

ESI for

Chemoselective alcoholysis/acetolysis of *trans*-ketals over *cis*-ketals: Total synthesis of the cellular second messenger, *D*-*myo*-inositol-1,4,5-trisphosphate

Adiyala Vidyasagar, Atchutarao Pathigoola and Kana M. Sureshan*

School of Chemistry,

Indian Institute of Science Education and Research, Thiruvananthapuram,

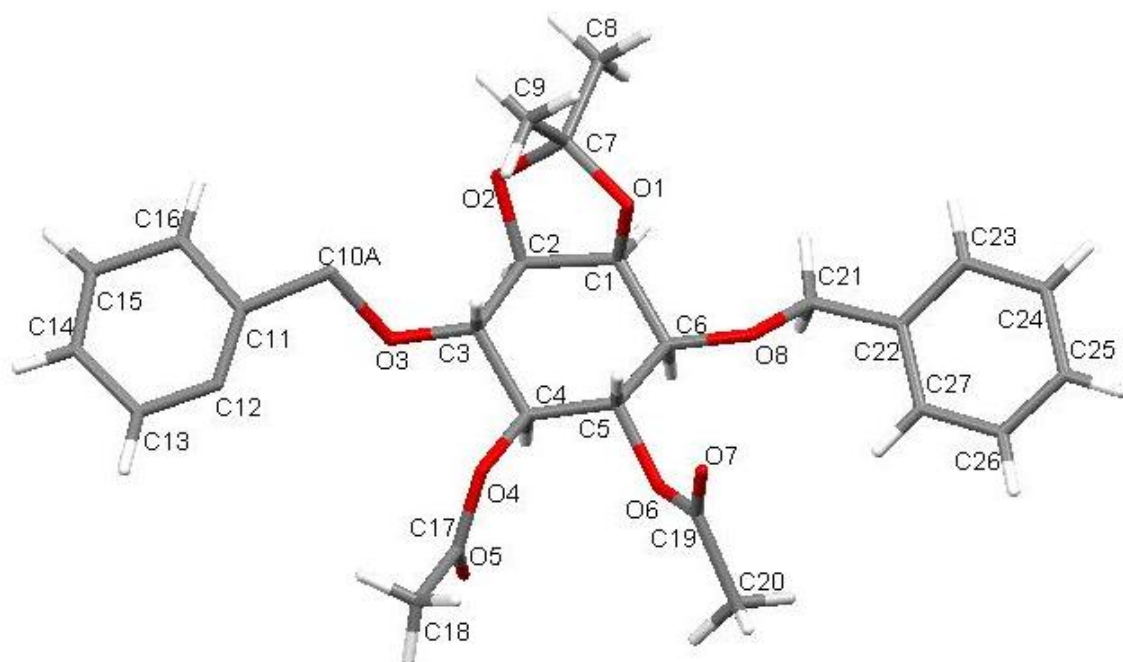
Thiruvananthapuram – 695016, India.

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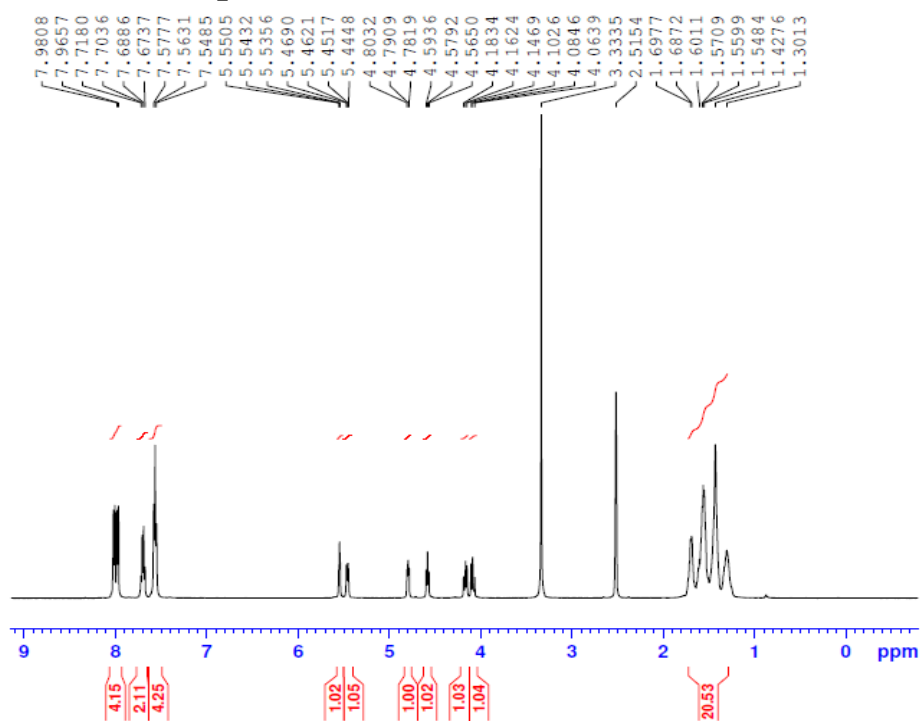
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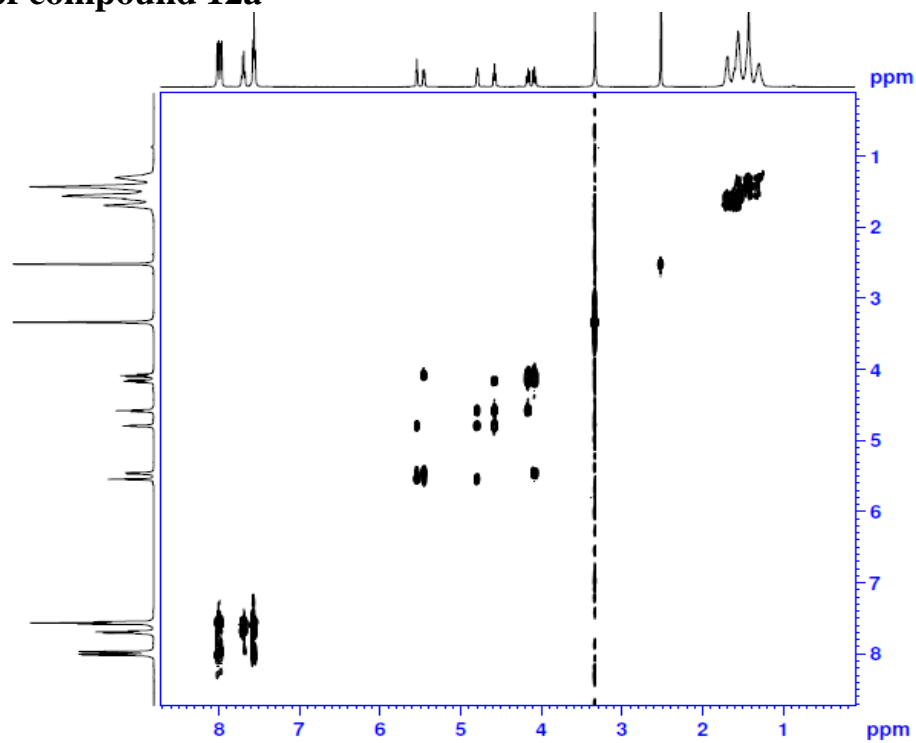
Crystal structure of 24a



¹H NMR of compound 12a

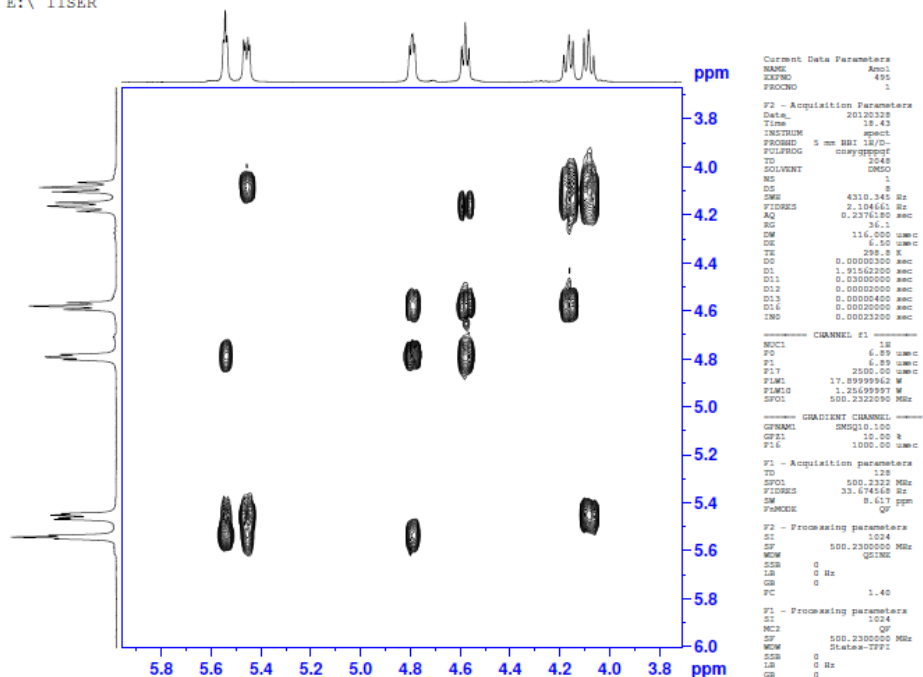


COSY of compound 12a



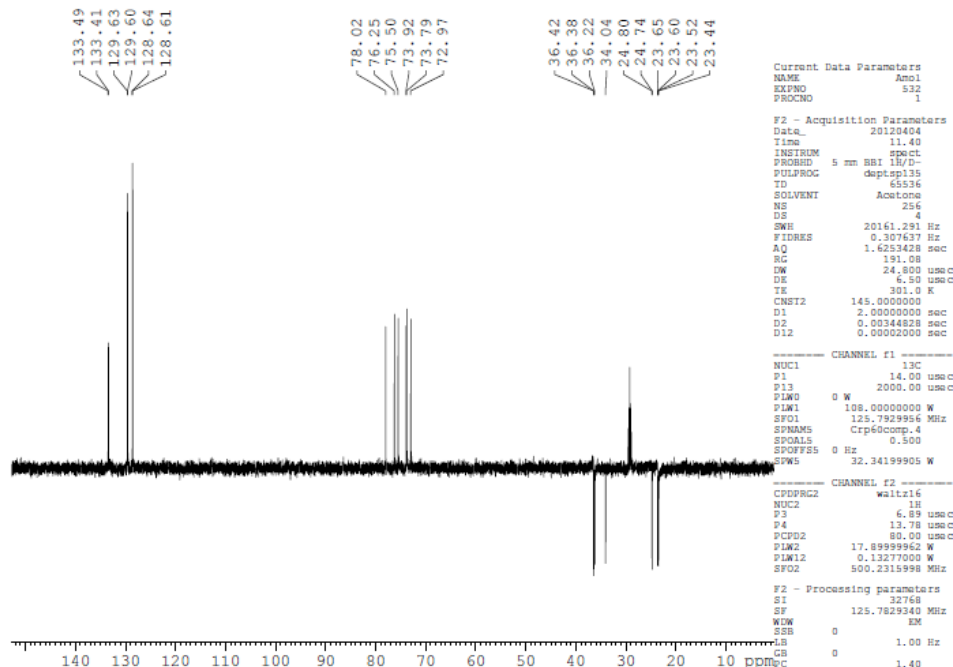
COSY of compound 12a(zoom)

KMS-B-V-29-DMSO
COSYGPW E:\ IISER



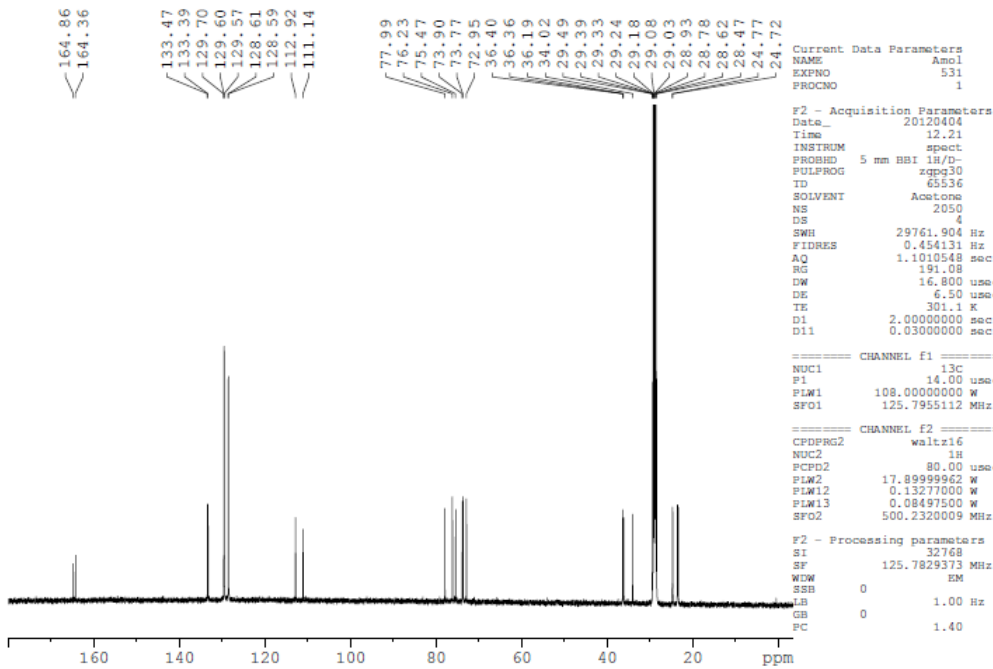
DEPT-135 of compound 12a

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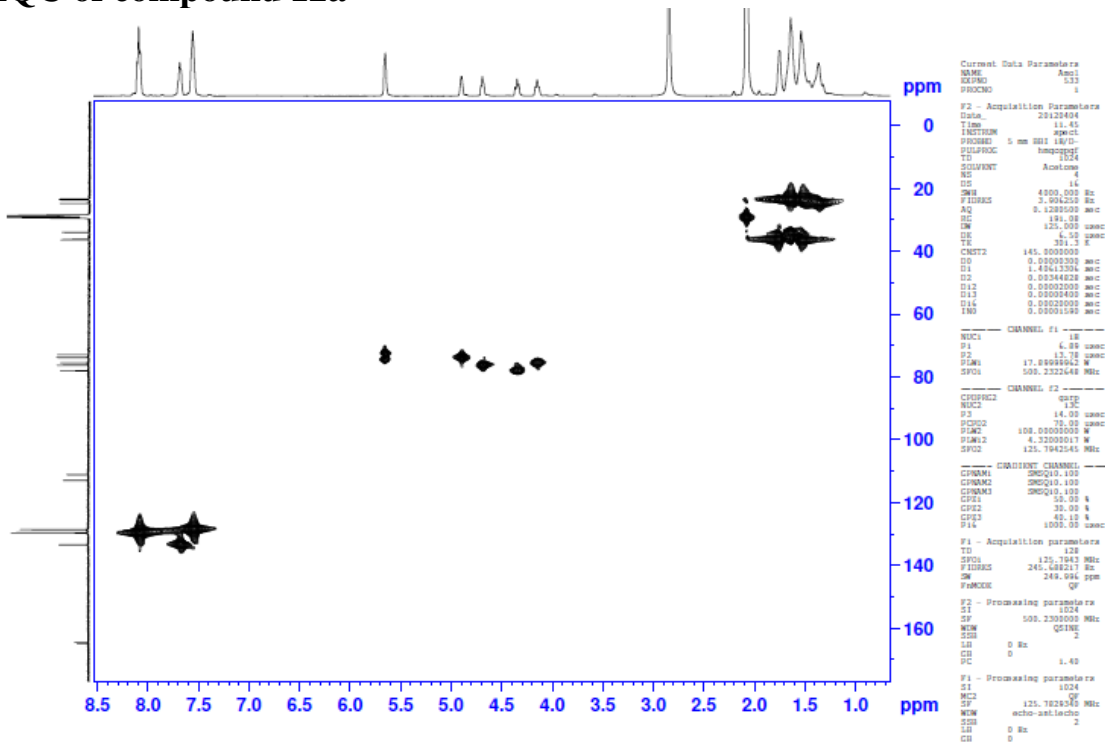


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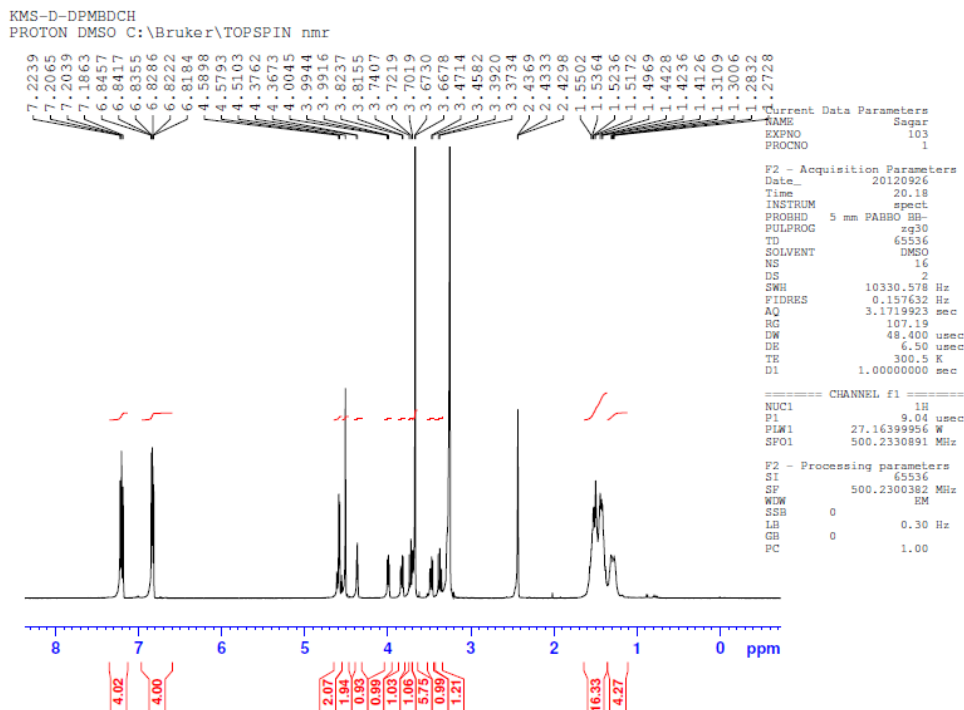
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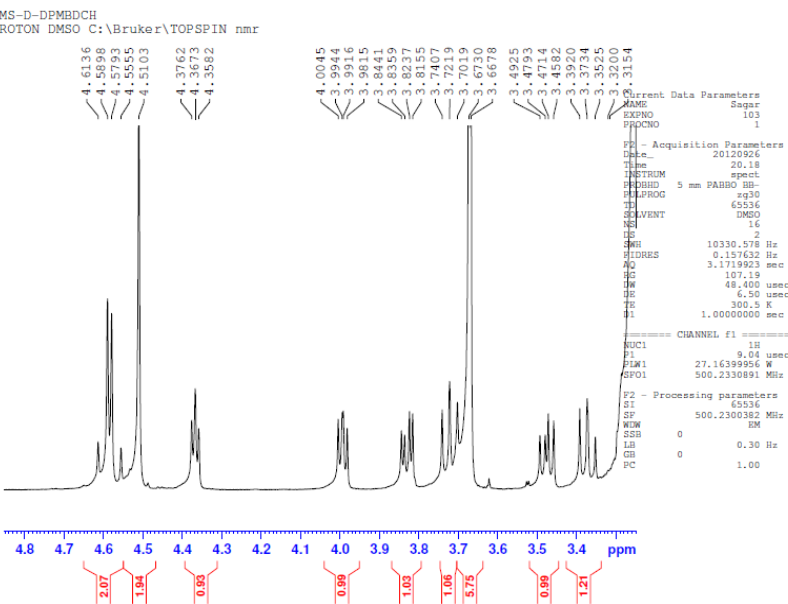
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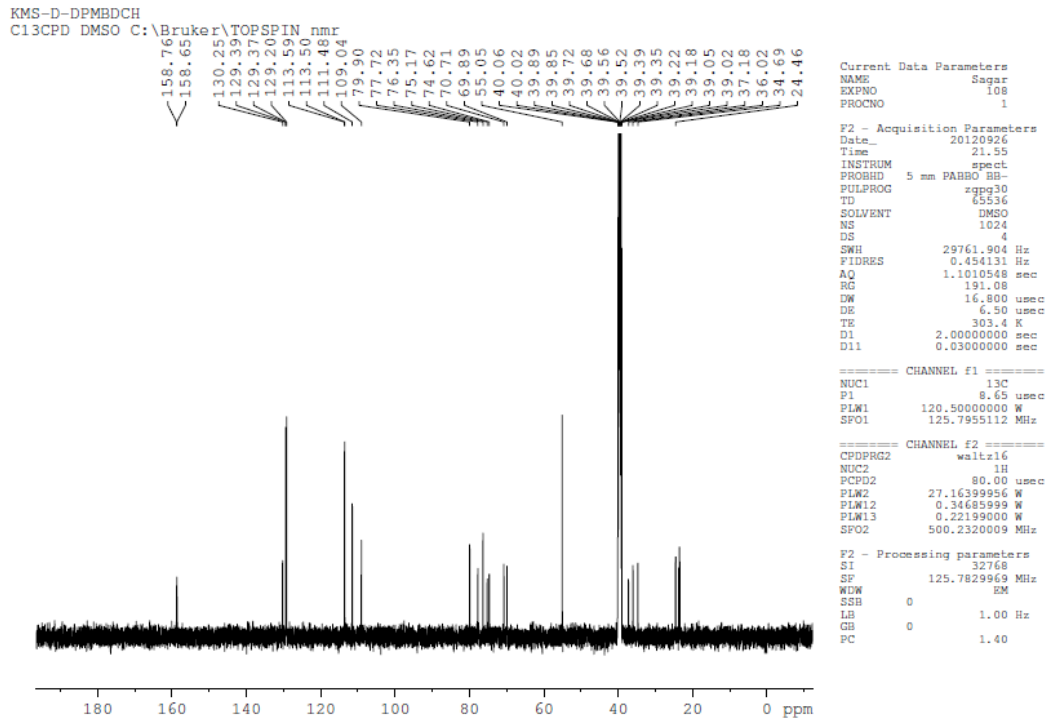
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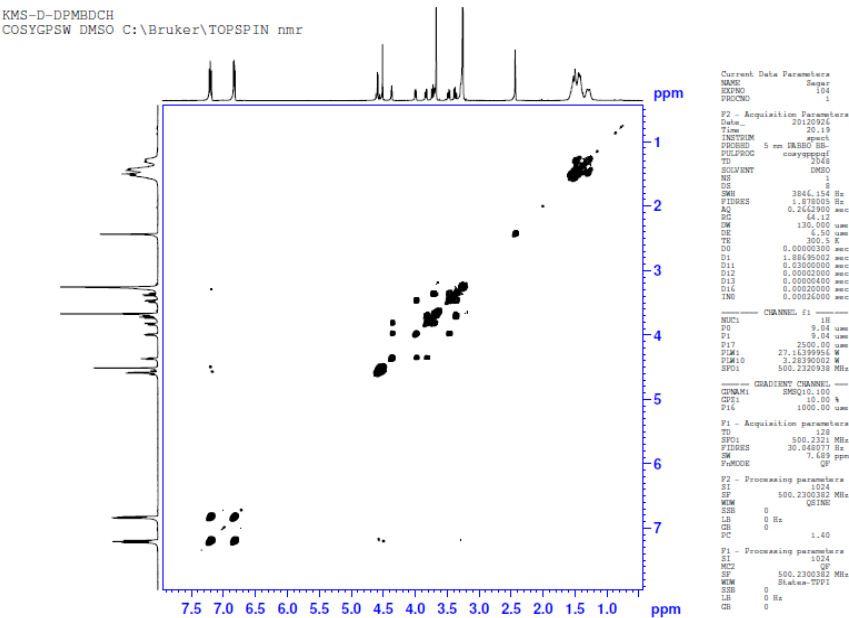
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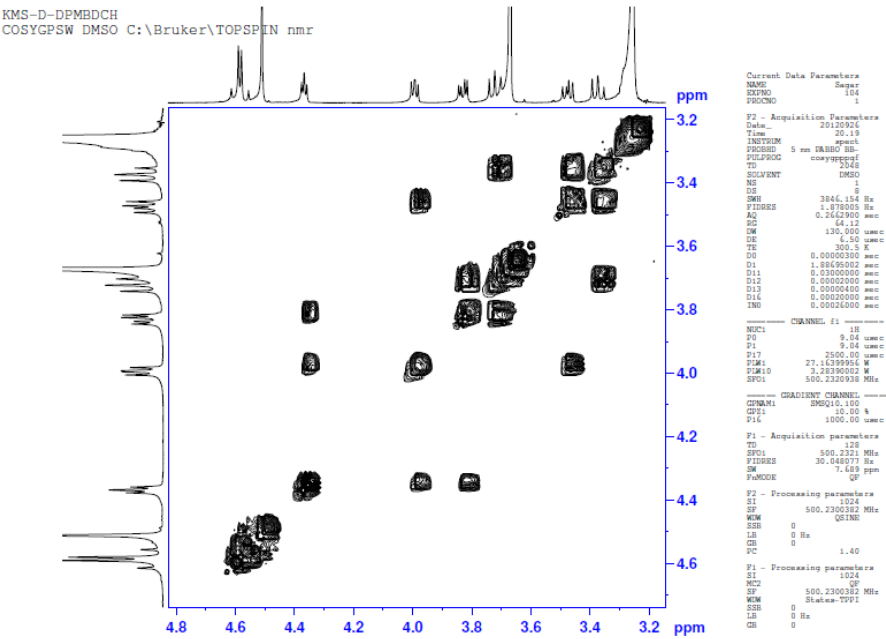
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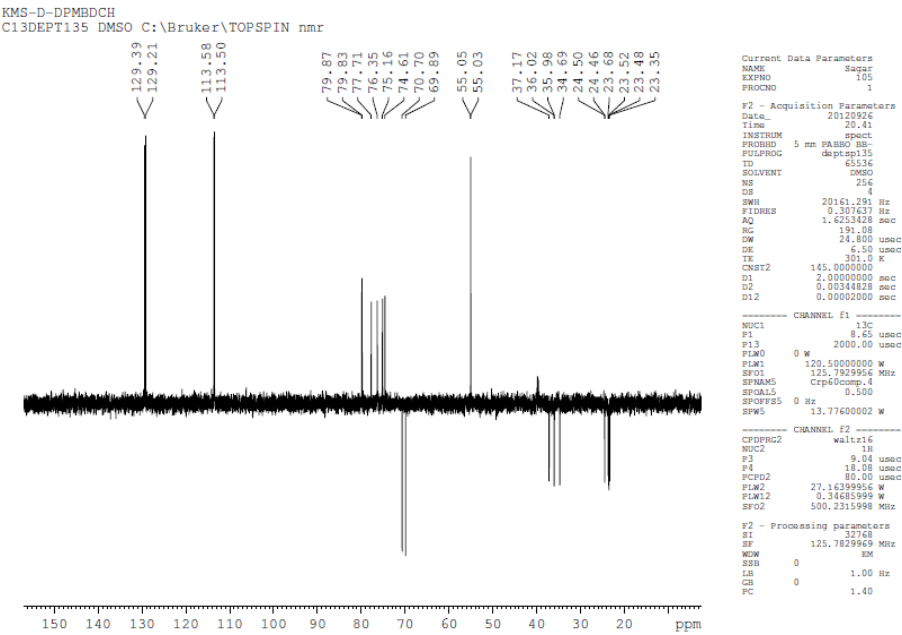
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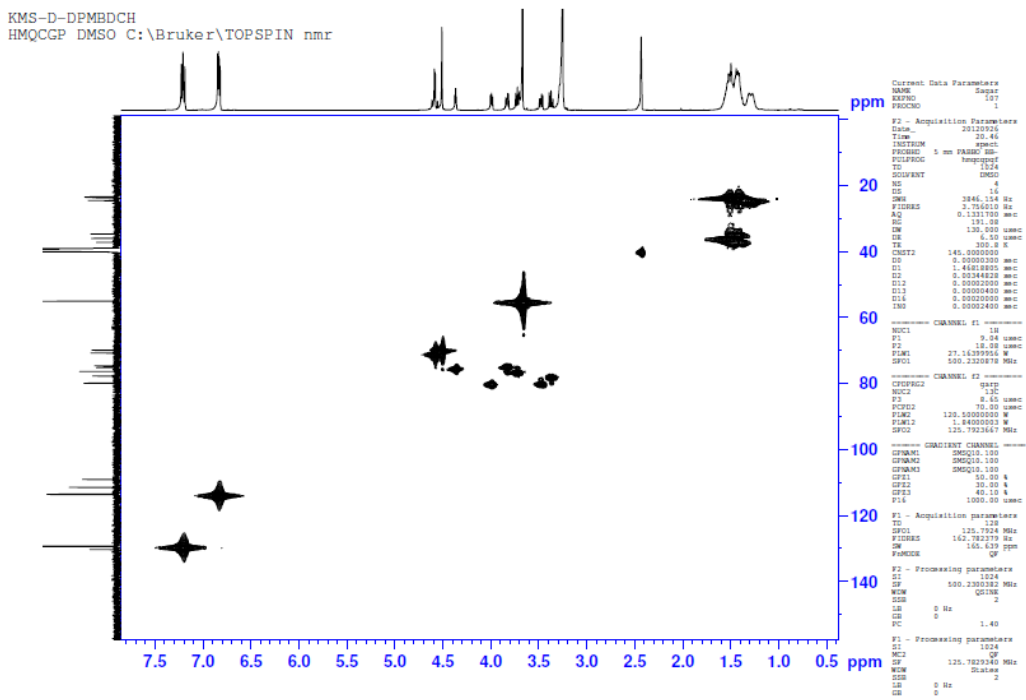
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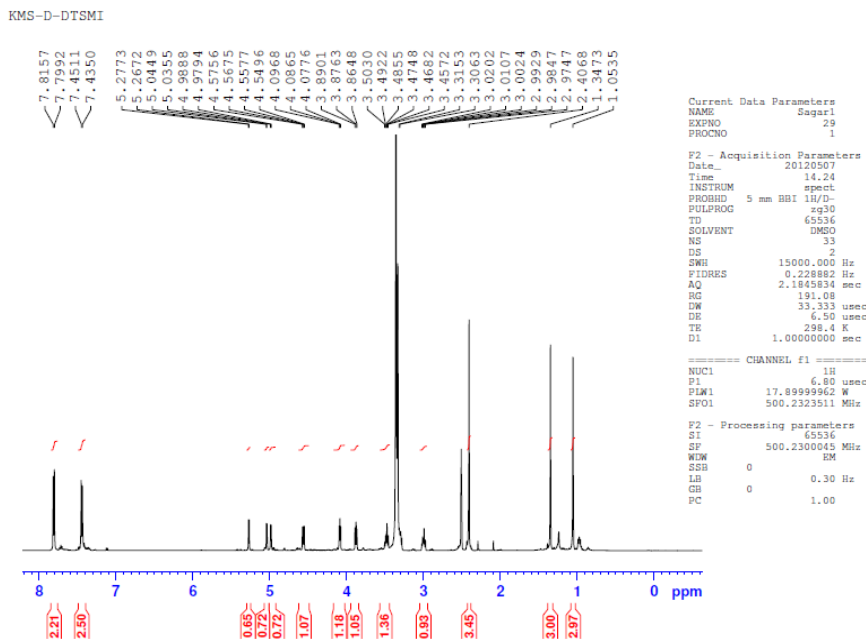
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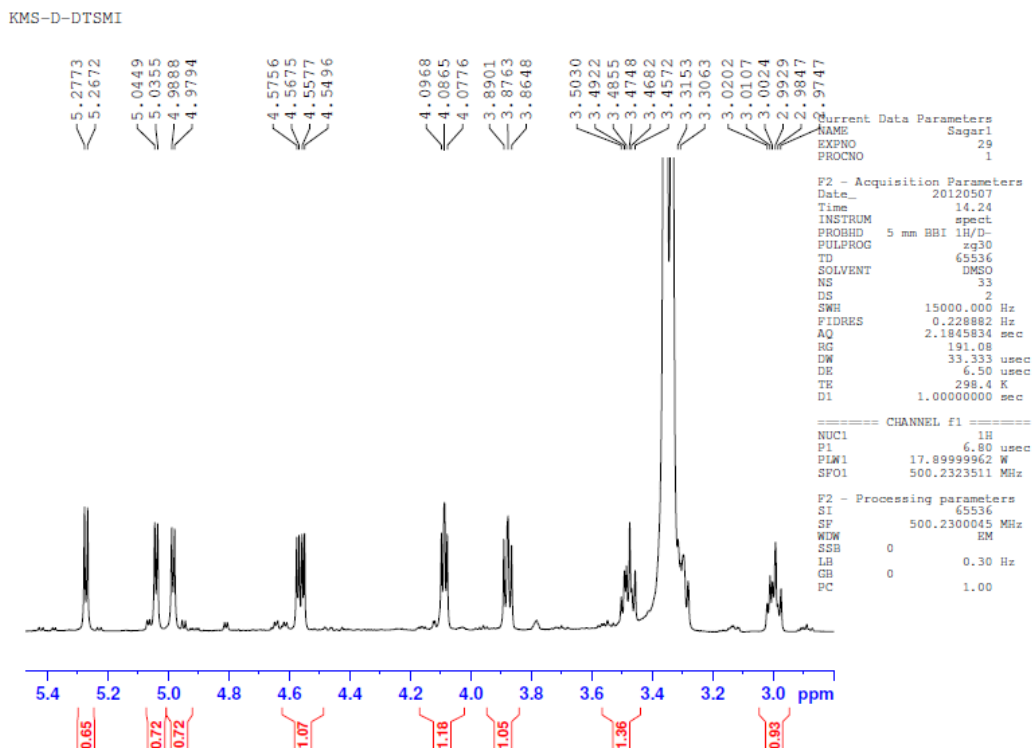
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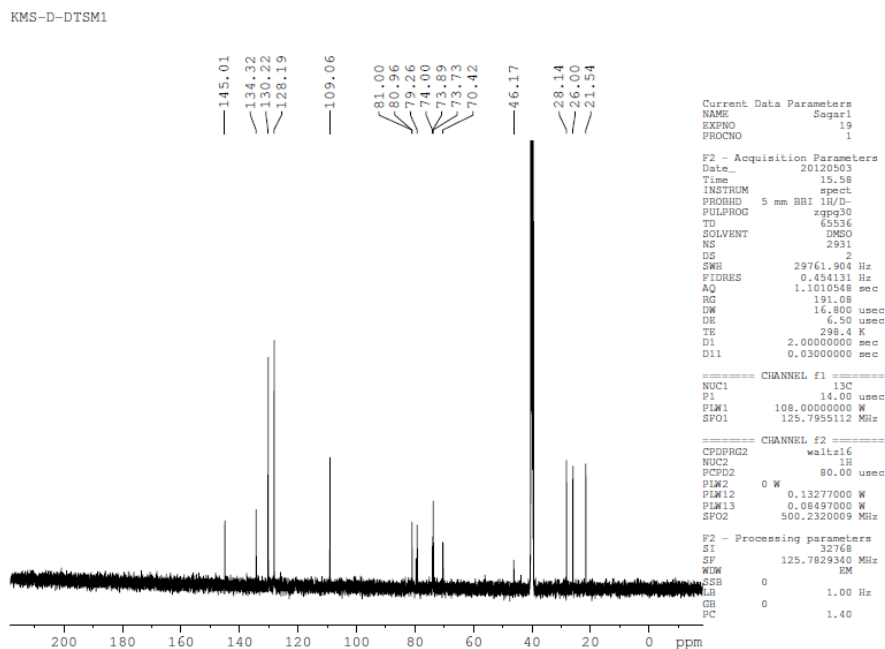
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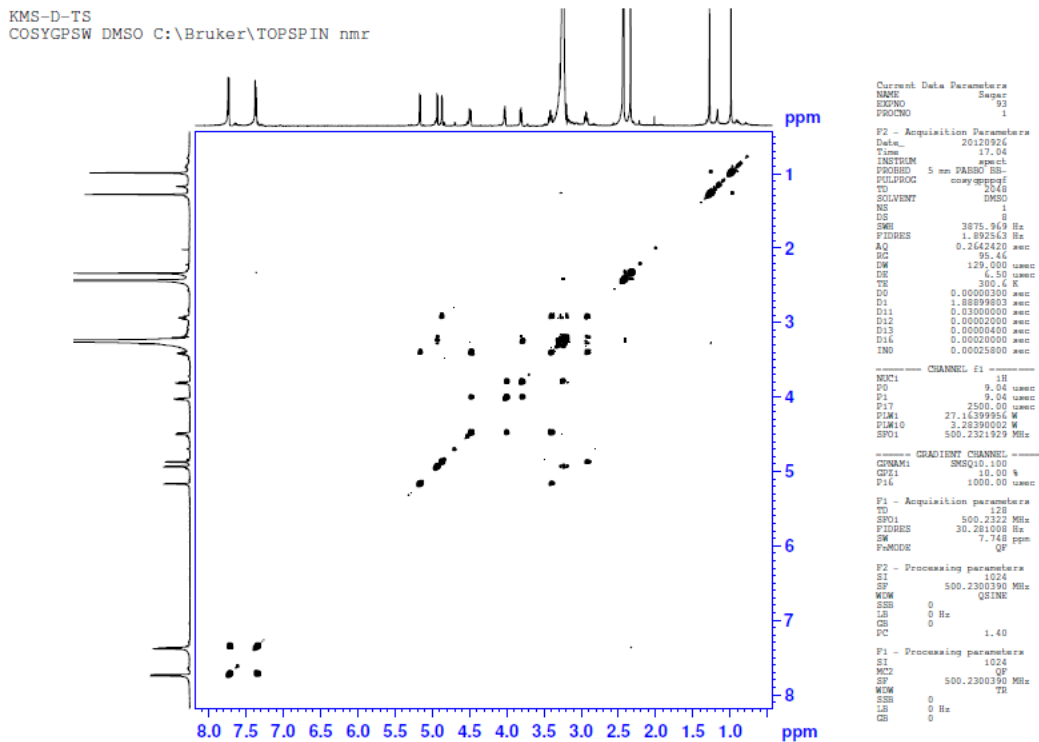
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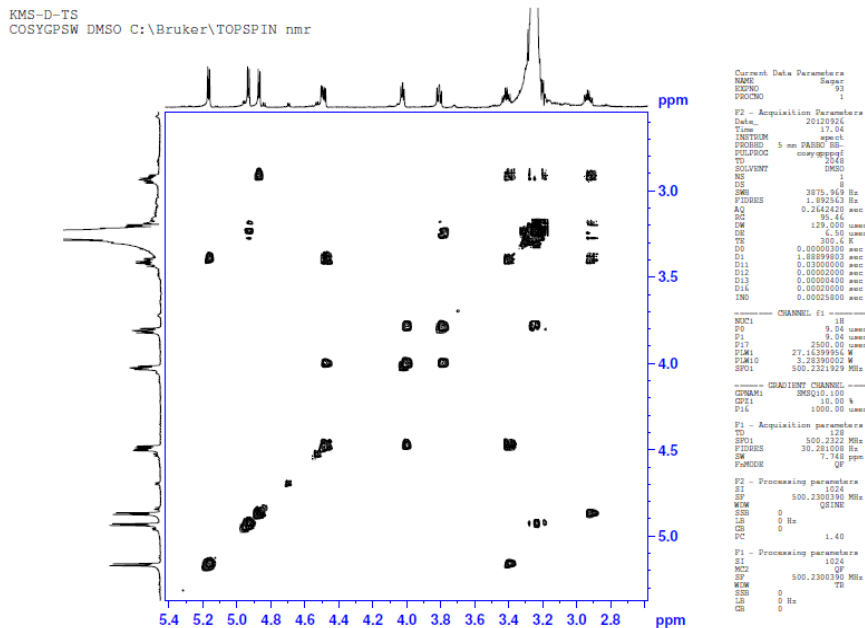
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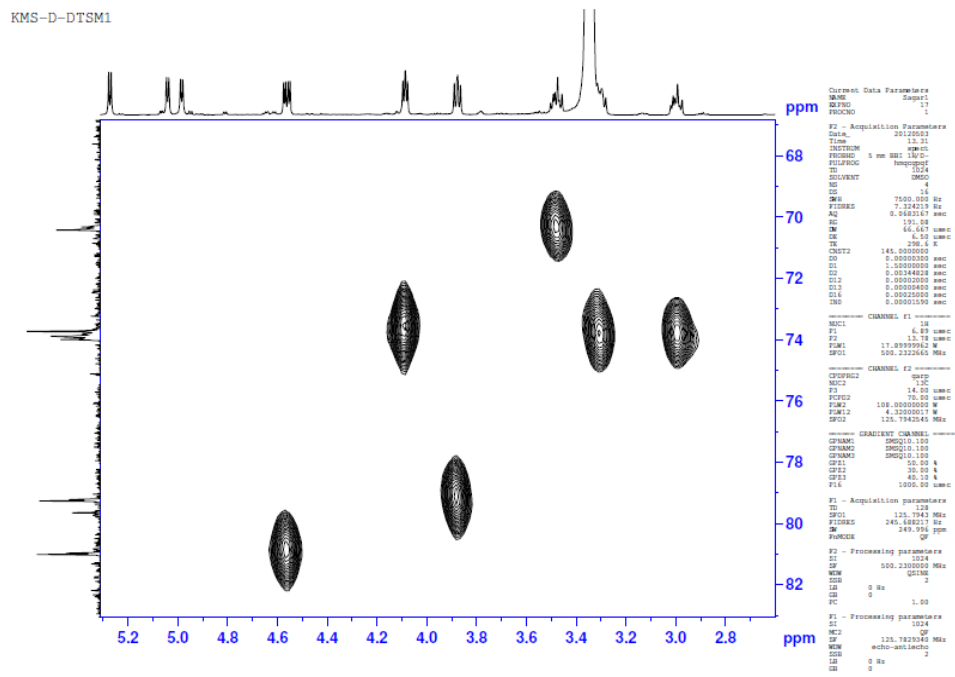
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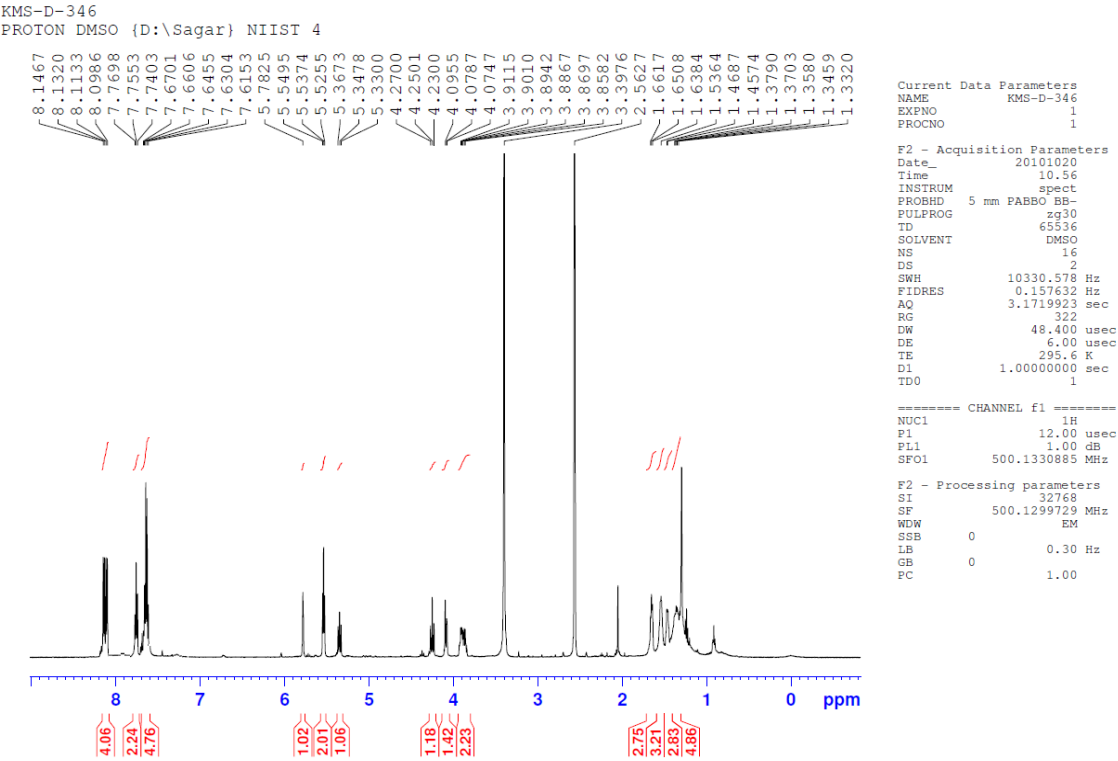
COSY of compound 16c (zoom)



HMQC of Compound 16c

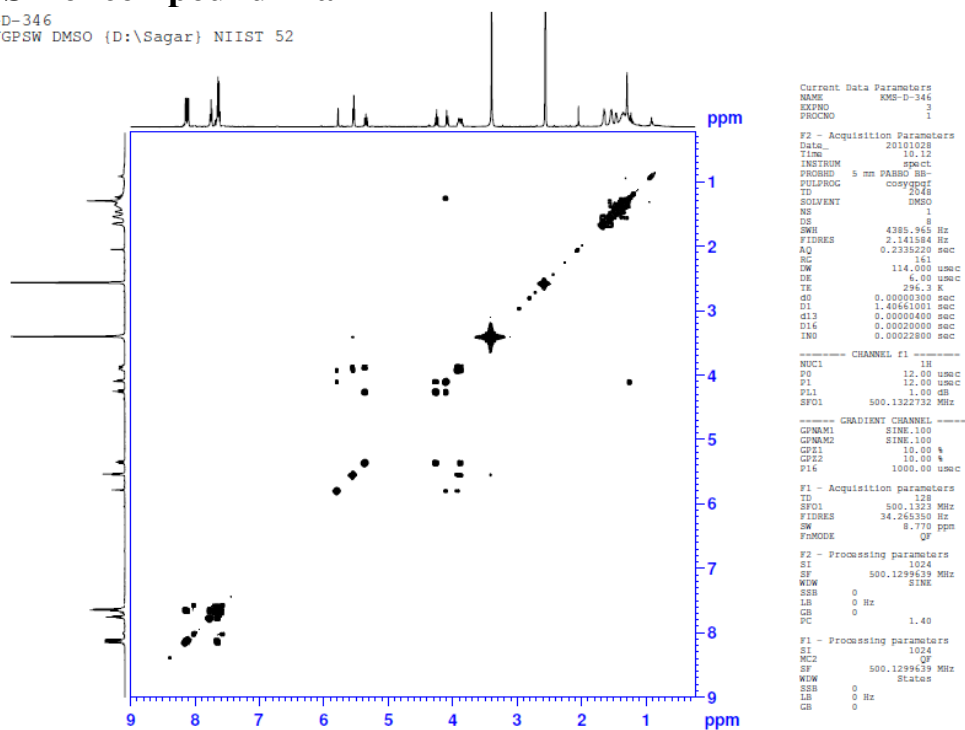


¹H NMR of compound 21a



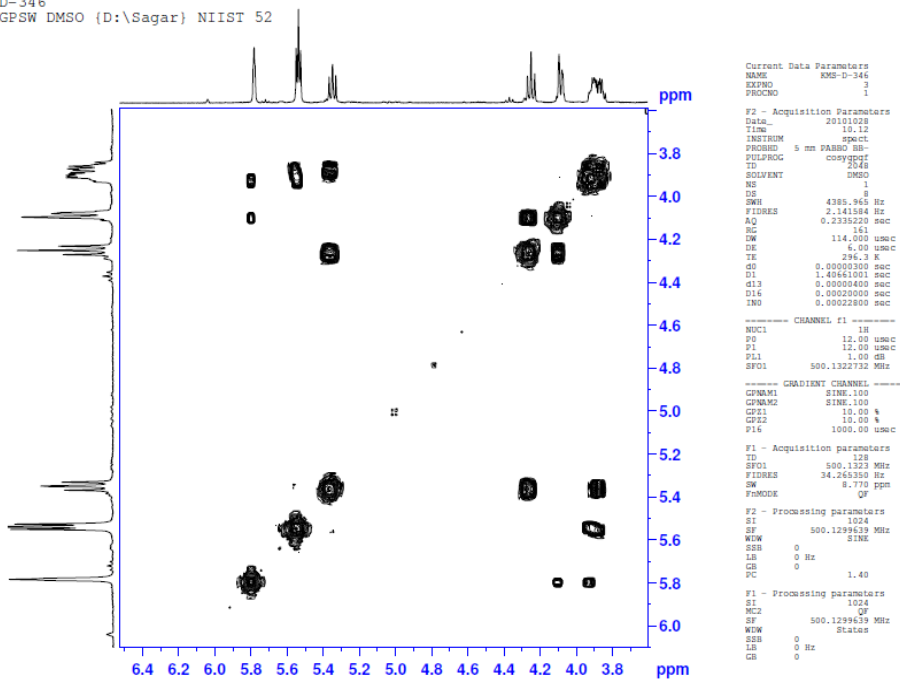
COSY of compound 21a

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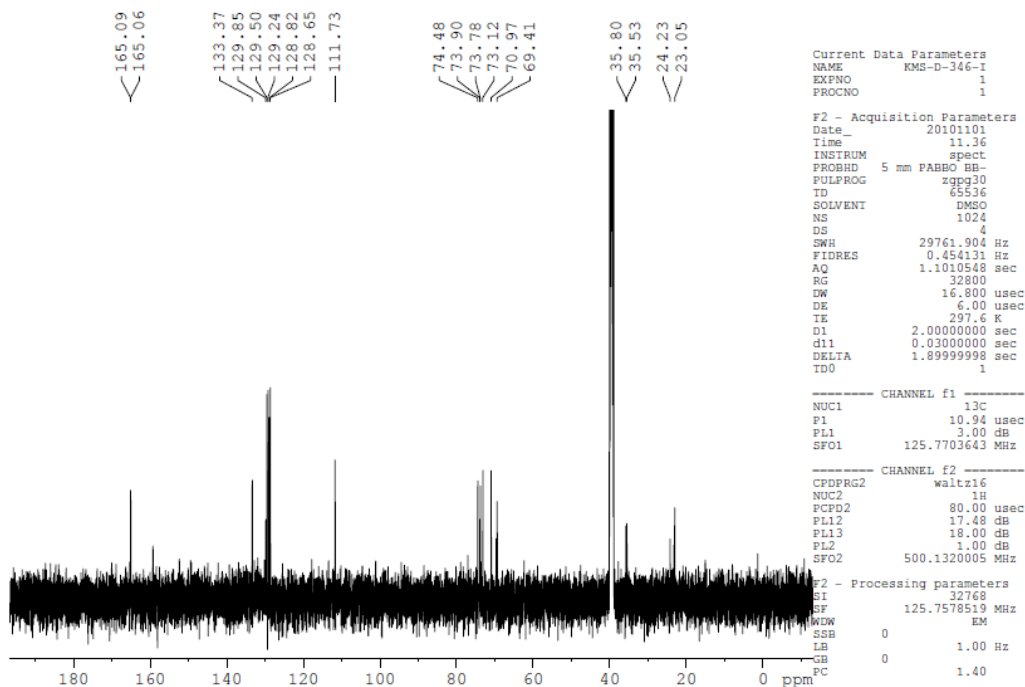
COSY of comound 21a (ZOOM)

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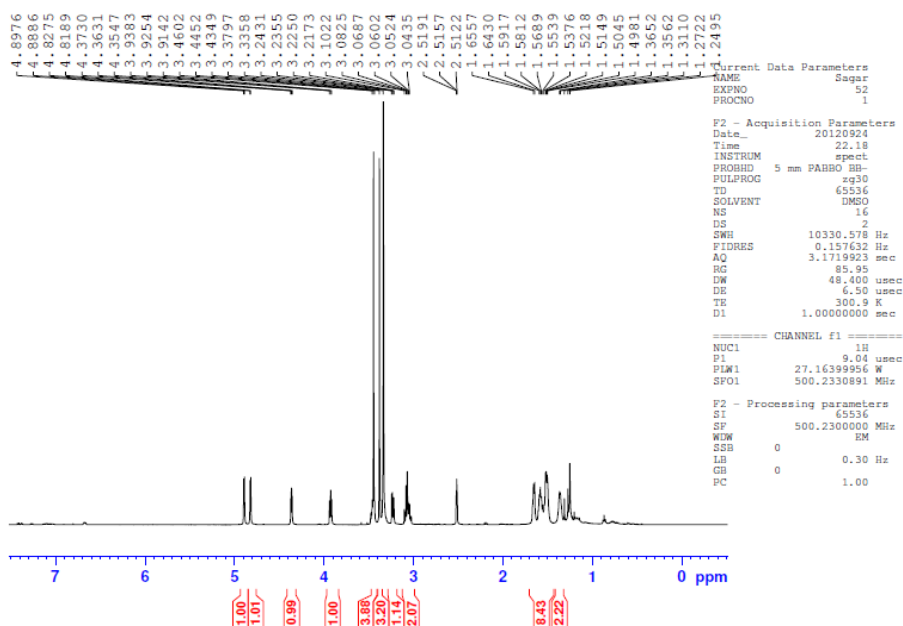
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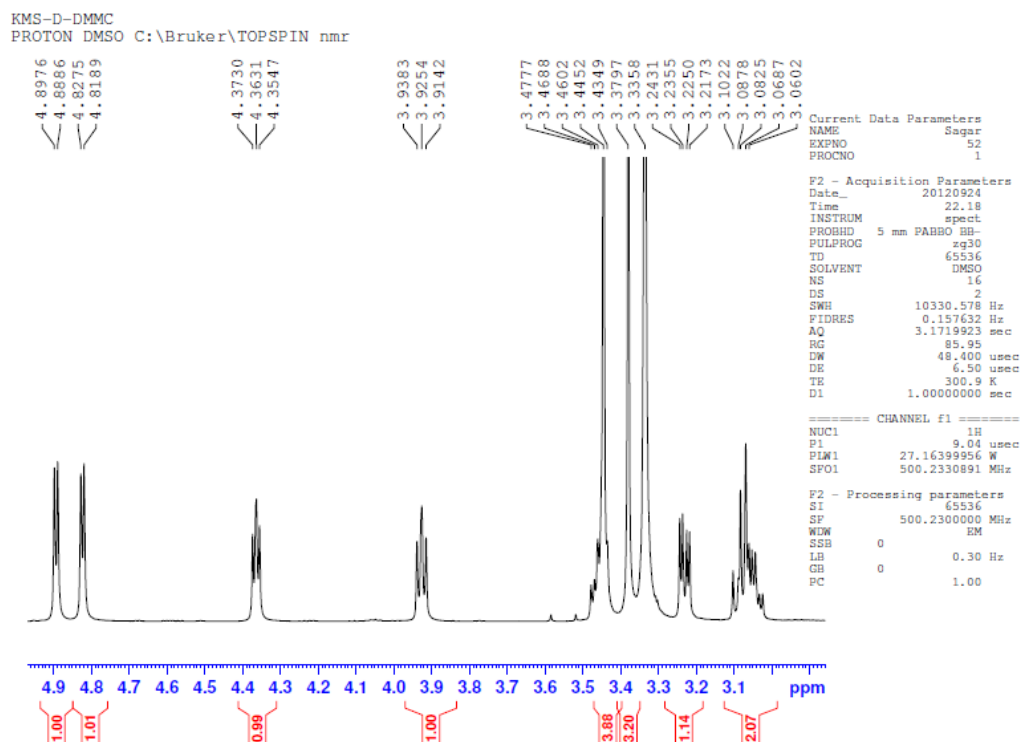


¹H NMR of compound 22b

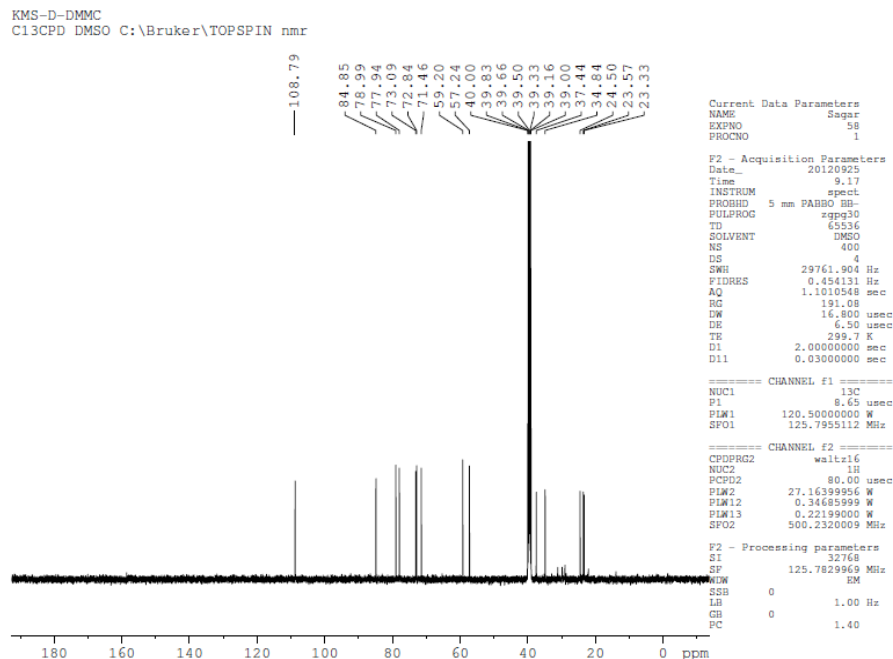
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PROTON DMSO C:\Bruker\TOPSPIN nmr



¹H NMR of compound 22b (zoom)

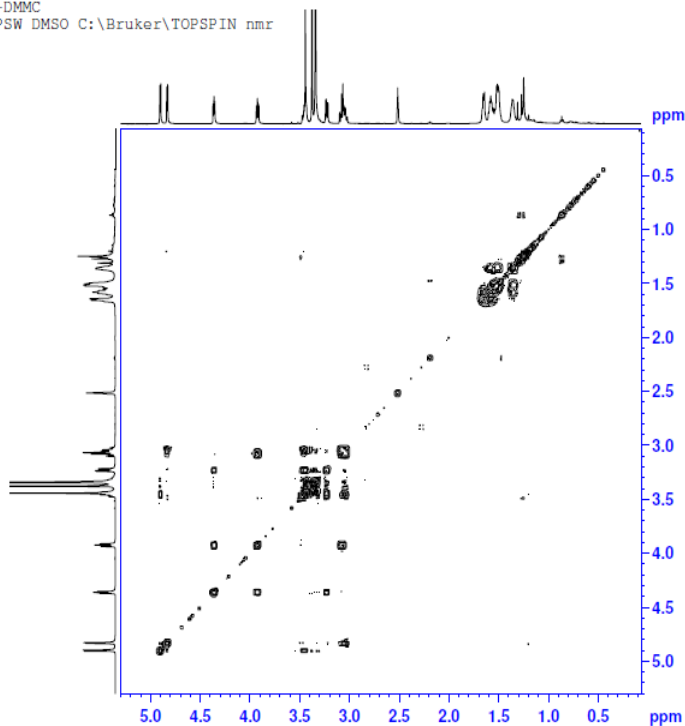


¹³C NMR of compound 22b



COSY of compound 22b

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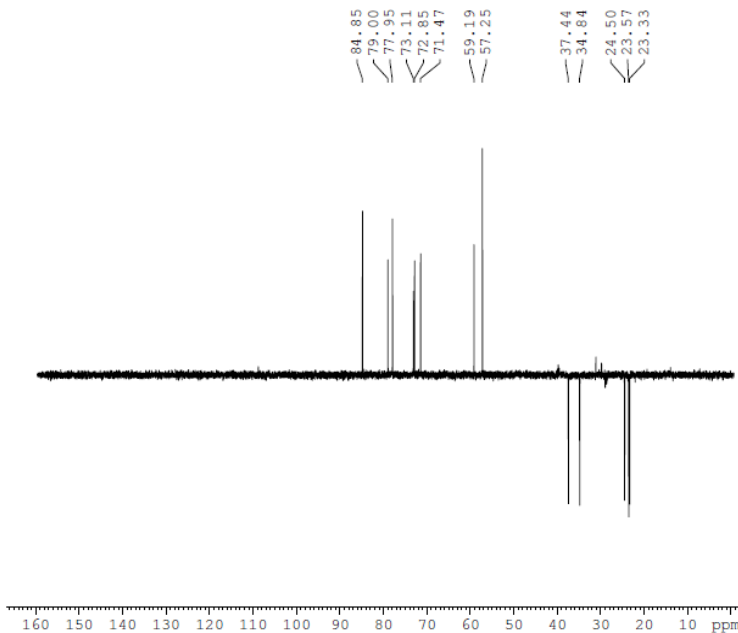


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PROCNO 1

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DEPT 135 of compound 22b

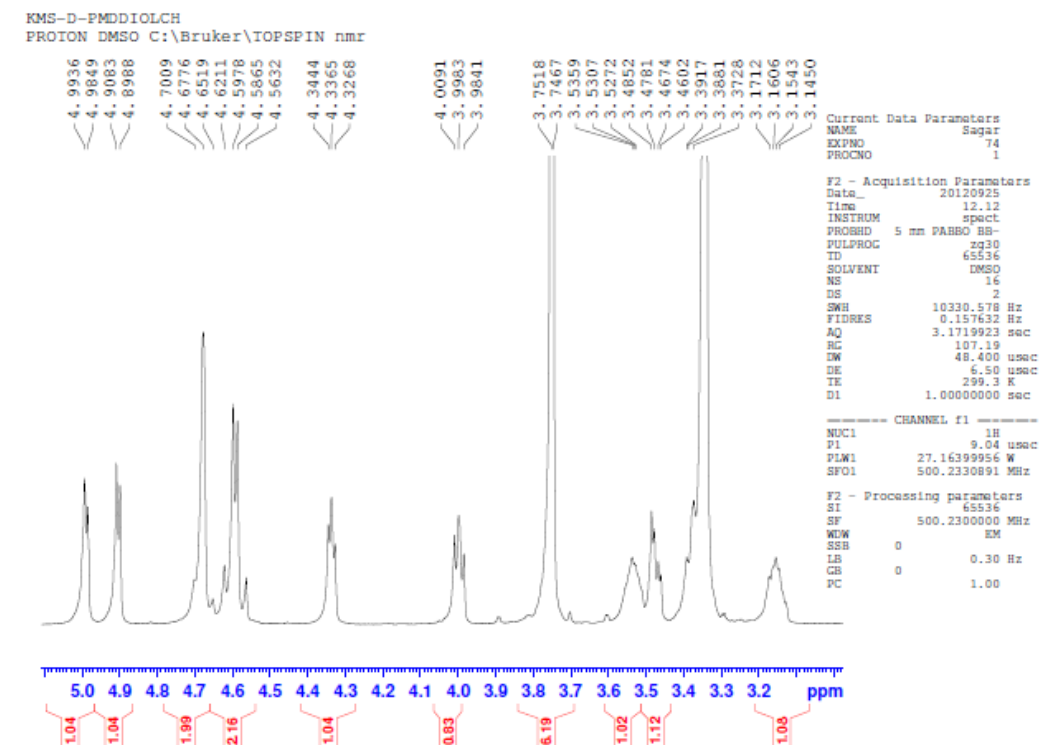
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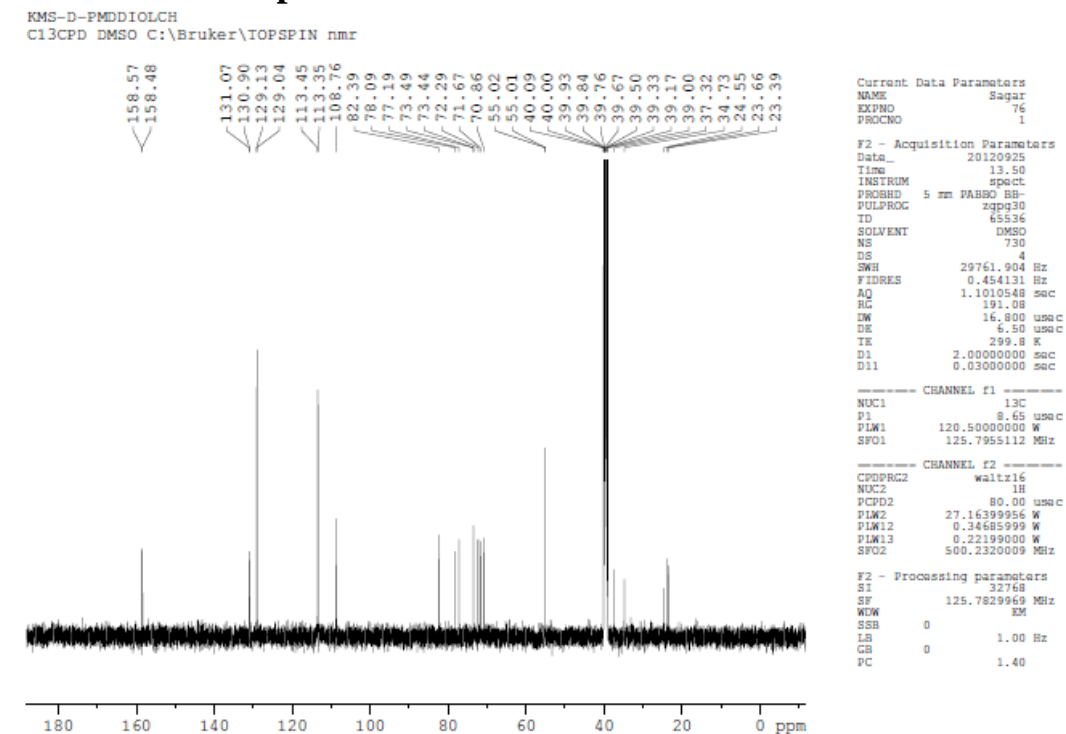
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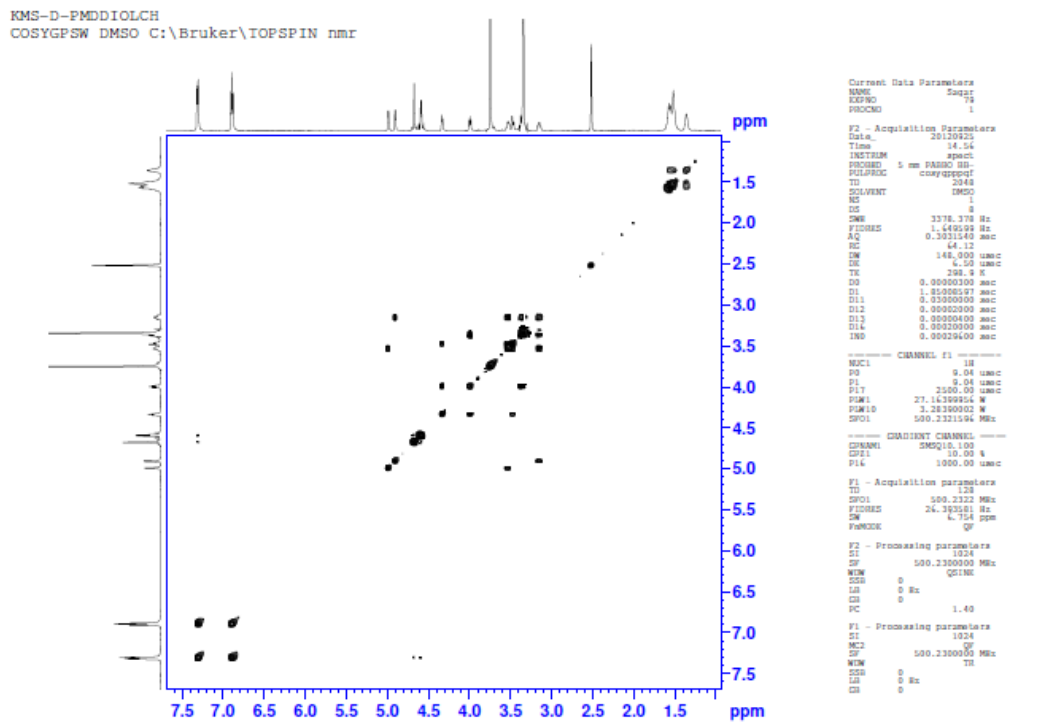
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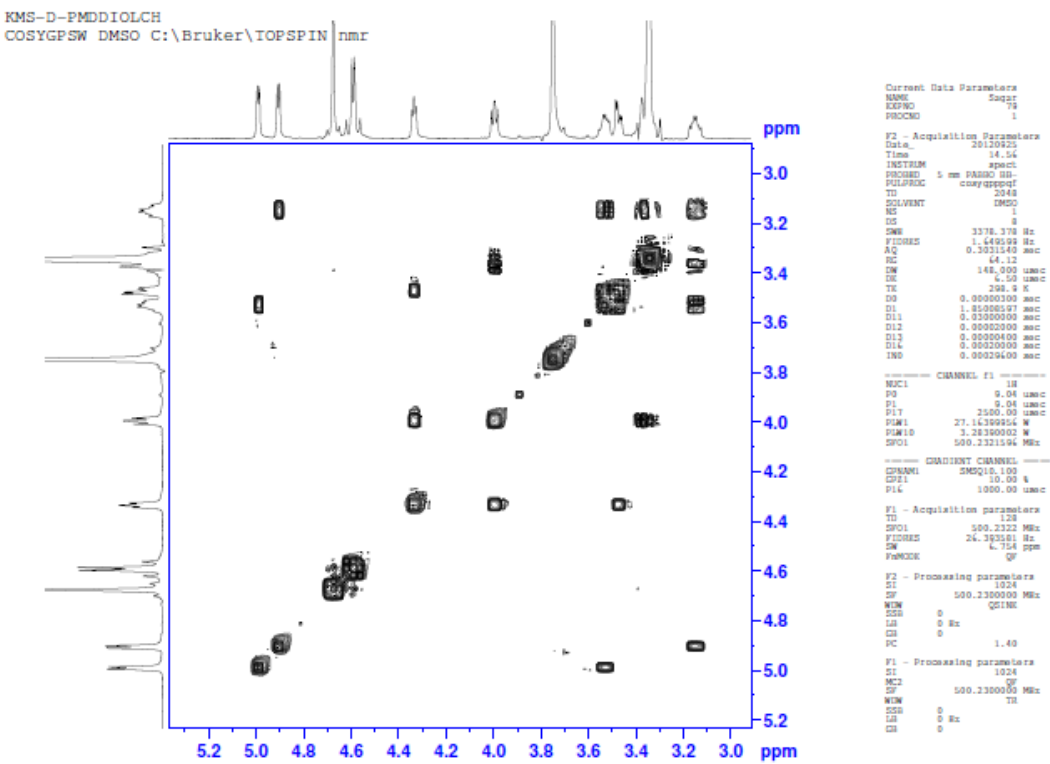
¹³C NMR of compound 22c



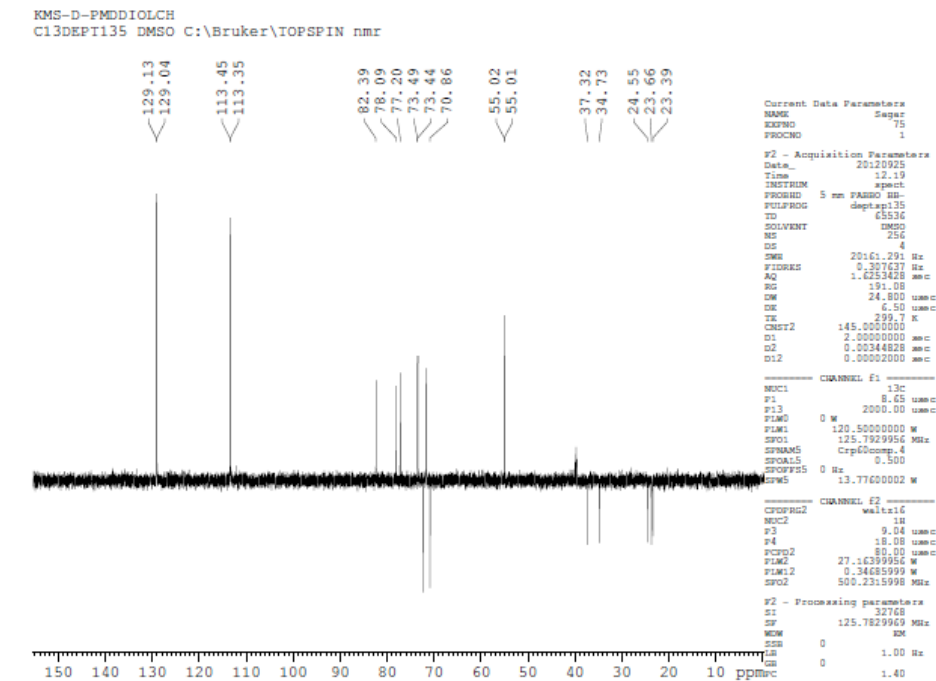
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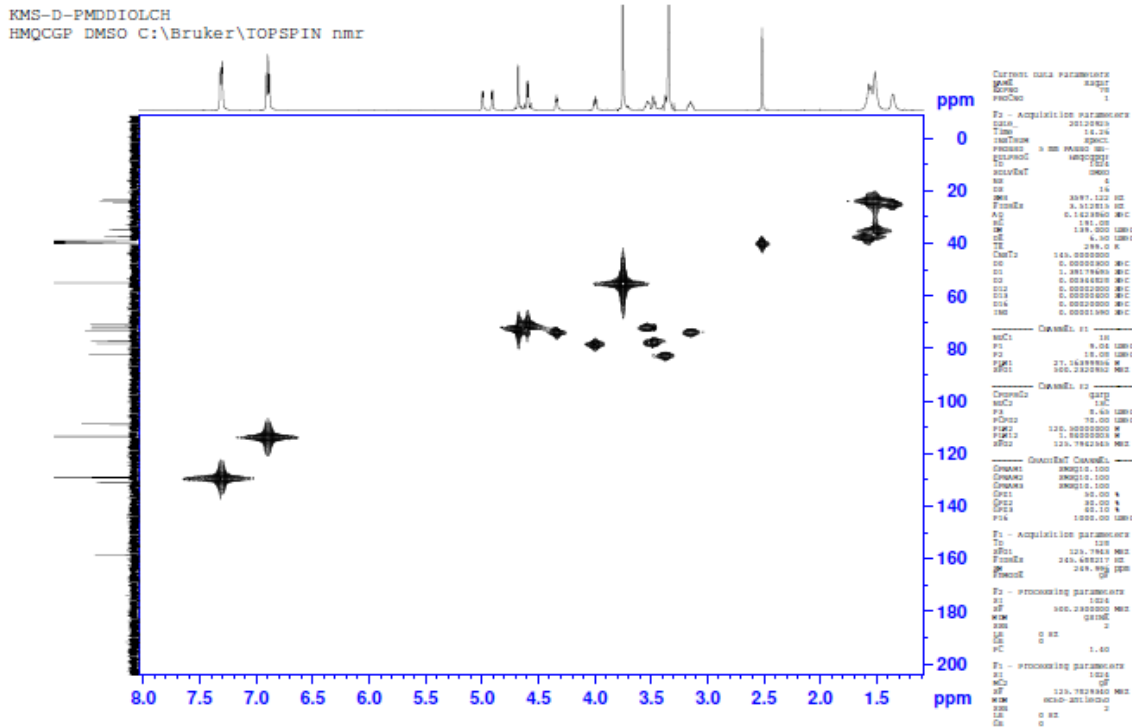
COSY of compound 22c (zoom)



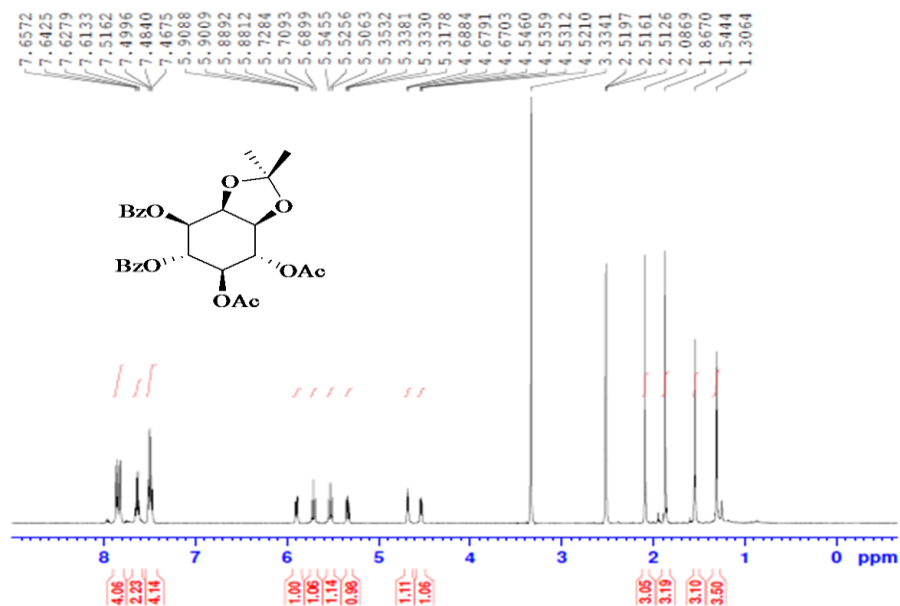
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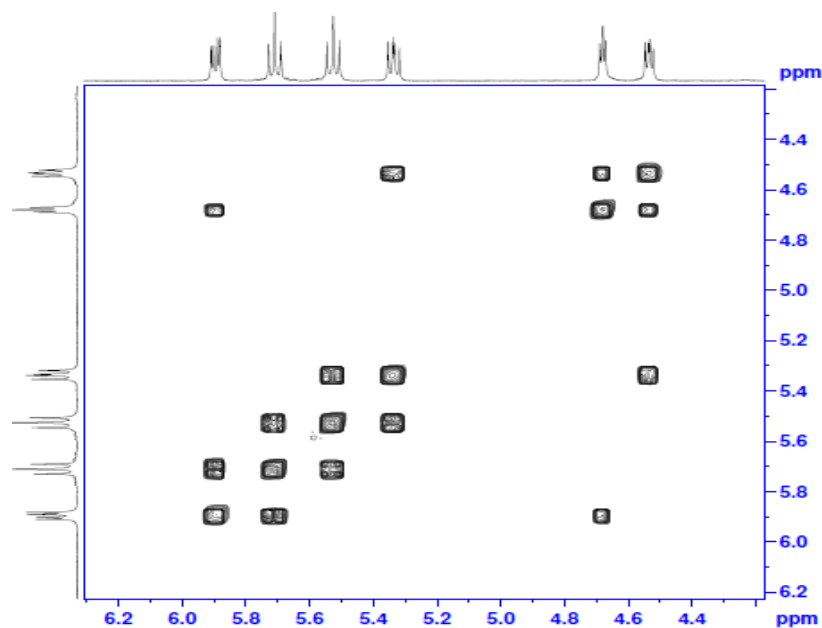
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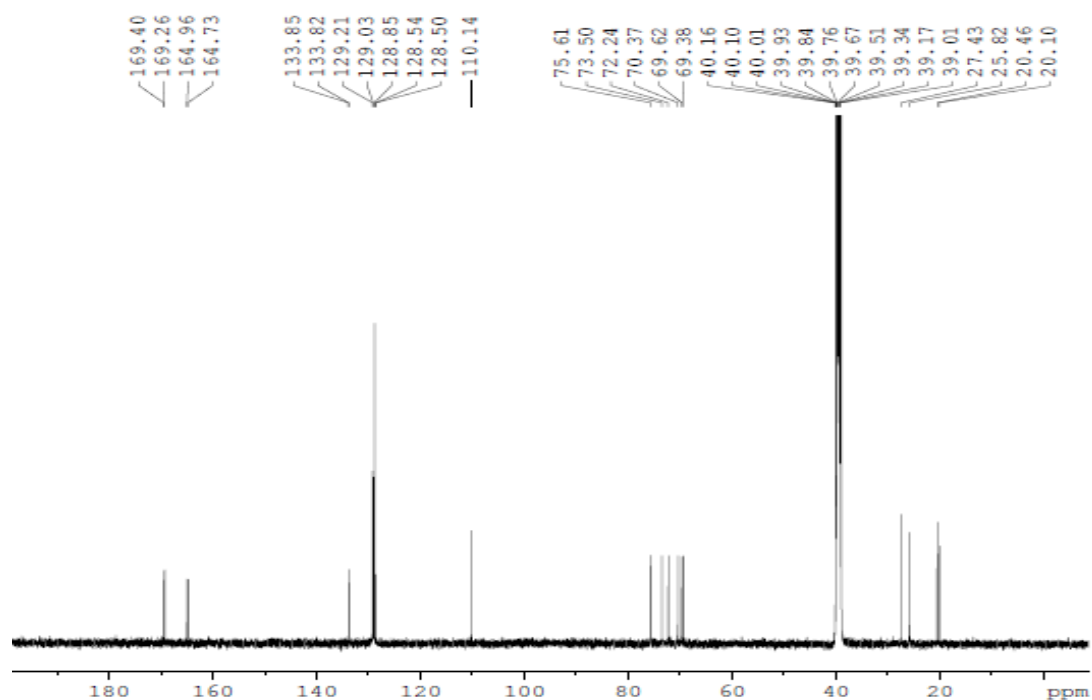
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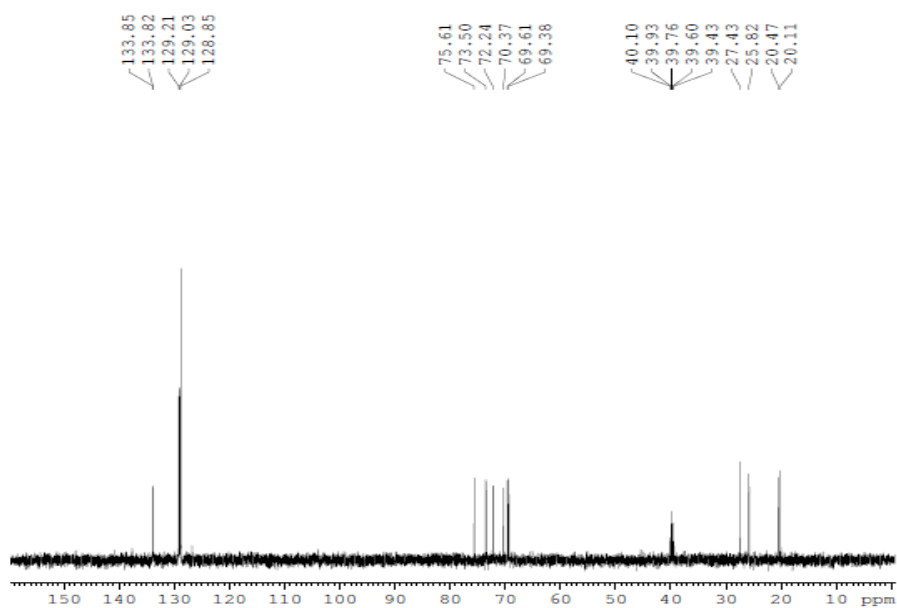
COSY of compound 23b



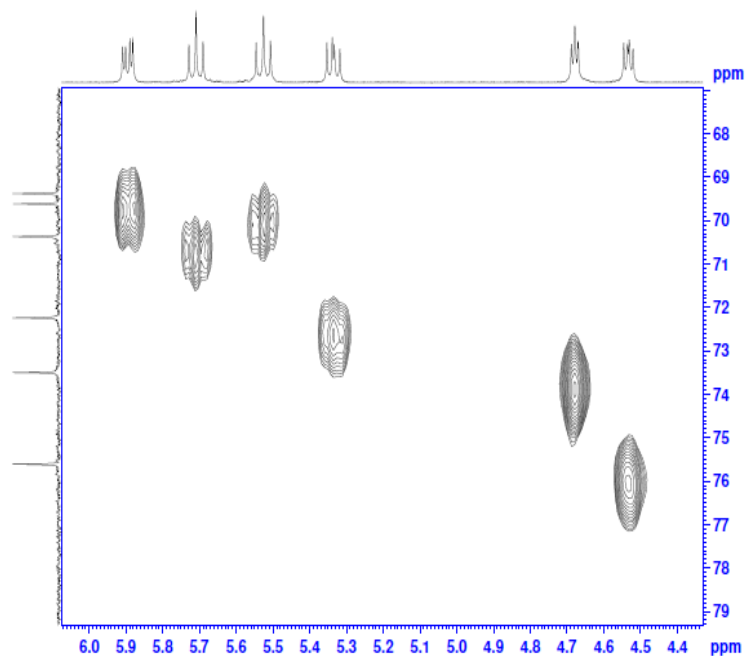
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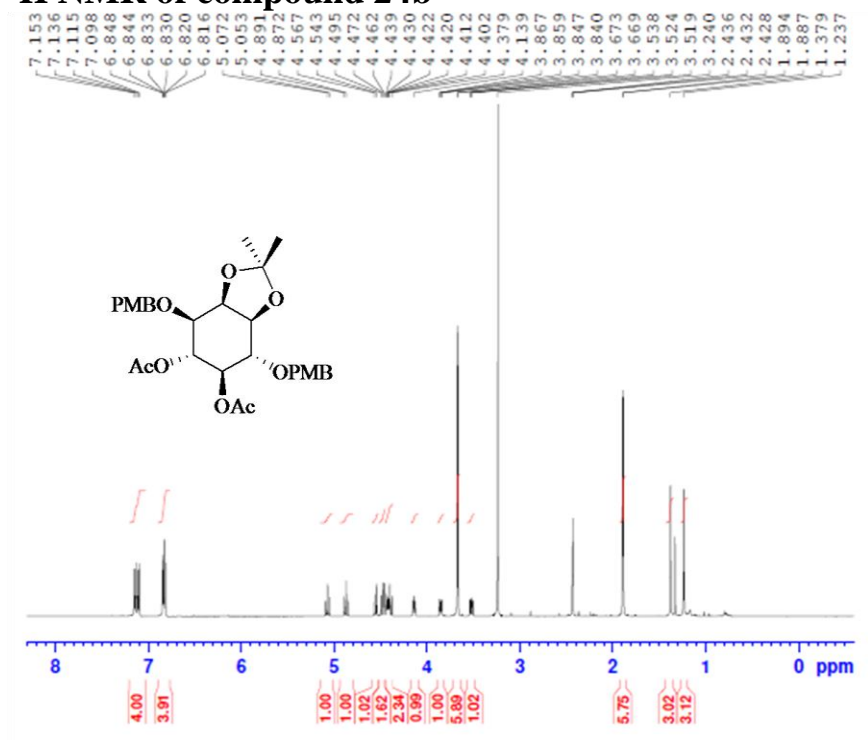
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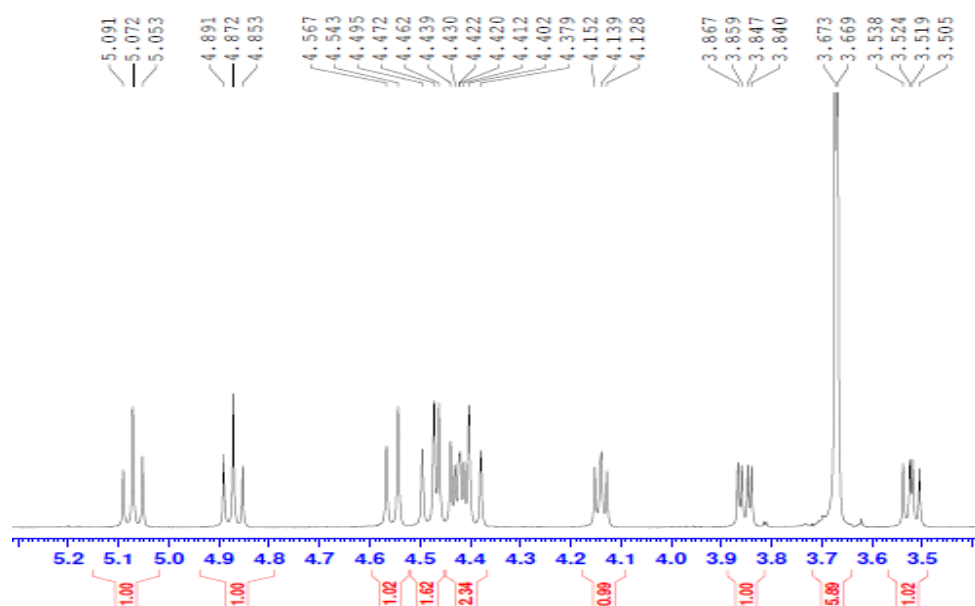
HMQC of compound 23b



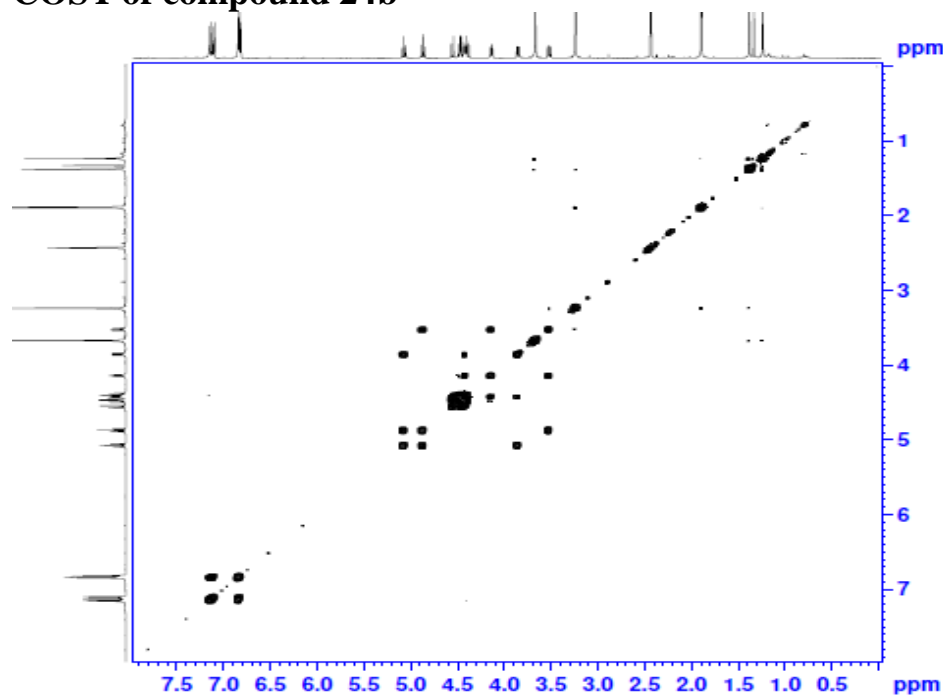
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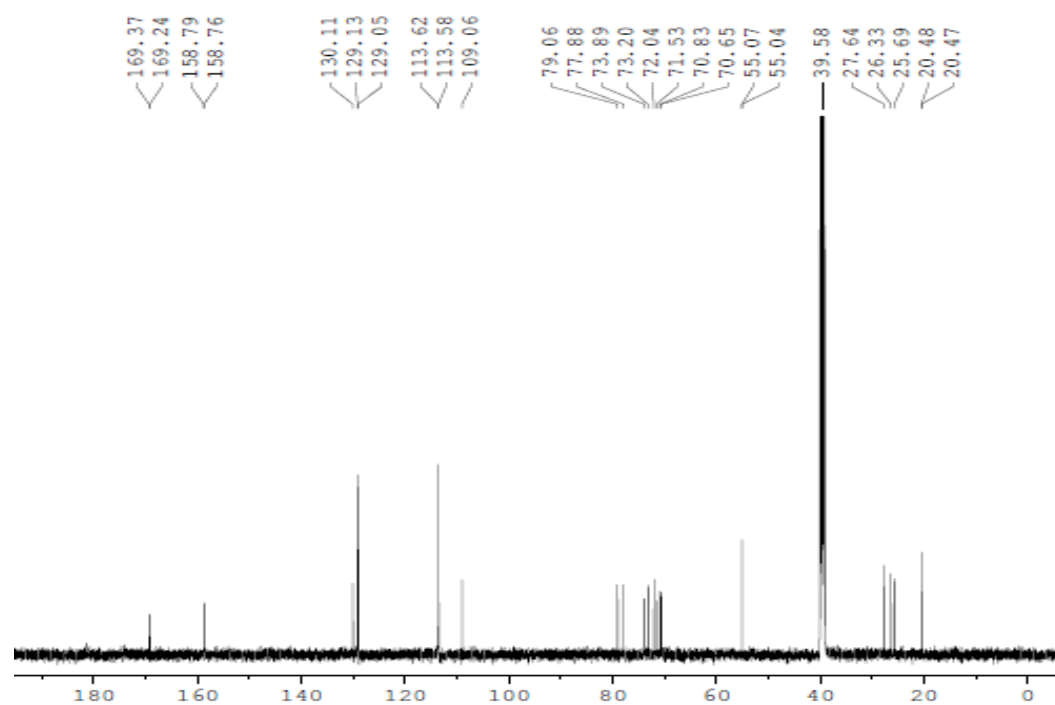
¹H NMR of compound 24b (zoom)



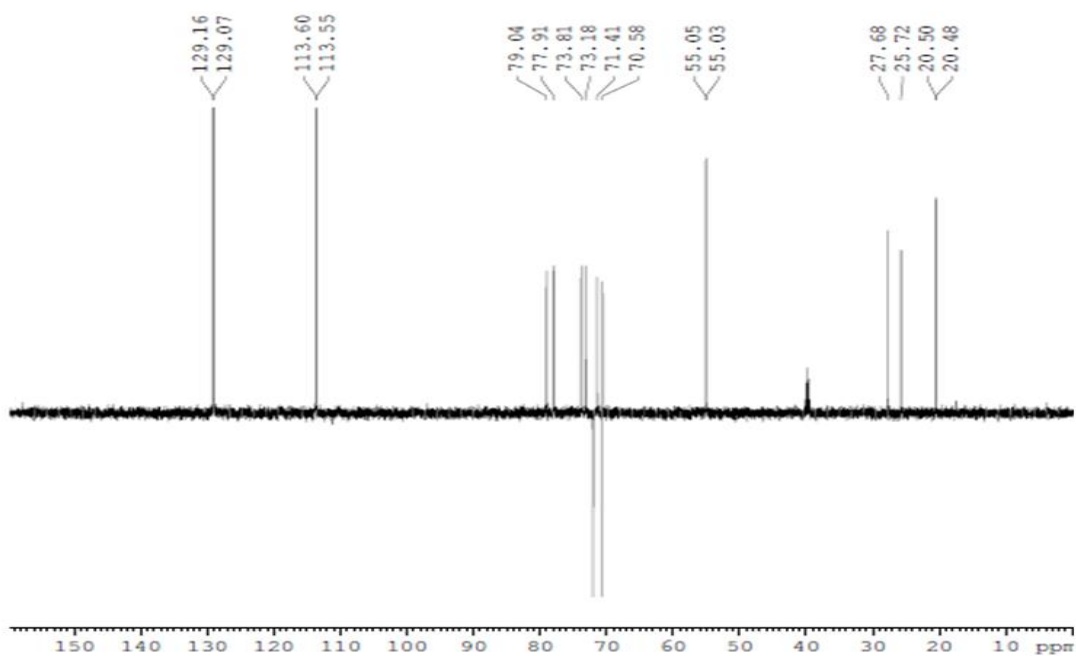
COSY of compound 24b



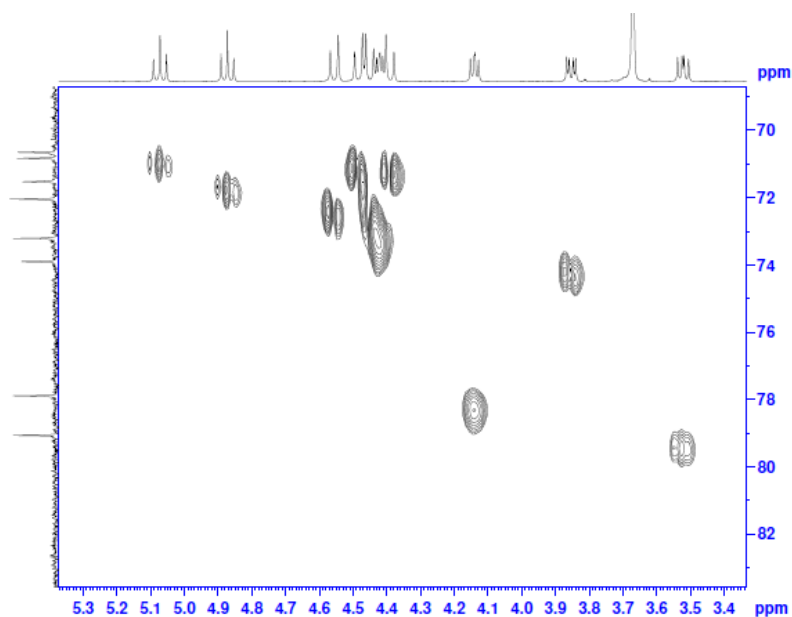
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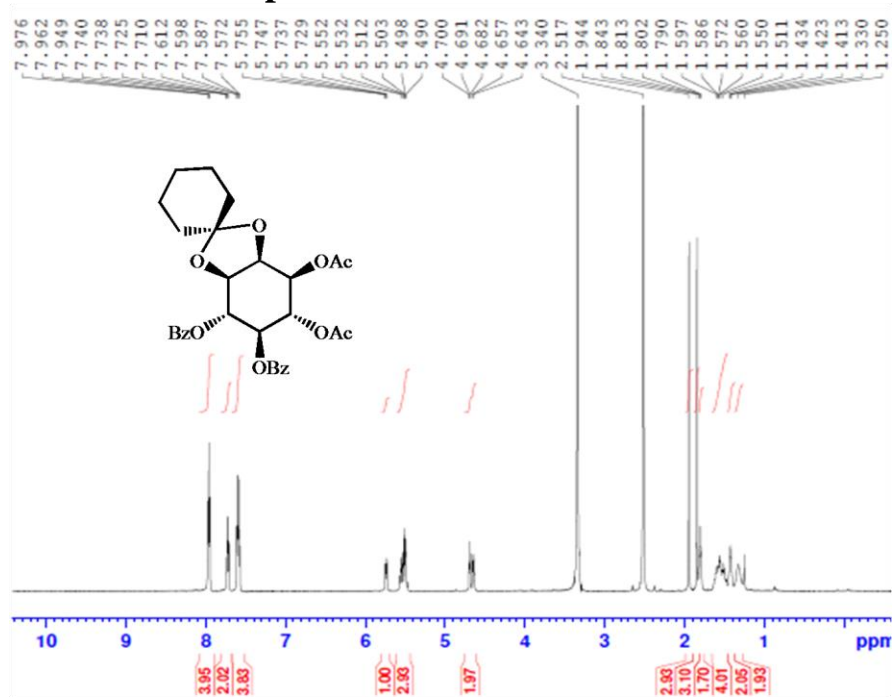
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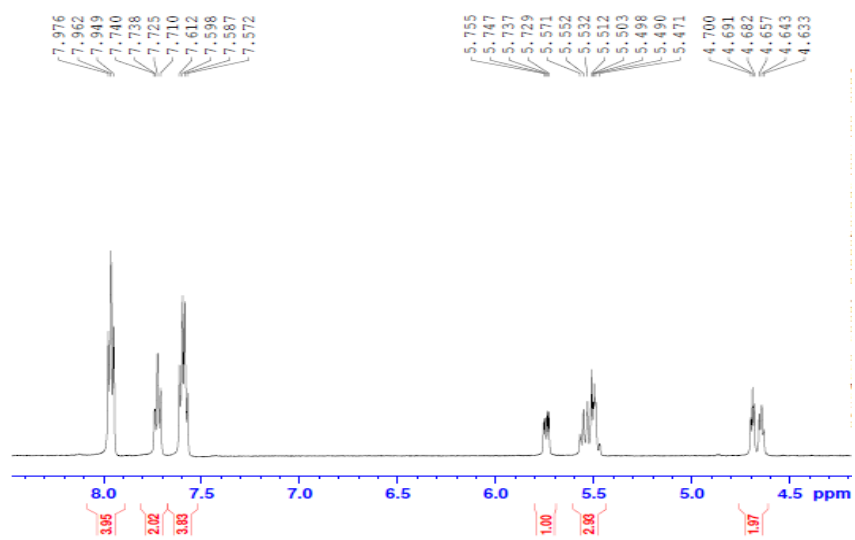
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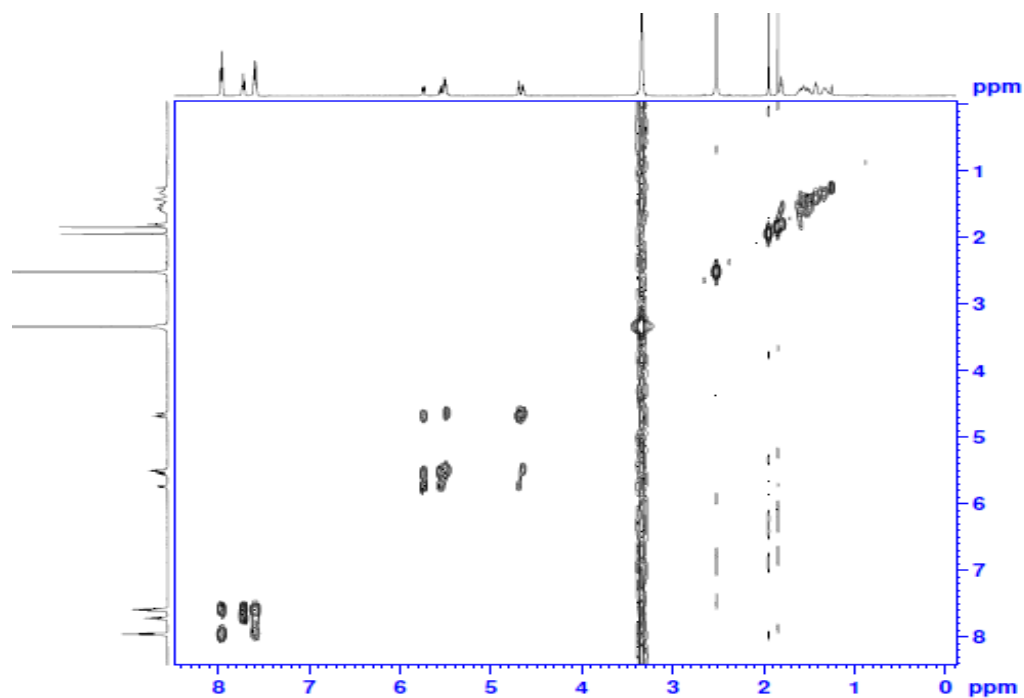
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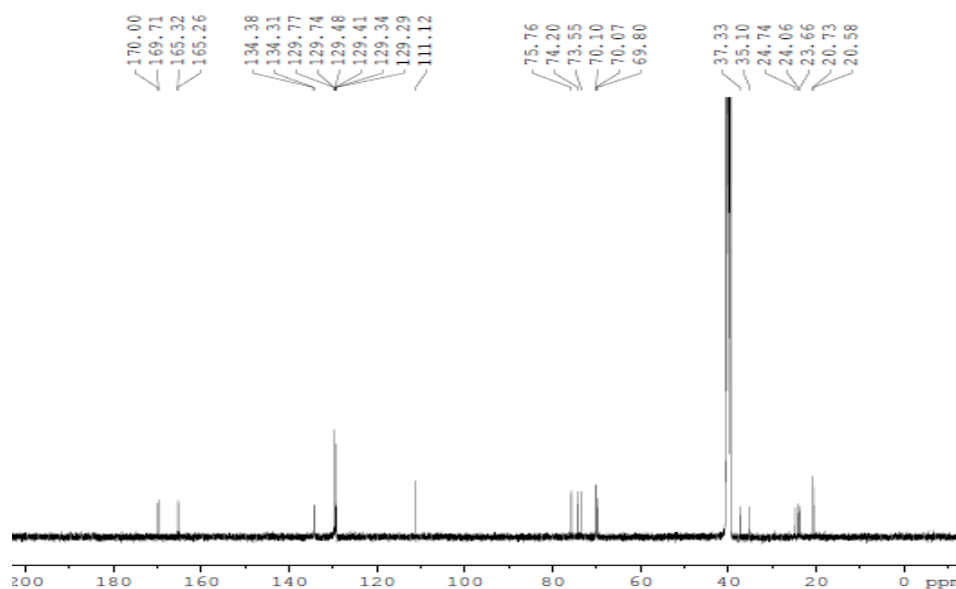
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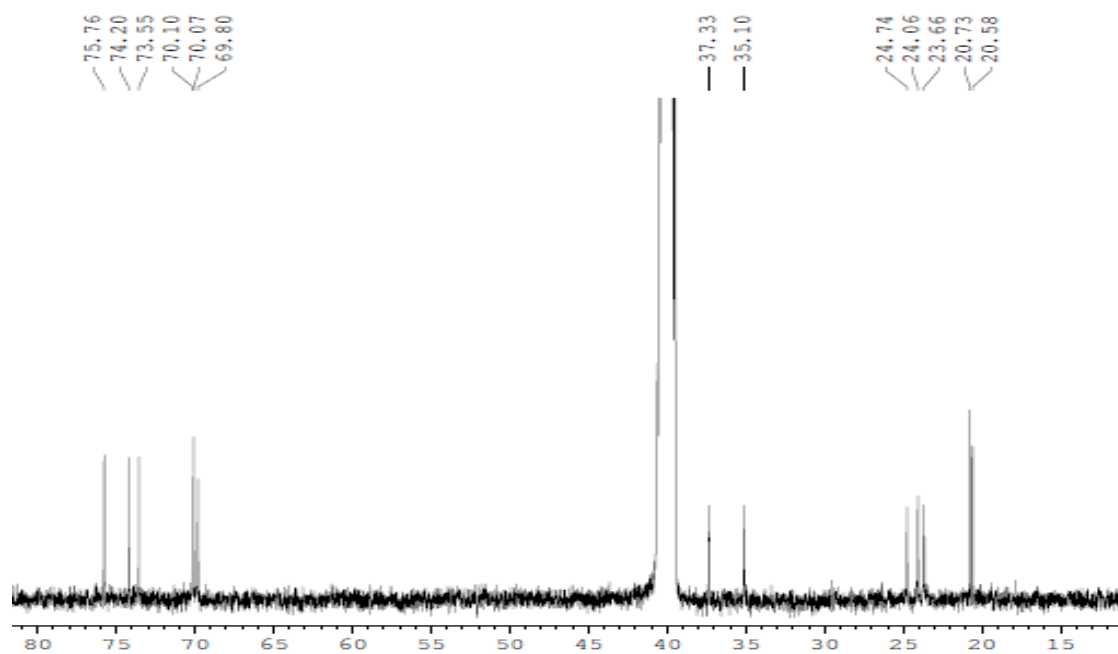
COSY of compound 26a



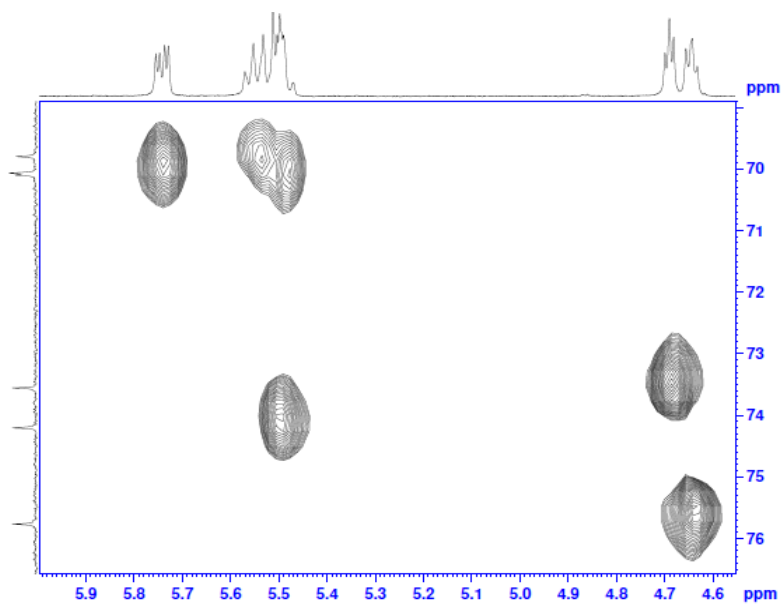
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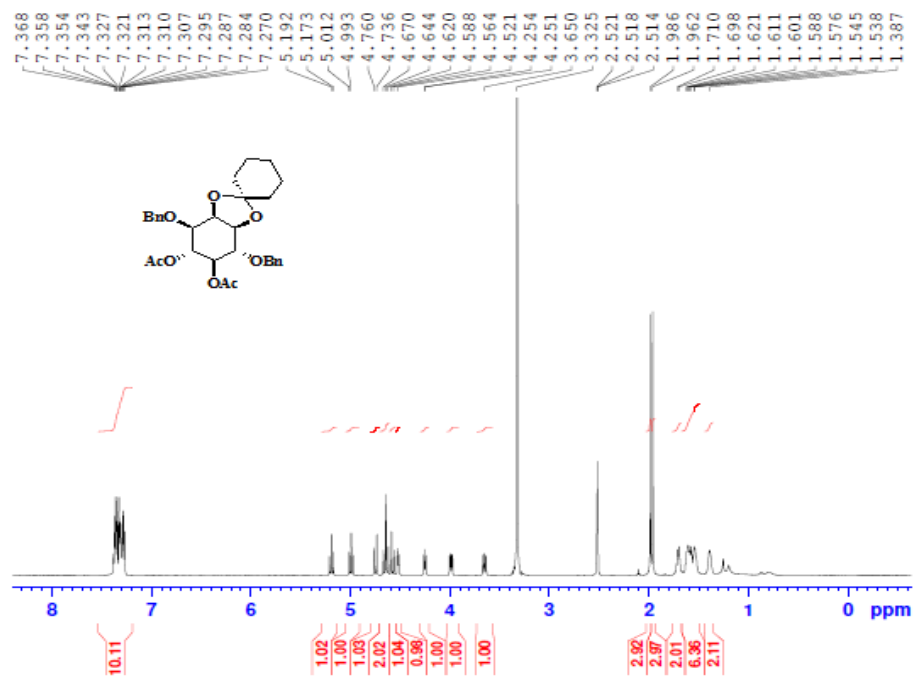
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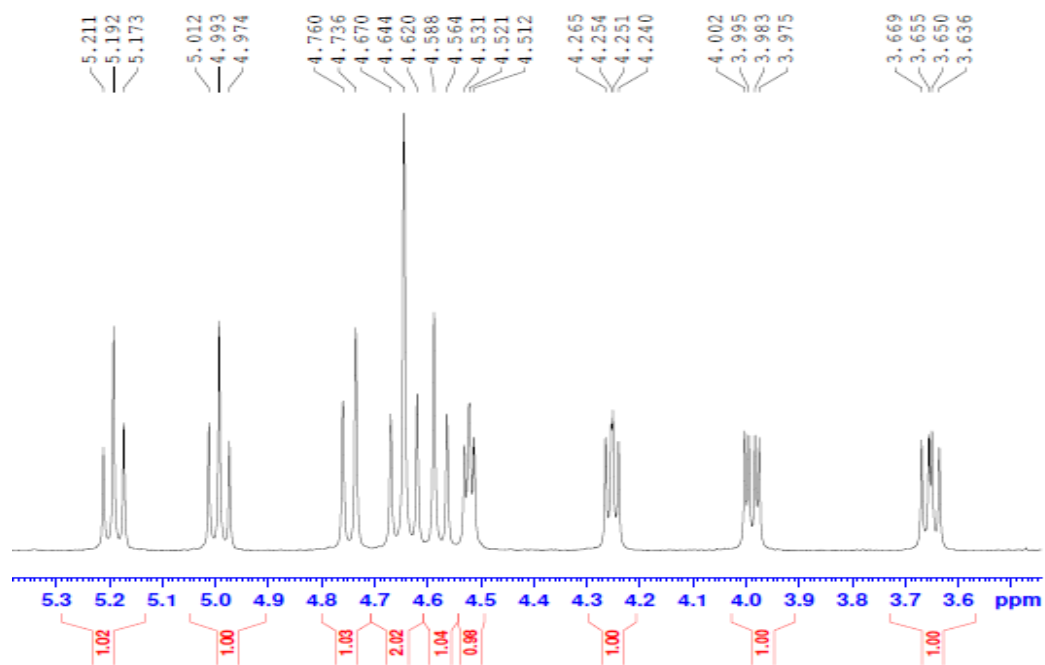
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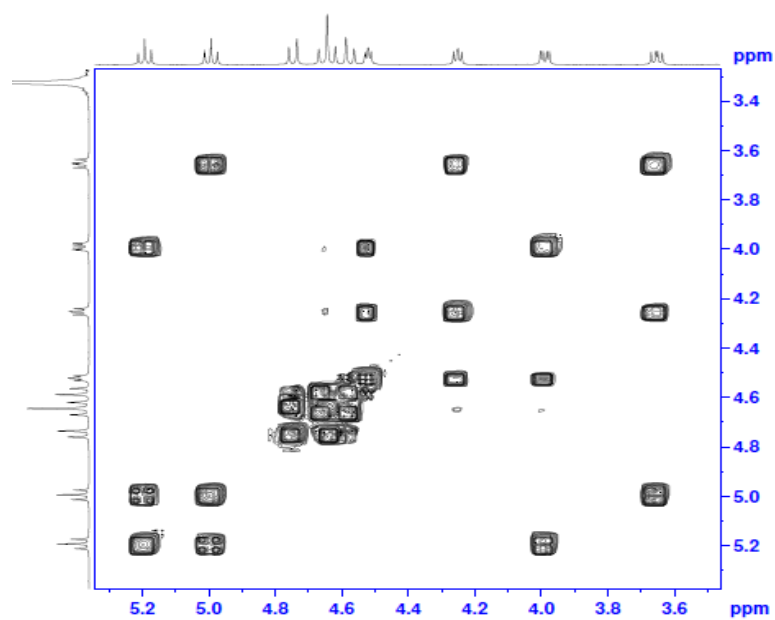
¹H NMR of compound 27a



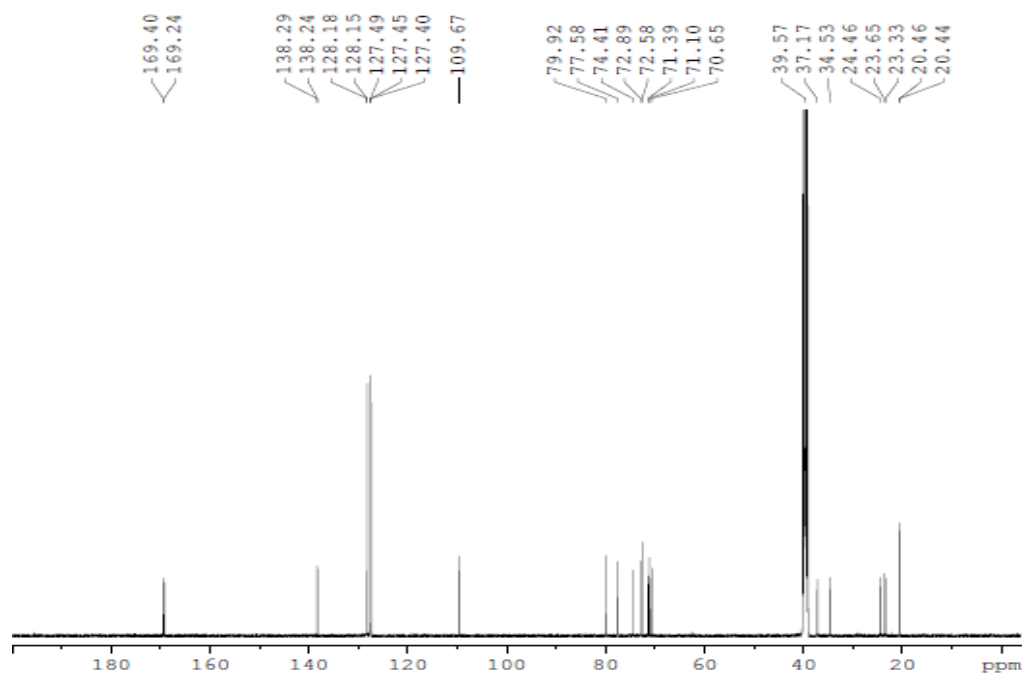
¹H NMR of compound 27a(zoom)



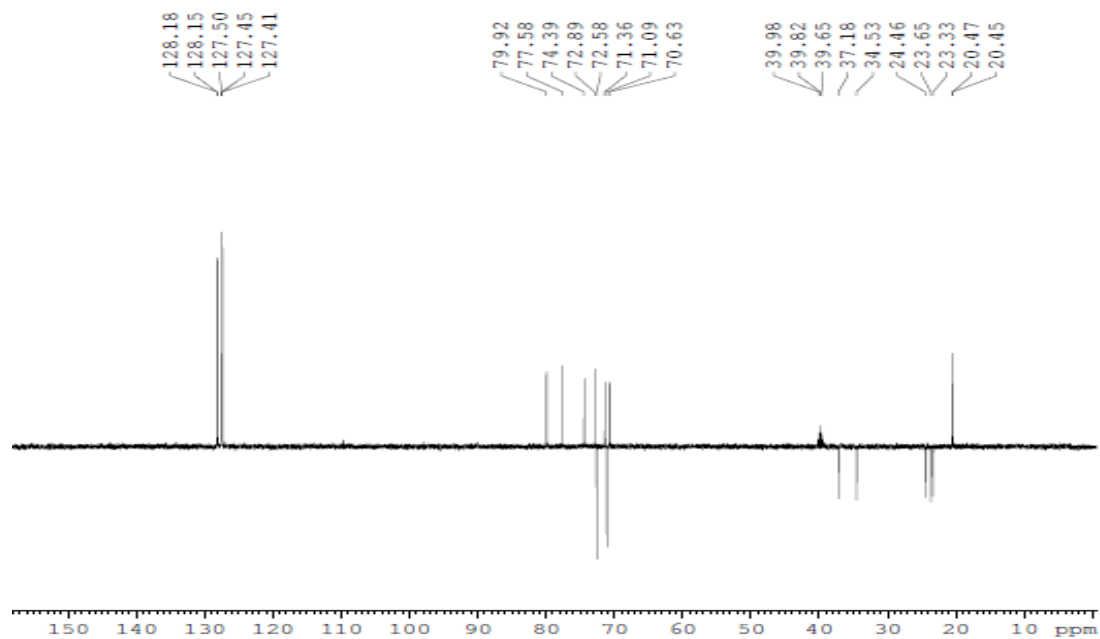
COSY of compound 27a



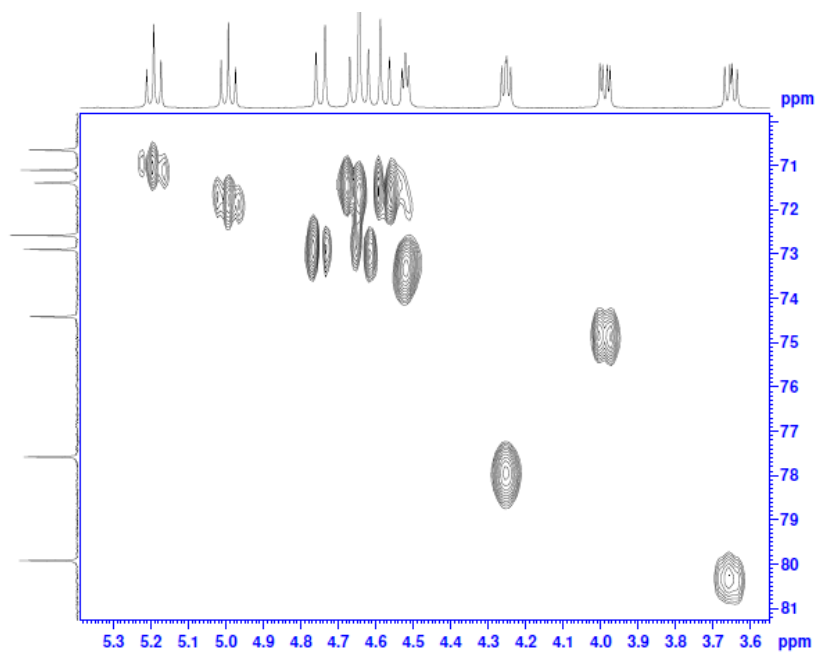
¹³C NMR of compound 27a



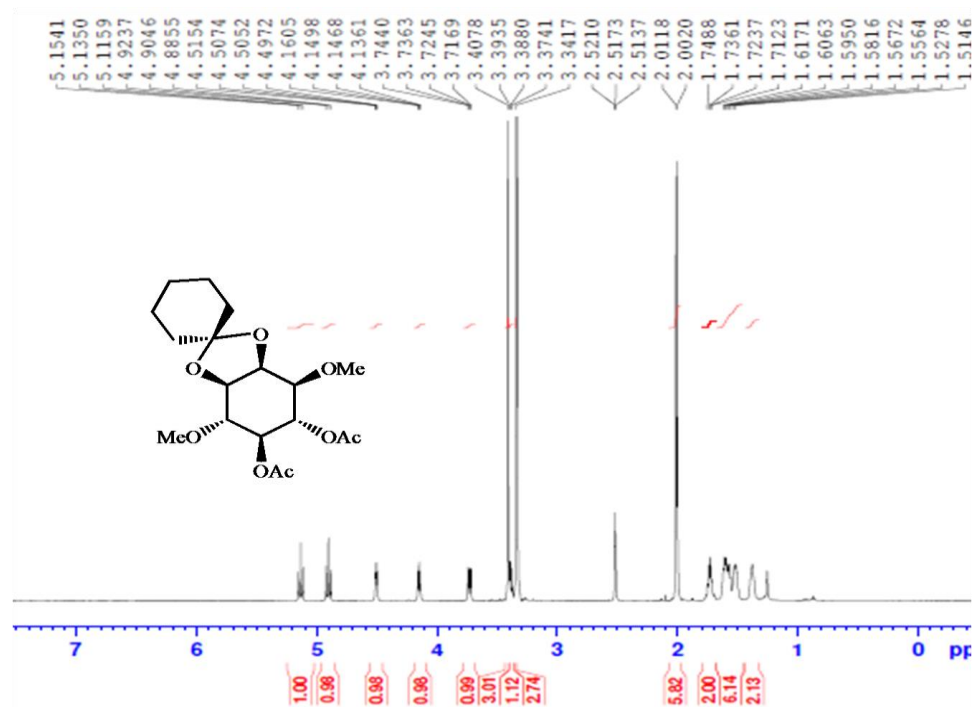
DEPT-135 of compound 27a



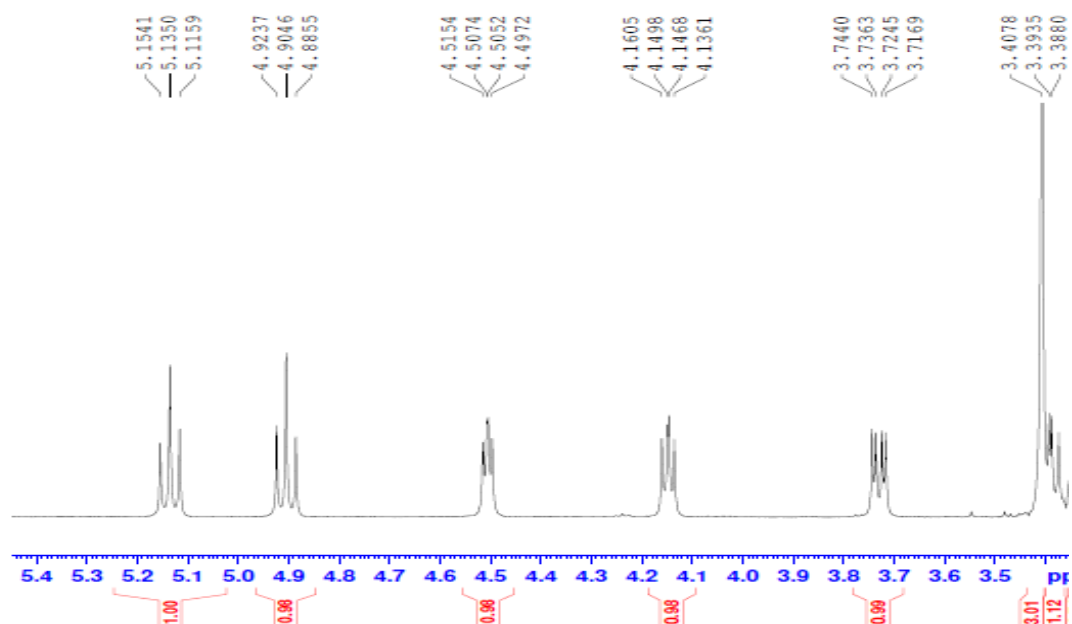
HMQC of compound 27a



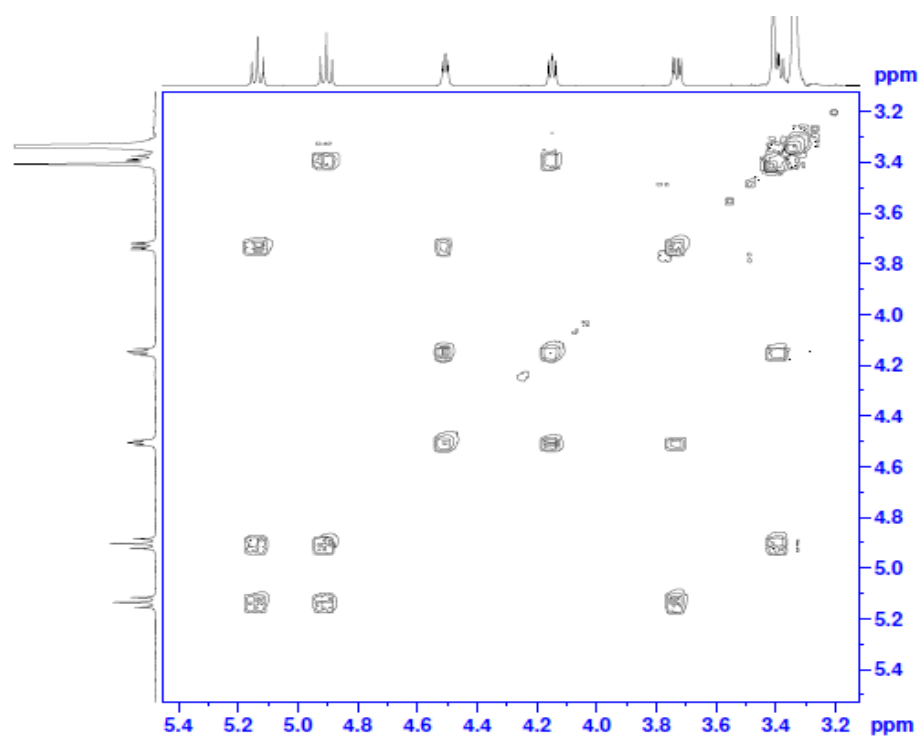
¹H NMR of compound 27b



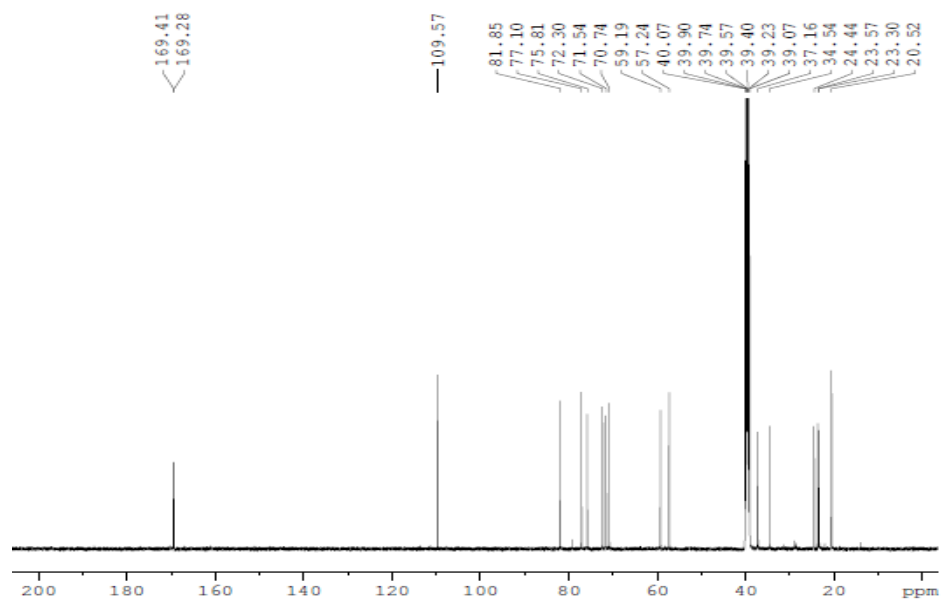
^1H NMR of compound 27b (zoom)



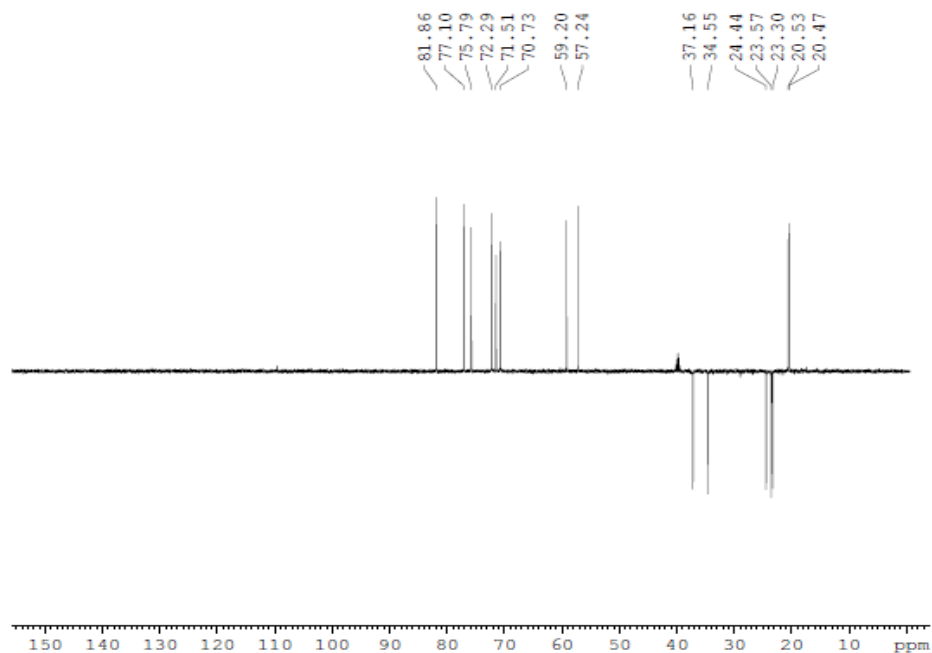
COSY of compound 27b



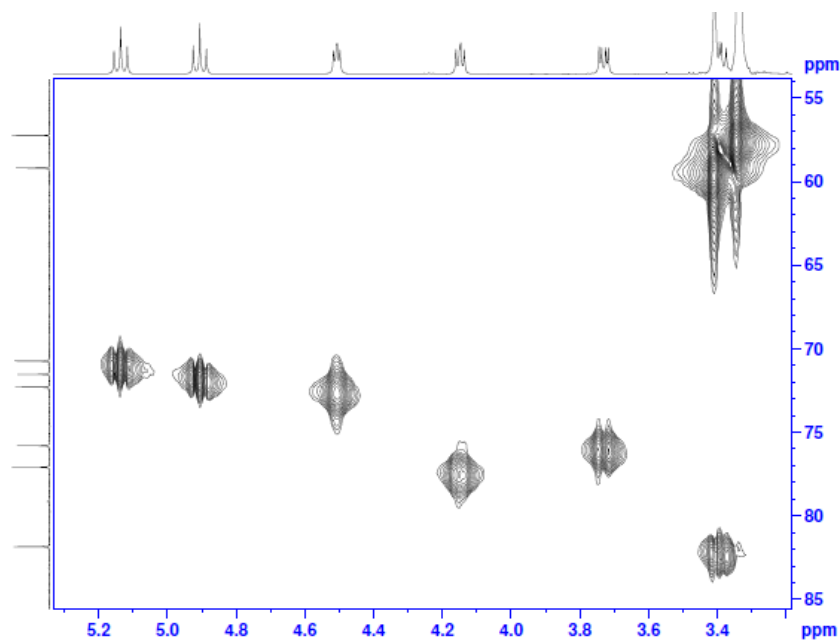
¹³C NMR of compound 27b



DEPT-135 of compound 27b

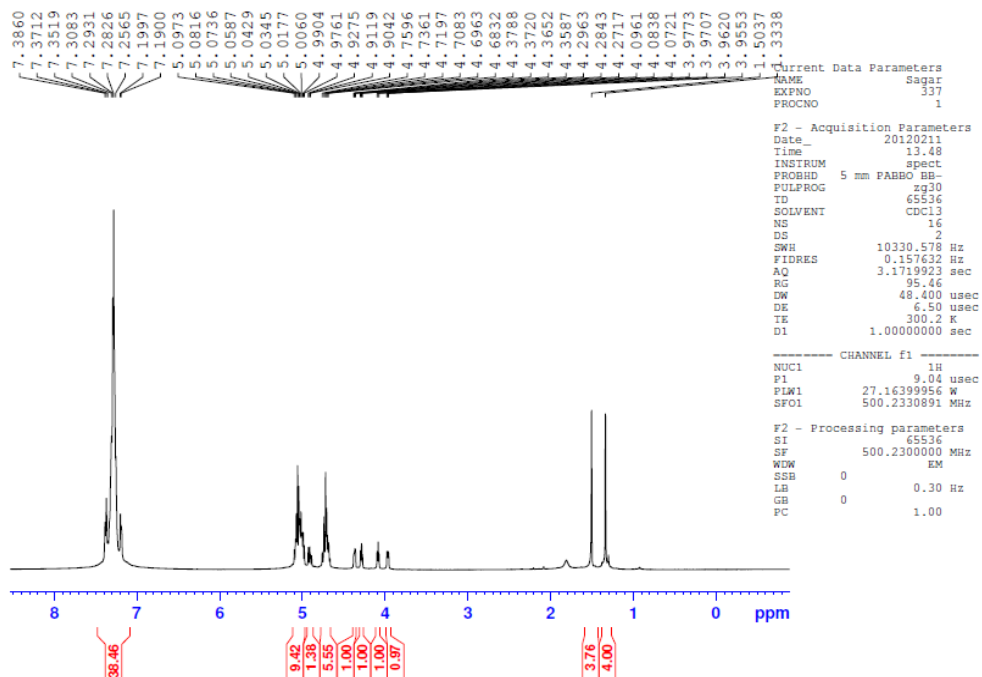


HMQC of compound 27b



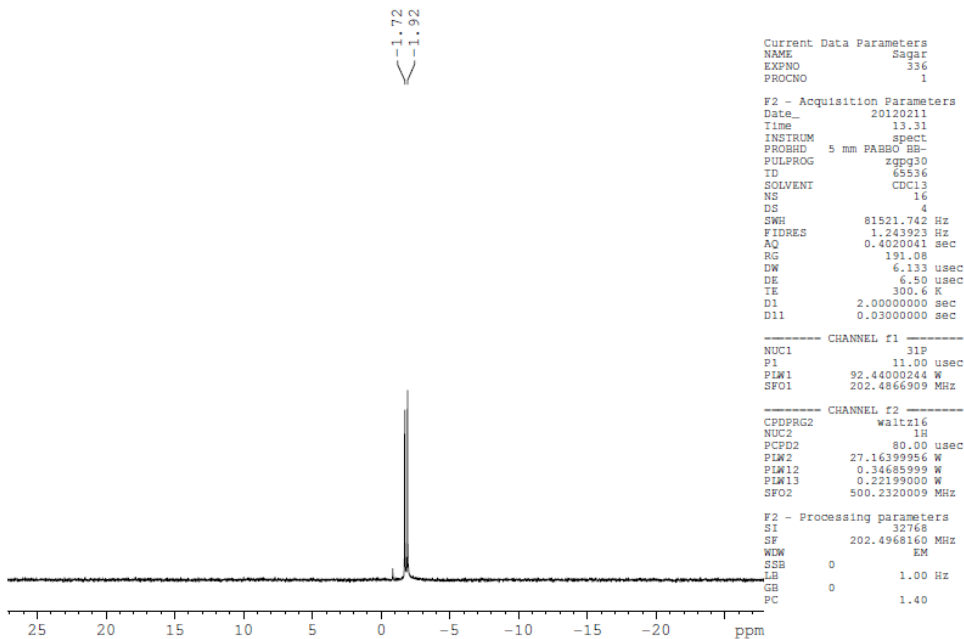
¹HNMR of compound (+)-35

KMS-D-500-1
PROTON CDCl₃ E:\ IISER



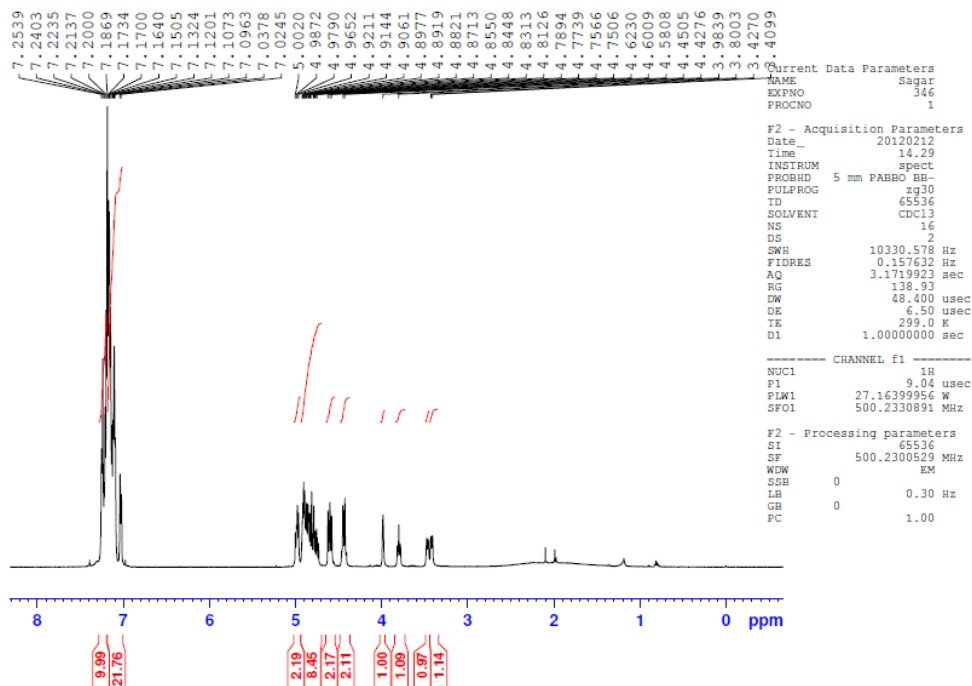
³¹P NMR of compound (+)-35

KMS-D-500-1
P31CPD CDCl3 E:\ IISER



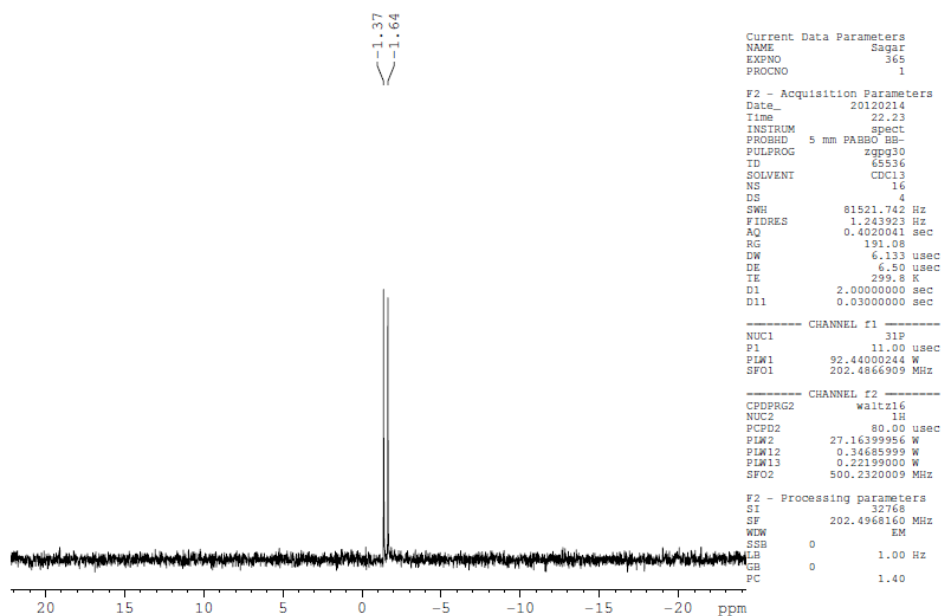
¹H NMR of compound (+)-36

KMS-D-501-2
PROTON CDCl3 E:\ IISER



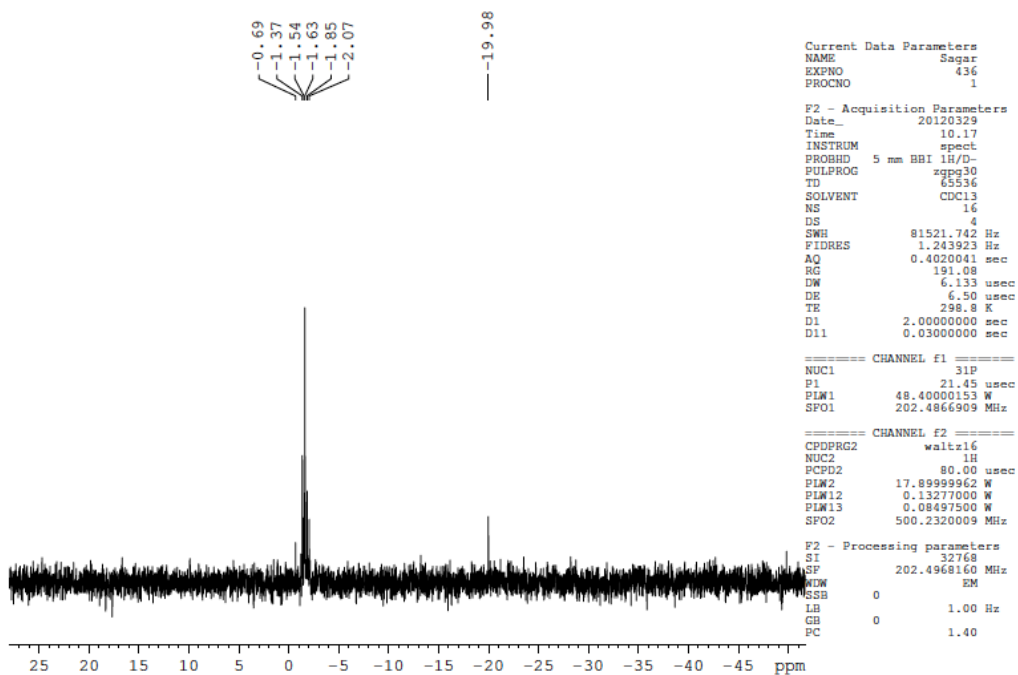
³¹P NMR of compound (+)-36

KMS-D-502-1
P31CPD CDCl3 E:\ IISER



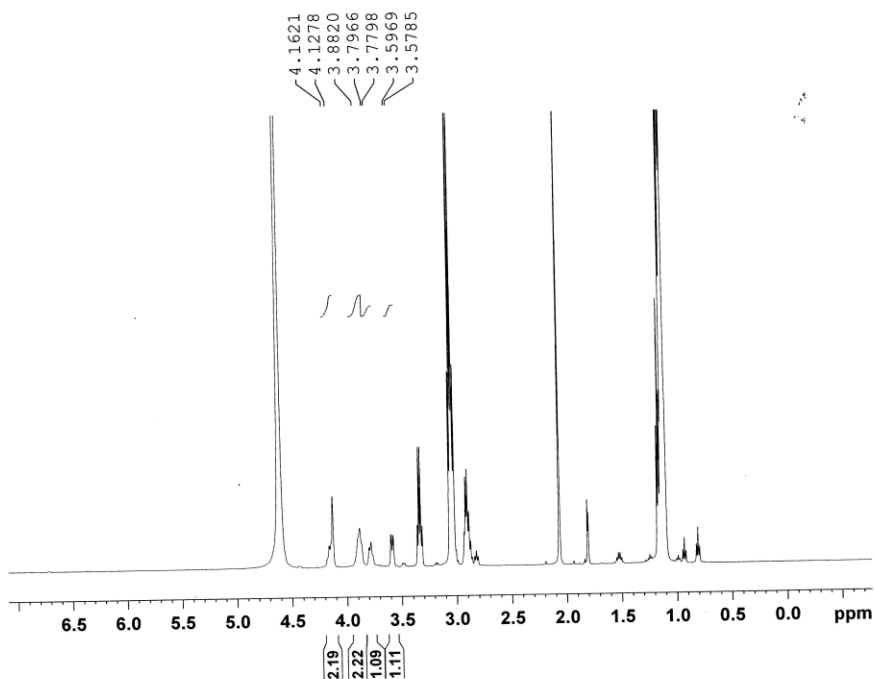
³¹P NMR of crude compound after POCl₃ phosphorylation

KMS-D-517-3
P31CPD CDCl3 E:\ IISER



¹H NMR of compound 5 (IP₃)

PROTON-IISER E:\ IISER



Current Data Parameters
NAME Sagar
EXPNO 477
PROCNO 1

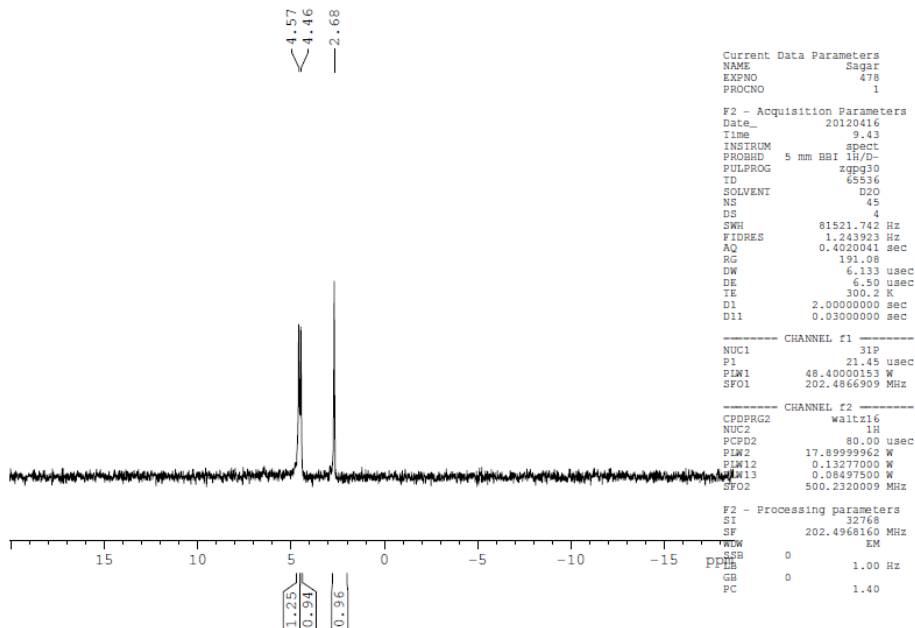
F2 - Acquisition Parameters
Date_ 20120416
Time 9.31
INSTRUM spect
PROBHD 5 mm BBI 1H/D-
PULPROG zg30
TD 65536
SOLVENT D2O
NS 68
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 85.95
DW 48.400 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 6.89 usec
PLW1 17.89999962 W
SFO1 500.2330891 MHz

F2 - Processing parameters
SI 65536
SF 500.2300520 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

³¹P NMR of compound 5(IP₃):

P31CPD E:\ IISER



Current Data Parameters
NAME Sagar
EXPNO 478
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120416
Time 9.43
INSTRUM spect
PROBHD 5 mm BBI 1H/D-
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 45
DS 4
SWH 81521.742 Hz
FIDRES 1.243923 Hz
AQ 0.4020041 sec
RG 191.08
DW 6.133 usec
DE 6.50 usec
TE 300.2 K
D1 2.00000000 sec
D11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 31P
P1 21.45 usec
PLW1 48.40000153 W
SFO1 202.4864909 MHz

===== CHANNEL f2 =====
CPDPRG2 waitz16
NUC2 1H
PCPD2 80.00 usec
PLW2 17.89999962 W
PLW12 0.13277000 W
PLW13 0.08497500 W
SFO2 500.2320009 MHz

F2 - Processing parameters
SI 32768
SF 202.4968160 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40