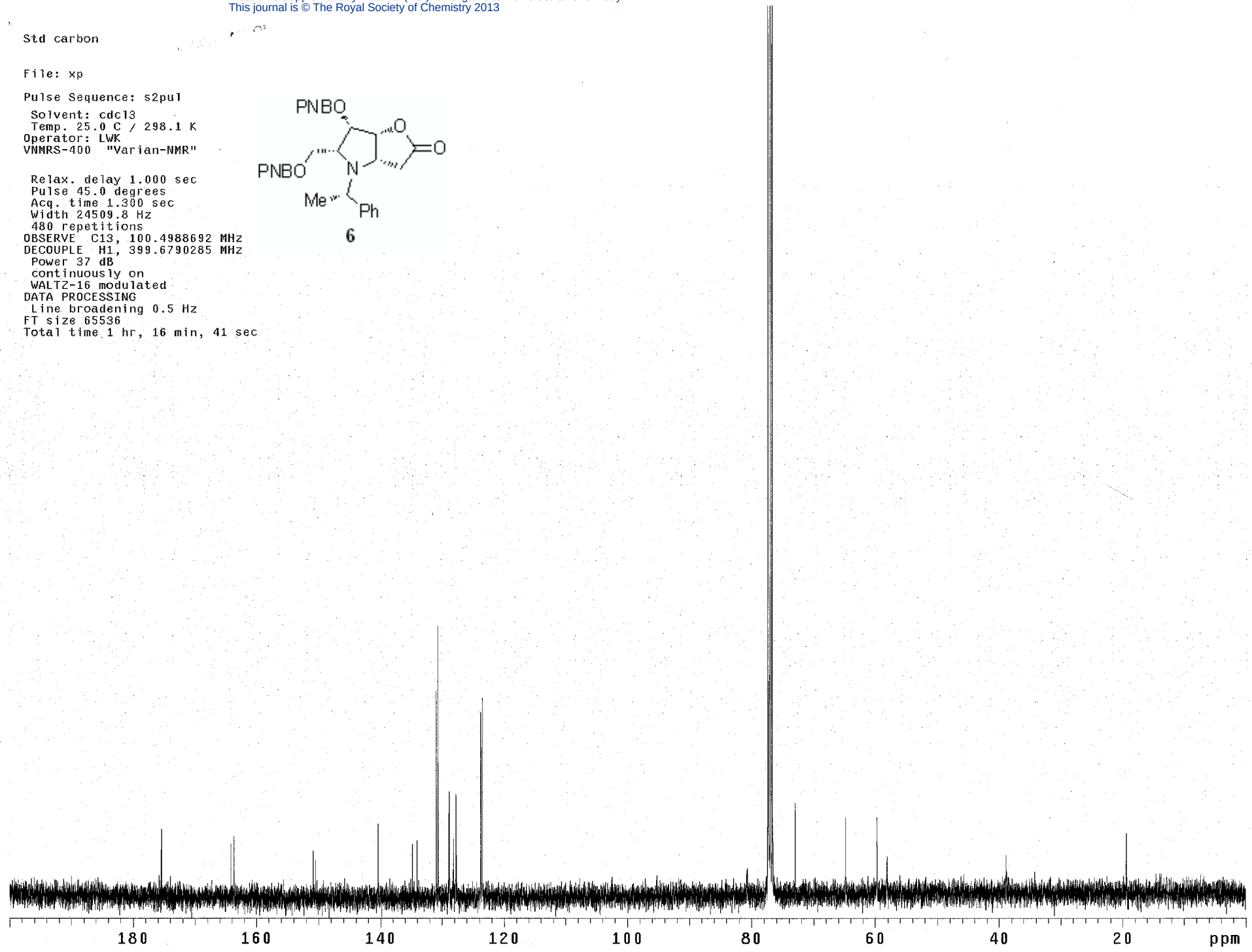

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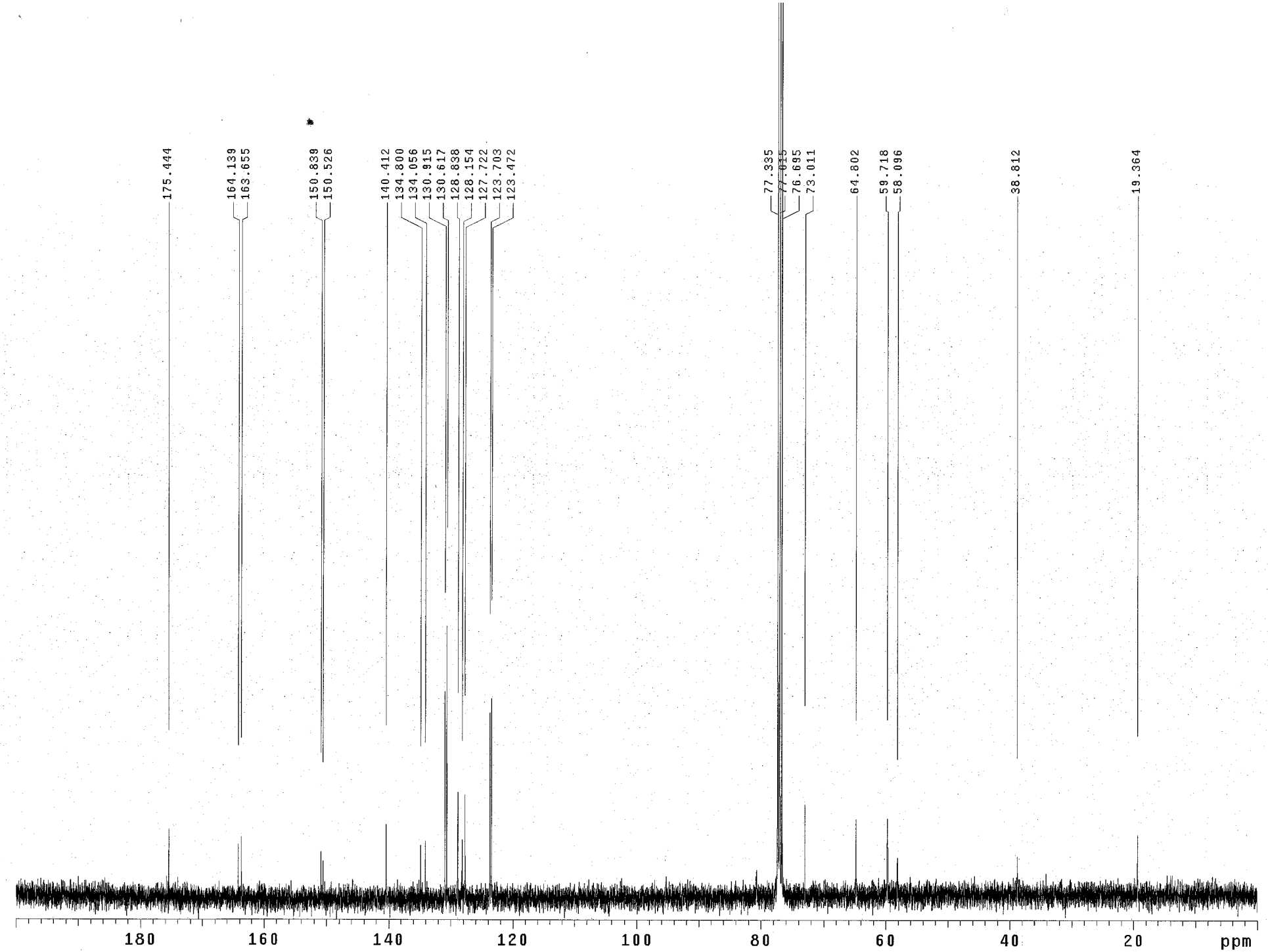

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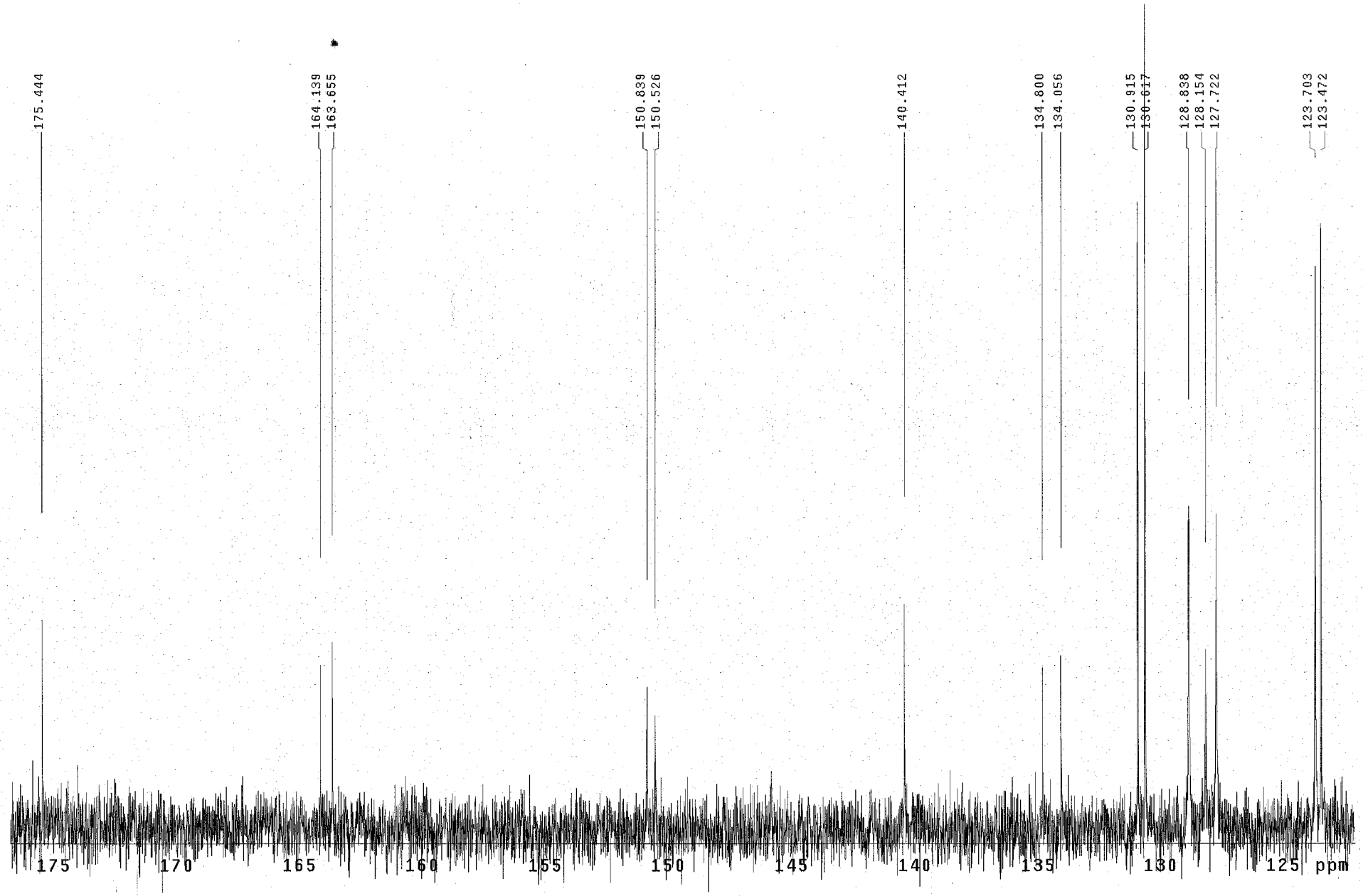

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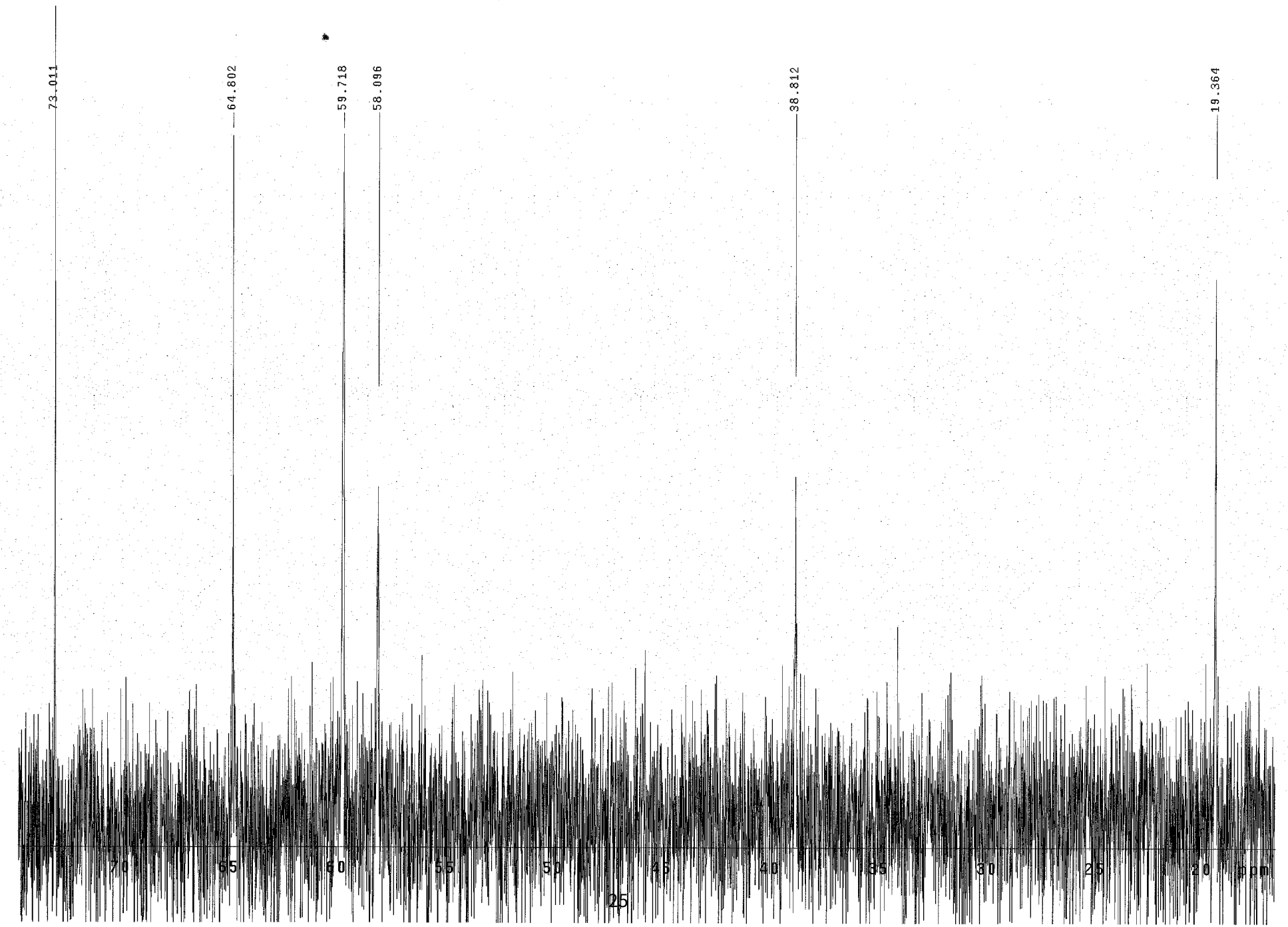

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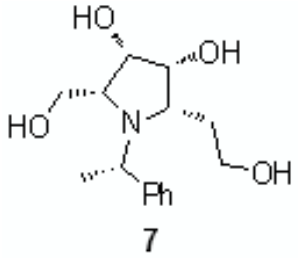
Std proton

File: xp

Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
Operator: LWK
VNMRS-400 "Varian-NMR"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.049 sec
 Width 6410.3 Hz
 8 repetitions
OBSERVE   H1, 399.7323984 MHz
DATA PROCESSING
 Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 30 sec



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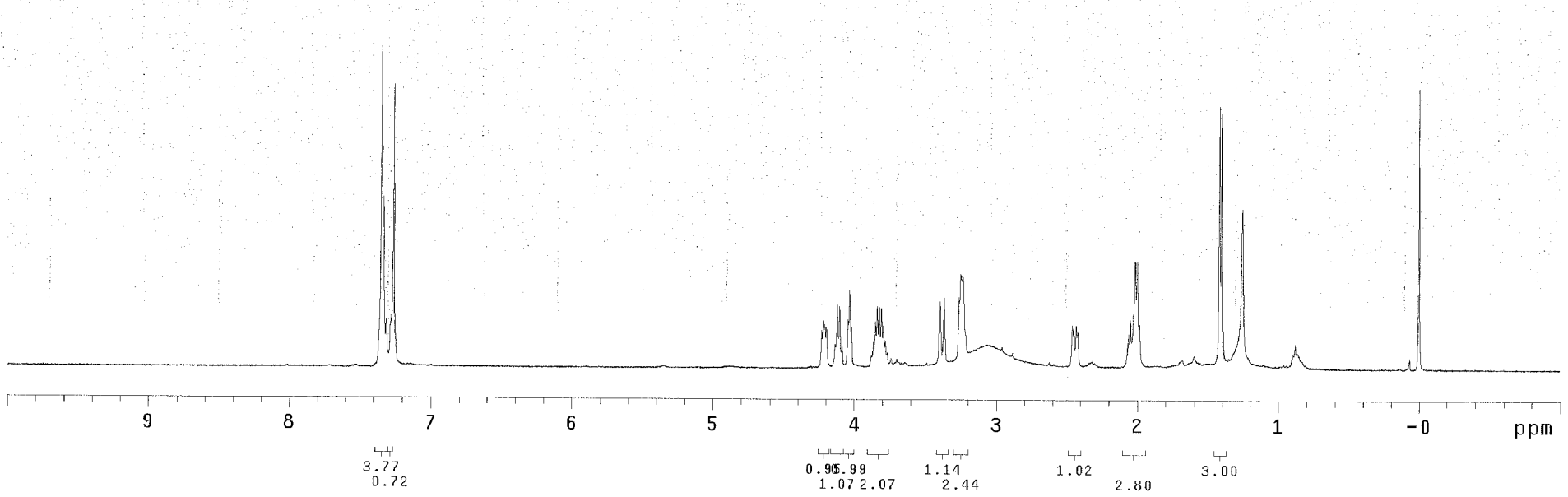

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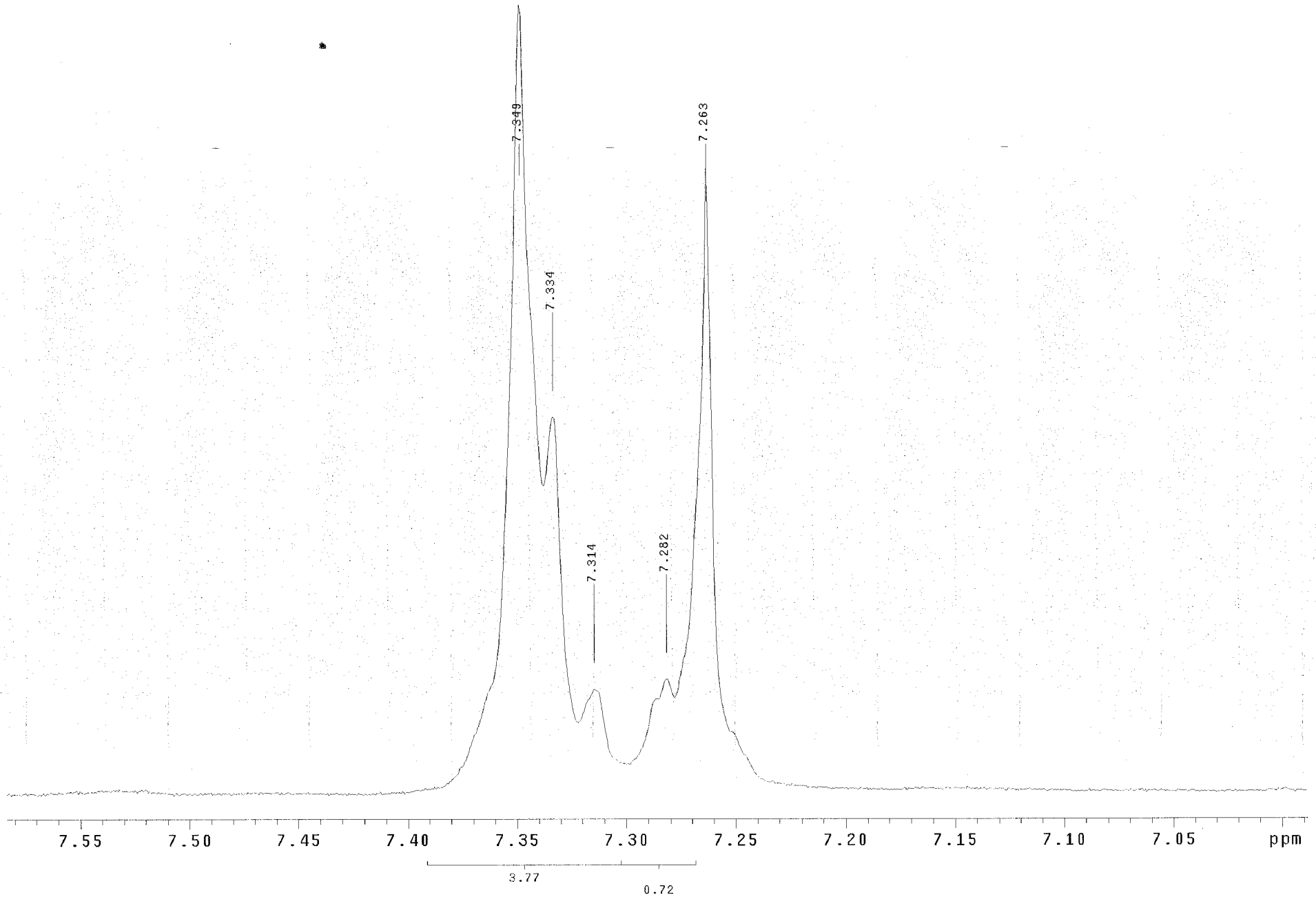

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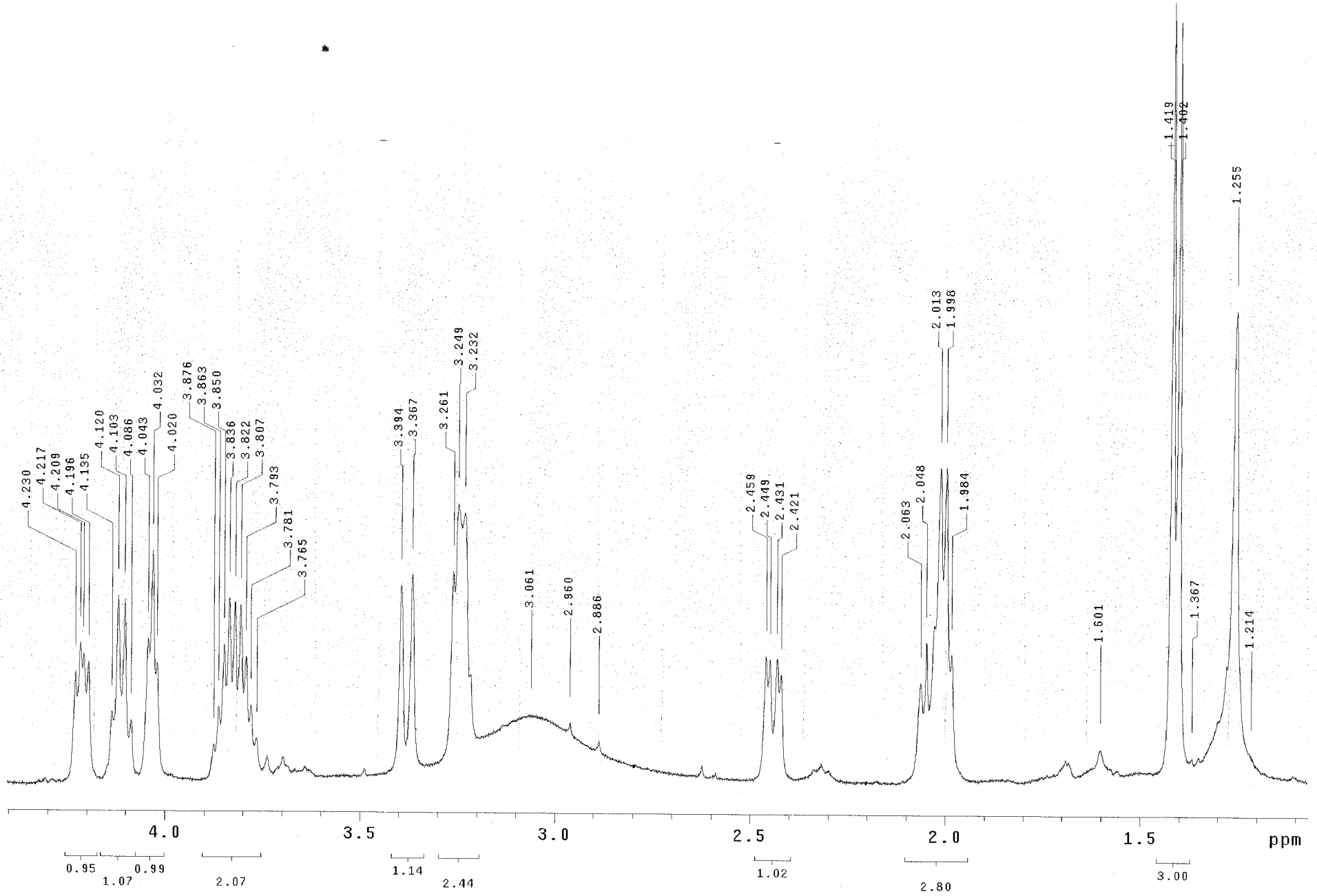

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Std carbon

File: xp

Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
Operator: LWK
VNMRS-400  "Varian-NMR"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24509.8 Hz
 172 repetitions
OBSERVE  C13, 100.5095164 MHz
DECOUPLE  H1, 399.7213720 MHz
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 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 16 min, 41 sec
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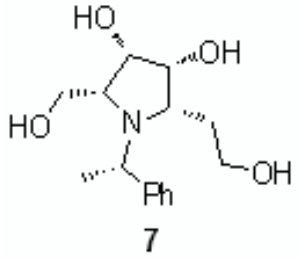

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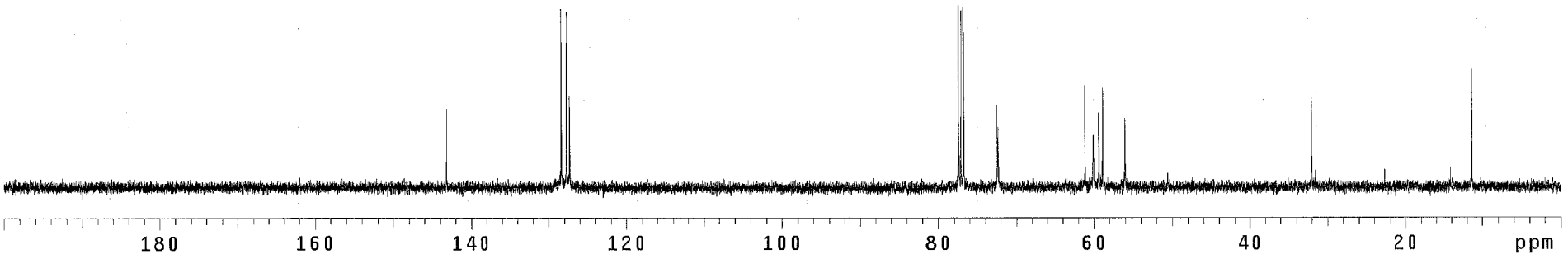

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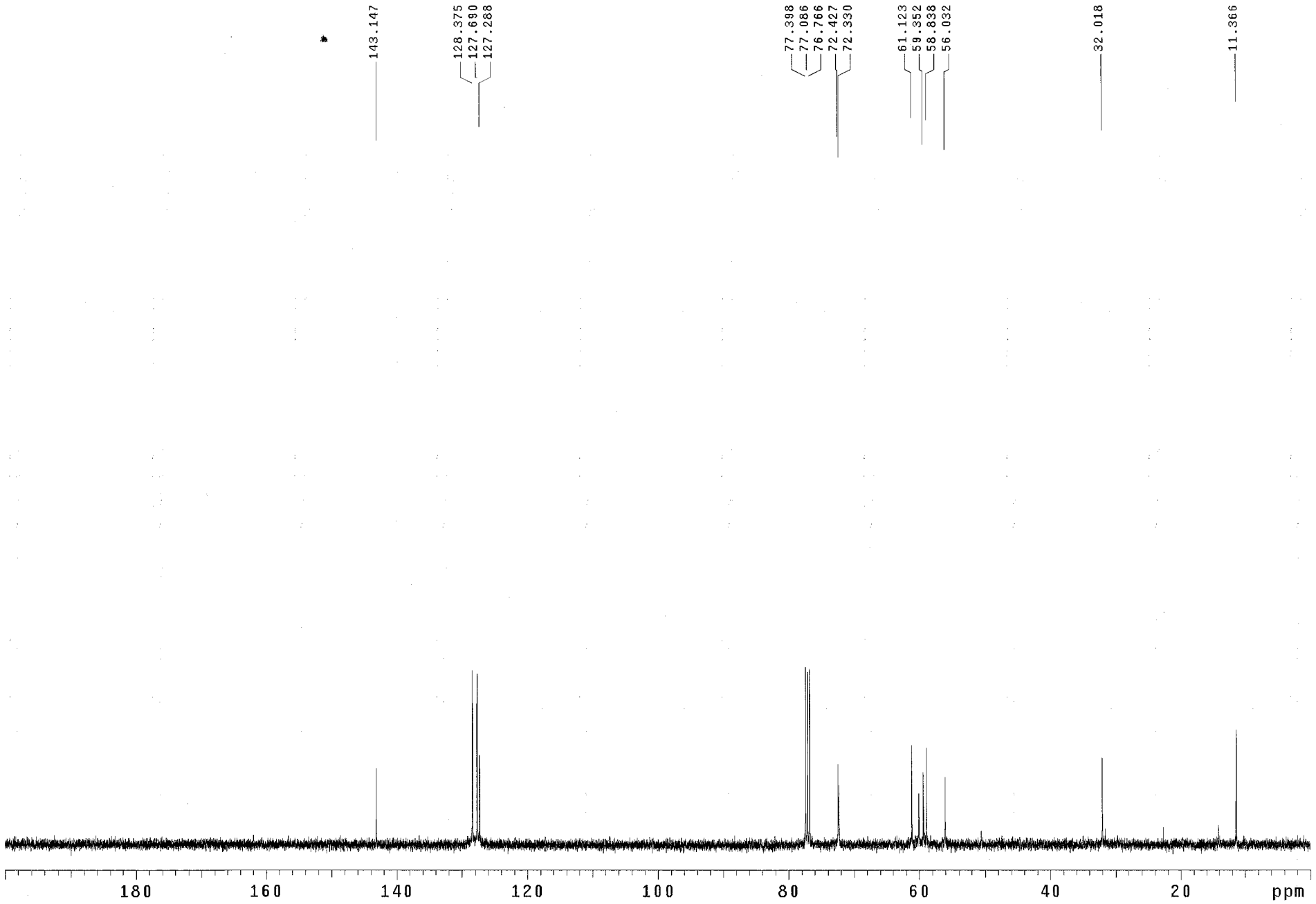

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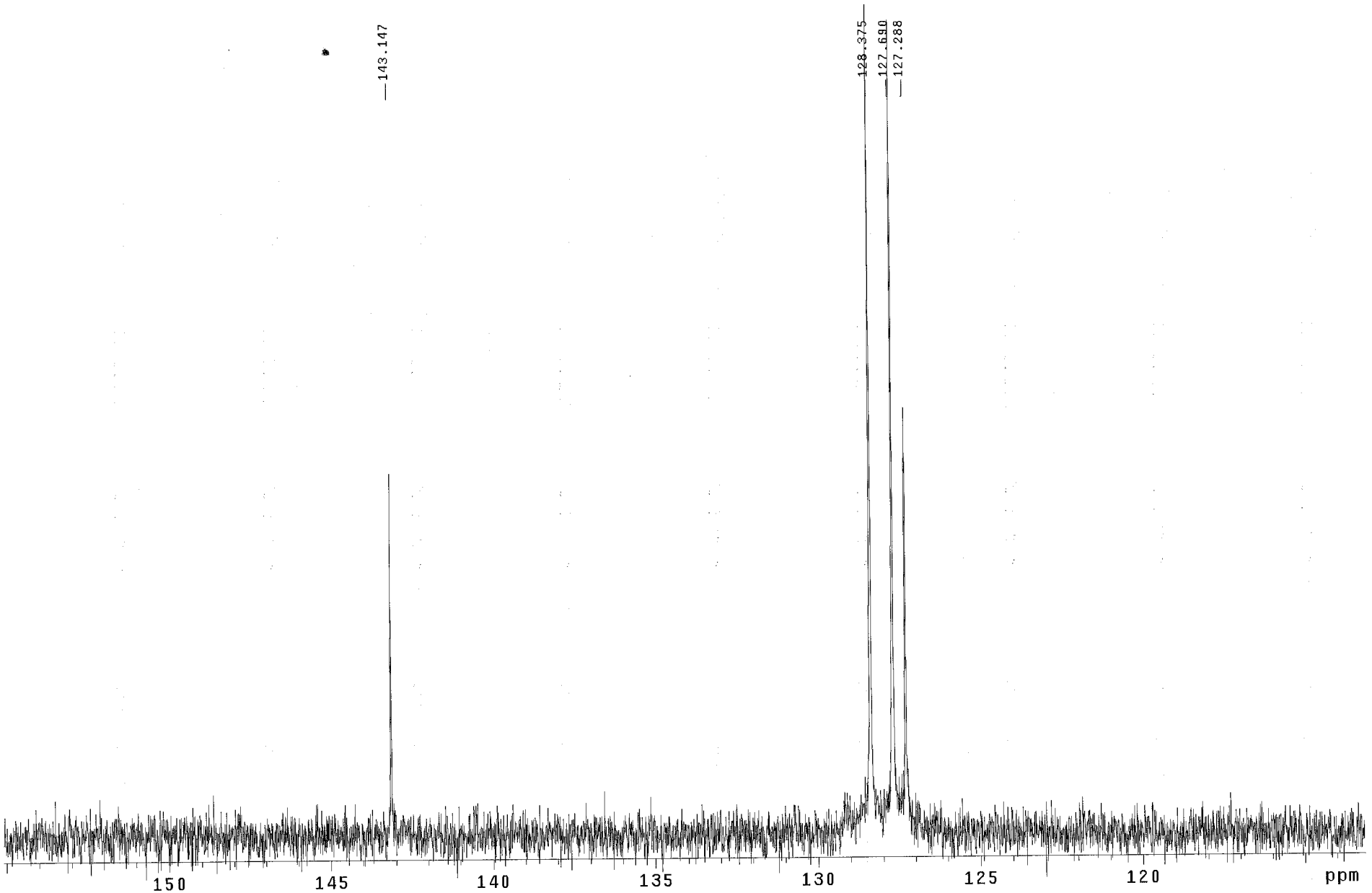

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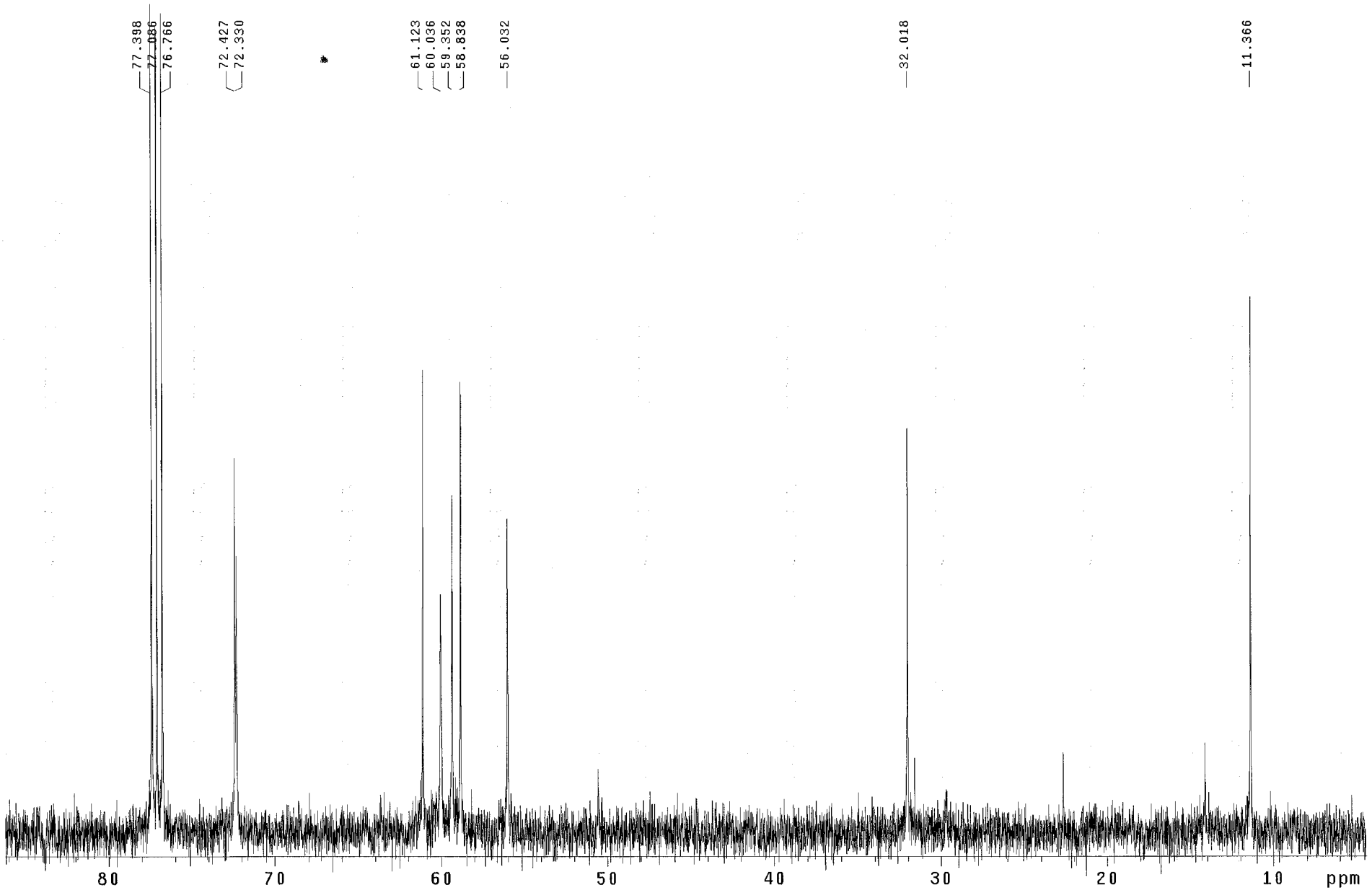

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Std proton

File: xp

Pulse Sequence: s2pul
 Solvent: d2o
 Ambient temperature
Operator: LWK
VNMRS-400 "Varian-NMR"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.049 sec
 Width 6410.3 Hz
 16 repetitions
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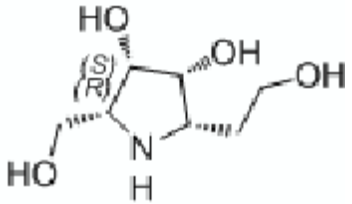
DATA PROCESSING
 Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 55 sec



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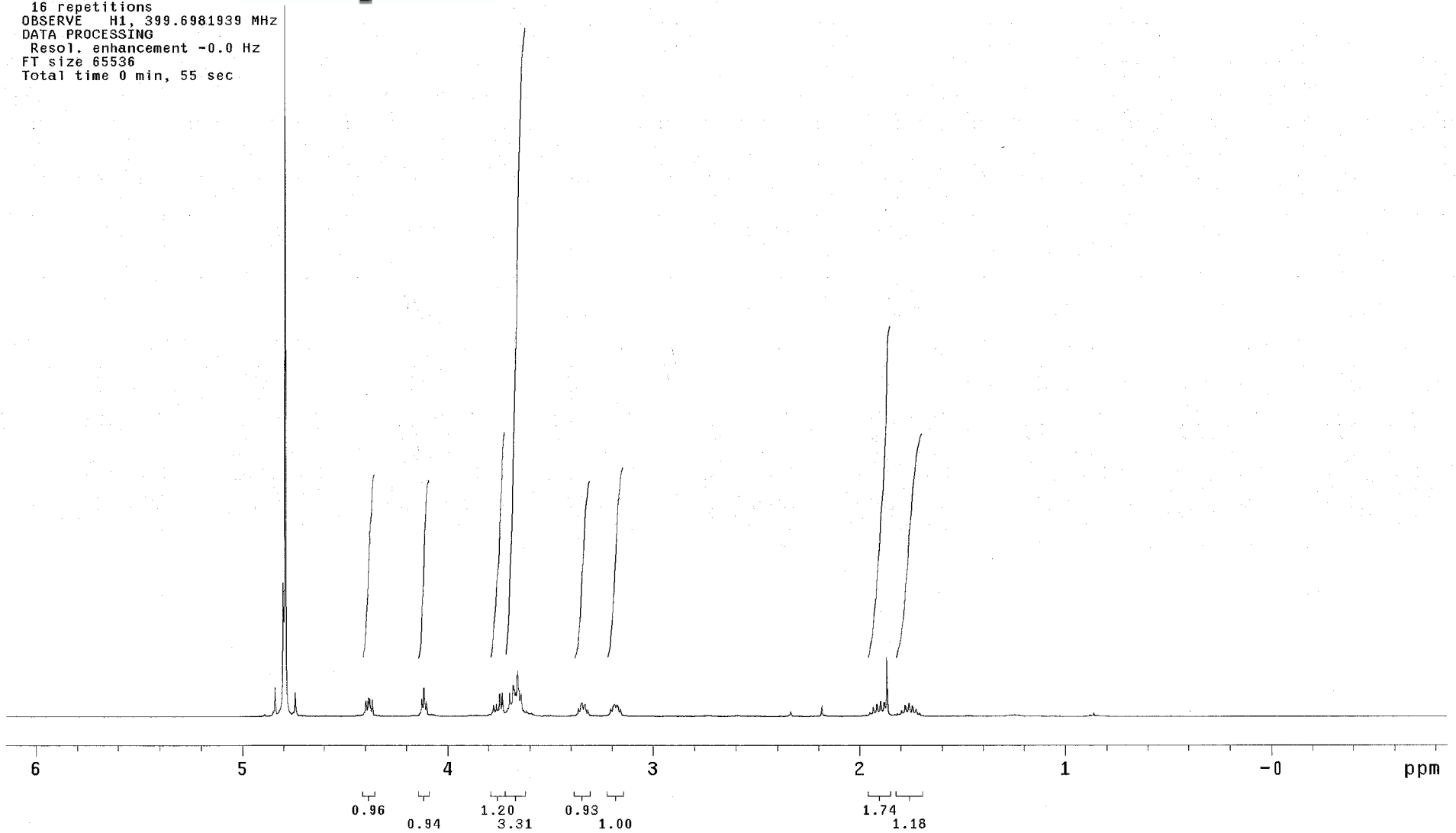

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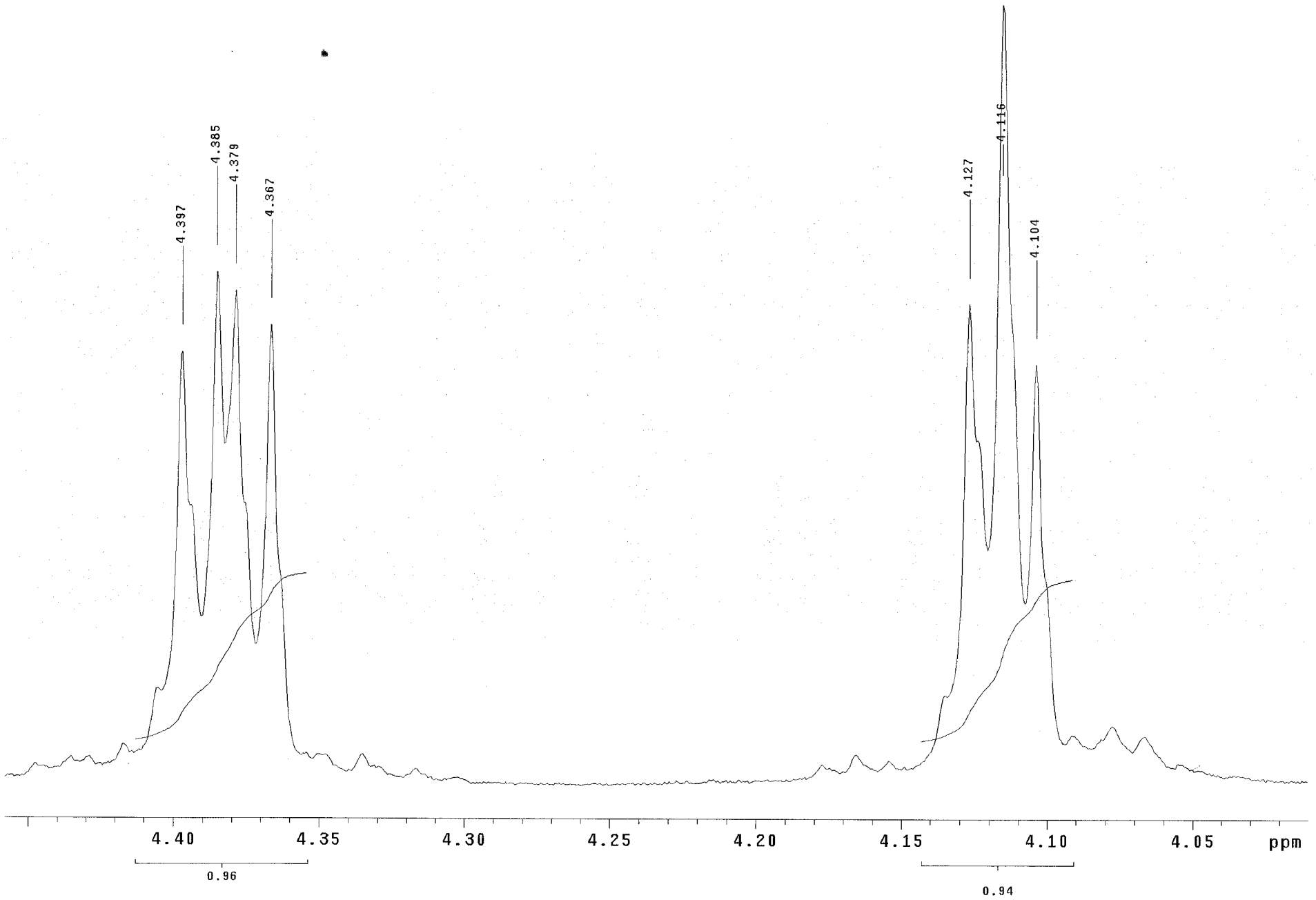

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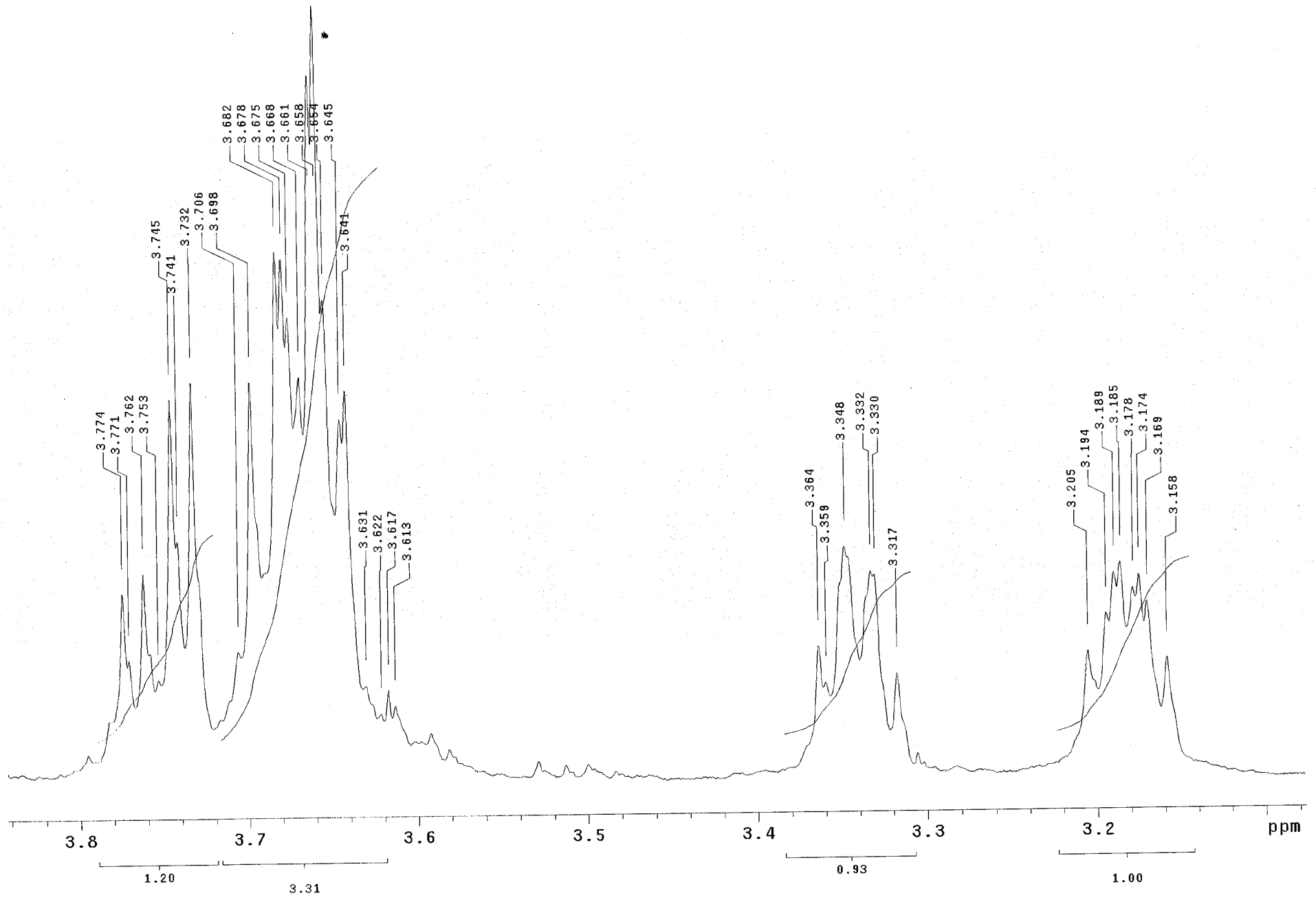

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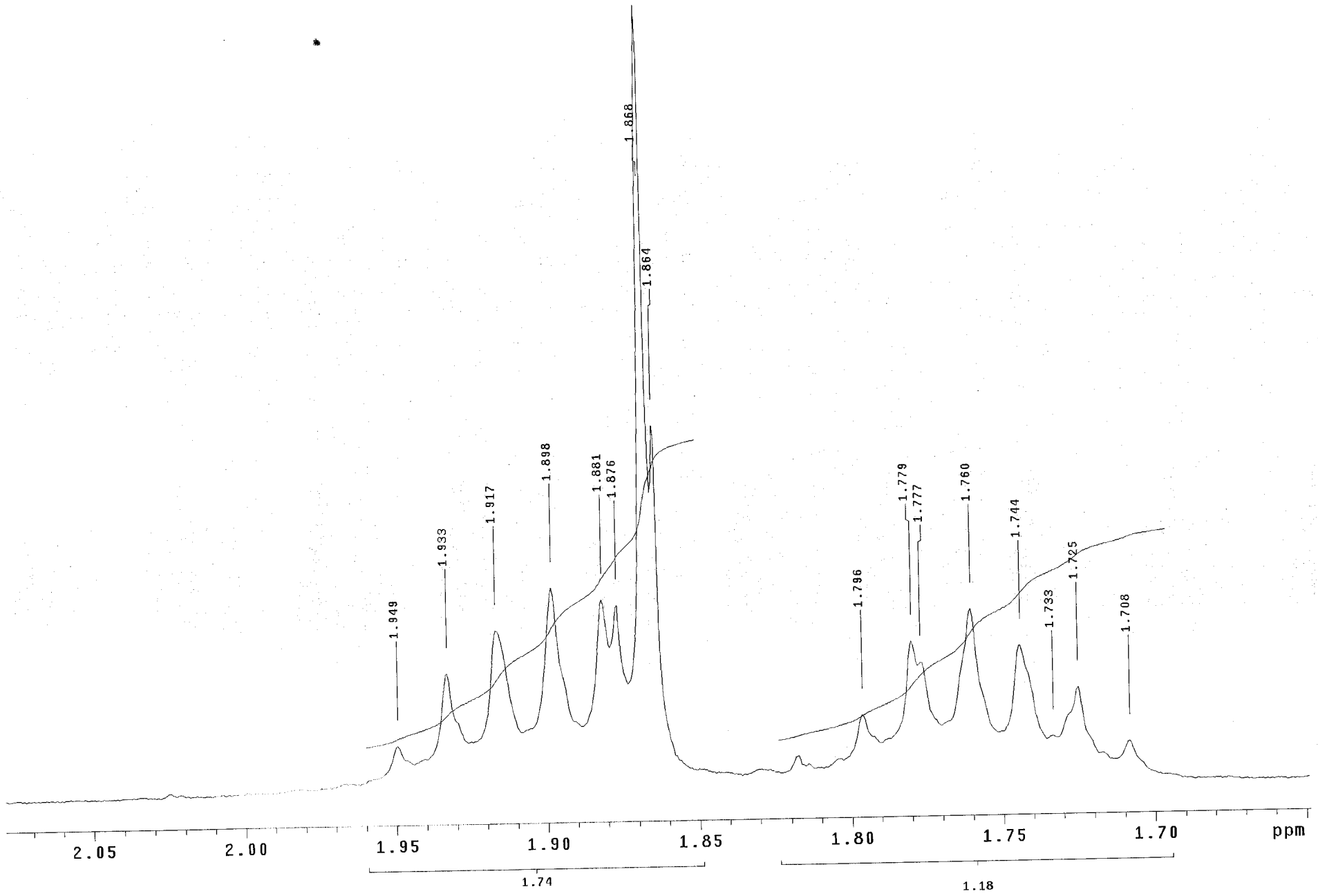

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Std carbon

File: xp

Pulse Sequence: s2pul
 Solvent: d2o
 Temp. 25.0 C / 298.1 K
Operator: LWK
VNMRS-400 "Varian-NMR"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24509.8 Hz
 384 repetitions
OBSERVE C13, 100.5042054 MH_
DECOUPLE H1, 399.7002504 MHz
 Power 37 dB
 continuously on
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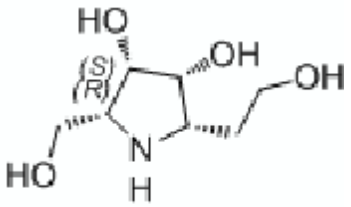
DATA PROCESSING
 Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 16 min, 41 sec



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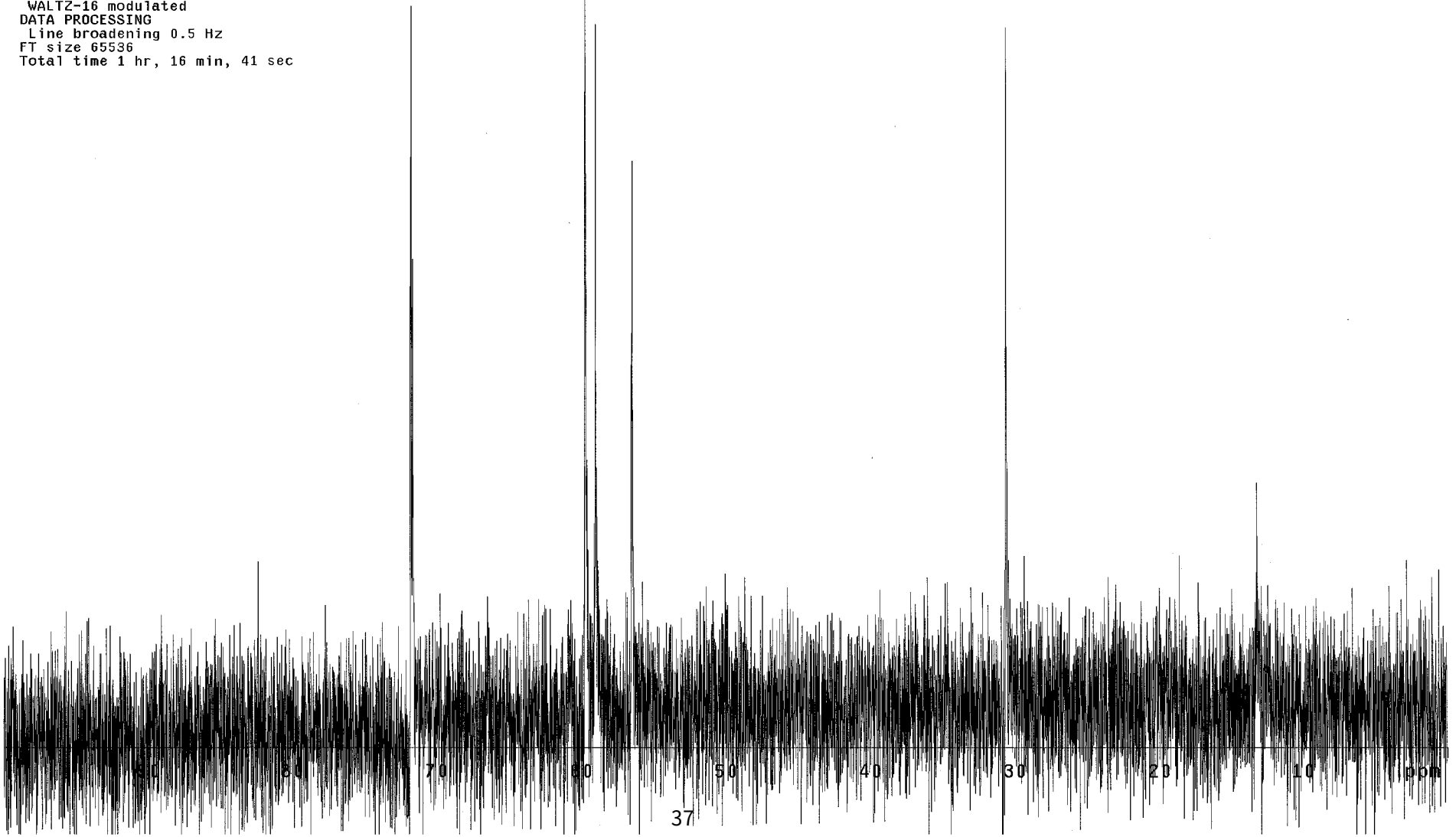

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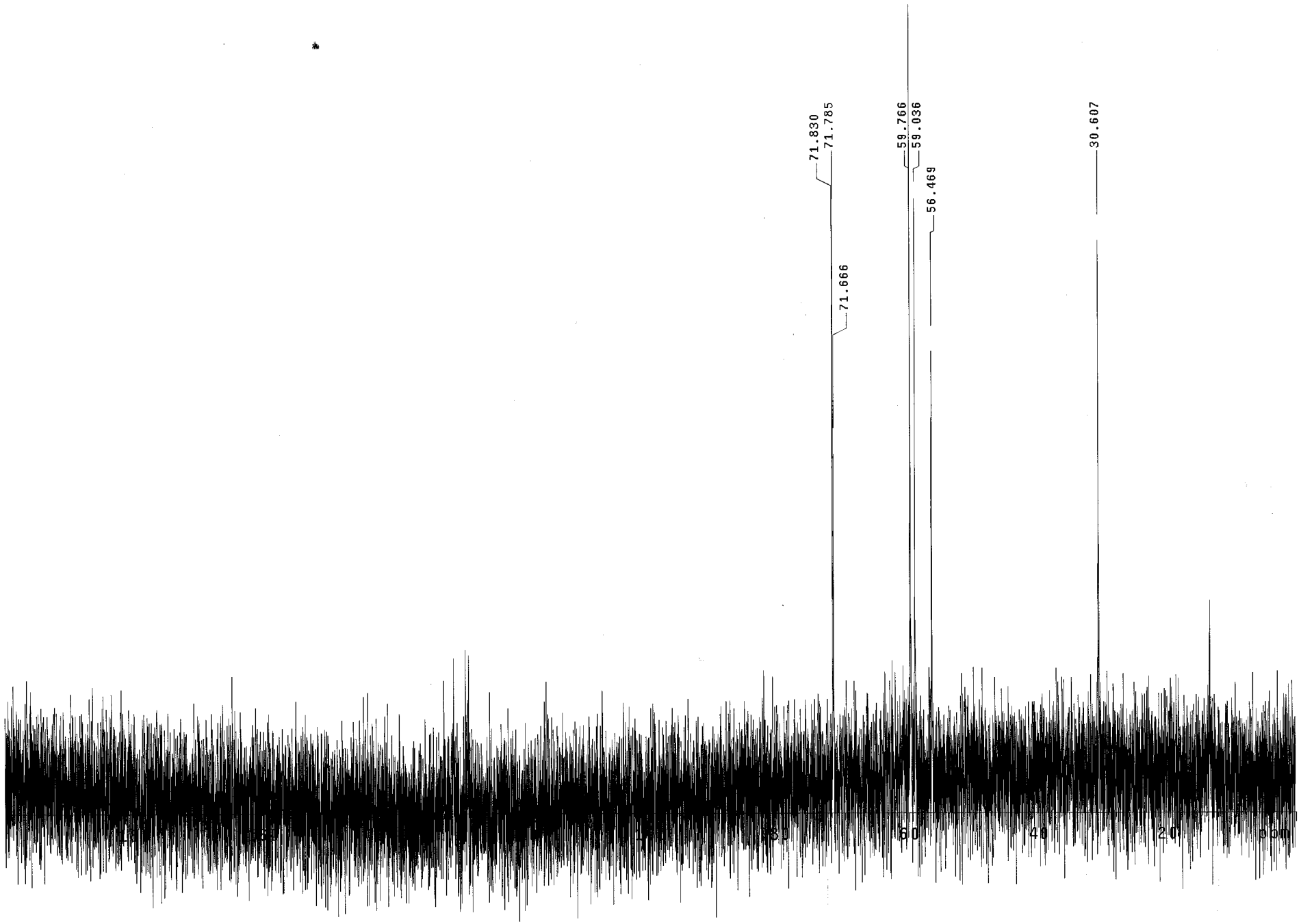

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Std proton

File: xp

Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
Operator: LWK
VNMRS-400 "Varian-NMR"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.049 sec
 Width 6410.3 Hz
 8 repetitions
OBSERVE H1, 399.7193691 MHz
DATA PROCESSING
 Resol. enhancement -0.0 Hz
FT size 65536
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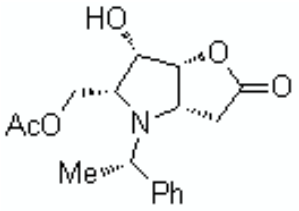

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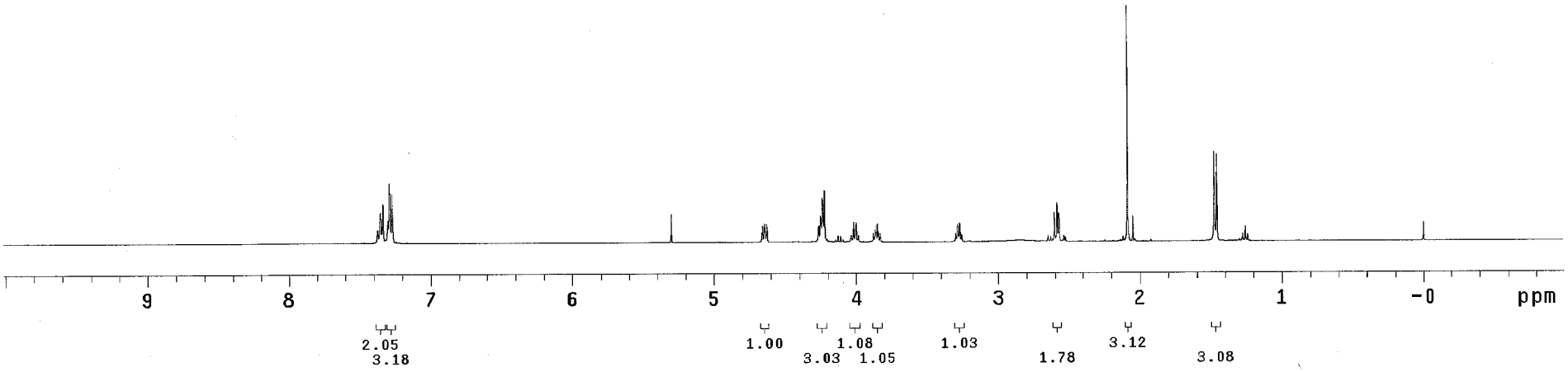

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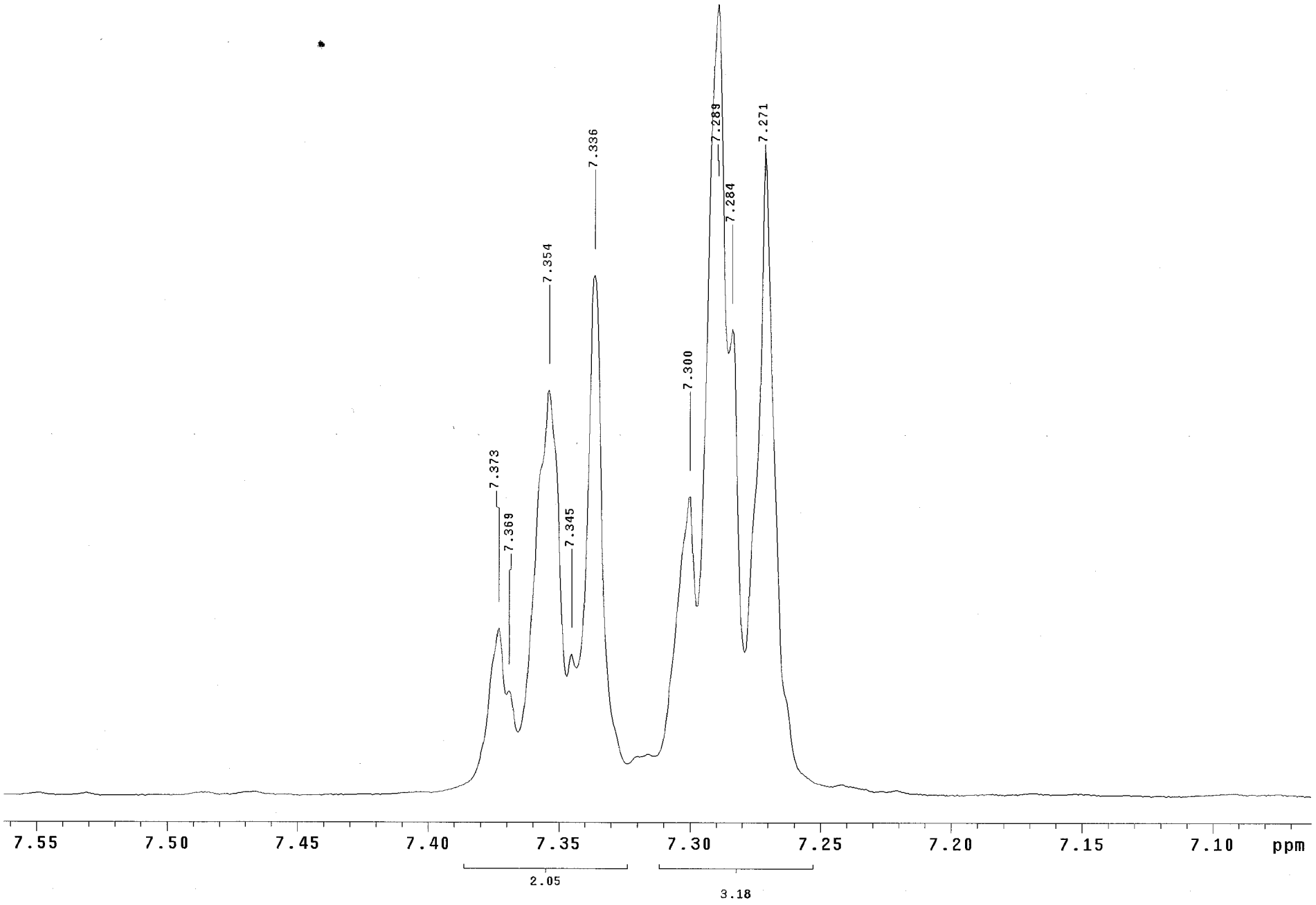

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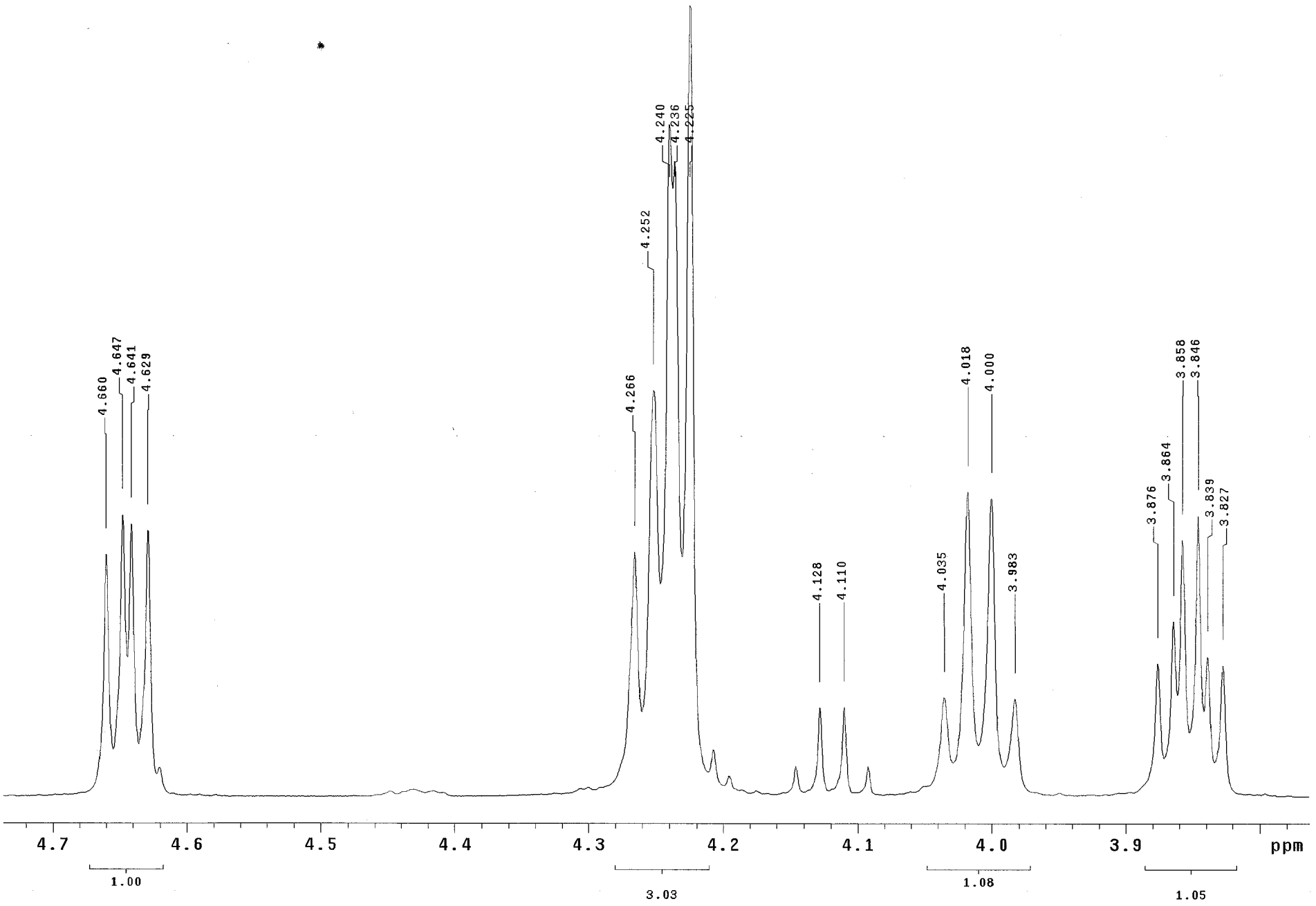

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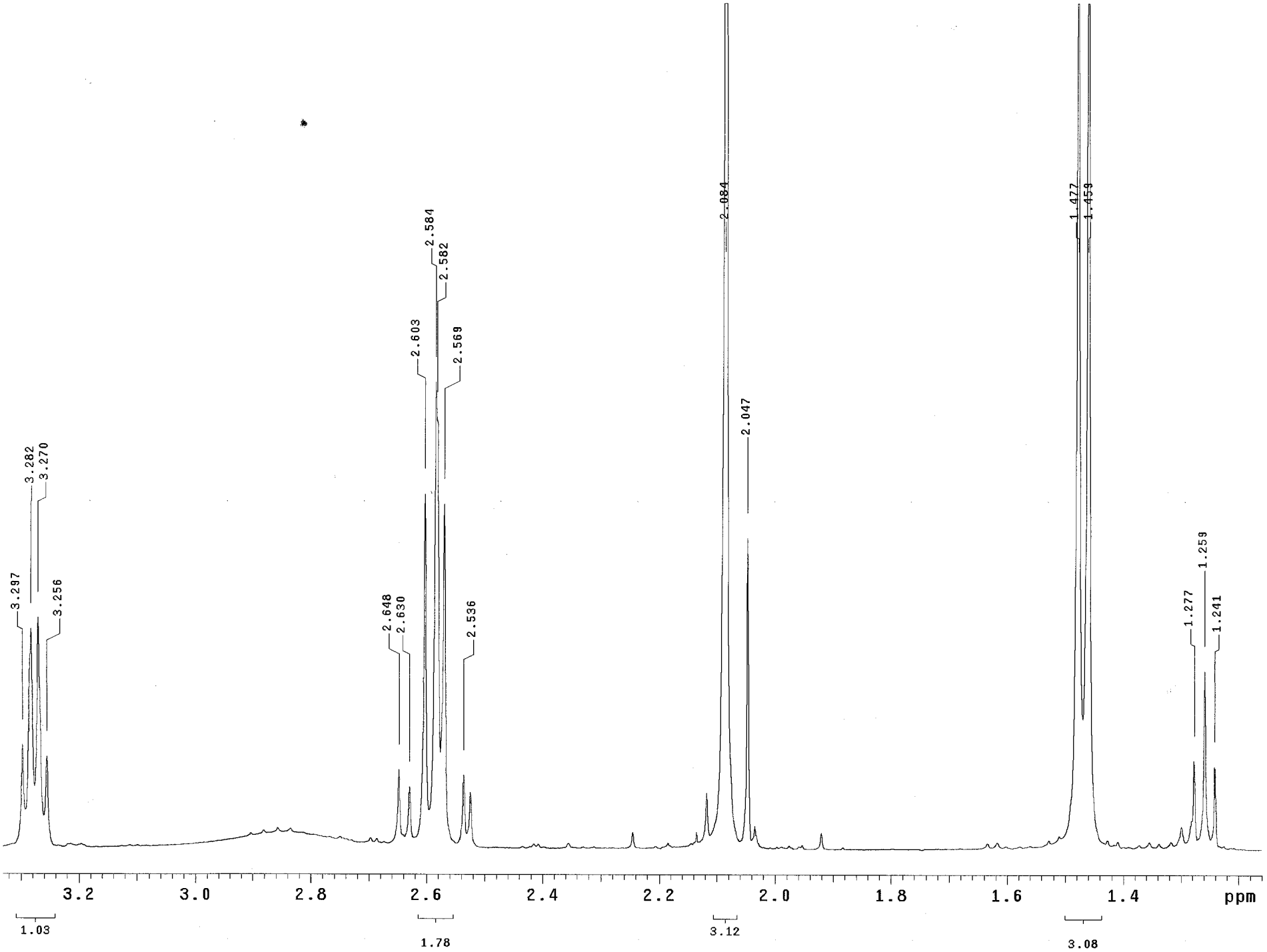

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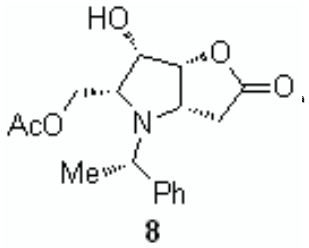

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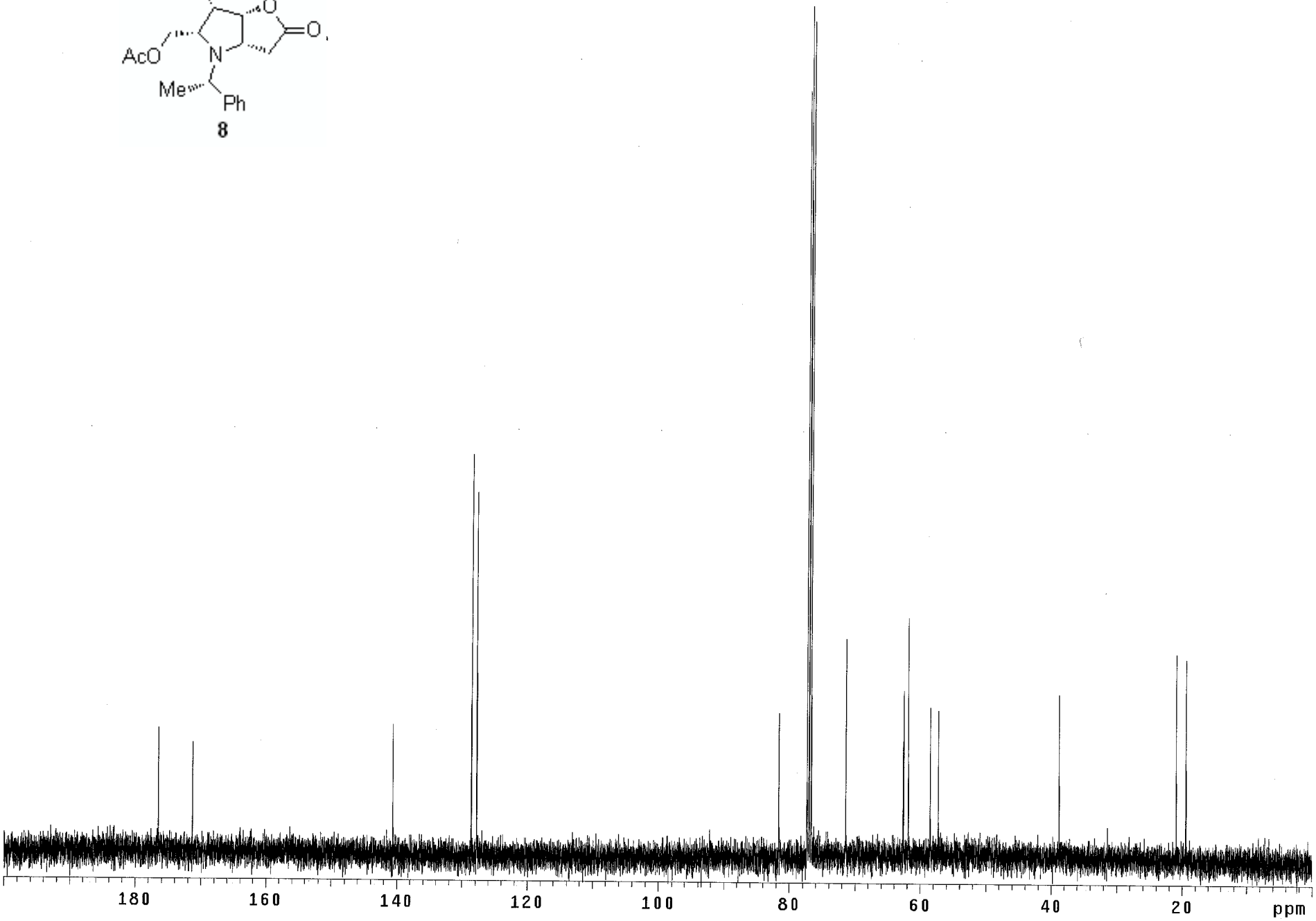

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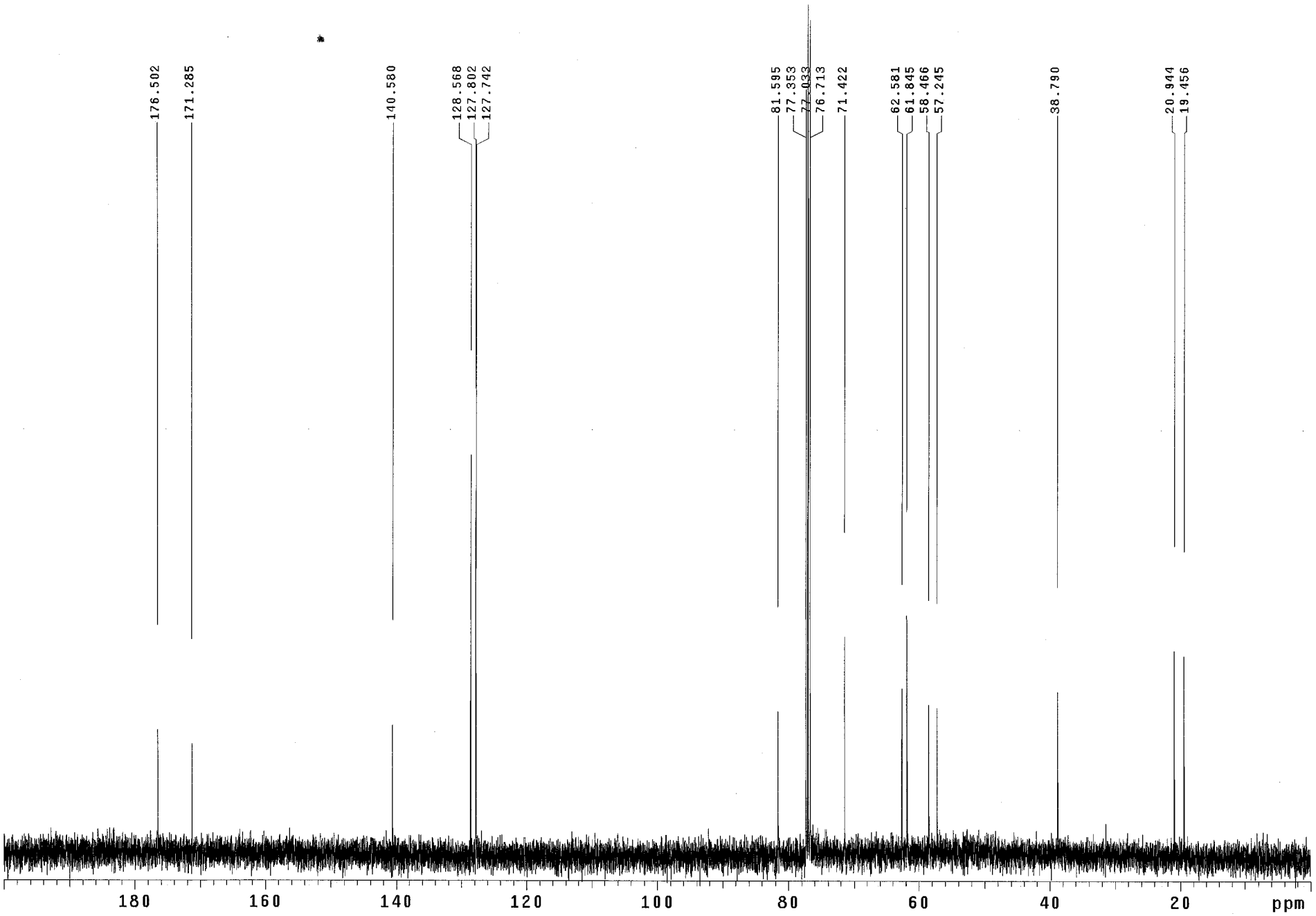

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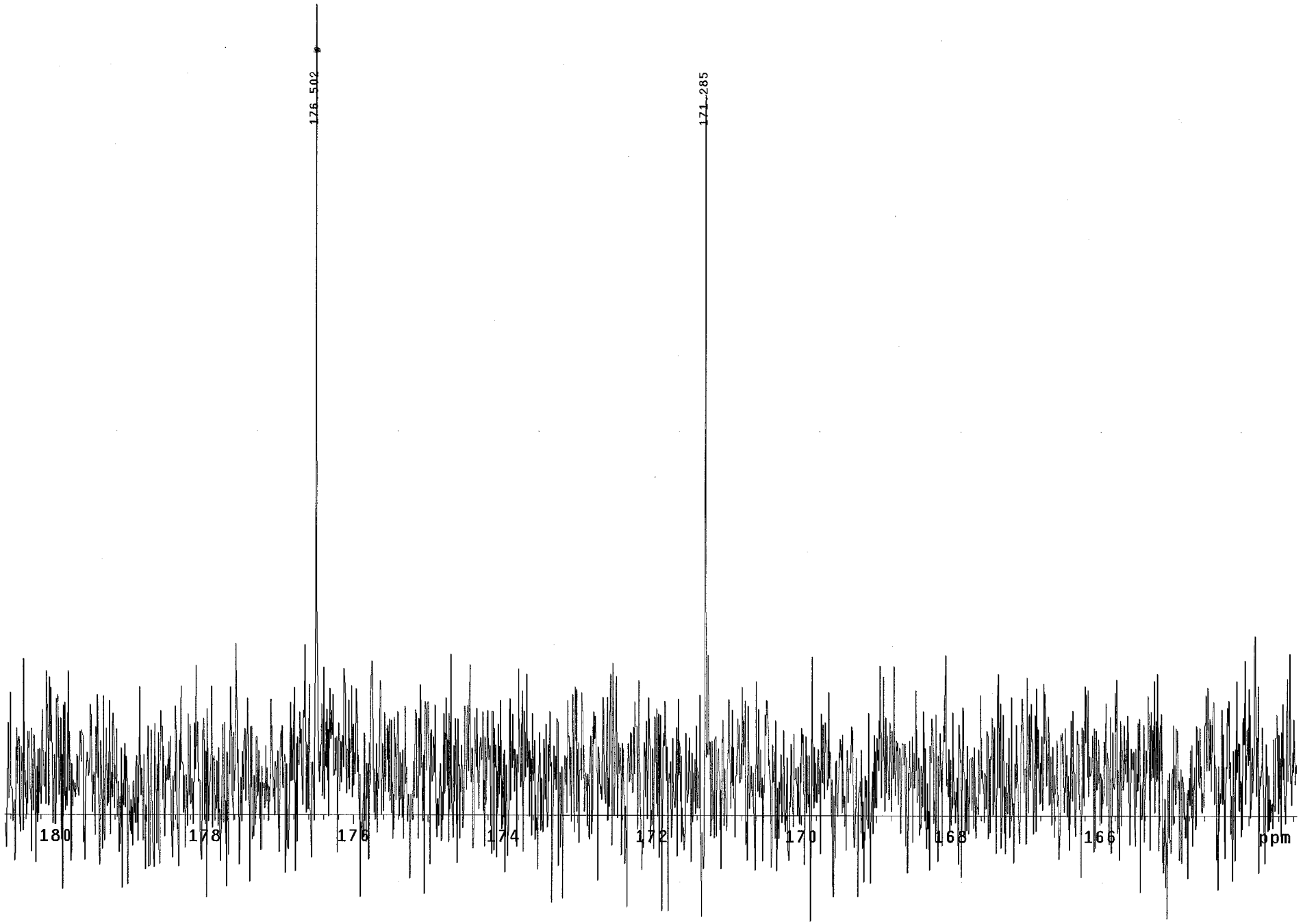

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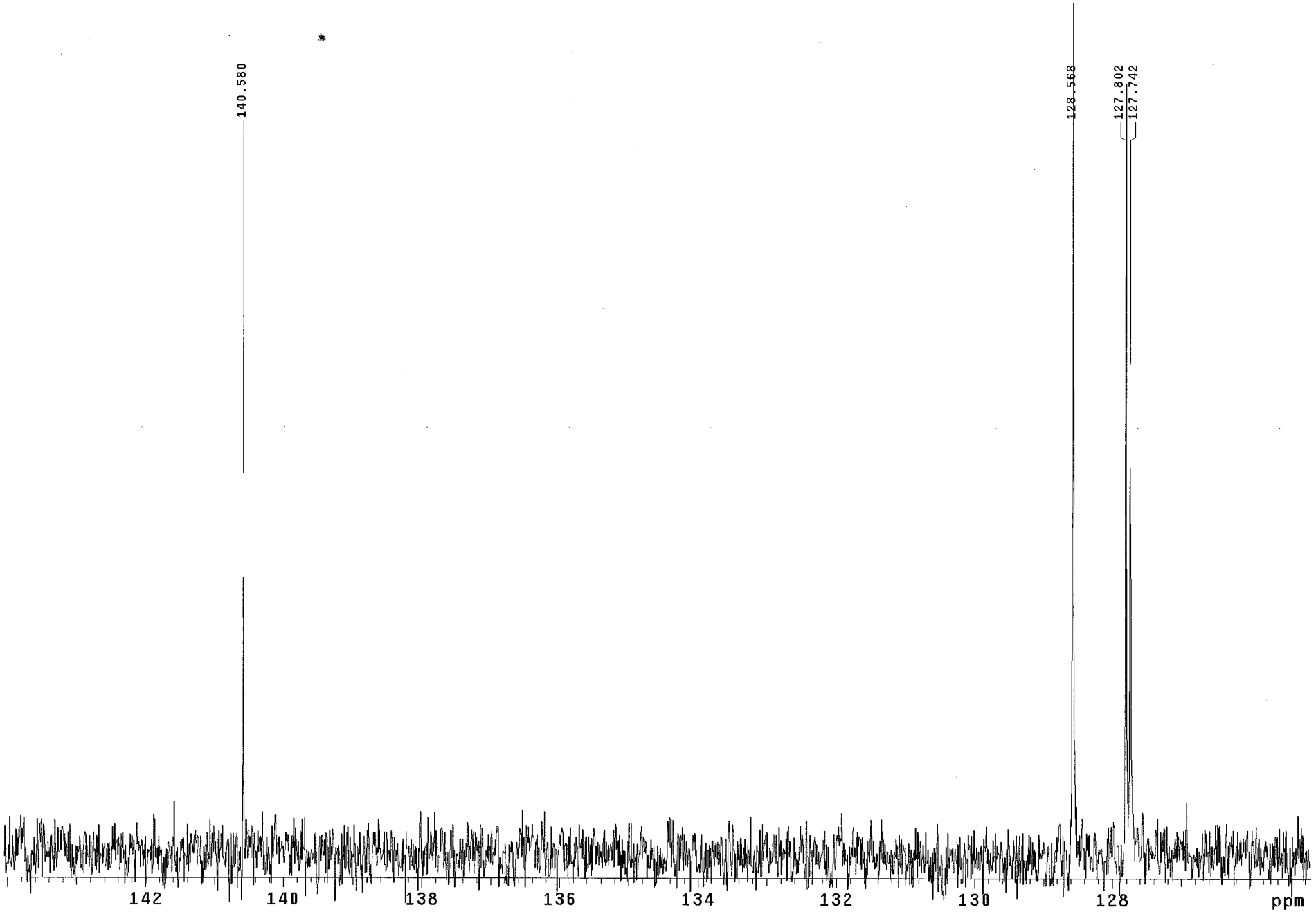

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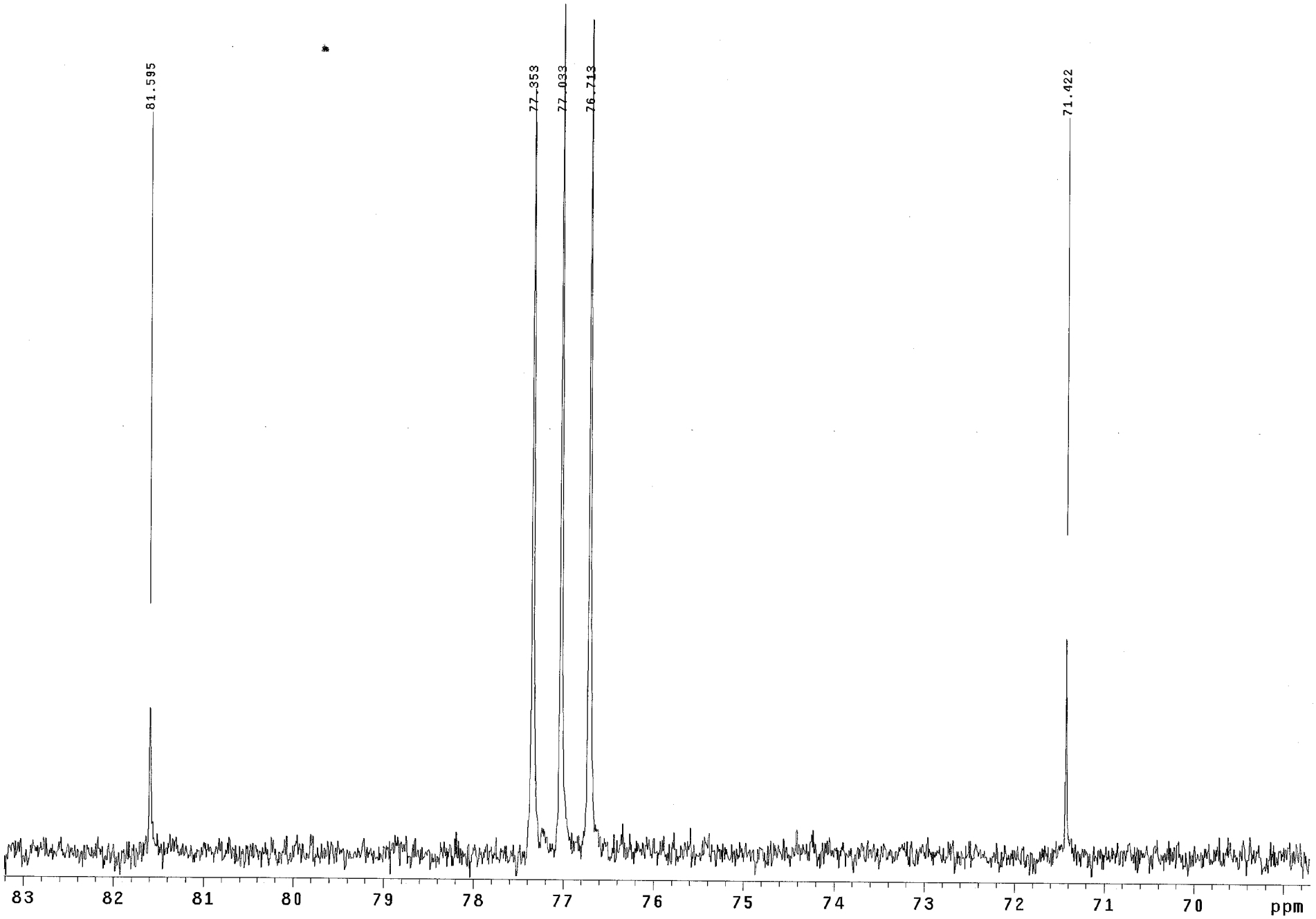

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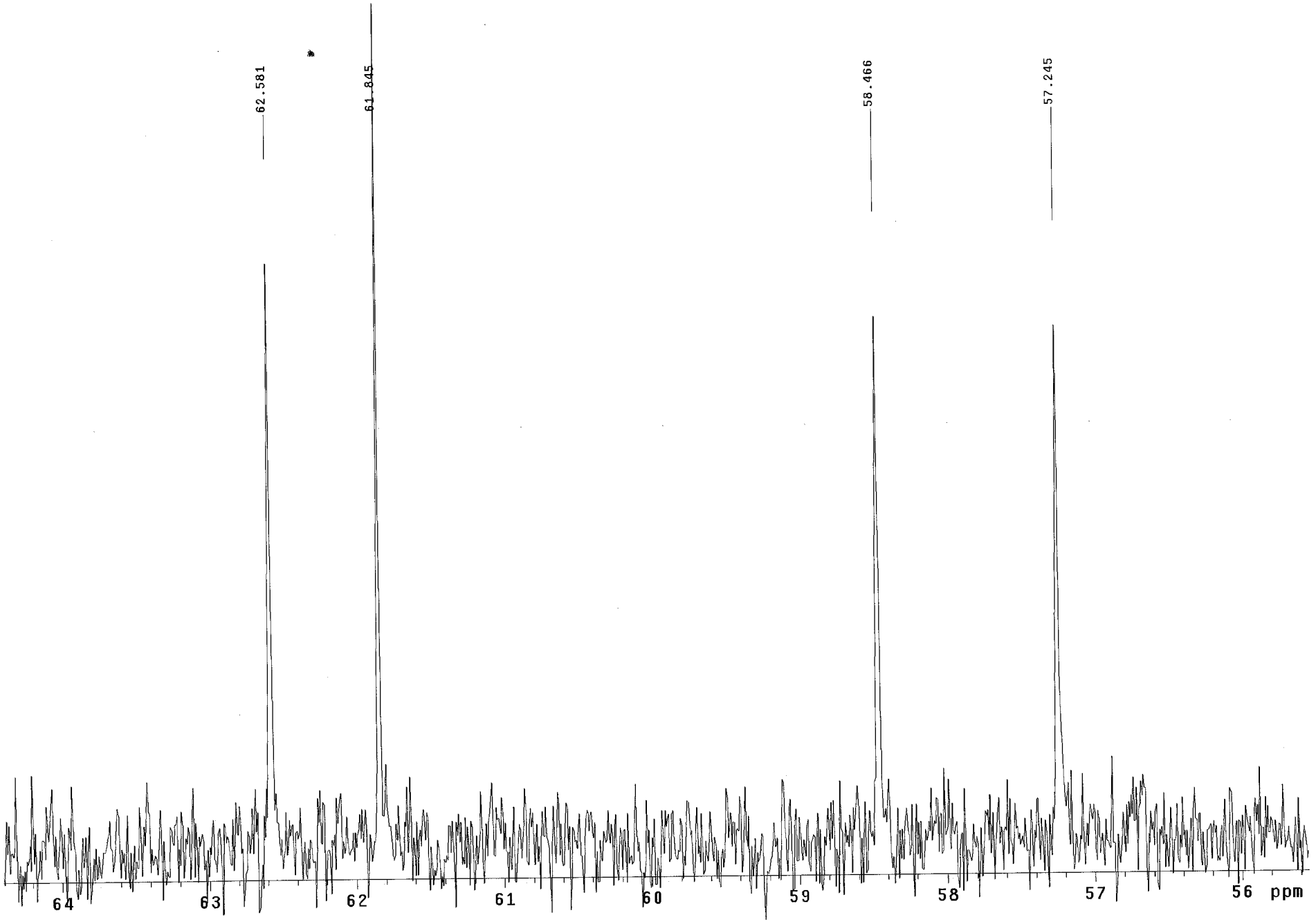

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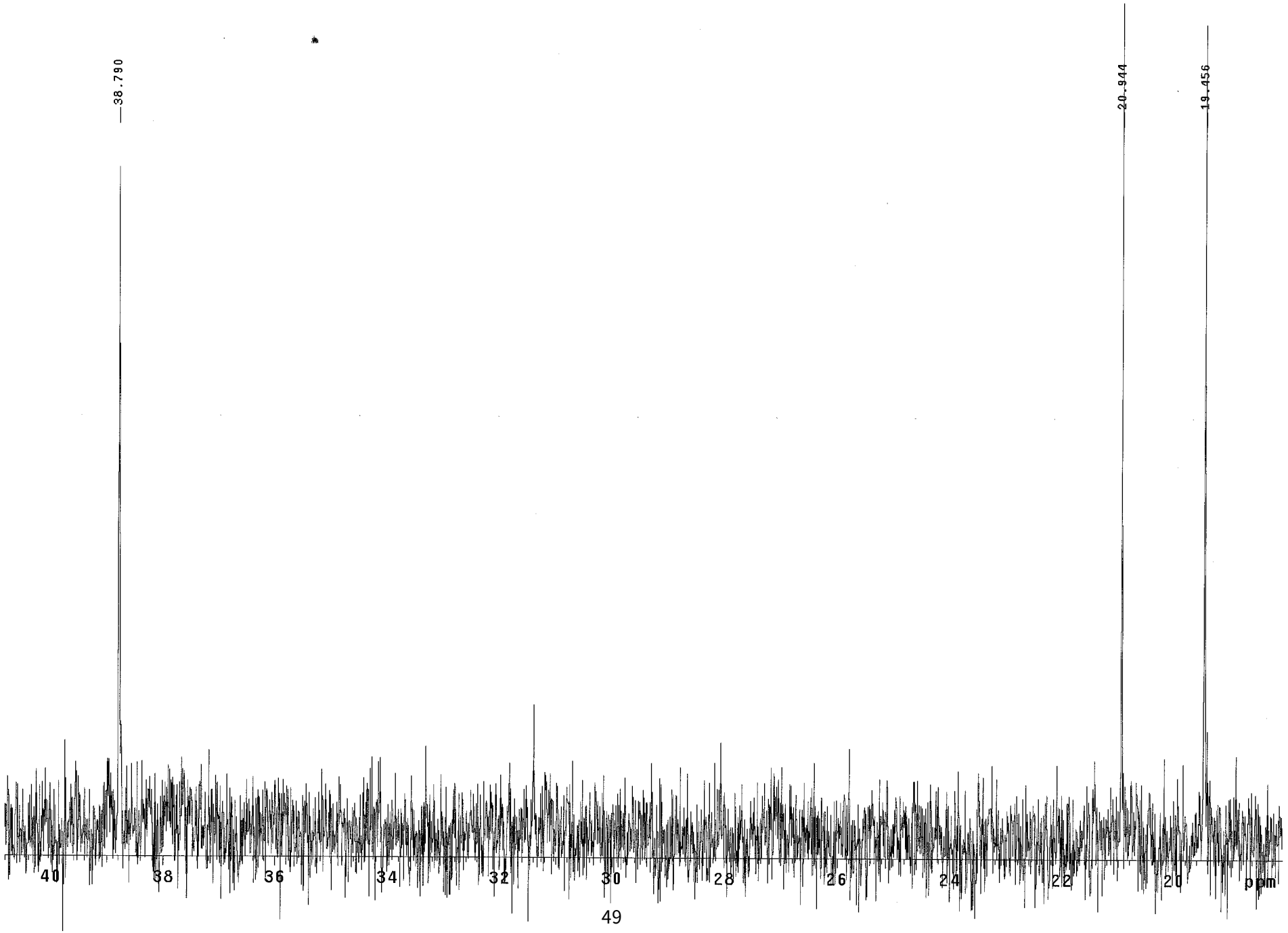

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Ambient temperature
Operator: LDH
VNMRS-400 "Varian-NMR"

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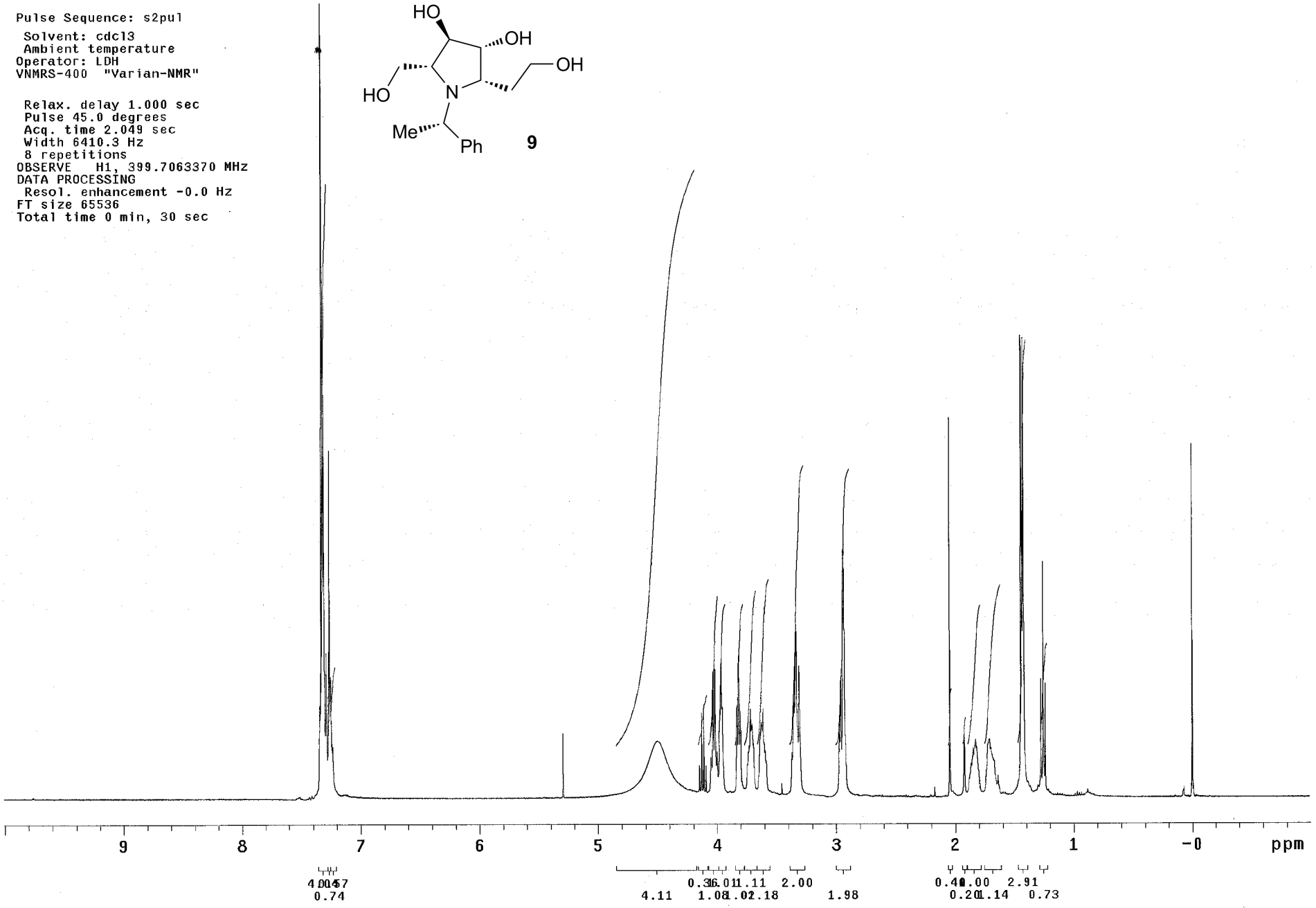

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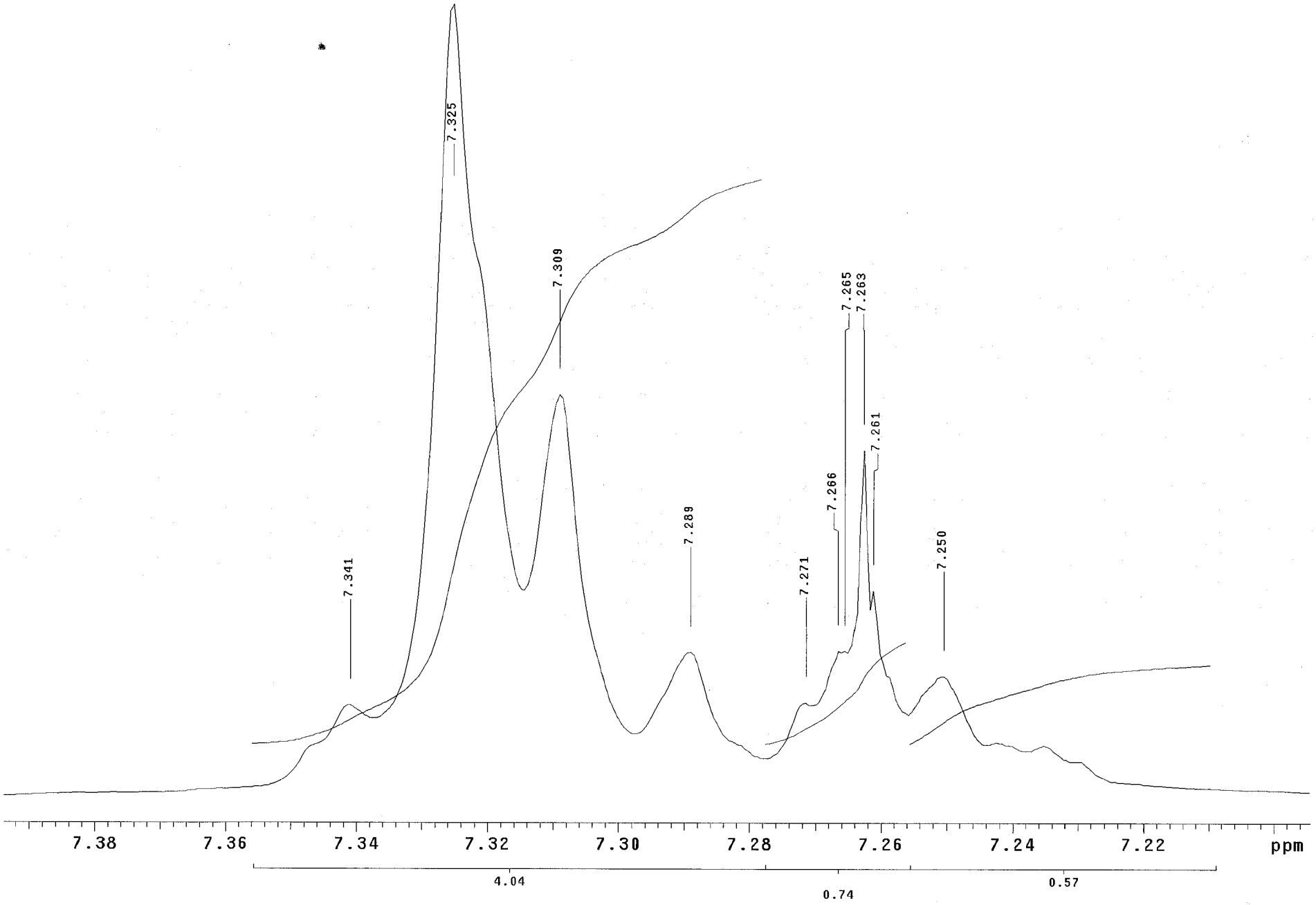

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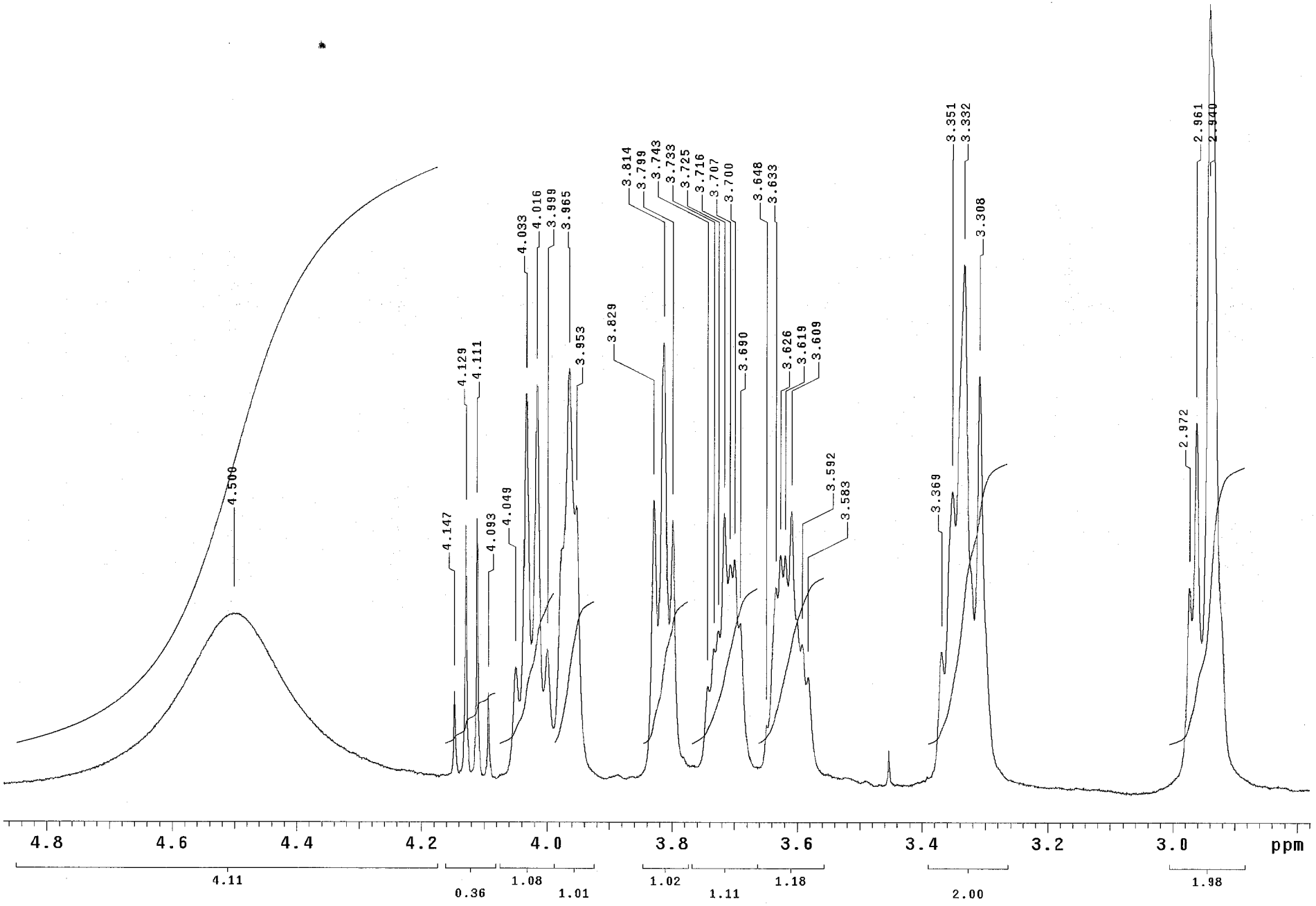

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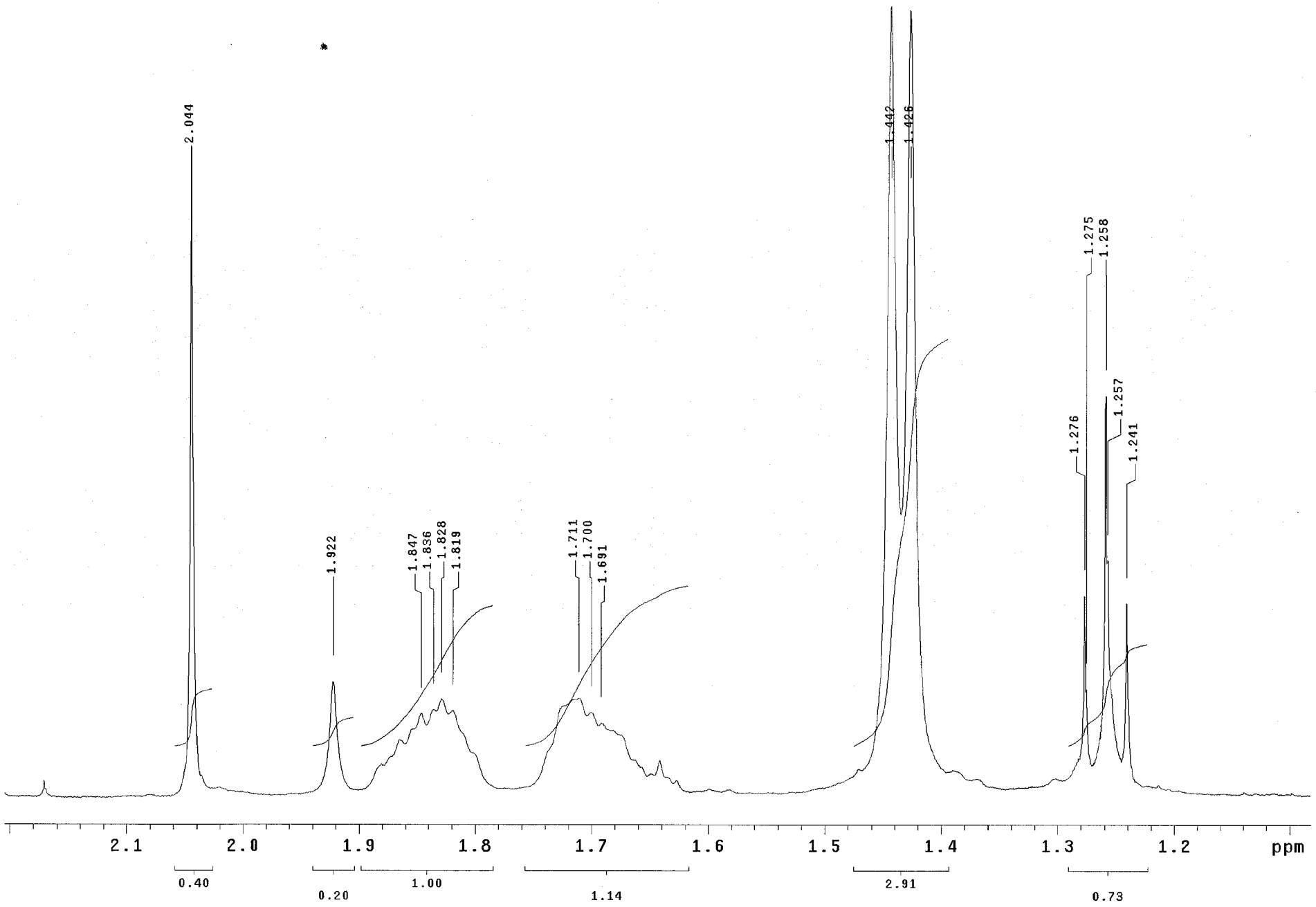

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Operator: LWK
VNMRS-400 "Varian-NMR"

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 460 repetitions
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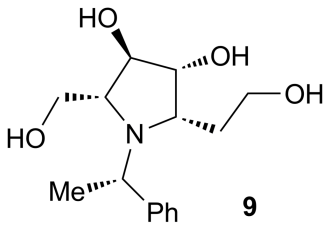

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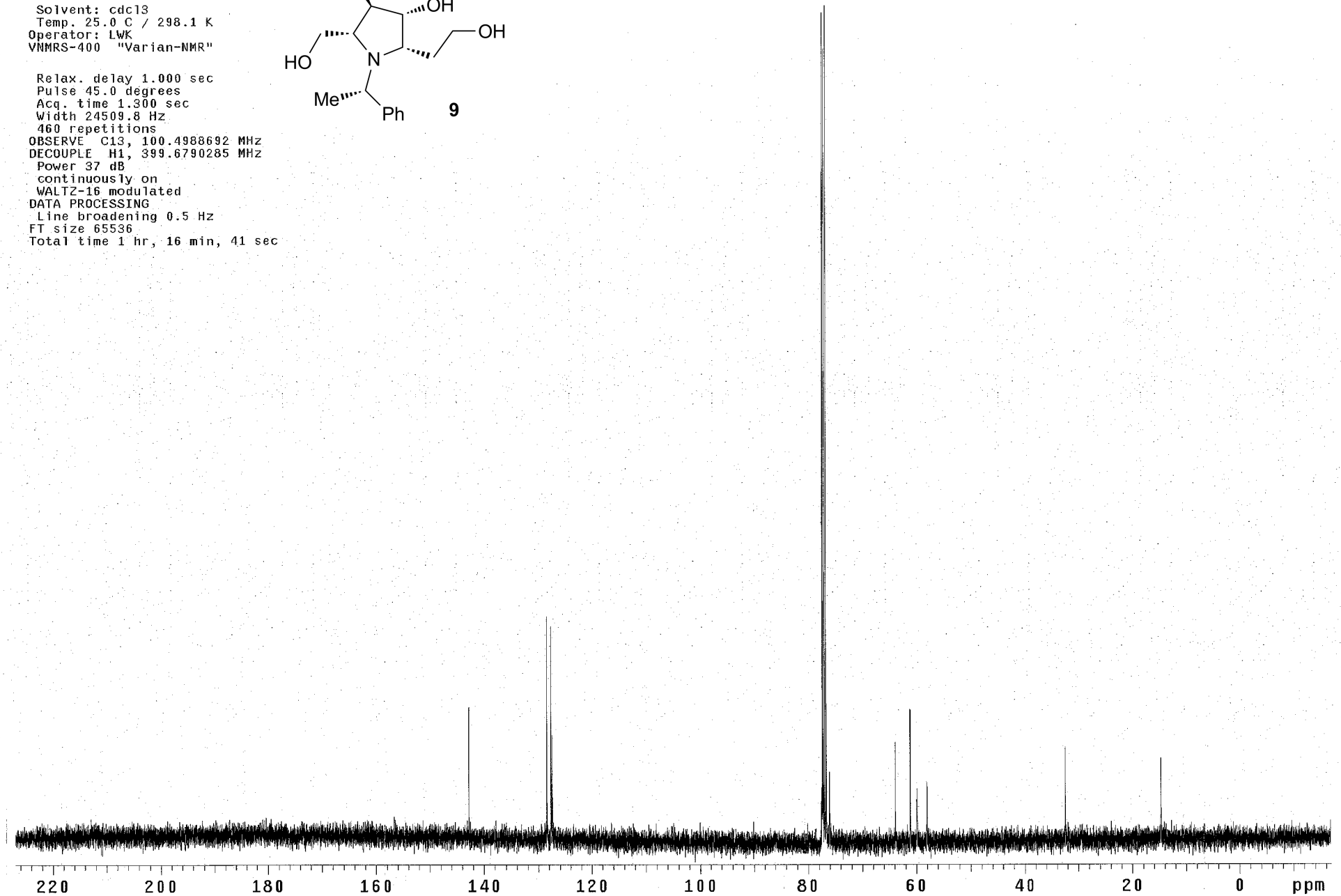

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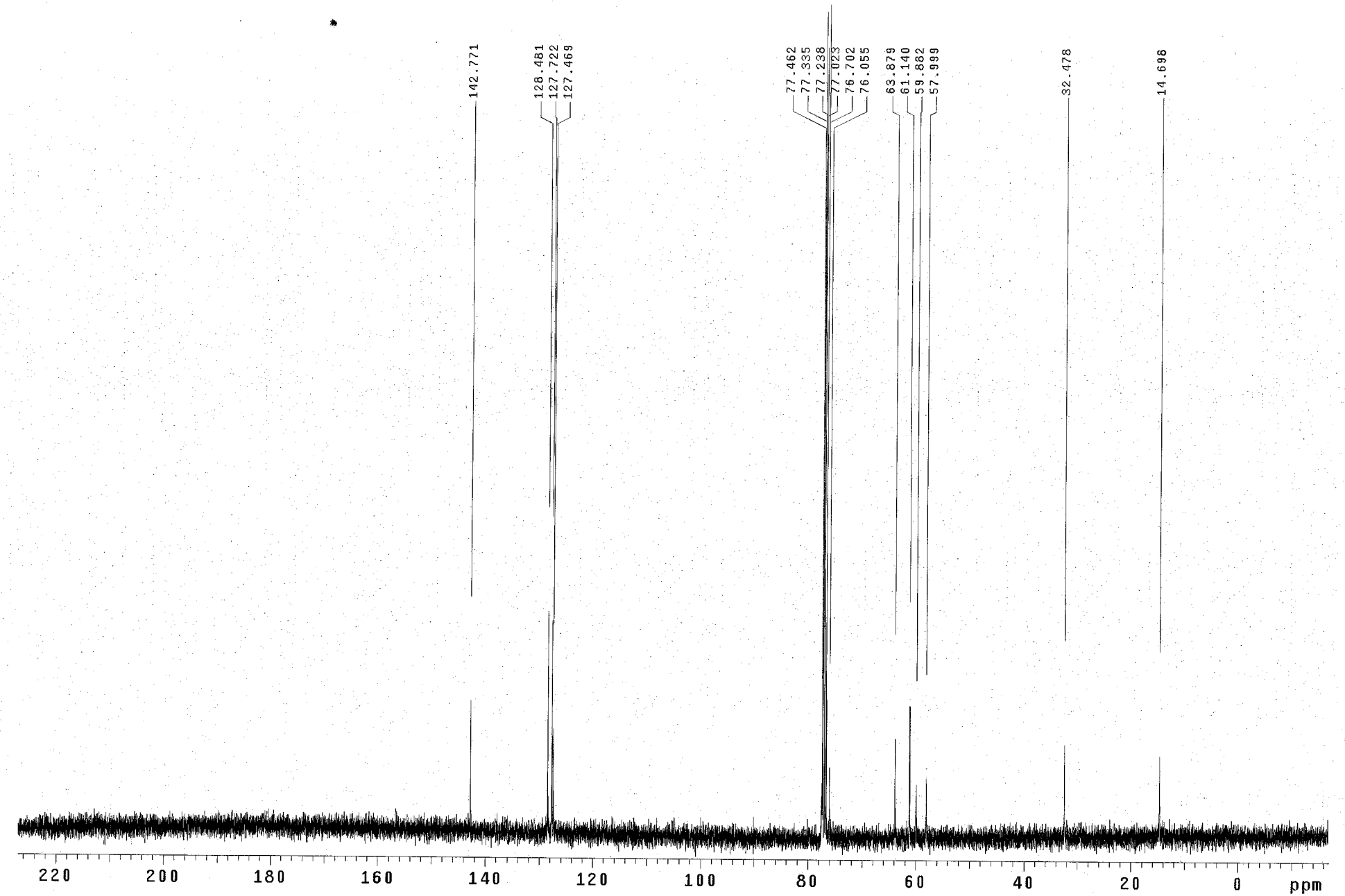

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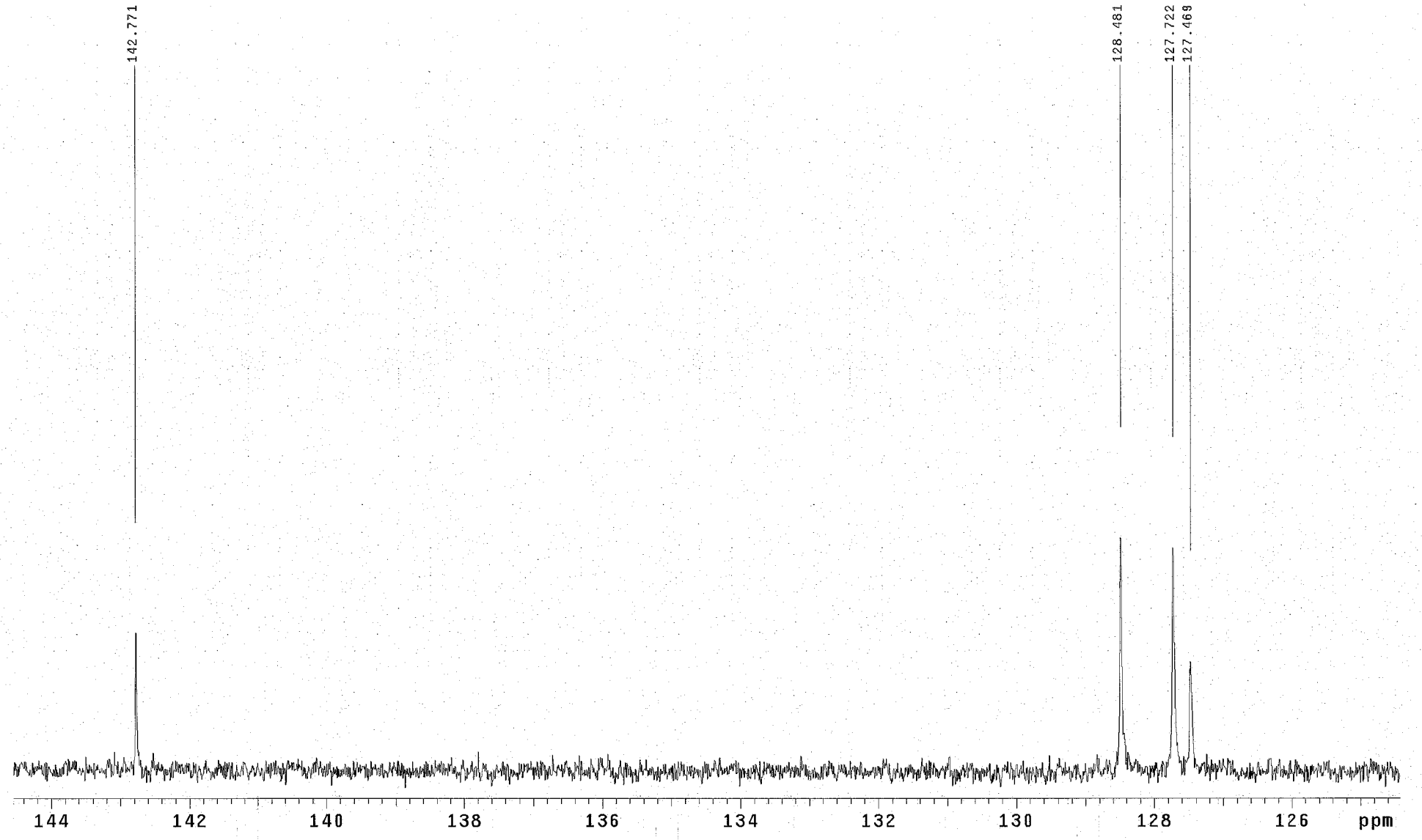

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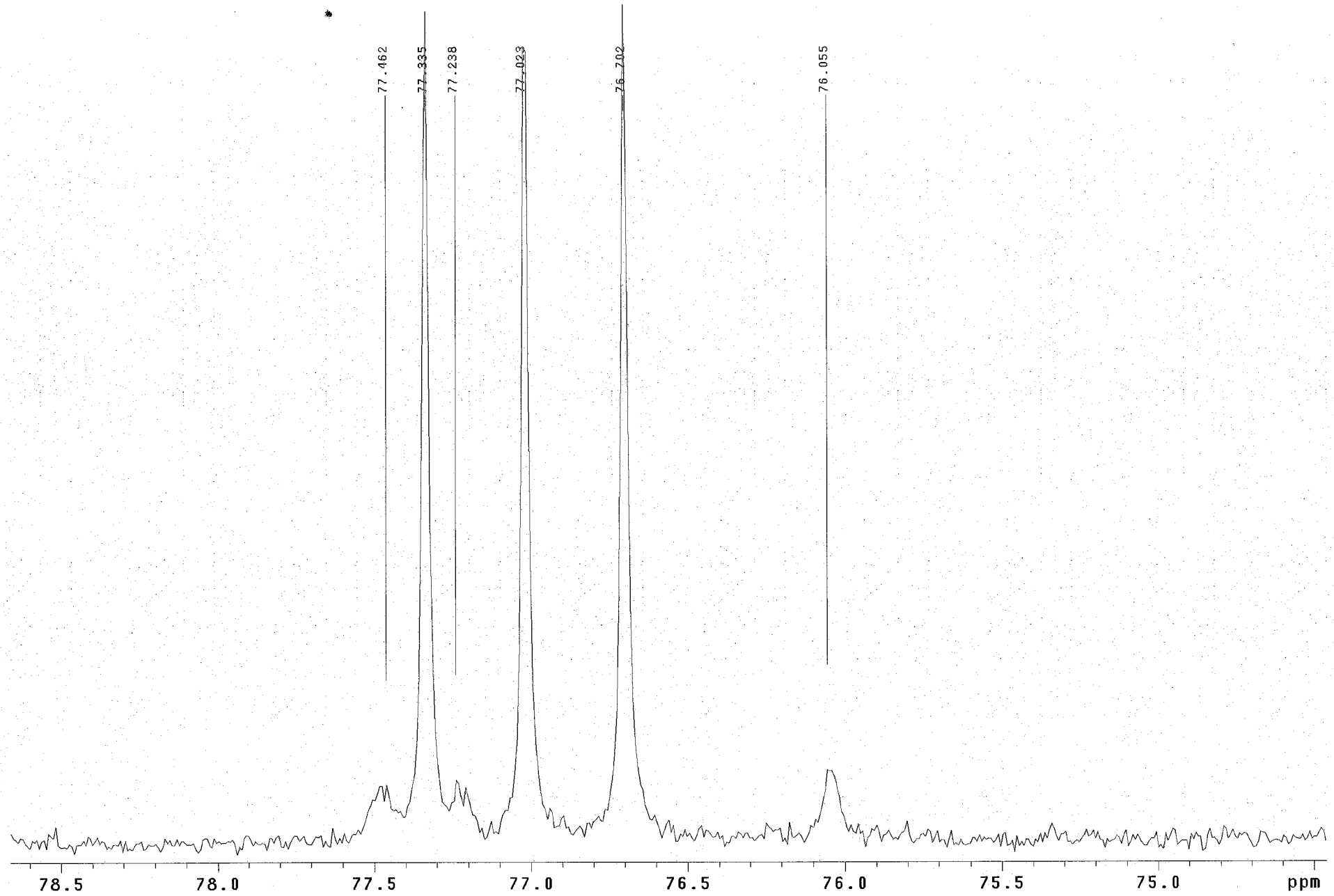

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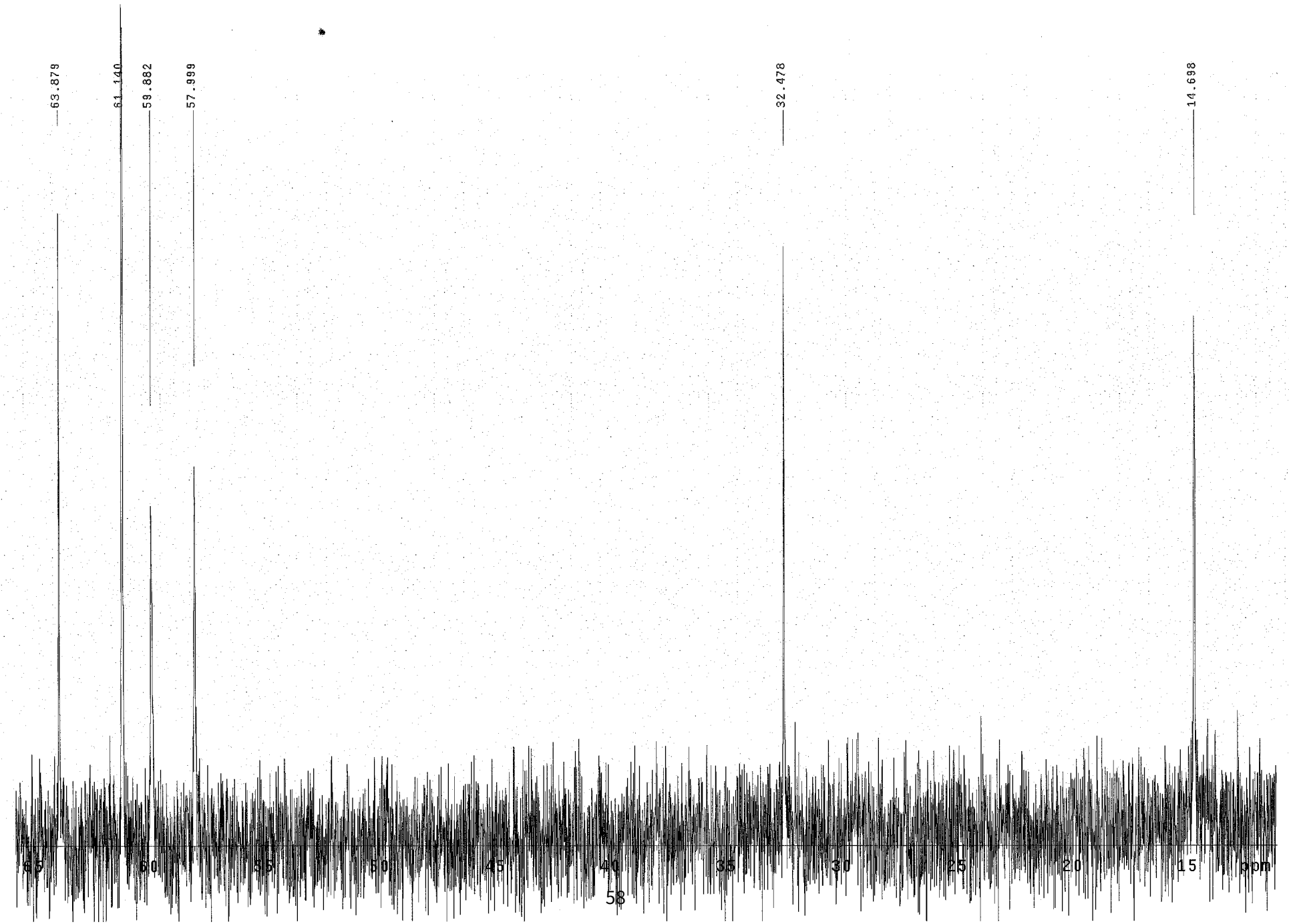

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Std proton

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 Ambient temperature
Operator: LWK
VNMRS-400 "Varian-NMR"

 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.049 sec
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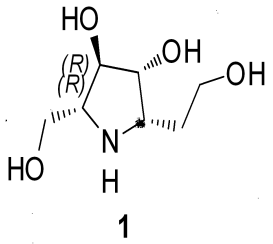

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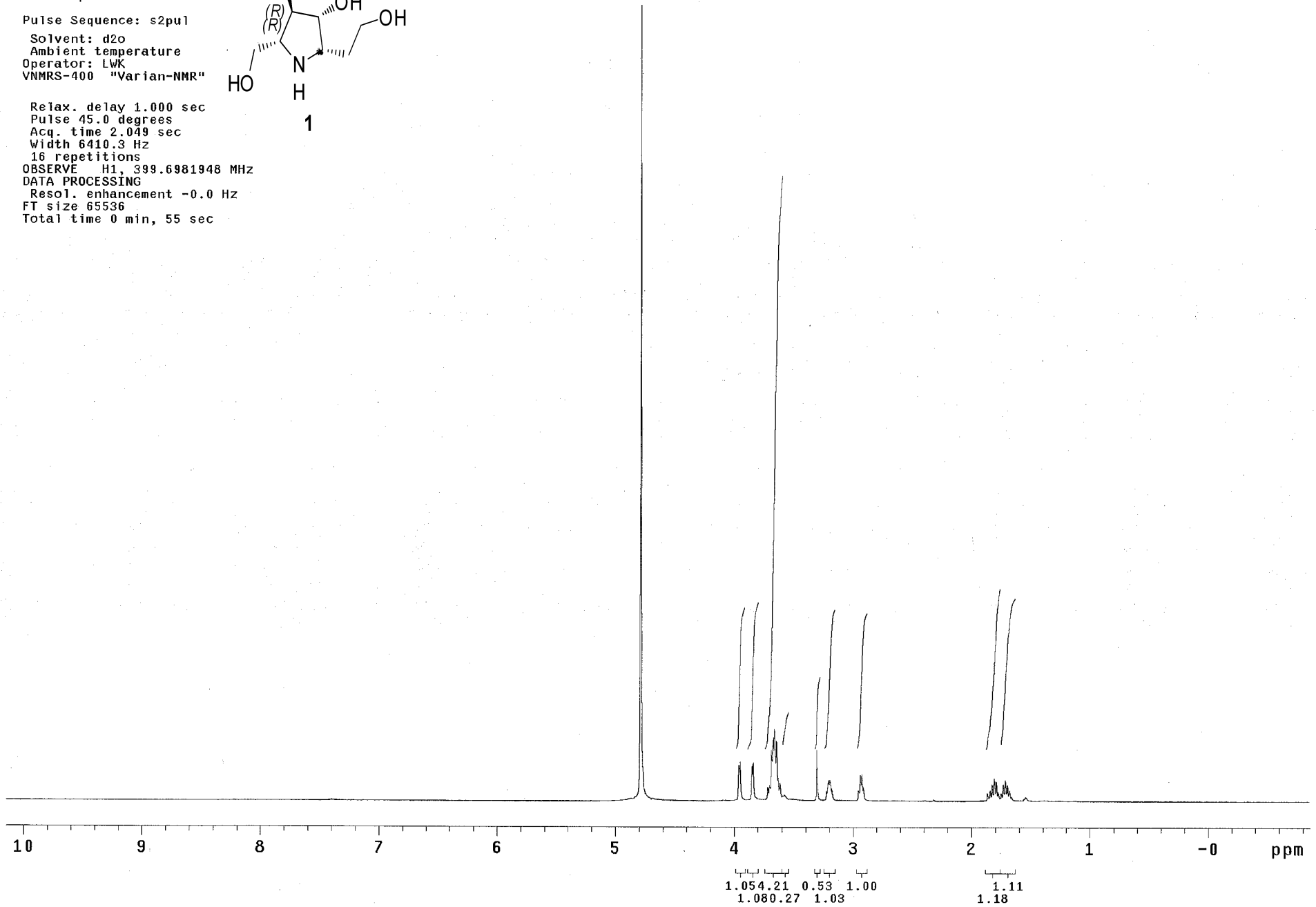

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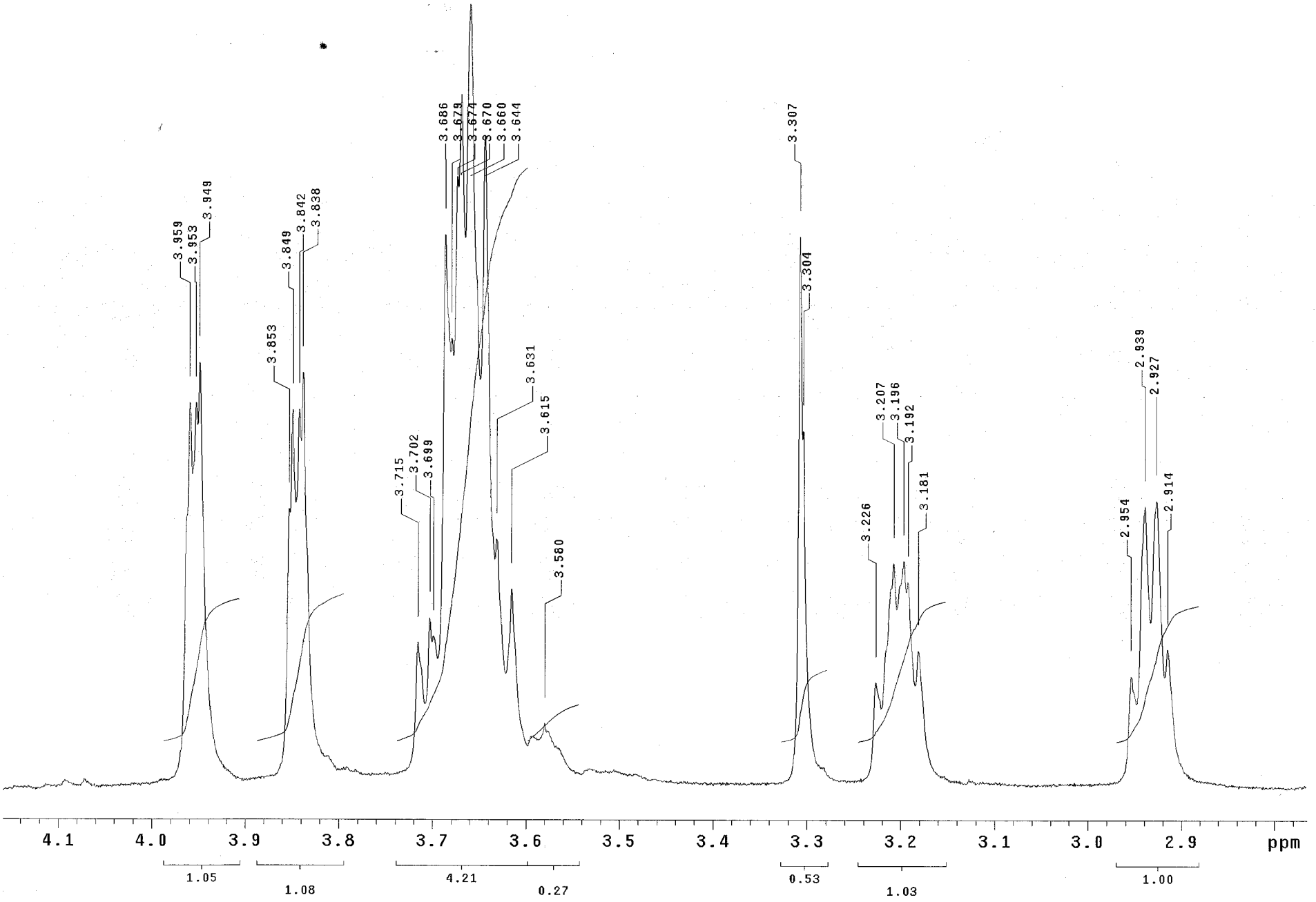

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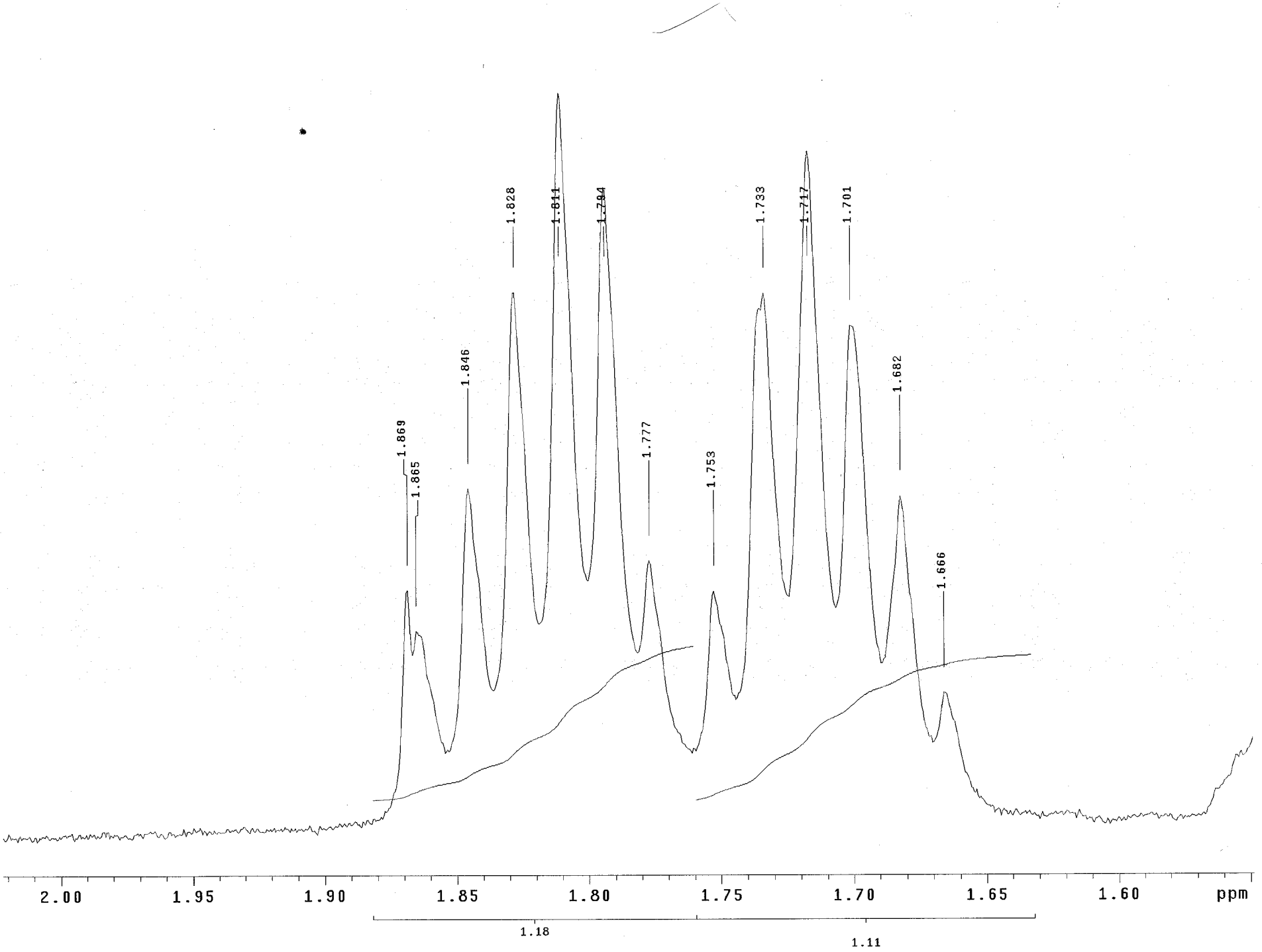

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Std carbon

File: xp

Pulse Sequence: s2pul
Solvent: d2o
Ambient temperature
Operator: LWK
VNMRS-400 "Varian-NMR"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
320 repetitions
OBSERVE C13, 100.5097747 MHz
DECOUPLE H1, 399.7223993 MHz
Power 37 dB
continuously on
WALTZ-16 modulated
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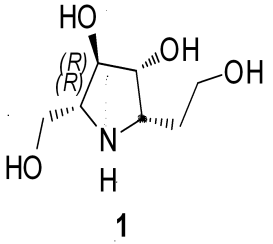

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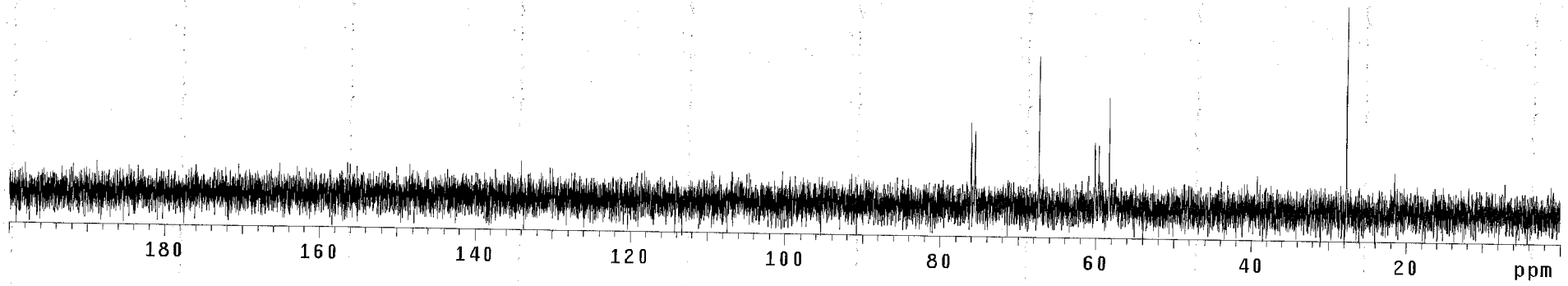

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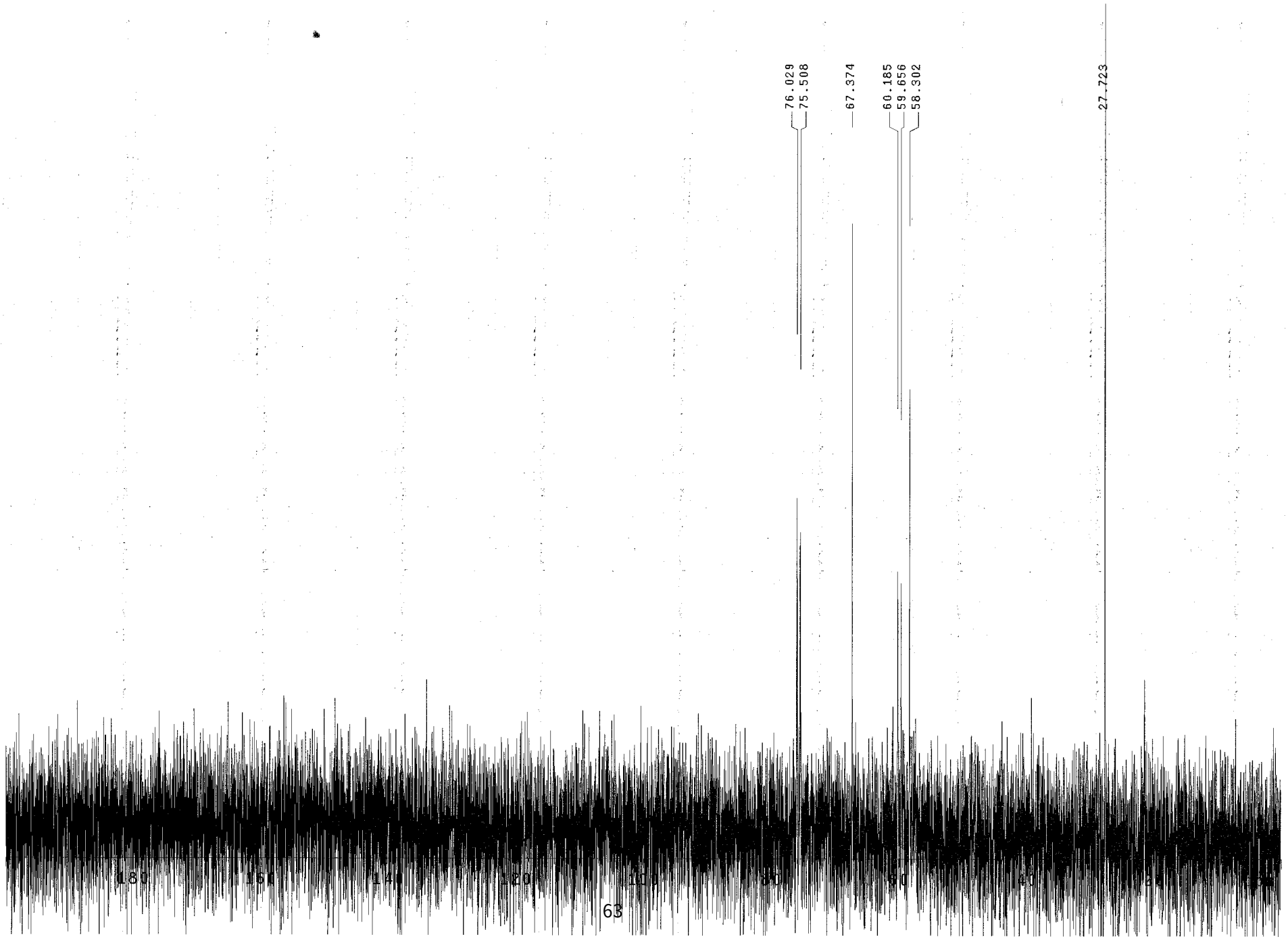

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2. X-Ray Crystal Structure of Compound **6**

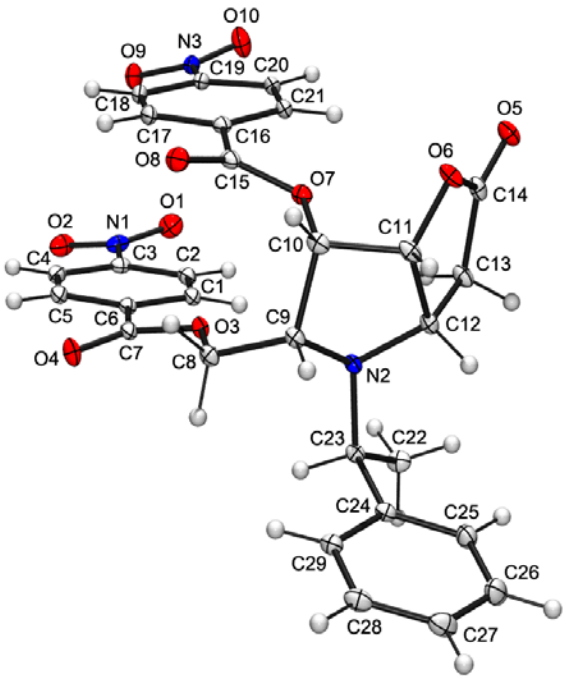

Figure 1. X-ray crystal structure of **6** with thermal ellipsoids drawn at the 30% probability level.

X-ray Crystallography. A single crystal of **6**, was picked from solutions by a nylon loop (Hampton Research Co.) on a hand made cooper plate mounted inside a liquid N_2 Dewar vessel at *ca.* –40 ºC and mounted on a goniometer head in a N_2 cryostream. Data collections were carried out on a Bruker SMART AXS diffractometer equipped with a monochromator in the Mo Kα (λ = 0.71073 Å) incident beam. The CCD data were integrated and scaled using the Bruker-SAINT software package, and the structure was solved and refined using SHEXTL V 6.12.[1] Hydrogen atoms were

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located in the calculated positions. Crystal data for **6**: $C_{29}H_{25}N_3O_{10}$, Monoclinic, *P2(1)/c*, Z = 4, a = 9.4026(2), b = 32.1434(7), c = 9.1405(2) Å, β = 110.9000(10) °, V = 2580.78(10) Å^3, μ = 0.114 mm^{-1}, ρ_{calcd} = 1.481 g/cm^3, R_1 = 0.0357, wR_2 = 0.0912 for 6427 unique reflections, 380 variables. The crystallographic data for **6** are listed in Table 1, and Tables 2 lists the selected bond distances and angles. CCDC-902981 for **6** contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

References

[1] G. M. Sheldrick, *SHELXTL/PC Version 6.12 for Windows XP*, Bruker AXS Inc., Madison, WI, 2001.

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Table 1. Crystal data and structure refinements for **6**.

	6
Empirical formula	$C_{29}H_{25}N_3O_{10}$
Formula weight	575.52
Temperature (K)	100(2)
Wavelength (Å)	0.71073
Crystal system/space group	Monoclinic, *P2(1)/c*
Unit cell dimensions	
a (Å)	9.4026(2)
b (Å)	32.1434(7)
c (Å)	9.1405(2)
α (°)	90
β (°)	110.9000(10)
γ (°)	90
Volume (Å^3)	2580.78(10)
Z	4
Calculated density (g/cm^{-3})	1.481
Absorption coefficient (mm^{-1})	0.114
Reflections collected	47551
Independent reflections [*R*(int)]	6427 [0.0309]
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	6427/0/380
Goodness-of-fit on F^2	1.014
Final *R* indices [$I > 2\text{sigma}(I)$]	$R_1 = 0.0357$, $wR_2 = 0.0912$
R indices (all data)	$R_1 = 0.0427$, $wR_2 = 0.0964$
Largest difference peak and hole (e/Å^3)	0.368 and –0.257

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Table 2. Selected bond distances (Å) and angles (º) for **6**.

Bond Distances (Å)	
N2-C23	1.4793(13)
N2-C9	1.4708(13)
N2-C12	1.4699(13)
C9-C8	1.5143(15)
C9-C10	1.5292(15)
C10-O7	1.4460(13)
C10-C11	1.5249(16)
C11-C12	1.5417(15)
C11-O6	1.4359(13)
O6-C14	1.3599(13)
C14-O5	1.2003(14)
C14-C13	1.5074(16)
C13-C12	1.5321(15)
Bond Angles (°)	
C12-N2-C9	107.84(8)
N2-C9-C10	102.02(8)
C9-C10-C11	100.23(9)
C10-C11-C12	104.87(8)
C12-C11-O6	107.77(9)
C11-O6-C14	110.86(8)
O6-C14-C13	110.77(9)
C14-C13-C12	105.31(9)