

Synthesis of halogenated reverse position C-Nucleosides against *Leishmania major* and *Leishmania tropica*.

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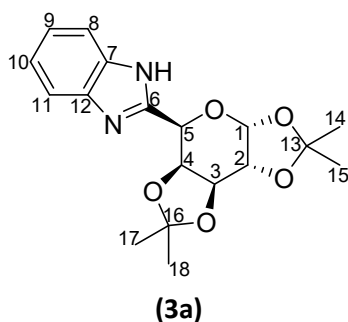
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1.	2-((3aR,5R,5aS,8aS,8bR)-2,2,7,7-tetramethyltetrahydro-5H-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-5-yl)-1H-benzo[d]imidazole (3a) :	2
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3.	5,6-dichloro-2-((3aR,5R,5aS,8aS,8bR)-2,2,7,7-tetramethyltetrahydro-5H-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-5-yl)-1H-benzo[d]imidazole (5a):	13
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•	FAB +ve low- and high- resolution Mass spectrometry of compound (6a)	23
5.	5-bromo-2-((3aR,5R,5aS,8aS,8bR)-2,2,7,7-tetramethyltetrahydro-5H-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-5-yl)-1H-benzo[d]imidazole (7a):	24
•	Mass spectrometry of compound (7a)	25
6.	2-((3a'R,5'R,5a'S,8a'S,8b'R)-tetrahydro-5'H-dispiro[cyclohexane-1,2'-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-7',1''-cyclohexan]-5'-yl)-1H-benzo[d]imidazole (3b)	26
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7.	5,6-difluoro-2-((3a'R,5'R,5a'S,8a'S,8b'R)-tetrahydro-5'H-dispiro[cyclohexane-1,2'-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-7',1''-cyclohexan]-5'-yl)-1H-benzo[d]imidazole (4b)	28
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8.	5,6-dichloro-2-((3a'R,5'R,5a'S,8a'S,8b'R)-tetrahydro-5'H-dispiro[cyclohexane-1,2'-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-7',1''-cyclohexan]-5'-yl)-1H-benzo[d]imidazole (5b):	32
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10.	5-bromo-2-((3a'R,5'R,5a'S,8a'S,8b'R)-tetrahydro-5'H-dispiro[cyclohexane-1,2'-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-7',1''-cyclohexan]-5'-yl)-1H-benzo[d]imidazole (7b) :	45
•	FAB +ve mood low and high resolution of compound 7b	46
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1. 2-((3a*R*,5*R*,5a*S*,8a*S*,8b*R*)-2,2,7,7-tetramethyltetrahydro-5H-bis([1,3]dioxolo) [4,5-b:4',5'-d]pyran-5-yl)-1H-benzo[d]imidazole (**3a**) :

- Spectral Analysis of (**3a**)



S. No.	H ¹ -NMR	Integration	J-value	C ¹³ -NMR
Galactose pyranose ring				
1	5.655-5.645 (<i>d</i>)	1	5 Hz (<i>axial-equatorial</i>)	-CH 96.5
2	4.416-4.402 (<i>dd</i>)	1	$J_1 = 2\text{Hz}$ (<i>di-equatorial</i>) $J_2 = 4.85\text{Hz}$ (<i>axial-equatorial</i>)	-CH 66.1
3	4.746-4.726 (<i>dd</i>)	1	$J_1 = 7.4\text{ Hz}$ (<i>di-axial</i>) $J_2 = 2\text{ Hz}$ (<i>di-equatorial</i>)	-CH 72.58
4	4.705-4.685 (<i>dd</i>)	1	$J_1 = 7.85\text{ Hz}$ (<i>di-axial</i>) $J_2 = 2\text{Hz}$ (<i>di-equatorial</i>)	-CH 70.54
5	5.216-5.213 (<i>d</i>)	1	$J = 1.5\text{ Hz}$ (<i>di-equatorial</i>)	-CH 70.80
Benzimidazole				
6	-	-	-	-C 152.43
7	-	-	-	-
8-11	7.591 (broad signal)	4	-	-
12	-	-	-	-
Iso-propyl acetyl group				
13	-	-	-	-C 109.42
14-15	1.427 (<i>s</i>) - 1.353(<i>s</i>)	3 each	-	-CH ₃ 26.01 -CH ₃ 24.88
16	-	-	-	-C 109.72
17-18	1.547 (<i>s</i>)- 1.300(<i>s</i>)	3 each	-	-CH ₃ 26.2 -CH ₃ 24.07
Imidazole				
NH (imidazole)	9.66 (rough broad)	1	-	-

Table S1: H¹NMR and C¹³ NMR of compound (**3a**) along with *J*-values.

- Single Crystal XRD of compound (**3a**)

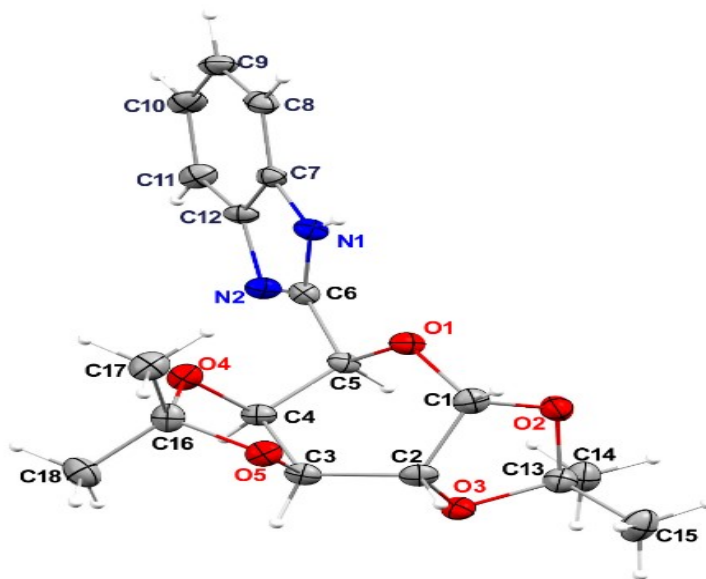


Figure S1: ORTEP view of compound (**3a**) at 24 % probability.

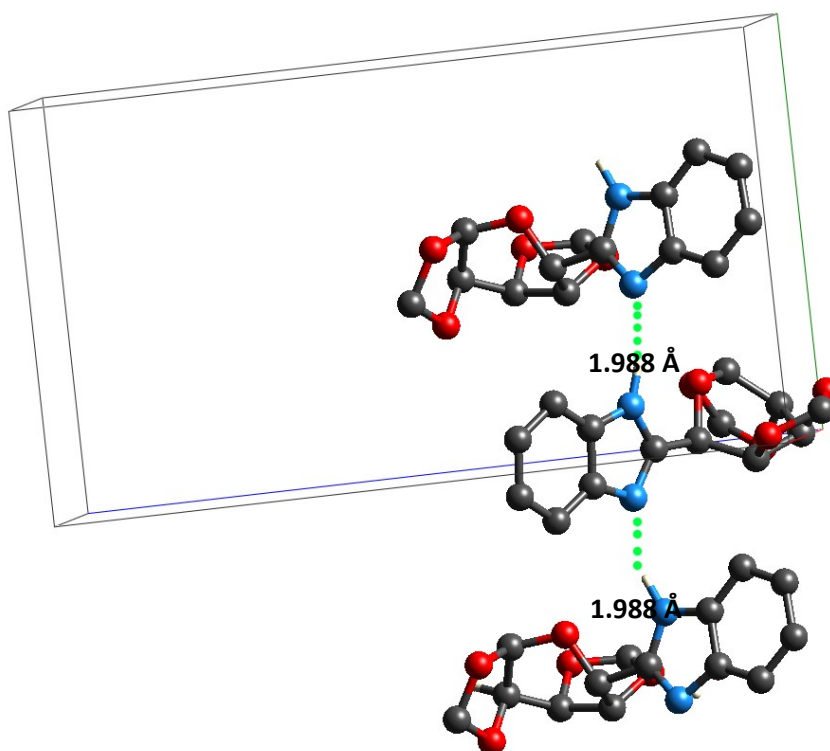


Figure S2: Hydrogen bonded imidazole ring in 1.988 Å along *a* of compound (**3a**) in a unit cell.

Identification code	UKGALH_0ma	
Empirical formula	C ₃₆ H ₄₄ N ₄ O ₁₀	
Formula weight	692.75	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P 21	
Unit cell dimensions	a = 9.9303(7) Å	α = 90°.
	b = 17.9109(12) Å	β = 94.298(4)°.
	c = 10.1057(7) Å	γ = 90°.
Volume	1792.4(2) Å ³	
Z	2	
Density (calculated)	1.284 Mg/m ³	
Absorption coefficient	0.781 mm ⁻¹	
F(000)	736	
Crystal size	0.110 x 0.100 x 0.030 mm ³	
Theta range for data collection	4.387 to 68.288°.	
Index ranges	-11 ≤ h ≤ 11, -21 ≤ k ≤ 21, -12 ≤ l ≤ 12	
Reflections collected	41507	
Independent reflections	6527 [R(int) = 0.0799]	
Completeness to theta = 67.679°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.977 and 0.919	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	6527 / 1 / 464	
Goodness-of-fit on F ²	1.064	
Final R indices [I > 2σ(I)]	R1 = 0.0374, wR2 = 0.0858	
R indices (all data)	R1 = 0.0443, wR2 = 0.0899	
Absolute structure parameter	0.27(19)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.179 and -0.249 e.Å ⁻³	
CCDC	2296171	

Table S2: Description of single crystal XRD analysis of compound (**3a**).

- FAB +ve high- and low-resolution mass of (**3a**):

File: UKGALH-FABP

Date Run: 07-31-2023 (Time Run: 09:59:40)

Sample: UMAIR /PROF. DR. SAMMER

Instrument: JEOL-600H-2

Inlet: My Inlet

Ionization mode: FAB+

Run By: HEJ-MASS-LAB

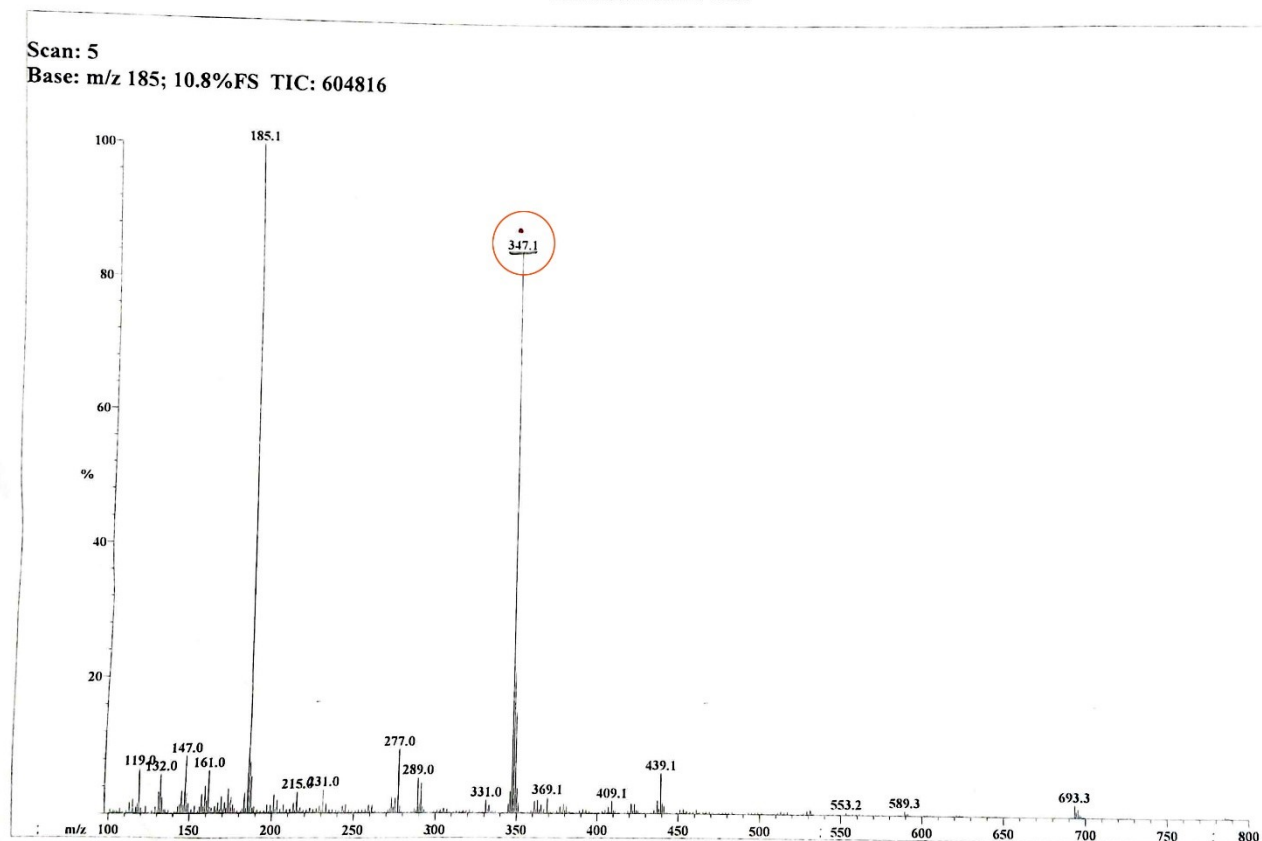
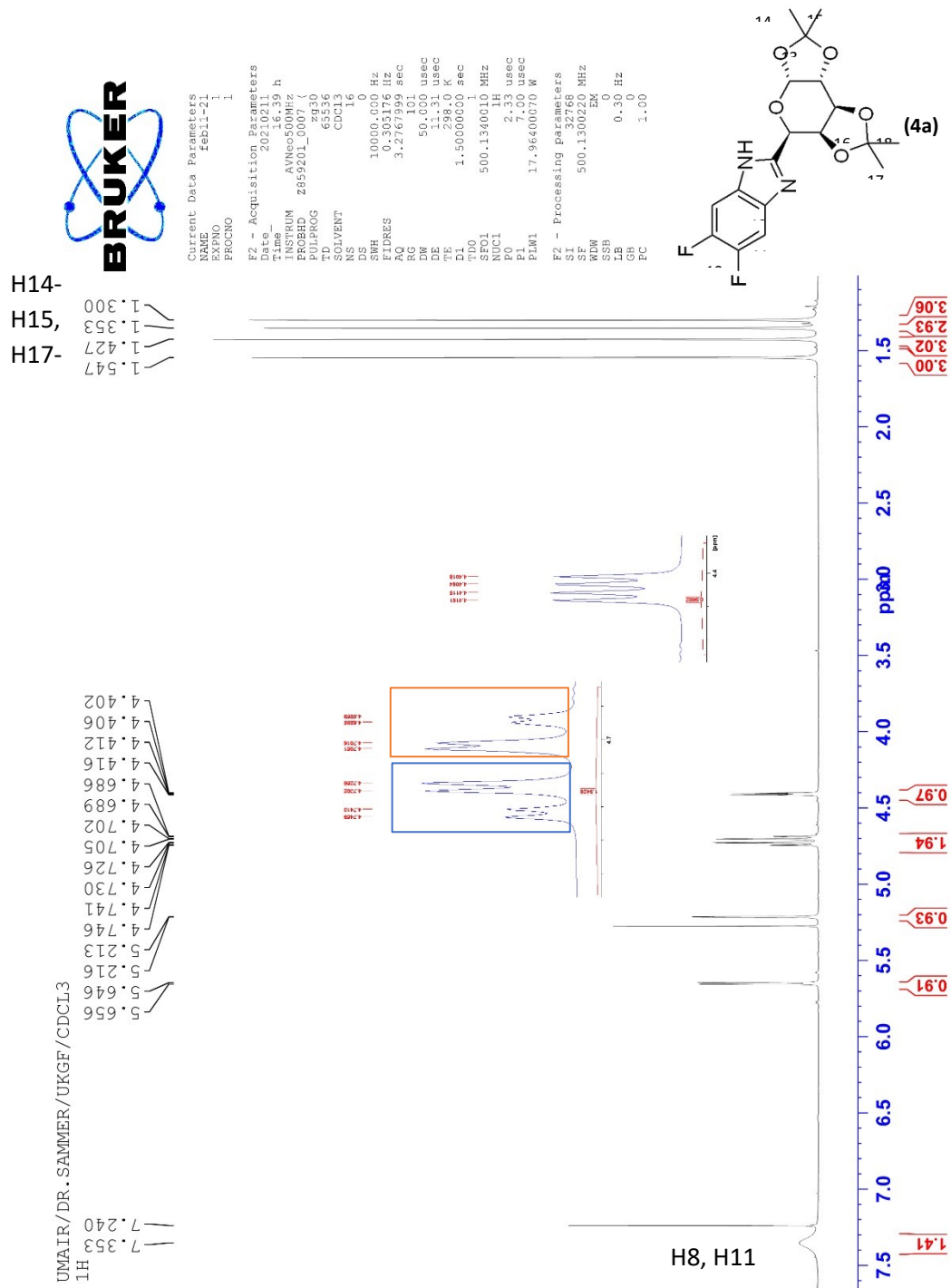


Figure-S3: Low resolution positive mode of FAB spectra of **3a**. Encircled is the m/z value 347.1.

High resolution FAB ($m+1$) positive mode at JEOL HX 110 Mass spectrometer of compound **3a** is **347.1607**, while theoretical mass was **347.1624** which corresponds to the composition of **C₁₈H₂₃O₅N₂**.

2. 5,6-difluoro-2-((3*aR*,5*R*,5*aS*,8*aS*,8*bR*)-2,2,7,7-tetramethyltetrahydro-5H-bis([1,3]dioxolo)[4,5-*b*:4',5'-*d*]pyran-5-yl)-1H-benzo[d]imidazole (4a):

- Spectral Analysis of compound (4a) :



¹H-NMR at 500MHz, in compound 5,6-difluoro-2-((3*aR*,5*R*,5*aS*,8*aS*,8*bR*)-2,2,7,7-tetrahydro-5H-bis([1,3]dioxolo)[4,5-*b*:4',5'-*d*]pyran-5-yl)-1H-benzo[d]imidazole (4a)



UMAIR/DR. SAMEER/UKGE/CDCL3

BB

152.43
149.06
148.94
147.02

109.69
109.42

72.58
70.81
70.52
65.99

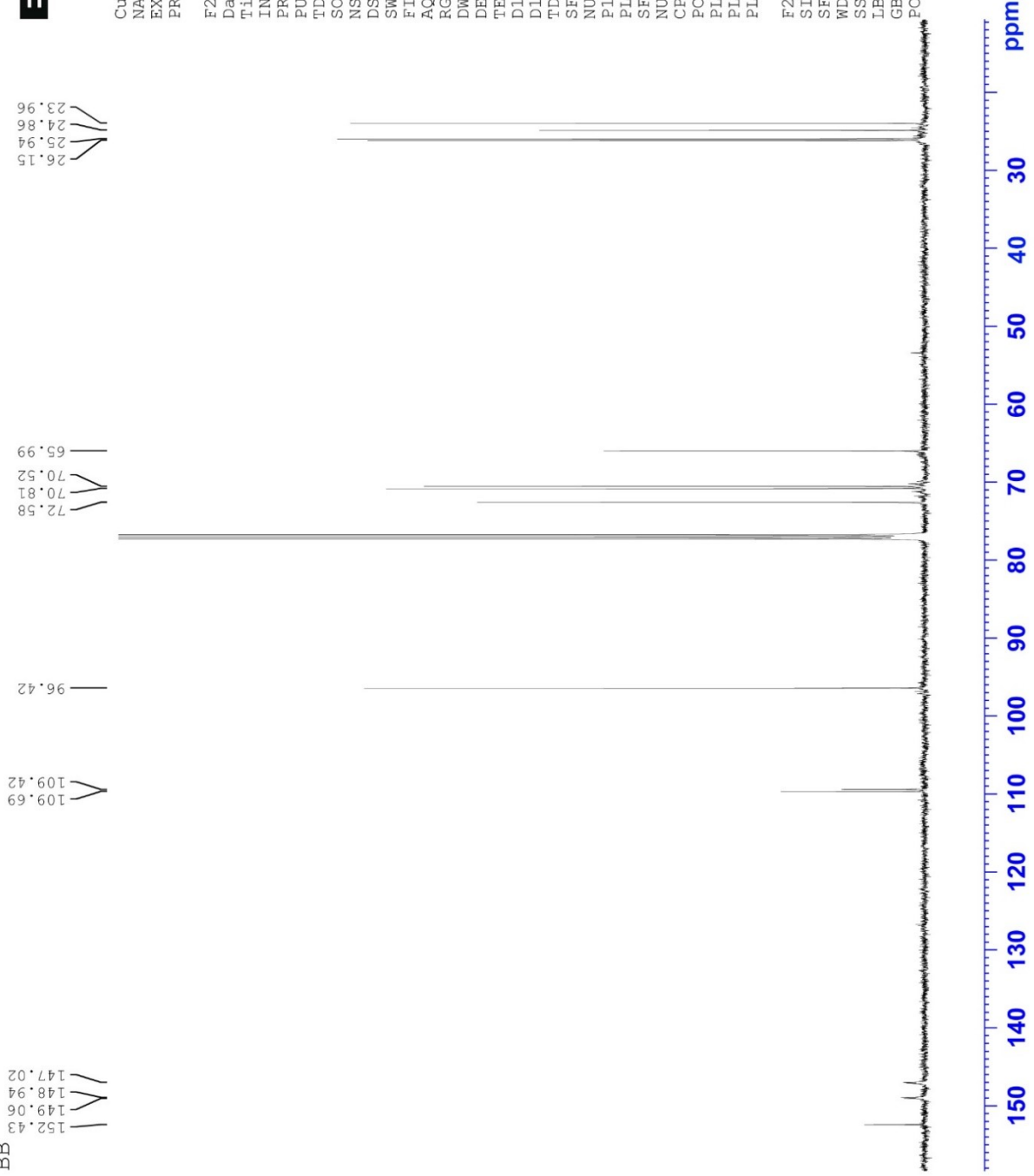
26.15
25.94
24.86
23.96

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EXPNO 1
PROCNO 1

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SOLVENT CDCL3
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FIDRES 1.838408 Hz
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DE 12.00 usec
TE 298.0 K
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D11 0.03000000 sec
TD0 16
SF01 125.7973986 MHz
NUC1 13C
P1 12.00 usec
PLW1 229.03999329 W
SF02 500.2320009 MHz
NUC2 1H
CPDPRG[2] waltz65
PCPD2 80.00 usec
PLW2 18.45899963 W
PLW12 0.18459000 W
PLW13 0.09284900 W

F2 - Processing parameters
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EM
SSB 0
LB 1.00 Hz
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500MHz, in
-difluoro-2-
2,2,7,7-
I-
,5'-d]pyran-
ole (4a)

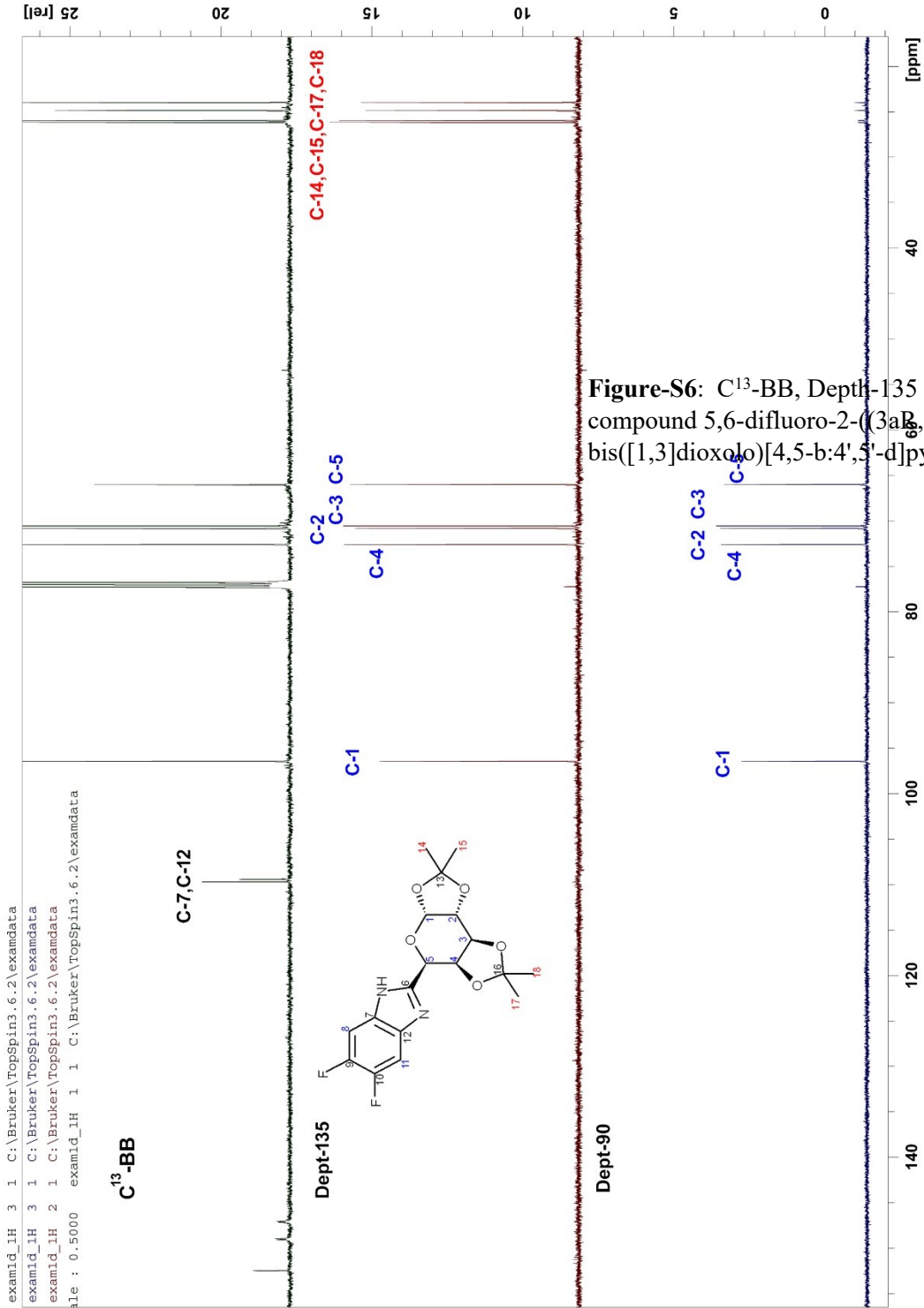


Figure-S6: C¹³-BB, Dept-135 and Dept-90 NMR at 500MHz of compound 5,6-difluoro-2-((3aR,5R,5aS,8aS,8bR)-2,2,7,7-tetrahydrobis[1,3]dioxolo[4,5-b':4',5'-d]pyran-5-yl)-1H-benzo[d]imidazole


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PROCNO        1

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SFO93         125.7688522 MHz
NUC93         13C
P94           15.00 usec
SFO94         125.7688522 MHz
NUC94         13C
P95           15.00 usec
SFO95         125.7688522 MHz
NUC95         13C
P96           15.00 usec
SFO96         125.7688522 MHz
NUC96         13C
P97           15.00 usec
SFO97         125.7688522 MHz
NUC97         13C
P98           15.00 usec
SFO98         125.7688522 MHz
NUC98         13C
P99           15.00 usec
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NUC99         13C
P100          15.00 usec
SFO100        125.7688522 MHz
NUC100        13C

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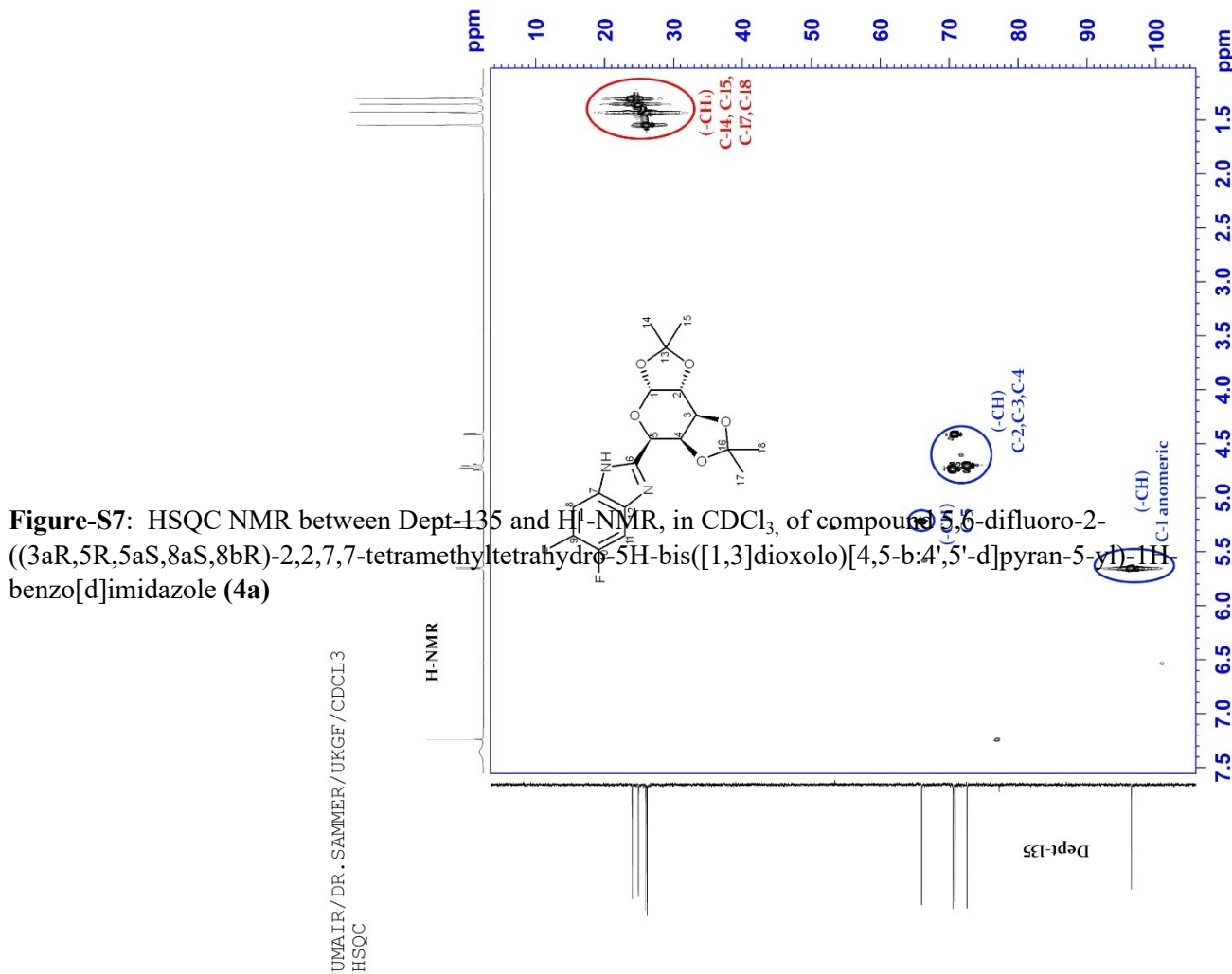


Figure-S7: HSQC NMR between Dept-135 and ¹H-NMR, in CDCl₃, of compound 5,6-difluoro-2-((3aR,5R,5aS,8aS,8bR)-2,2,7,7-tetramethyltetrahydro-5H-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-5-yl)-1H-benzo[d]imidazole (**4a**)

- Single Crystal XRD analysis of (**4a**)

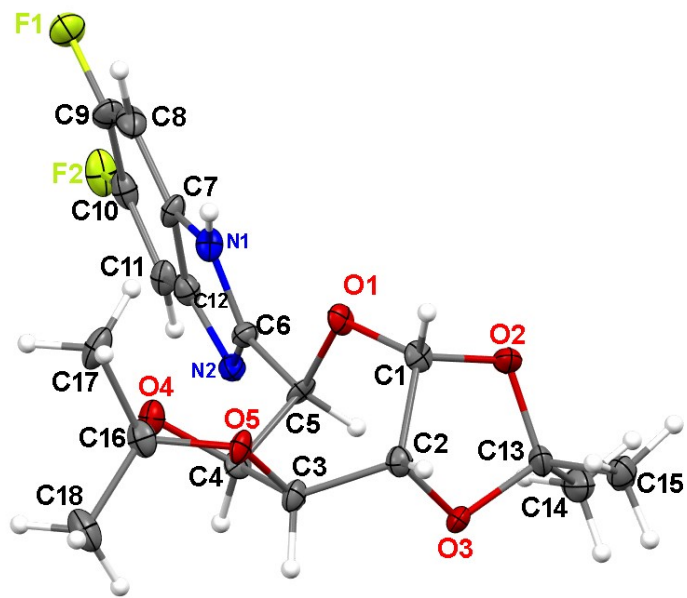


Figure-S7: ORTEP view of the compound (4a) at 40% probability.

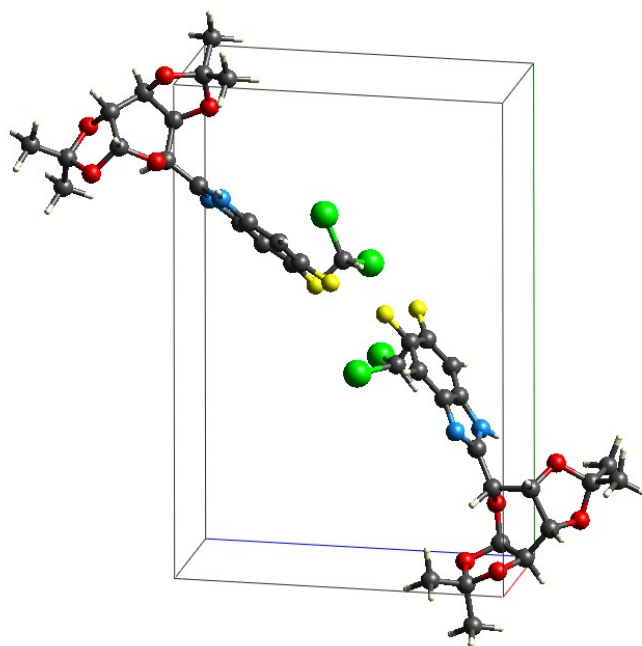
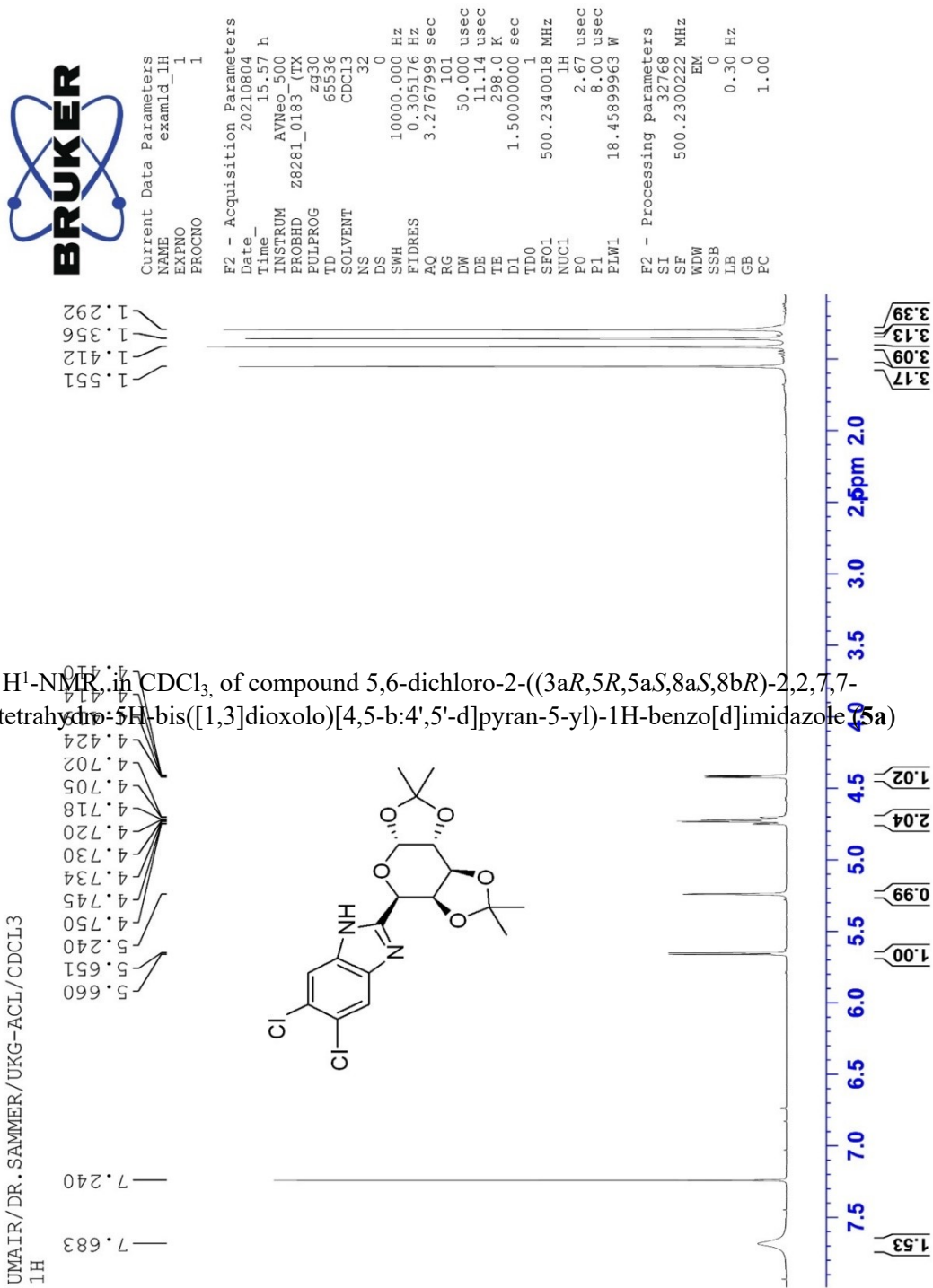


Figure S8: Crystal packing of compound (4a) in a unit cell, with two DCM molecules in unit cell.

Identification code	4a	
Empirical formula	C ₁₉ H ₂₂ C ₁₂ F ₂ N ₂ O ₅	
Formula weight	467.28	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P 21	
Unit cell dimensions	a = 10.0713(12) Å	α = 90°.
	b = 17.542(2) Å	β = 106.352(7)°.
	c = 12.5246(15) Å	γ = 90°.
Volume	2123.2(4) Å ³	
Z	4	
Density (calculated)	1.462 Mg/m ³	
Absorption coefficient	3.215 mm ⁻¹	
F(000)	968	
Crystal size	0.310 x 0.310 x 0.024 mm ³	
Theta range for data collection	3.678 to 68.288°.	
Index ranges	-12 ≤ h ≤ 12, -21 ≤ k ≤ 21, -15 ≤ l ≤ 15	
Reflections collected	40977	
Independent reflections	7405 [R(int) = 0.1428]	
Completeness to theta = 67.679°	99.6 %	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	7405 / 1 / 550	
Goodness-of-fit on F ²	1.053	
Final R indices [I > 2σ(I)]	R1 = 0.0825, wR2 = 0.1923	
R indices (all data)	R1 = 0.1147, wR2 = 0.2122	
Absolute structure parameter	0.128(14)	
Extinction coefficient	0.0050(7)	
Largest diff. peak and hole	1.299 and -0.975 e.Å ⁻³	
CCDC	2300614	

Table S3: Crystal packing of compound (**4a**) in a unit cell, with two DCM molecules in unit cell.

3. 5,6-dichloro-
bis([1,3
Spectra



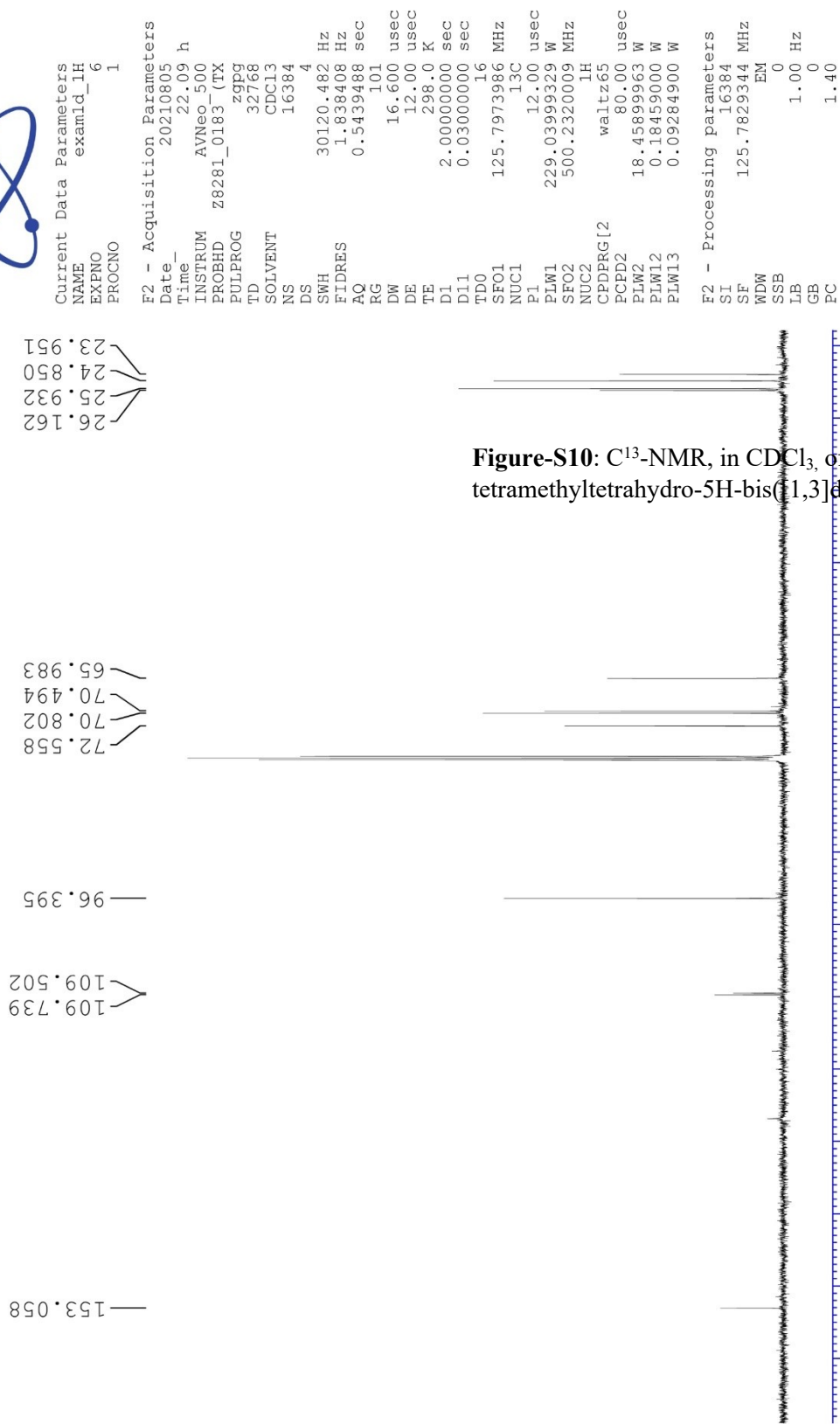


Figure-S10: C^{13} -NMR, in $CDCl_3$, of compound 5,6-dichloro-2-(tetramethyltetrahydro-5H-bis(1,3)dioxolo)[4,5-b:4',5'-d]pyran-5-ylidene

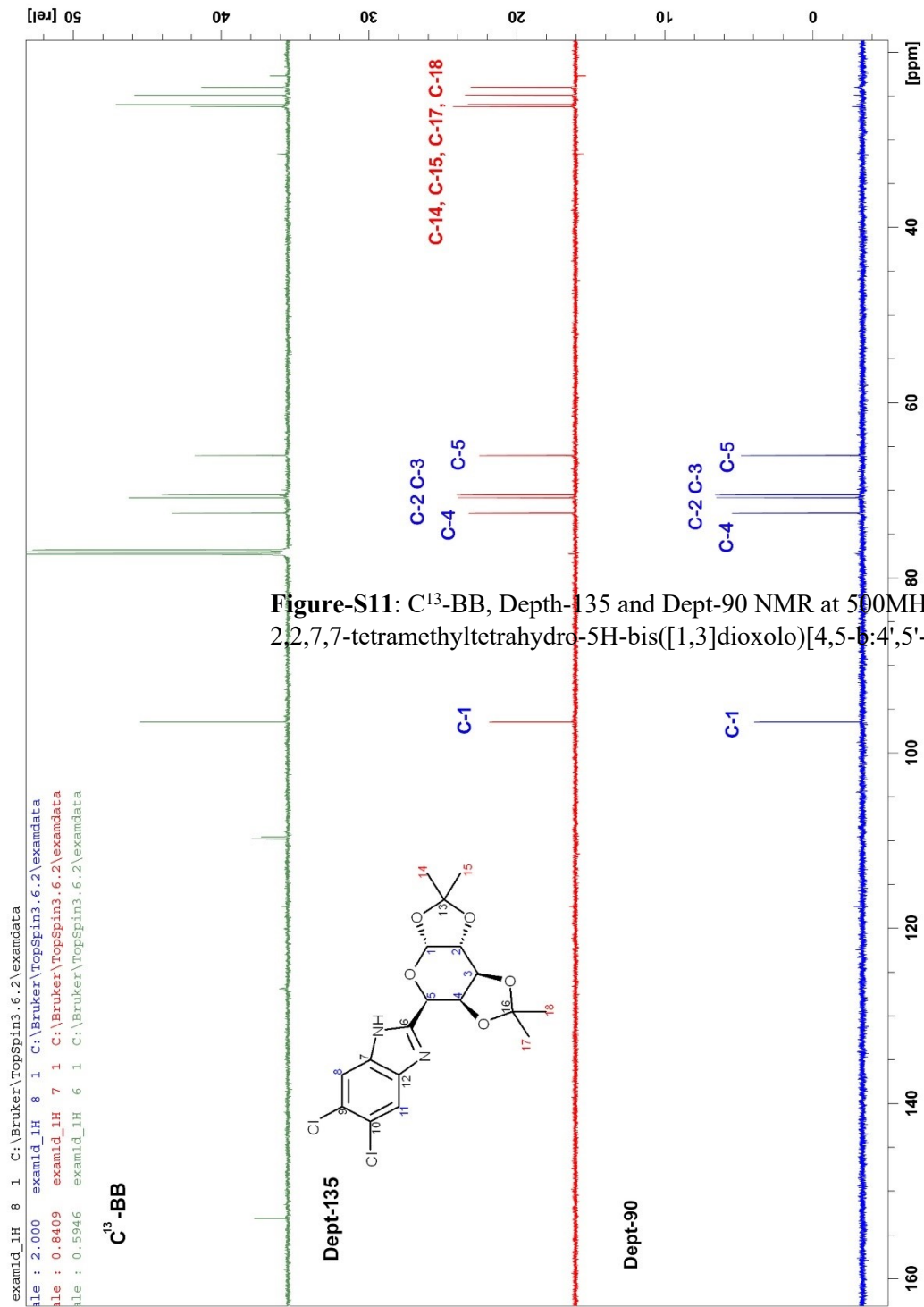


Figure-S11: C¹³-BB, Dept-135 and Dept-90 NMR at 500MHz 5,6-dichloro-2,2,7,7-tetramethyltetrahydro-5H-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-5



Current Data Parameters
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 EXPNO 2
 PROCNO 1

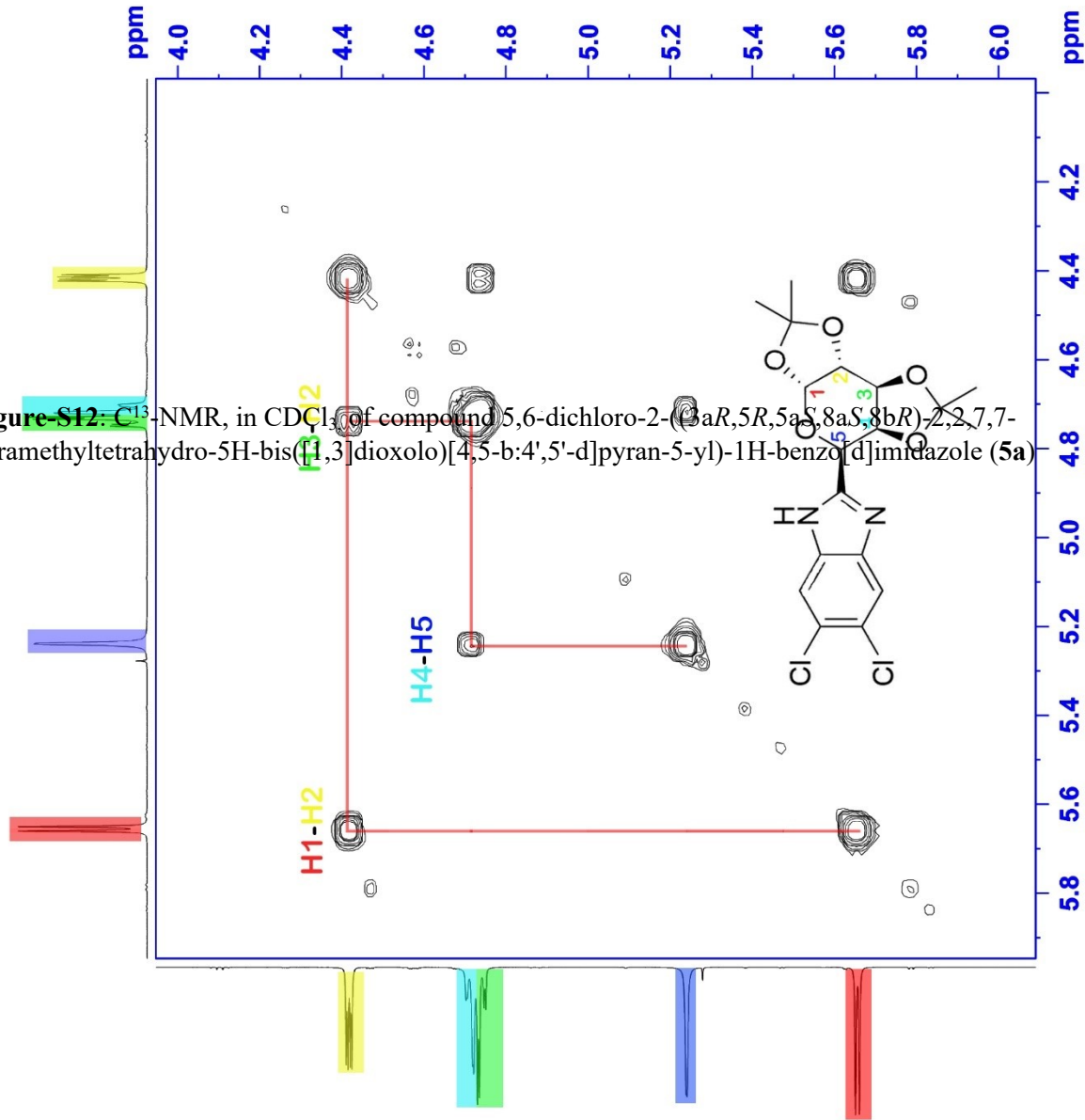
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 PULPROG zgpg30
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 SOLVENT CDCl3
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 DS 8
 SWH 5882.353 Hz
 FIDRES 5.744485 Hz
 AQ 0.1740800 sec
 RG 101
 DW 85.000 usec
 DE 6.50 usec
 TE 298.0 K
 D0 0.00000300 sec
 D1 1.50000000 sec
 D13 0.00000400 sec
 D16 0.00020000 sec
 INO 0.00017000 sec
 TDel 1
 SFO1 500.2330014 MHz
 NUC1 1H
 P0 8.00 usec
 P1 8.00 usec
 PLW1 18.4589963 W
 GPNAM[1] SMSQ10.100
 GPZ1 10.00 %
 F16 1000.00 usec

F1 - Acquisition parameters
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 SFO1 500.233 MHz
 FIDRES 45.955883 Hz
 SW 11.759 ppm
 FnmODE QF

F2 - Processing parameters
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 SF 500.2300222 MHz
 WDW QNONE
 SSB 0
 LB 0 Hz
 GB 0
 FC 1.40

F1 - Processing parameters
 SI 1024
 MC2 QF
 SF 500.2300222 MHz
 WDW QNONE
 SSB 0
 LB 0 Hz
 GB 0

Figure S12: ¹³C-NMR, in CDCl₃, of compound 5,6-dichloro-2-((3*aR*,5*R*,5*aS*,8*aS*,8*bR*)-2,2,7,7-tetramethyltetrahydro-5*H*-bis([1,3]dioxolo)[4,5-*b*:4',5'-*d*]pyran-5-yl)-1*H*-benzo[*d*]imidazole (5a)



UMAIR/DR. SAMMER/UKG-ACL/CDCl₃
HSQC



Current Data Parameters
NAME exam1c_1h
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
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Time 0.06 h
Date_ 20210905
Time 0.06 h
PROBHD 28281 0183 TX
PULPROG zgpg30
F1FLPROG zgpg30
TD 1024
SOLVENT CDCl₃
NS 32
DS 4
SWH 5882.353 Hz
FIDRES 11.488971 Hz
AQ 0.0870400 sec
RG 101
DW 85.000 usec
DE 1.300 usec
TE 298.0 K
CNST2 145.0000000
D0 0.0000000 sec
D1 1.5000000 sec
D2 0.0000000 sec
D3 0.0000000 sec
D4 0.0000000 sec
D5 0.0000000 sec
D6 0.0000000 sec
D7 0.0000000 sec
D8 0.0000000 sec
D9 0.0000000 sec
D10 0.0000000 sec
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D16 0.0000000 sec
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D19 0.0000000 sec
D20 0.0000000 sec
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D88 0.0000000 sec
D89 0.0000000 sec
D90 0.0000000 sec
D91 0.0000000 sec
D92 0.0000000 sec
D93 0.0000000 sec
D94 0.0000000 sec
D95 0.0000000 sec
D96 0.0000000 sec
D97 0.0000000 sec
D98 0.0000000 sec
D99 0.0000000 sec
D100 0.0000000 sec

ZGPGTNS
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NUC1 1H
F1 8.00 usec
P1 16.00 usec
PCPD2 10.00 usec
PLW1 18.45899963 W
SFO2 125.7948829 MHz
NUC2 13C
CPDPRG2 garp
P3 12.00 usec
PCPD3 10.00 usec
PLW2 229.03999329 W
PLW12 6.73089981 W
GPNAM[1] SWSQ10.100 %
GPNAM[2] SWSQ10.100 %
GPZ2 20.10 %
PI6 1000.00 usec

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FIDRES 196.293976 Hz
SW 199.735 ppm
FMODE Echo-Antiecho

F2 - Processing parameters
SI 1024
SF 500.2300222 MHz
WDW EM
SSB 2
LB 0 Hz
GB 1.40

F1 - Processing parameters
SI 1024
SF 125.792976 MHz
WDW EM
SSB 2
LB 0 Hz
GB 1.40

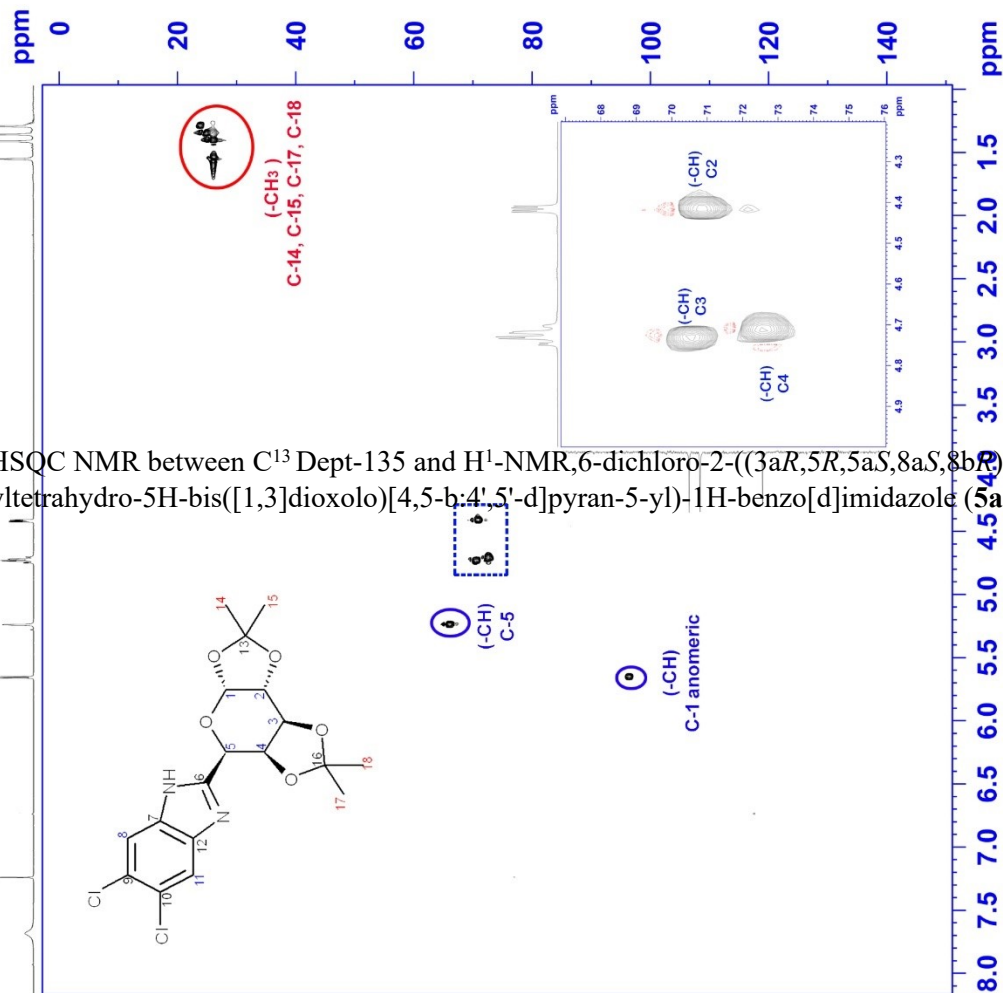


Figure-S13: 2D-HSQC NMR between C¹³ Dept-135 and H¹-NMR, 6-dichloro-2-((3aR,5R,5aS,8aS,8bR)-2,2,7,7-tetramethyltetrahydro-5H-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-5-yl)-1H-benzo[d]imidazole (5a)

- FAB +ve low- and high- resolution Mass spectrometry of compound (**5a**) :

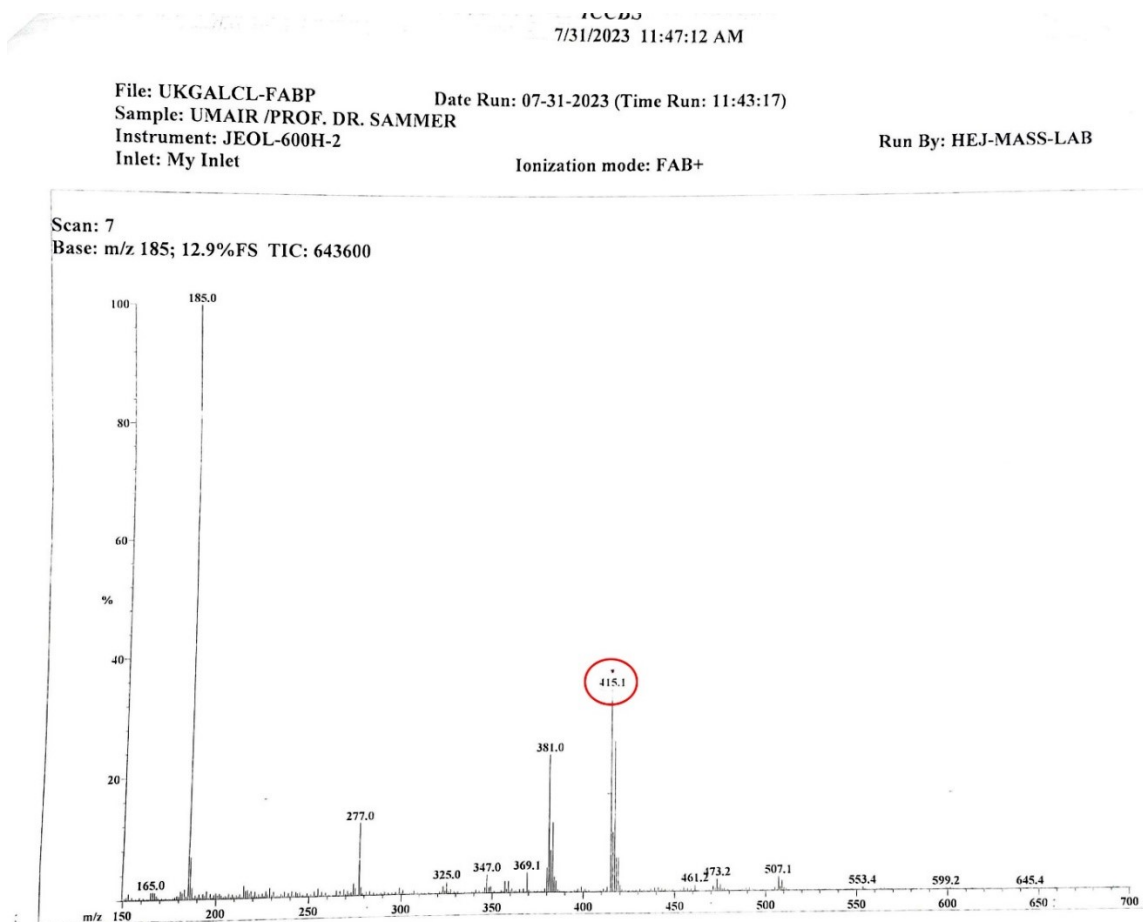


Figure-S14: Low resolution positive mode of FAB spectra of **5a**. Encircled is the m/z value 415.1.

High resolution FAB ($m+1$) positive mode at JEOL HX 110 Mass spectrometer of compound **5a** is **415.0808**, while theoretical mass was 415.0828 which corresponds to the composition of $C_{18}H_{21}O_5N_2Cl_2$.

4. Spectra
tetram
benzo[c

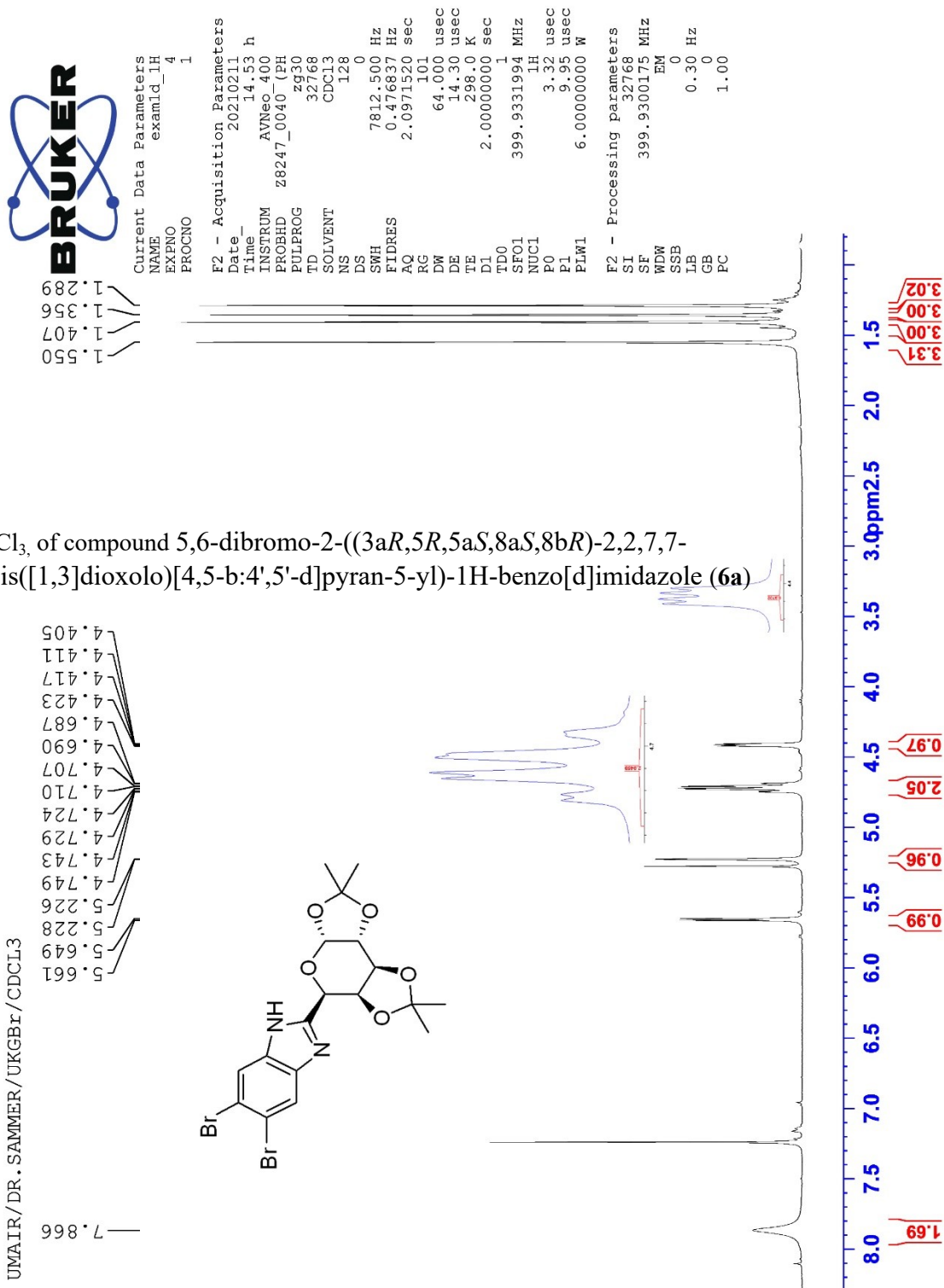


Figure-S15: $^1\text{H-NMR}$, in CDCl_3 , of compound 5,6-dibromo-2-((3aR,5R,5aS,8aS,8bR)-2,2,7,7-tetramethyltetrahydro-5H-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-5-yl)-1H-benzo[d]imidazole (6a)



Current Data Parameters
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EXPNO 1
PROCNO 1

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PROBHD Z8281_0183_1FX
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 16384
DS 4
SWH 30120.482 Hz
FIDRES 1.838408 Hz
AQ 0.5439488 sec
RG 101
DM 16.600 usec
DE 12.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 16
SF01 125.7973986 MHz
NUC1 13C
P1 12.00 usec
PLW1 229.03999329 W
SF02 500.2320009 MHz
NUC2 1H
CPDPRG2 waltz65
PCPD2 80.00 usec
PLW2 18.4589963 W
PLW12 0.18459000 W
PLW13 0.09284900 W

F2 - Processing parameters
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WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

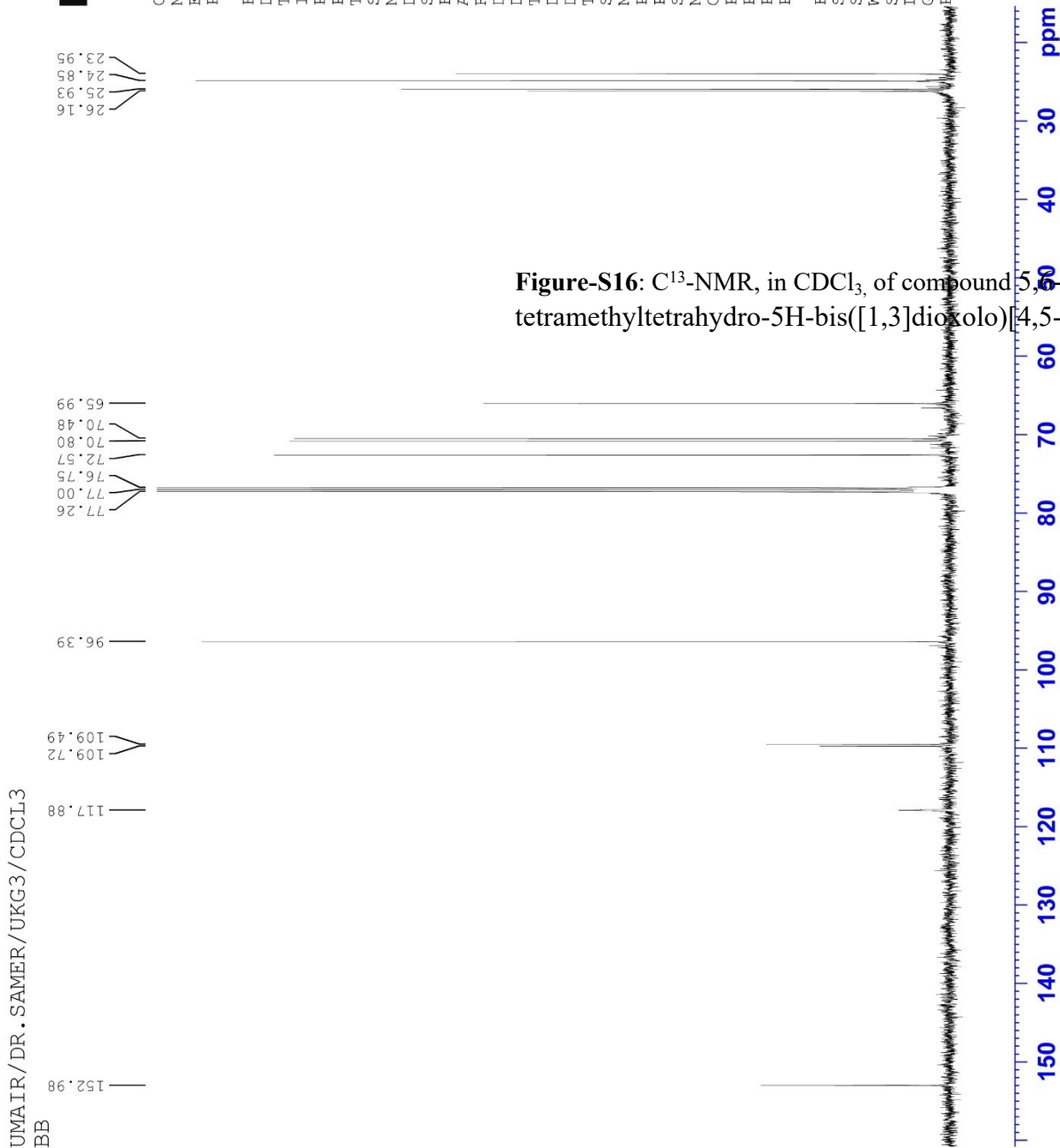


Figure-S16: ¹³C-NMR, in CDCl₃, of compound 5,5-dibromo-2-((3aR,4S)-tetramethyltetrahydro-5H-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-5-yl)pyridine

UMAIR/DR. SAMER/UKG3/CDCL3
HSQC



Current Data Parameters
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 EXPNO 3
 PROCNO 1

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 P1 12.00 usec
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 SOLVENT CDCL3
 NS 32
 DS 5892.358 Hz
 WDW EM
 FIDRES 11.668971 Hz
 AQ 0.0870400 sec
 RG 101
 DW 85.000 usec
 DE 6.50 usec
 TE 300.2 K
 CHST2 145.0000000
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 D1 1.50000000 sec
 D4 0.00172414 sec
 D11 0.00000000 sec
 D13 0.00000000 sec
 D16 0.00000000 sec
 D21 0.00020000 sec
 D21 0.00345000 sec
 INO 0.00001990 sec
 Tdau
 SFOPTNS
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 NU1 1H
 P1 8.00 usec
 P2 16.00 usec
 F28 1000.00 usec
 SFO2 500.1324999 MHz
 SFO2 125.7548829 MHz
 NUC2 13C
 CPDPRG2 garp
 P3 12.00 usec
 P4 24.00 usec
 P5 24.00 usec
 PLW2 229.03999329 MHz
 PLW2 6.73089981 M
 GRNAM1 SMSQ10.100
 GR21 SMSQ10.100 %
 GR22 SMSQ10.100 %
 PL6 1000.00 usec

F1 - Acquisition parameters
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 SFO1 500.1324999 MHz
 FIDRES 196.2693976 Hz
 SN 199.735 PPM
 FMODE Echo-Antiecho

F2 - Processing parameters
 SF 500.2300220 MHz
 WDW OSGINE
 SSB 2
 LB 0 Hz
 GB 0
 PC 1.40

F1 - Processing parameters
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 echo-antiecho
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 GR22 SMSQ10.100 MHz
 LB 0 Hz
 GB 0

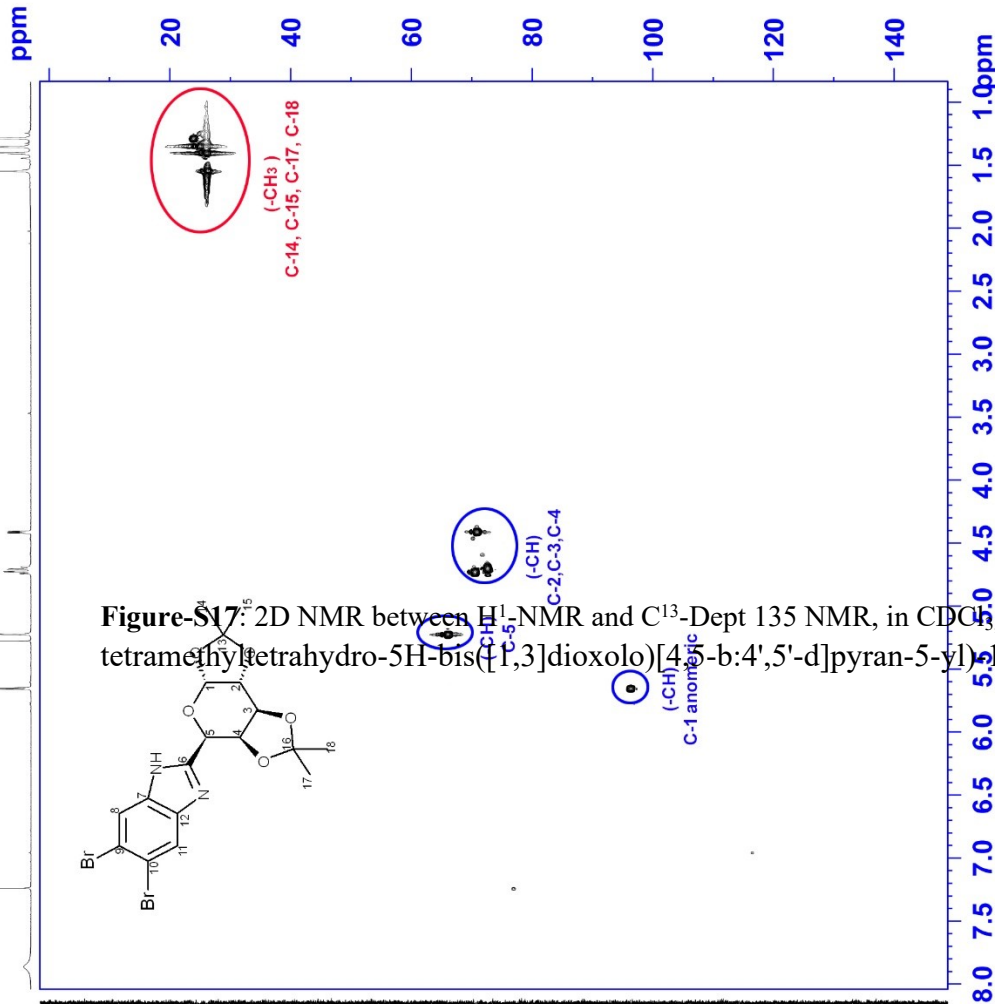
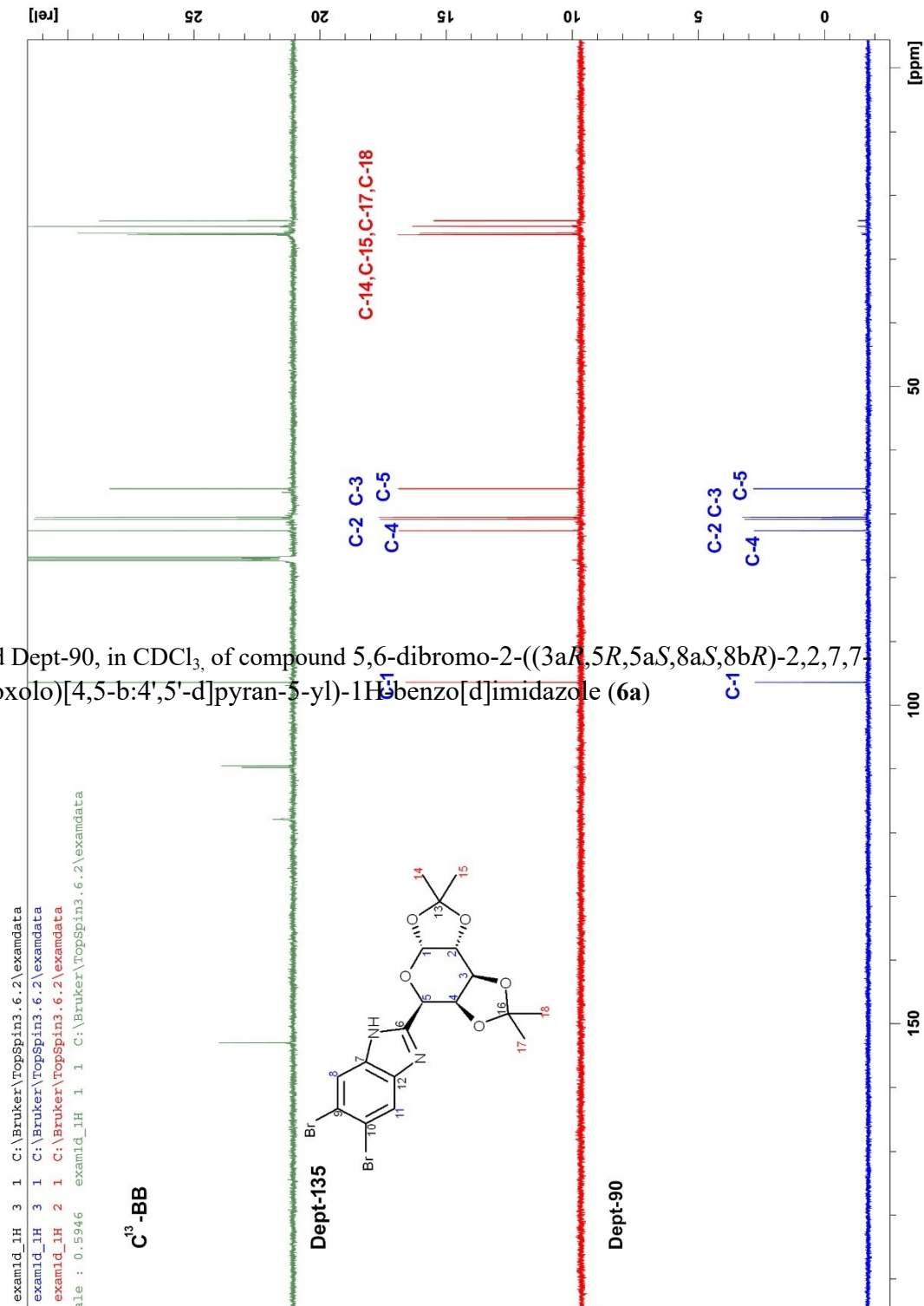


Figure-S17: 2D NMR between ^1H -NMR and ^{13}C -Dept 135 NMR, in CDCl_3 , of compound tetramethyl tetrahydro-5H-bis(1,3-dioxolo)[4,5-b:4',5'-d]pyran-5-yl]-H-ben

Figure-S18: C^{13} NMR BB, Dept-135 and Dept-90, in $CDCl_3$, of compound 5,6-dibromo-2-((3*aR*,5*R*,5*aS*,8*aS*,8*bR*)-2,2,7,7-tetramethyltetrahydro-5H-bis([1,3]dioxolo)[4,5-*b*:4',5'-*d*]pyran-5-yl)-1*H*-benzo[*d*]imidazole (**6a**)



- FAB +ve low- and high- resolution Mass spectrometry of compound (6a)

7/31/2023 12:18:13 PM

File: UKG3-FABP

Date Run: 07-31-2023 (Time Run: 12:16:21)

Sample: UMAIR /PROF. DR. SAMMER

Instrument: JEOL-600H-2

Inlet: My Inlet

Ionization mode: FAB+

Run By: HEJ-MASS-LAB

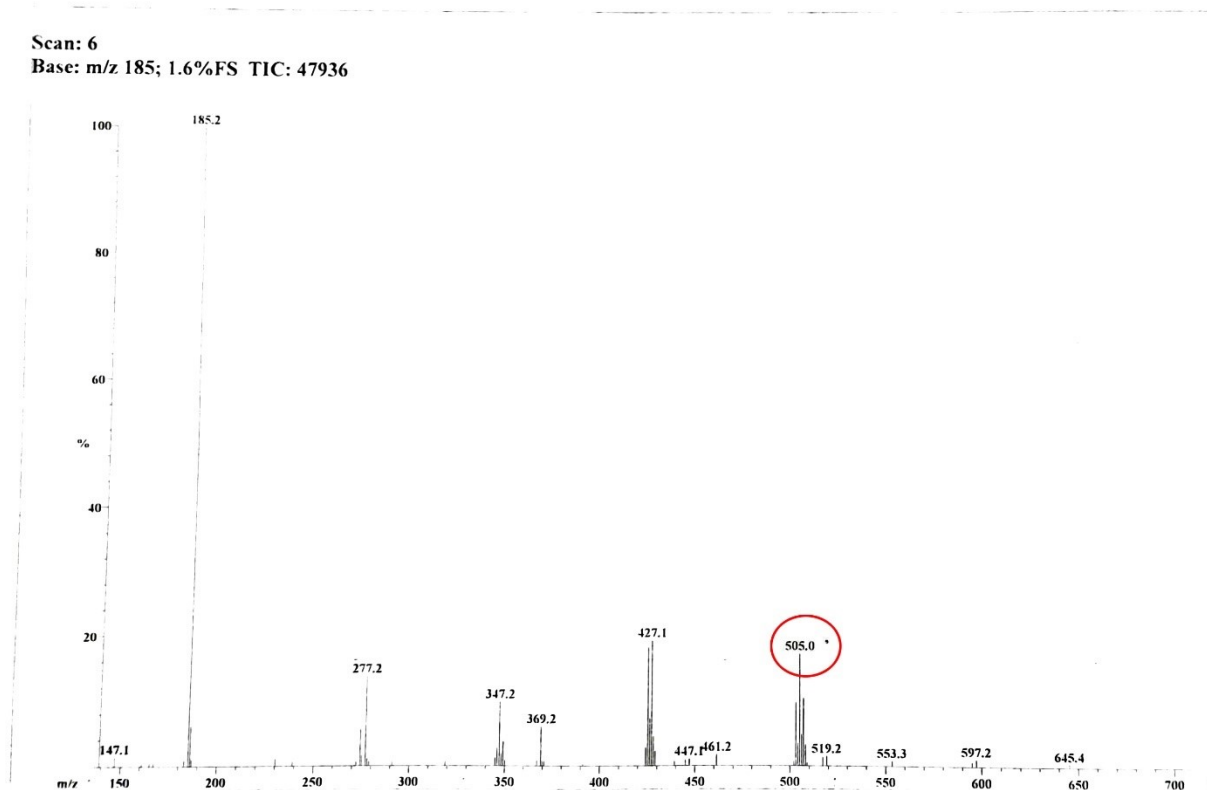


Figure-S19: Low resolution positive mode of FAB spectra of **6a**. Encircled is the m/z value **505.0**.

High resolution FAB positive mode at JEOL HX 110 Mass spectrometer of compound **6a** is **504.0808**, while theoretical mass was 504.1828 which corresponds to the composition of $C_{18}H_{21}O_5N_2Br_2$.

AVANCE NEO 400 MHz
Lab # 115

5. 5-bromo-2-((3aR,5R,5aS,8aS,8bR)-2,2,7,7-tetramethyltetrahydro-5H-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-5-yl)-1H-benzo[d]imidazole (7a):

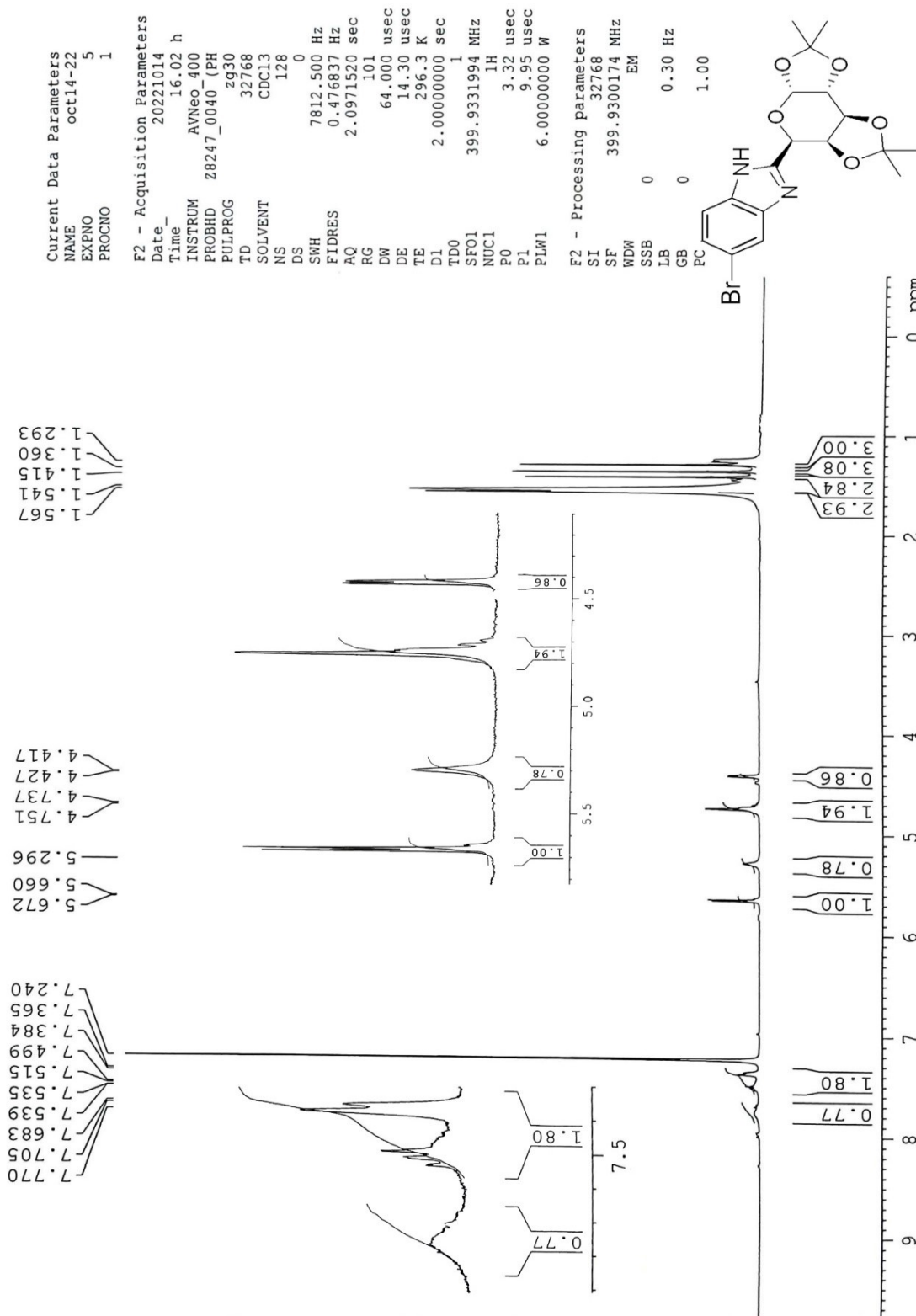


Figure-S20: ¹H NMR, in CDCl₃, of compound 5-bromo-2-((3aR,5R,5aS,8aS,8bR)-2,2,7,7-tetramethyltetrahydro-5H-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-5-yl)-1H-benzo[d]imidazole (7a):

- Mass spectrometry of compound (**7a**)

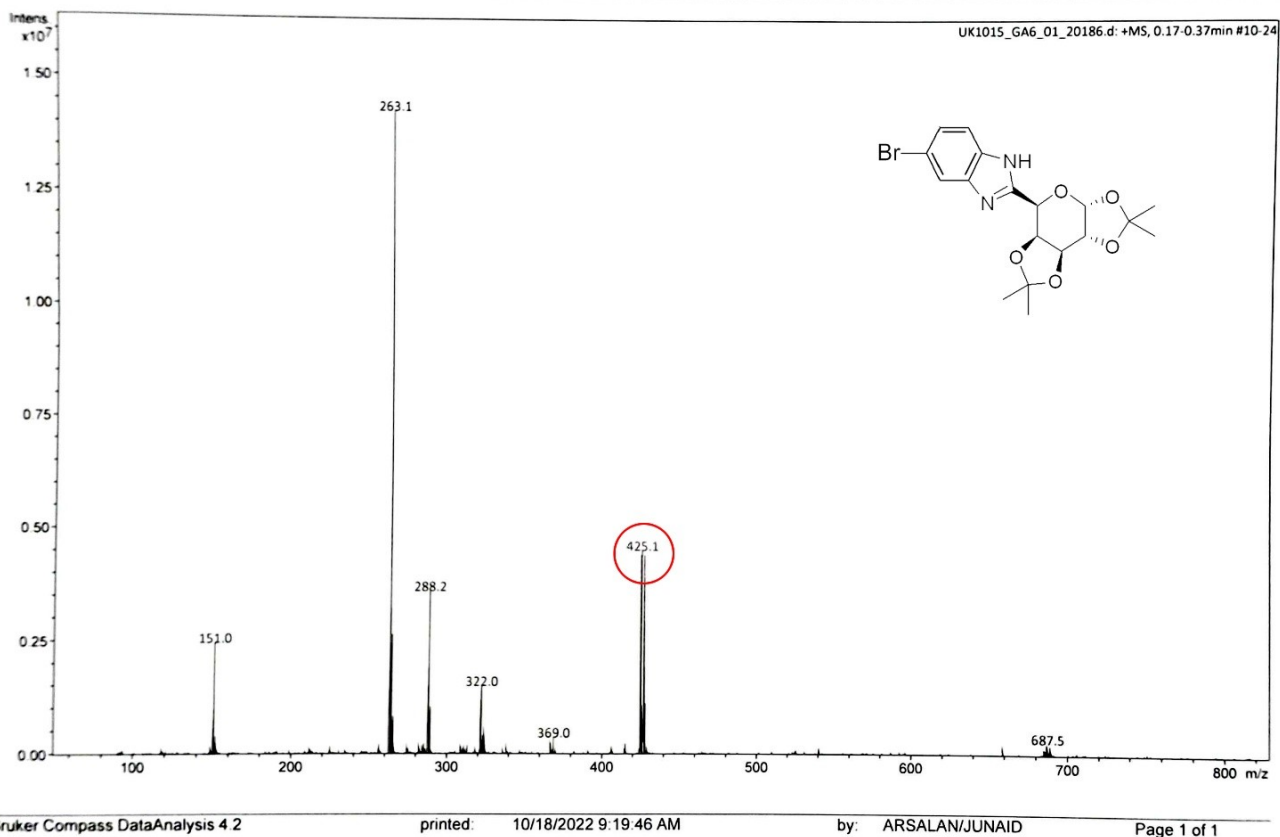


Figure-S21: LR-ESI MS positive of 5-bromo-2-((3aR,5R,5aS,8aS,8bR)-2,2,7,7-tetramethyltetrahydro-5H-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-5-yl)-1H-benzo[d]imidazole (**7a**):

High resolution FAB positive mode (m+1) + ve mode at JEOL HX 110 Mass spectrometer of compound **7a** is **425.0808**, while theoretical mass was 425.1808 which corresponds to the composition of **C₁₈H₂₂BrN₂O₅**.

6. 2-((3a'*R*,5'*R*,5a'*S*,8a'*S*,8b'*R*)-tetrahydro-5'*H*-dispiro[cyclohexane-1,2'-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-7',1''-cyclohexan]-5'-yl)-1*H*-benzo[d]imidazole (3b)

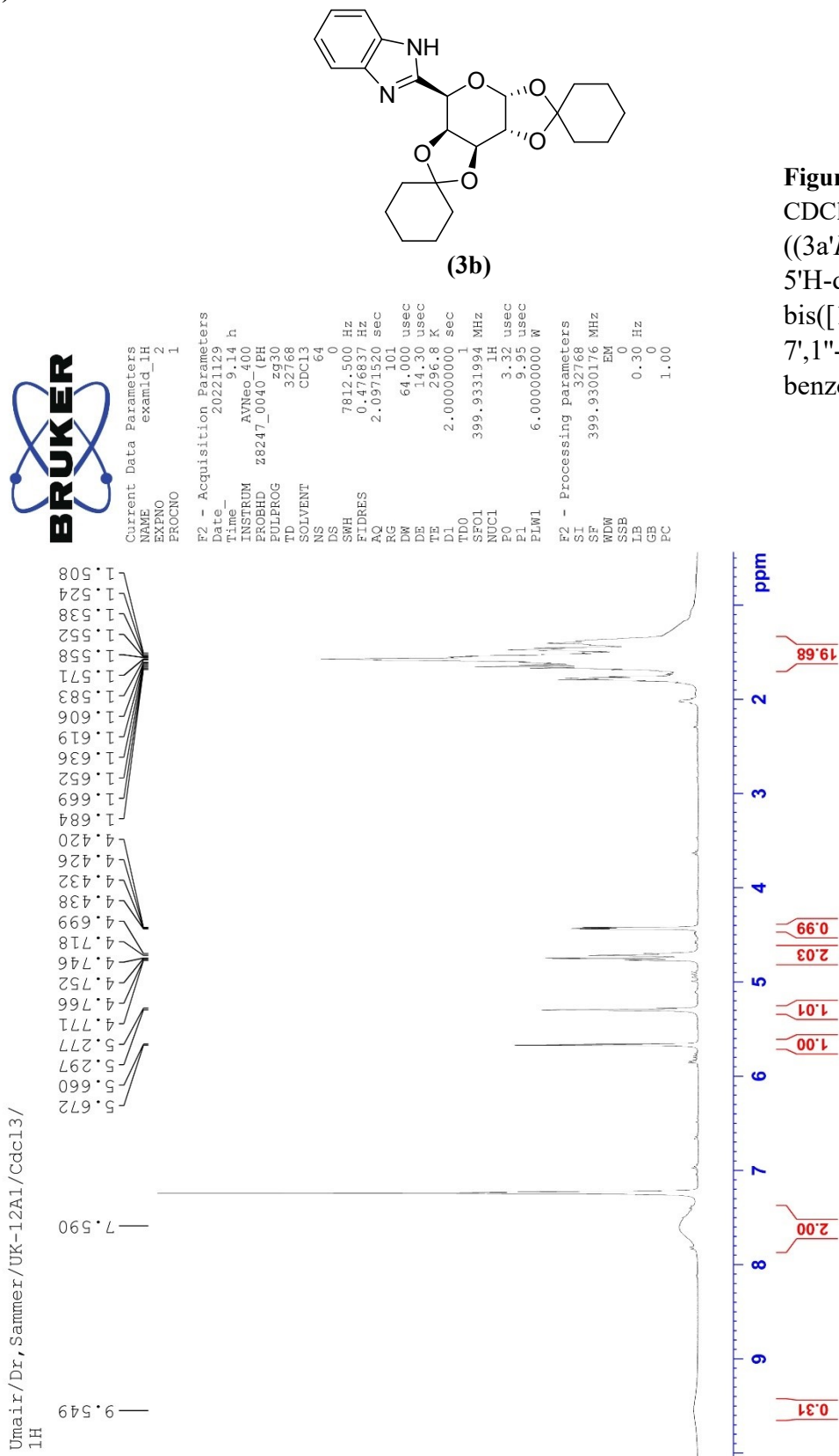


Figure-S22: ^1H -NMR at 500MHz CDCl_3 , of compound 2-((3a'*R*,5'*R*,5a'*S*,8a'*S*,8b'*R*)-tetrahydro-5'*H*-dispiro[cyclohexane-1,2'-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-7',1''-cyclohexan]-5'-yl)-1*H*-benzo[d]imidazole

- FAB +ve low- and high- resolution Mass spectrometry of compound (**3b**)

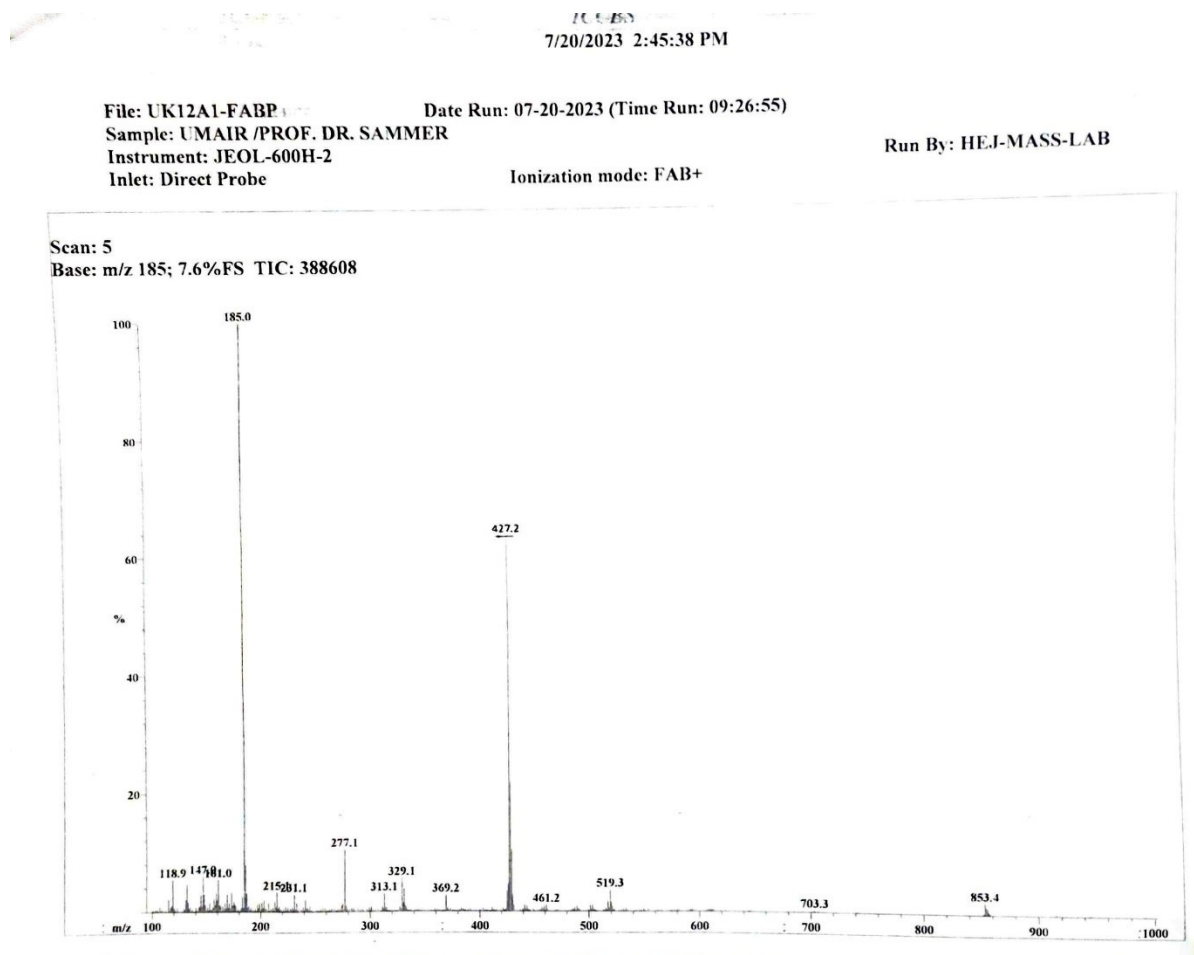
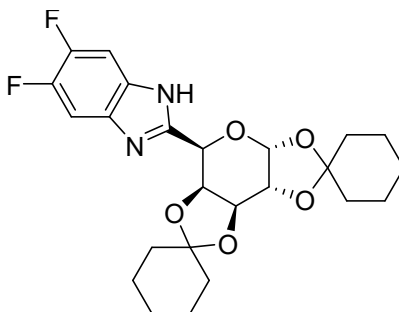


Figure S23: Low resolution FAB (+ve) of compound (**3b**) having m/z= 427.2

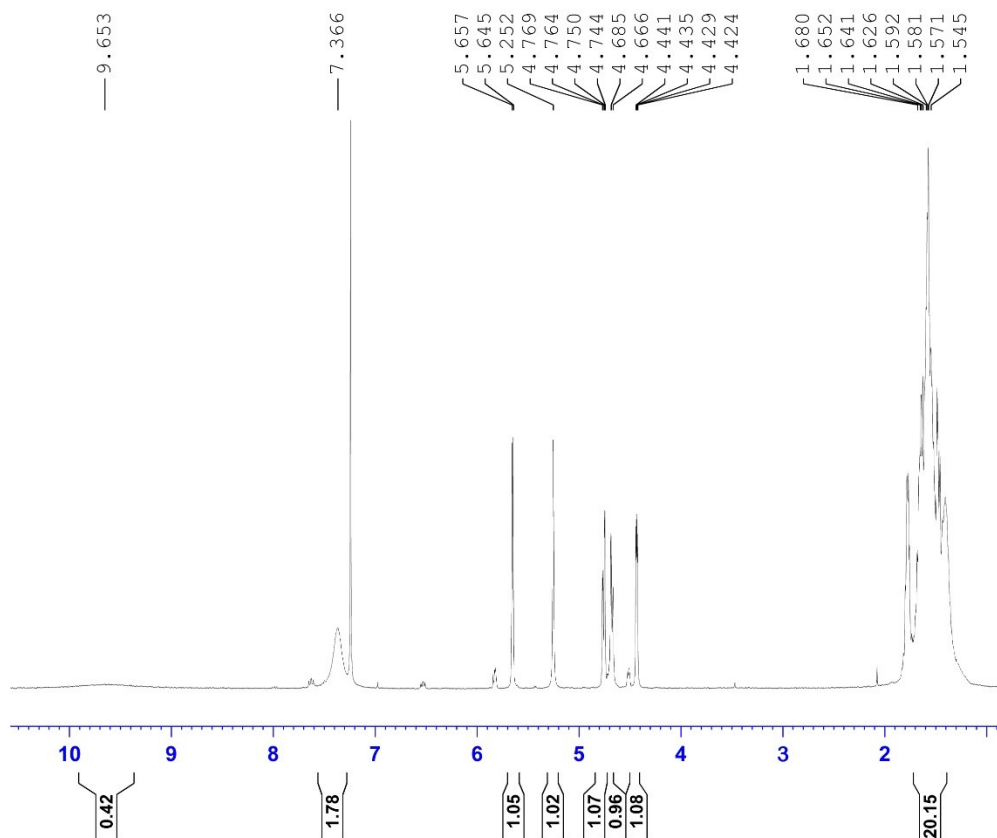
High resolution FAB (m+1) positive mode at JEOL HX 110 Mass spectrometer of compound **3b** is 427.2233, while theoretical mass was 427.2233 which corresponds to the composition of $C_{24}H_{31}N_2O_5$.

7. 5,6-difluoro-2-((3a'R,5'R,5a'S,8a'S,8b'R)-tetrahydro-5'H-dispiro[cyclohexane-1,2'-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-7',1''-cyclohexan]-5'-yl)-1H-benzo[d]imidazole (4b)



(4b)

Umair / Dr. Sammer / UK-I2A4R / CDCL3
1H



Current Data Parameters
NAME examid_1H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20230729
Time 14.48 h
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PROBHD Z114854_0013 (
PULPROG zg30
TD 32768
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NS 16
DS 0
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FIDRES 0.500288 Hz
AQ 1.9988480 sec
RG 101
DW 61.000 usec
DE 12.86 usec
TE 299.3 K
D1 2.0000000 sec
TDO 1
SFO1 400.1332010 MHz
NUC1 1H
PO 4.67 usec
PI 14.00 usec
PLW1 13.21300030 W

F2 - Processing parameters
SI 16384
SF 400.1300175 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

Figure-S24: $^1\text{H-NMR}$, in CDCl_3 , of compound 5,6-difluoro-2-((3a'R,5'R,5a'S,8a'S,8b'R)-tetrahydro-5'H-dispiro[cyclohexane-1,2'-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-7',1''-cyclohexan]-5'-yl)-1H-benzo[d]imidazole (4b)

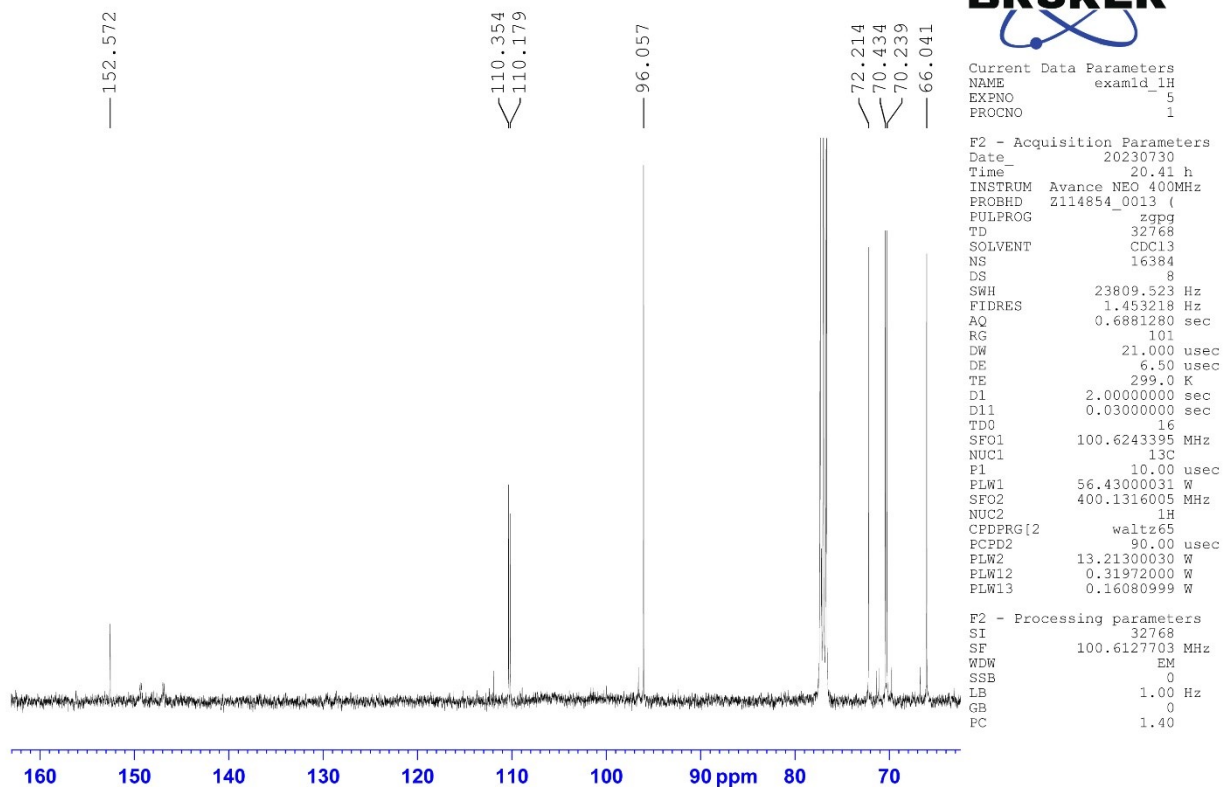


Figure-S25: C^{13} -NMR, in $CDCl_3$, of compound 5,6-difluoro-2-((3a'R,5'R,5a'S,8a'S,8b'R)-tetrahydro-5'H-dispiro[cyclohexane-1,2'-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-7',1''-cyclohexan]-5'-yl)-1H-benzo[d]imidazole (**4b**)

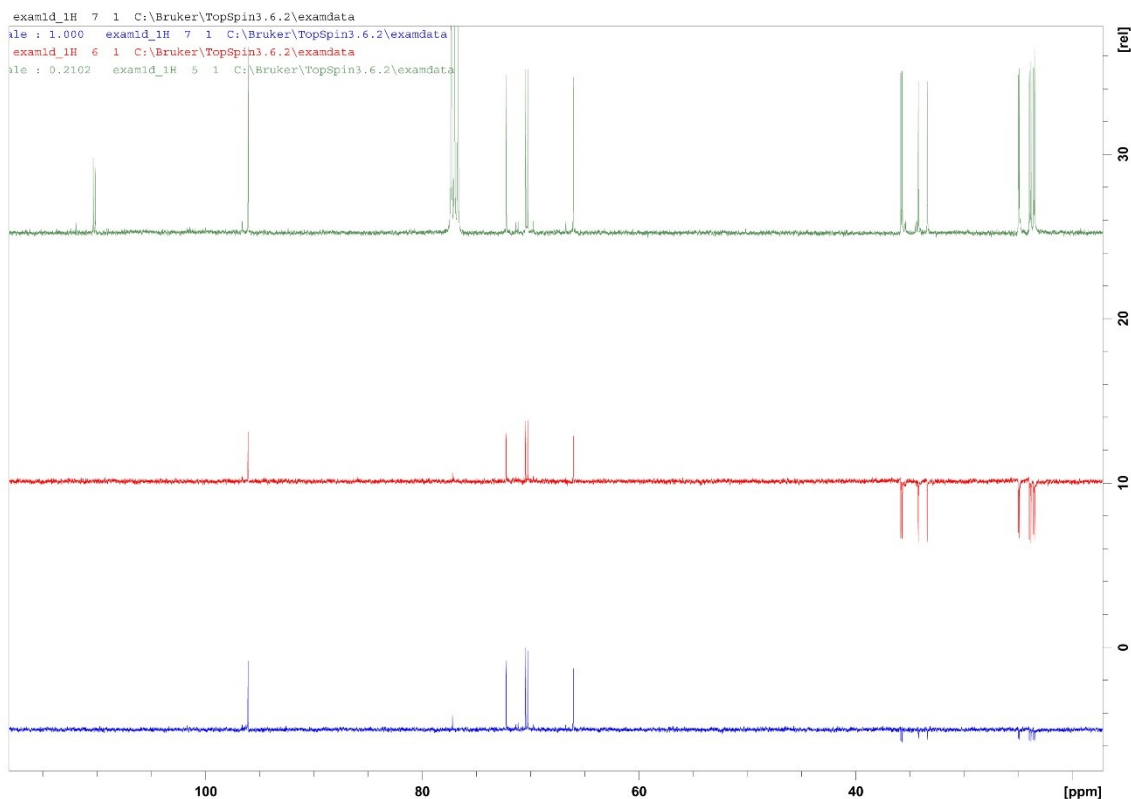


Figure-S26: C^{13} -NMR (BB, Dept-135, Dept-90), in $CDCl_3$, of compound 5,6-difluoro-2-((3a'R,5'R,5a'S,8a'S,8b'R)-tetrahydro-5'H-dispiro[cyclohexane-1,2'-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-7',1''-cyclohexan]-5'-yl)-1H-benzo[d]imidazole (**4b**)

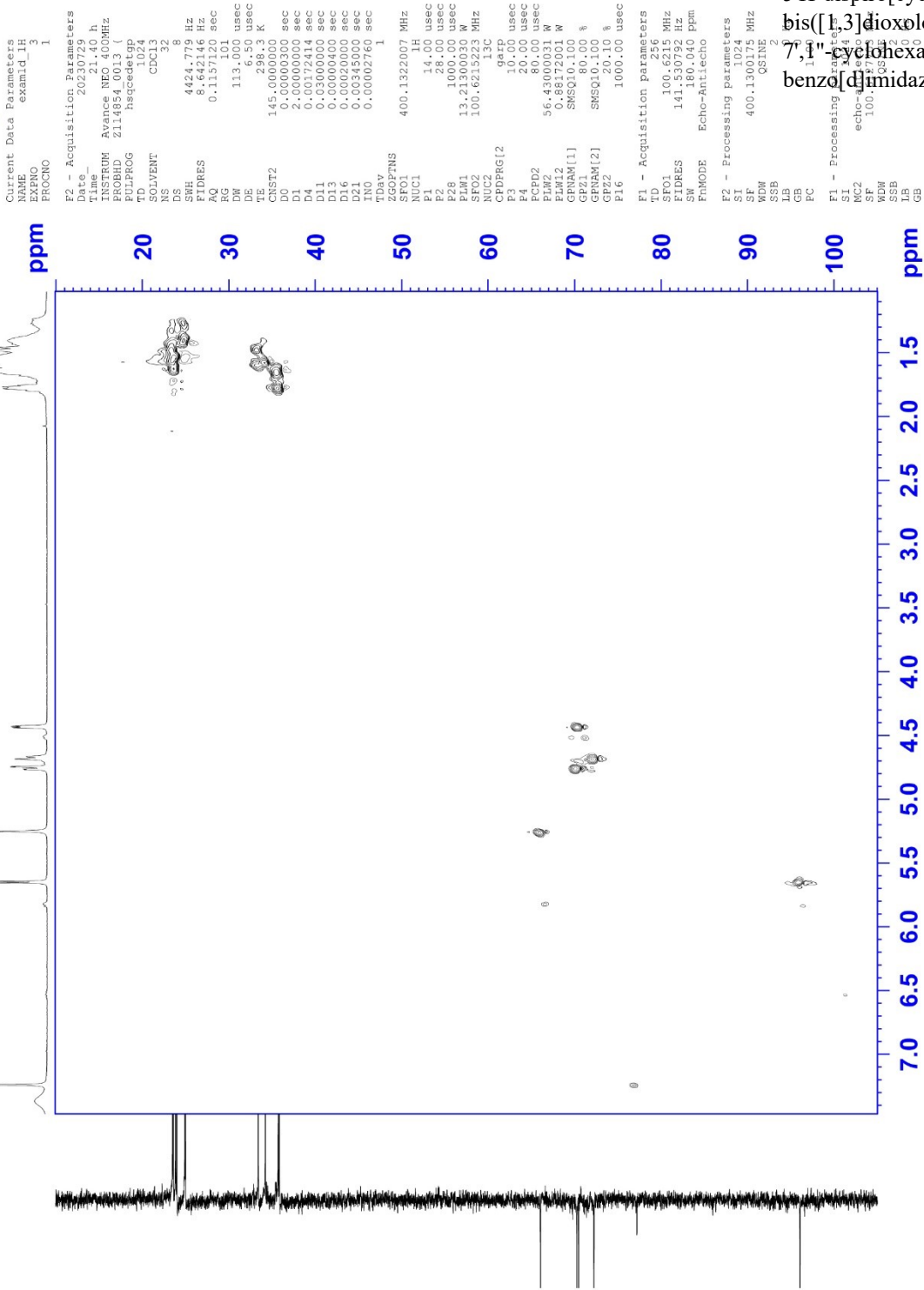


Figure-S27: 2D HSQC [¹H-NMR and ¹³C-NMR (Dept-135)], in CDCl₃, of compound 5,6-difluoro-2-((3a'R,5'R,5a'S,8a'S,8b'R)-tetrahydro-5'H-dispiro[cyclohexane-1,2'-bis([1,3-dioxolo][4,5-b:4',5'-d]pyran-7,4''-cyclohexan)-5'-yl)-1H-benzo[*d*]imidazole (**4b**)

- FAB +ve mood low and high resolution of compound 4b

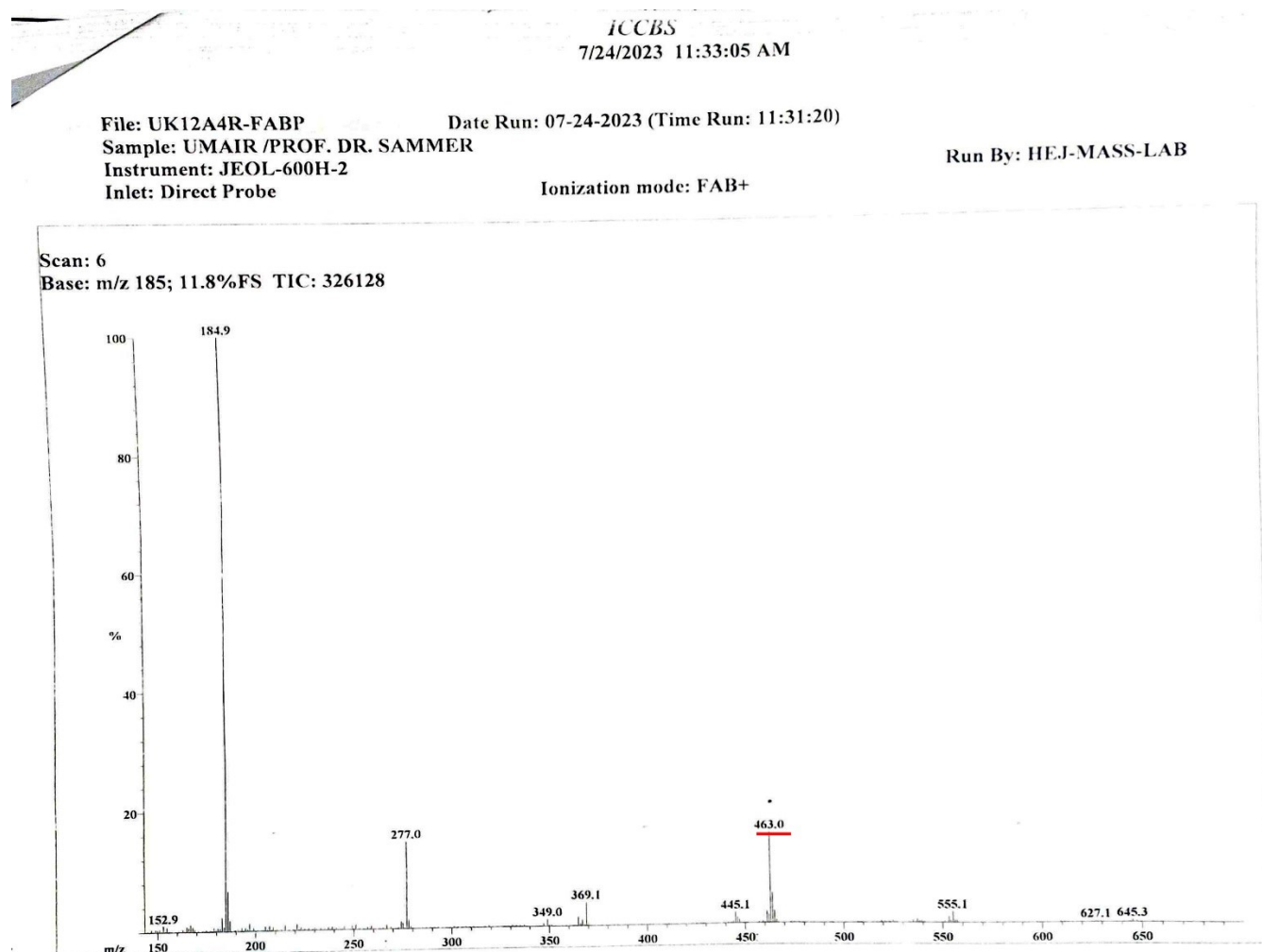


Figure S28: LR-FAB (+ve) shows the m/z value of 463.0 of compound (**4b**)

High resolution FAB positive mode at JEOL HX 110 Mass spectrometer of compound **4b** is **463.2059**, while theoretical mass was 463.2045 which corresponds to the composition of **C₂₄H₂₉N₂O₅F₂**.

8. 5,6-dichloro-2-((3a'*R*,5'*R*,5a'*S*,8a'*S*,8b'*R*)-tetrahydro-5'*H*-dispiro[cyclohexane-1,2'-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-7',1''-cyclohexan]-5'-yl)-1*H*-benzo[d]imidazole (5b):

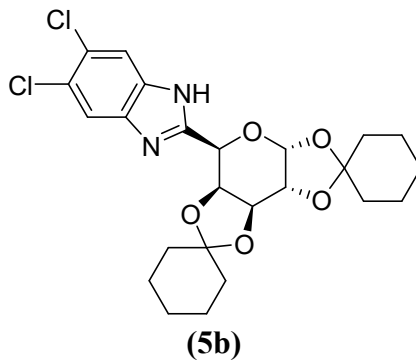
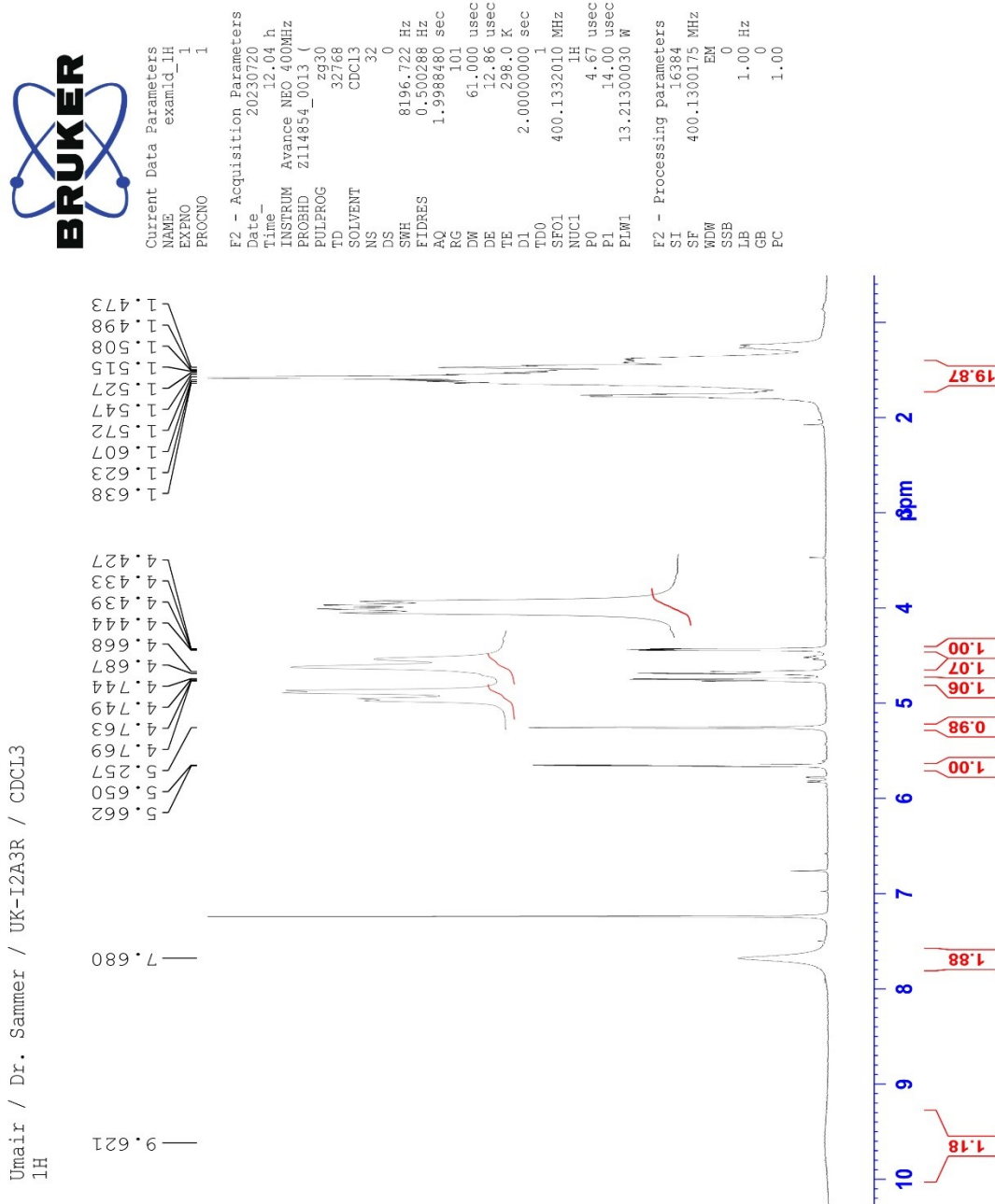


Figure-S29: ¹H-NMR at 500 MHz in CDCl₃, of compound 5,6-dichloro-2-((3a'*R*,5'*R*,5a'*S*,8a'*S*,8b'*R*)-tetrahydro-5'*H*-dispiro[cyclohexane-1,2'-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-7',1''-cyclohexan]-5'-yl)-1*H*-benzo[d]imidazole (5b)



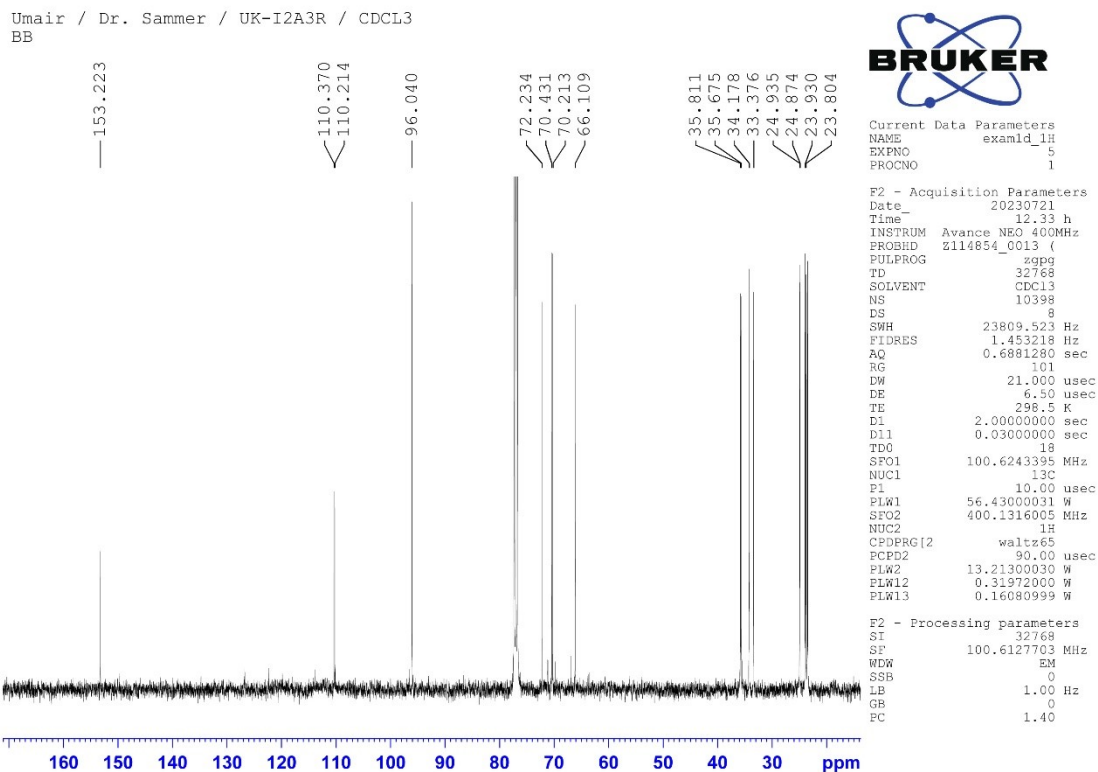


Figure-S30: ¹³C-NMR Broad band, in CDCl₃, of compound 5,6-dichloro-2-((3a'*R*,5'*R*,5a'*S*,8a'*S*,8b'*R*)-tetrahydro-5'*H*-dispiro[cyclohexane-1,2'-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-7',1''-cyclohexan]-5'-yl)-1*H*-benzo[d]imidazole (**5b**)

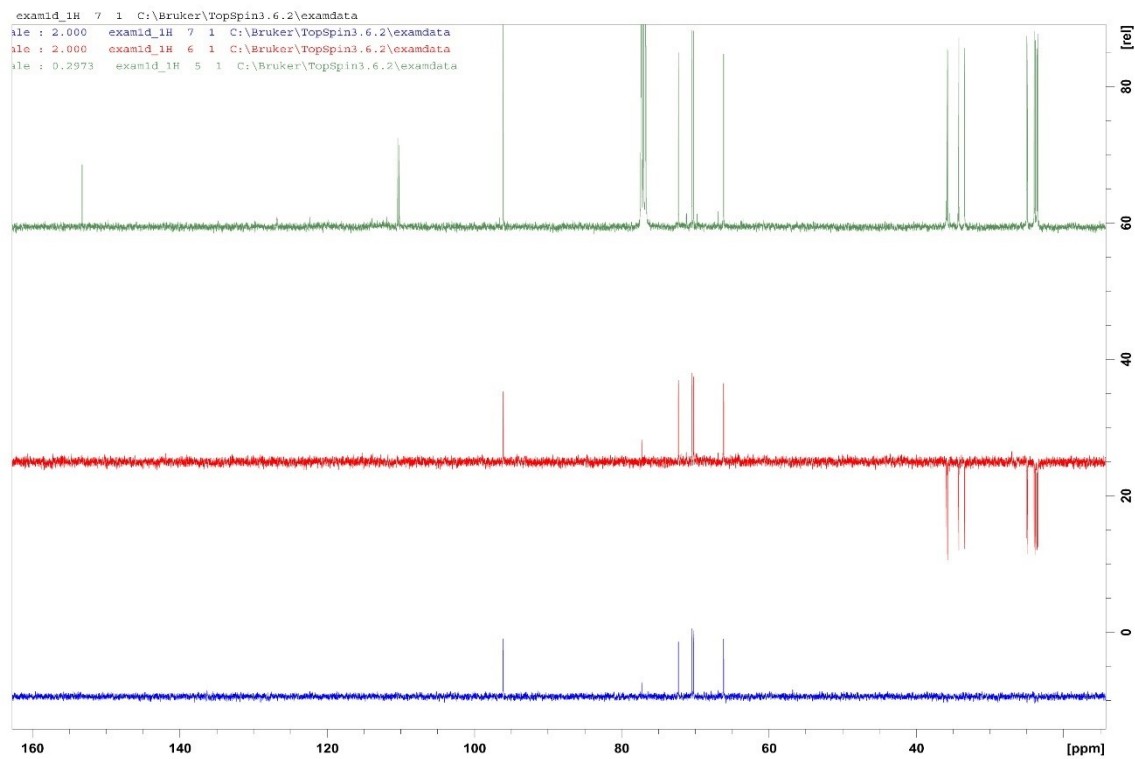


Figure-S31: ¹³C-NMR BB, DEPT-135, DEPT-90, in CDCl₃, of compound 5,6-dichloro-2-((3a'*R*,5'*R*,5a'*S*,8a'*S*,8b'*R*)-tetrahydro-5'*H*-dispiro[cyclohexane-1,2'-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-7',1''-cyclohexan]-5'-yl)-1*H*-benzo[d]imidazole (**5b**)



Current Data Parameters
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 PROCNO 1

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 Time 17:59 h
 INSTRUM Advance NEO 400MHz
 PROBHD Z11484_0013
 P1PRG1 zgpg30
 TD 1024
 SOLVENT CDCL3
 NS 32
 DS 4

SH 3968.254 Hz
 FIDRES 7.750496 Hz
 AQ 0.1280240 sec
 RG 101
 DW 126.00 usec
 DE 6.50 usec
 TE 298.15 K
 CNST2 145.0000000
 D0 0.0000000 sec
 D1 0.0000000 sec
 DA 0.0172314 sec
 D11 0.030000000 sec
 D13 0.000004000 sec
 D16 0.000200000 sec
 D18 0.000000000 sec
 INO 0.0002760 sec

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 ZGPTNS 400.1319606 MHz
 SFO1 400.1319606 MHz
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 P2 28.00 usec
 P28 1000.00 usec
 SFO2 100.6215230 MHz
 SFO3 100.6215230 MHz
 NUC2 13C
 CPDPRG2 garp
 P3 10.00 usec
 P4 10.00 usec
 P5 10.00 usec
 P6 10.00 usec
 P7 10.00 usec
 P8 10.00 usec
 P9 10.00 usec
 P10 10.00 usec
 P11 10.00 usec
 P12 10.00 usec
 P13 10.00 usec
 P14 10.00 usec
 P15 10.00 usec
 P16 1000.00 usec

F1 - Acquisition Parameters
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 SFO1 100.6215 MHz
 FIDRES 141.530792 Hz
 SWH 180.040 Ppm
 F2 - Processing parameters
 SI 1024
 SF 400.130015 MHz
 DE 6.50 usec
 QSIN2
 LB 0 Hz
 GB 0
 FC 1.00

F1 - Processing parameters
 SI 1024
 MC2 echo-antiecho
 PE 100.615230 MHz
 P1 14.00 usec
 P2 28.00 usec
 P3 10.00 usec
 P4 10.00 usec
 P5 10.00 usec
 P6 10.00 usec
 P7 10.00 usec
 P8 10.00 usec
 P9 10.00 usec
 P10 10.00 usec
 P11 10.00 usec
 P12 10.00 usec
 P13 10.00 usec
 P14 10.00 usec
 P15 10.00 usec
 P16 1000.00 usec

F2 - Processing parameters
 SI 1024
 SF 400.130015 MHz
 DE 6.50 usec
 QSIN2
 LB 0 Hz
 GB 0
 FC 1.00

F1 - Processing parameters
 SI 1024
 MC2 echo-antiecho
 PE 100.615230 MHz
 P1 14.00 usec
 P2 28.00 usec
 P3 10.00 usec
 P4 10.00 usec
 P5 10.00 usec
 P6 10.00 usec
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 P9 10.00 usec
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 P13 10.00 usec
 P14 10.00 usec
 P15 10.00 usec
 P16 1000.00 usec

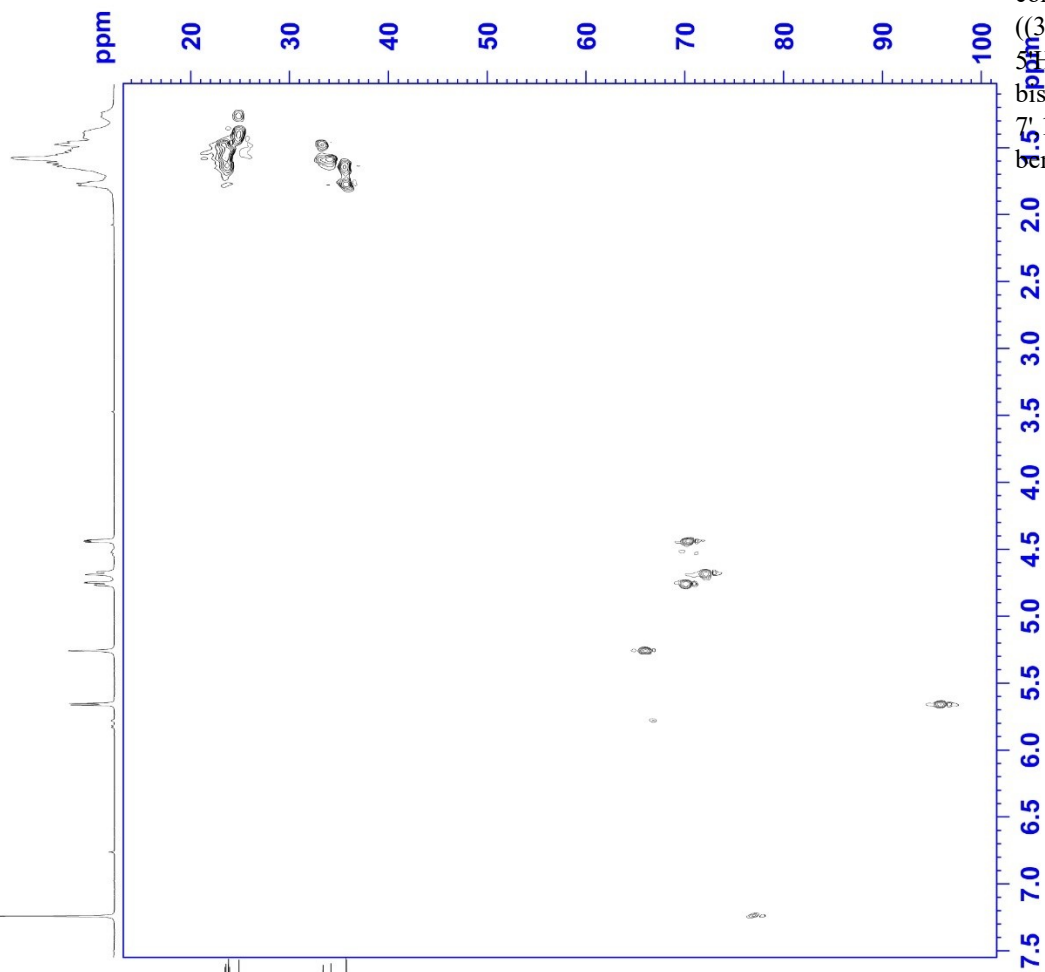


Figure-S32: 2D HSQC NMR spectrum of compound **5b** (3a'R,5'R,5a'S,8a'S,8b'R)-tetrakis[1,3]dioxolo[4,5-b:4',5'-b']-1,2,3,4-tetrahydro-1H-benzo[d]imidazole (**5b**)

- Single crystal XRD analysis of compound **5a**

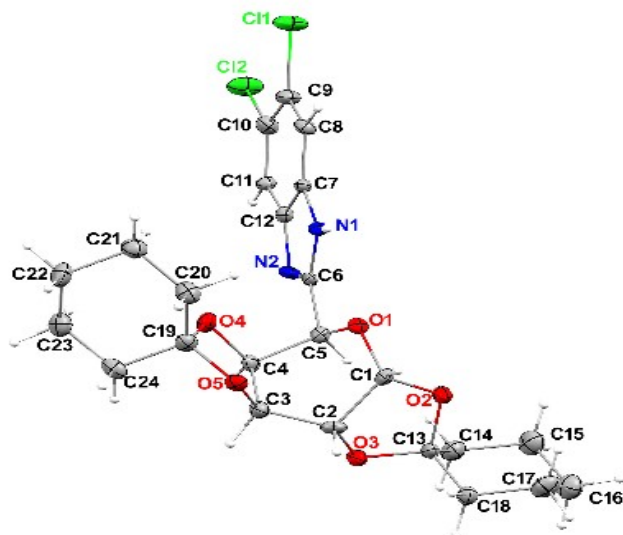


Figure-S33: ORTEP view of compound **5b**.

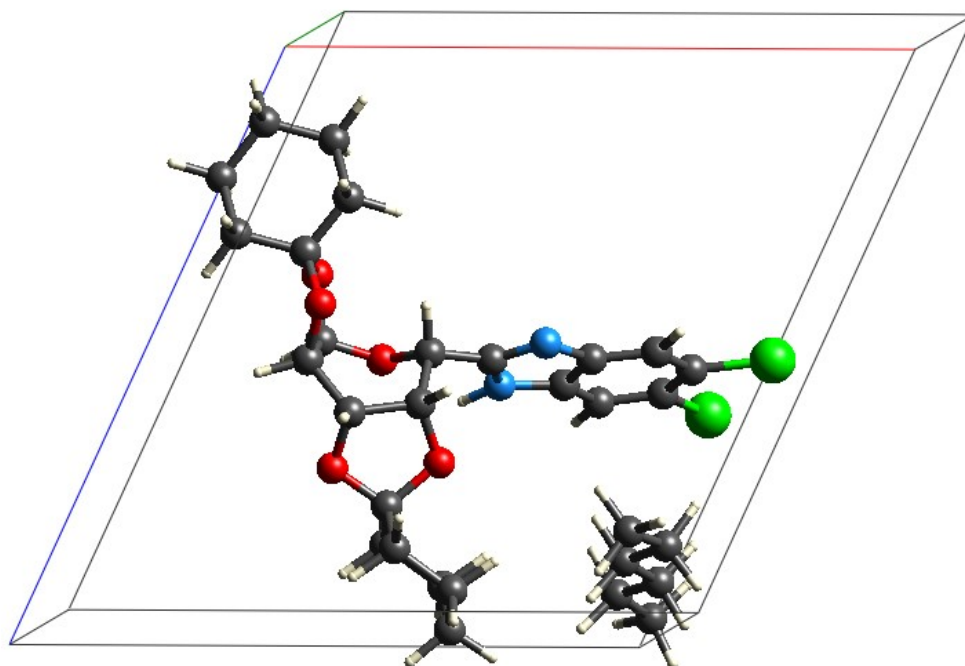


Figure-S34: Compound **5b** crystallized with *n*-hexane in unit cell, crystallized with one hexane molecule in unit cell.

Identification code	UK12A3	
Empirical formula	C ₂₄ H ₂₉ Cl ₂ N ₂ O ₅	
Formula weight	1163.11	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P 21	
Unit cell dimensions	a = 13.3850(7) Å	α = 90°.
	b = 17.9218(10) Å	β = 114.647(3)°.
	c = 13.9875(8) Å	γ = 90°.
Volume	3049.7(3) Å ³	
Z	2	
Density (calculated)	1.267 Mg/m ³	
Absorption coefficient	2.239 mm ⁻¹	
F(000)	1240	
Crystal size	0.32 x 0.22 x 0.15 mm ³	
Theta range for data collection	3.476 to 68.243°.	
Index ranges	-16 ≤ h ≤ 16, -21 ≤ k ≤ 21, -16 ≤ l ≤ 16	
Reflections collected	36114	
Independent reflections	11081 [R(int) = 0.2431]	
Completeness to theta = 67.679°	99.9 %	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	11081 / 1 / 714	
Goodness-of-fit on F ²	0.974	
Final R indices [I > 2σ(I)]	R1 = 0.0677, wR2 = 0.1269	
R indices (all data)	R1 = 0.1386, wR2 = 0.1545	
Absolute structure parameter	0.071(17)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.414 and -0.399 e.Å ⁻³	
CCDC	2296170	

Table S4: Crystal data and structure refinement for (5b)

File: UK12A3R-FAB Date Run: 07-24-2023 (Time Run: 11:17:46)
Sample: UMAIR /PROF. DR. SAMMER
Instrument: JEOL-600H-2
Inlet: Direct Probe

Run By: HEJ-MASS-LAB

Ionization mode: FAB+

Scan: 3

Base: m/z 185; 18.8%FS TIC: 361440

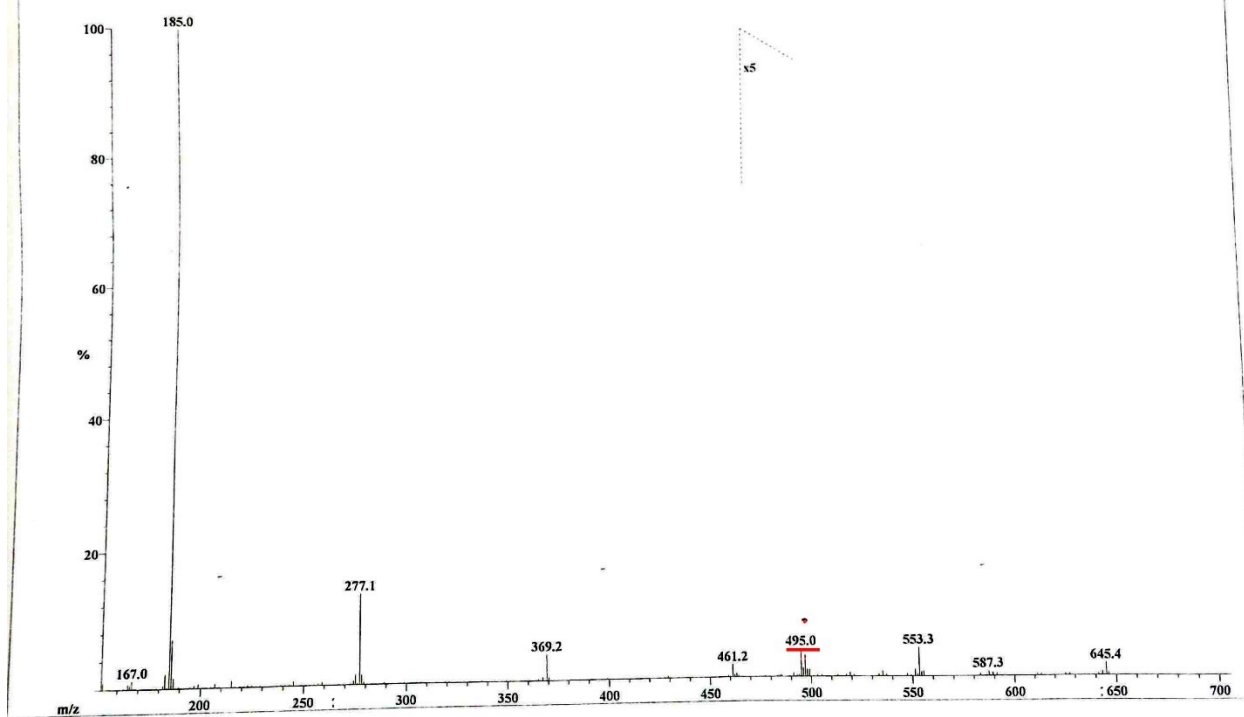
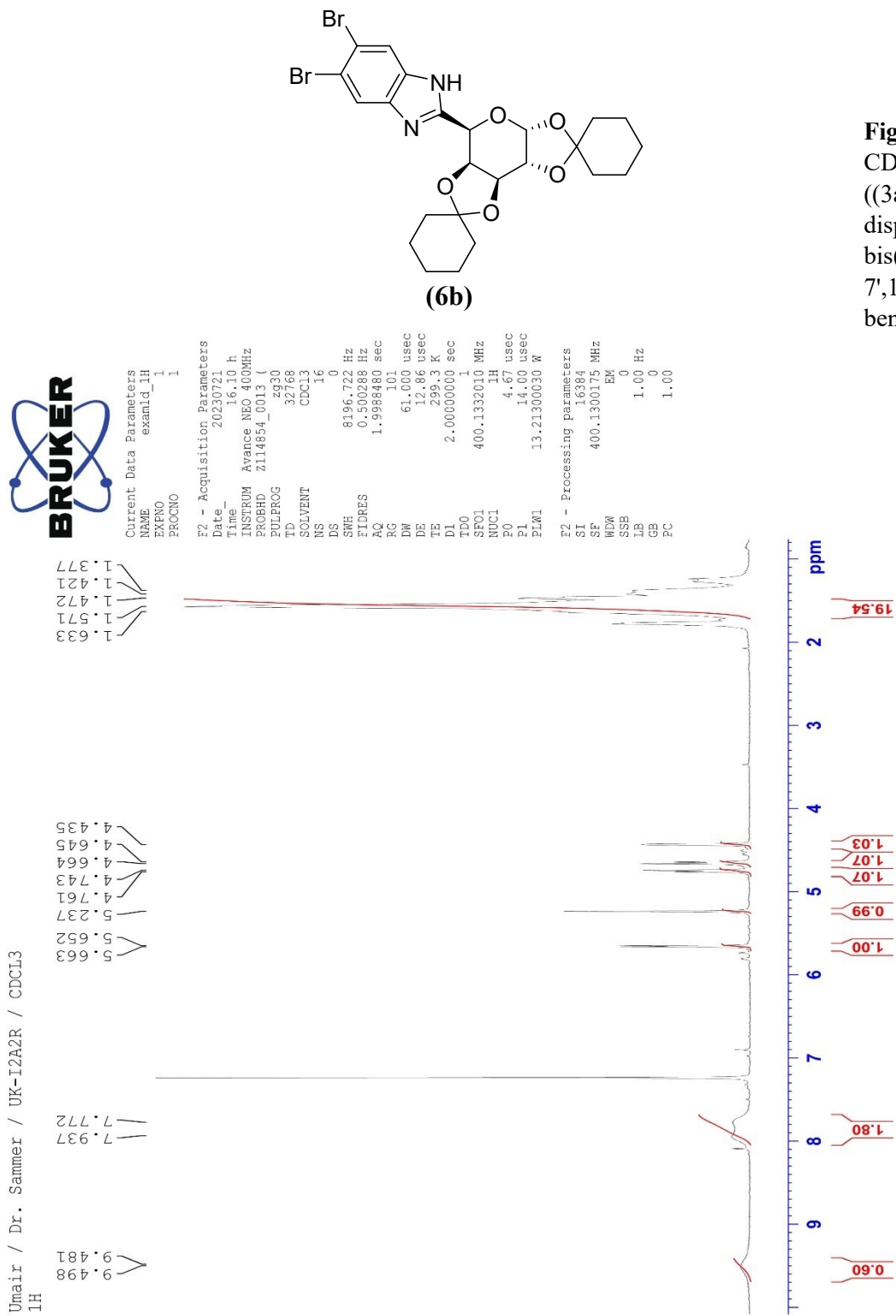


Figure S35: LR-FAB (+ve) shows the m/z value of 495.0 of compound (**5b**)

High resolution FAB positive mode at JEOL HX 110 Mass spectrometer of compound **5b** is 495.1475, while theoretical mass was 495.1454 which corresponds to the composition of $C_{24}H_{29}N_2O_5Cl_2$.

9. Spectral properties of 5,6-dibromo-2-(((3a'R,5'R,5a'S,8a'S,8b'R)-tetrahydro-5'H-dispiro[cyclohexane-1,2'-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-7',1''-cyclohexan]-5'-yl)-1H-benzo[d]imidazole (6b)





Current Data Parameters
 NAME examid_1
 EXNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date 20230721
 Time 18.14 h
 INSTRUM Avance NEO 400MHz
 PROBHD Z114854.0013 (cosyypgf)
 PULPROG 2048
 TD 2048
 SOLVENT CDCL3
 NS 16
 DS 4
 SWH 4424.779 Hz
 FIDRES 4.321073 Hz
 AQ 0.2314240 sec
 RG 113.000 usec
 DE 6.50 usec
 TE 299.0 K
 DO 0.0000300 sec
 D1 1.5000000 sec
 D13 0.0000400 sec
 D16 0.0002000 sec
 INO 0.00022600 sec
 TDel 1
 SFO1 400.1322007 MHz
 NUC1 1H
 P0 14.00 usec
 F1 14.00 usec
 F1M1 13.21300030 W
 GPMAM[1] SMSQ10.100
 GFZ1 10.00 %
 F16 1000.00 usec

F1 - Acquisition Parameters
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 SFO1 400.1322 MHz
 FIDRES 34.568584 Hz
 SWH 11.058 ppm
 F2 - Acquisition Parameters
 TD 1024
 SFO1 400.1300175 MHz
 FIDRES 0 Hz
 SWH 1.00 Hz
 F1 - Acquisition Parameters
 TD 1024
 SFO1 400.1300175 MHz
 FIDRES 0 Hz
 SWH 0 Hz

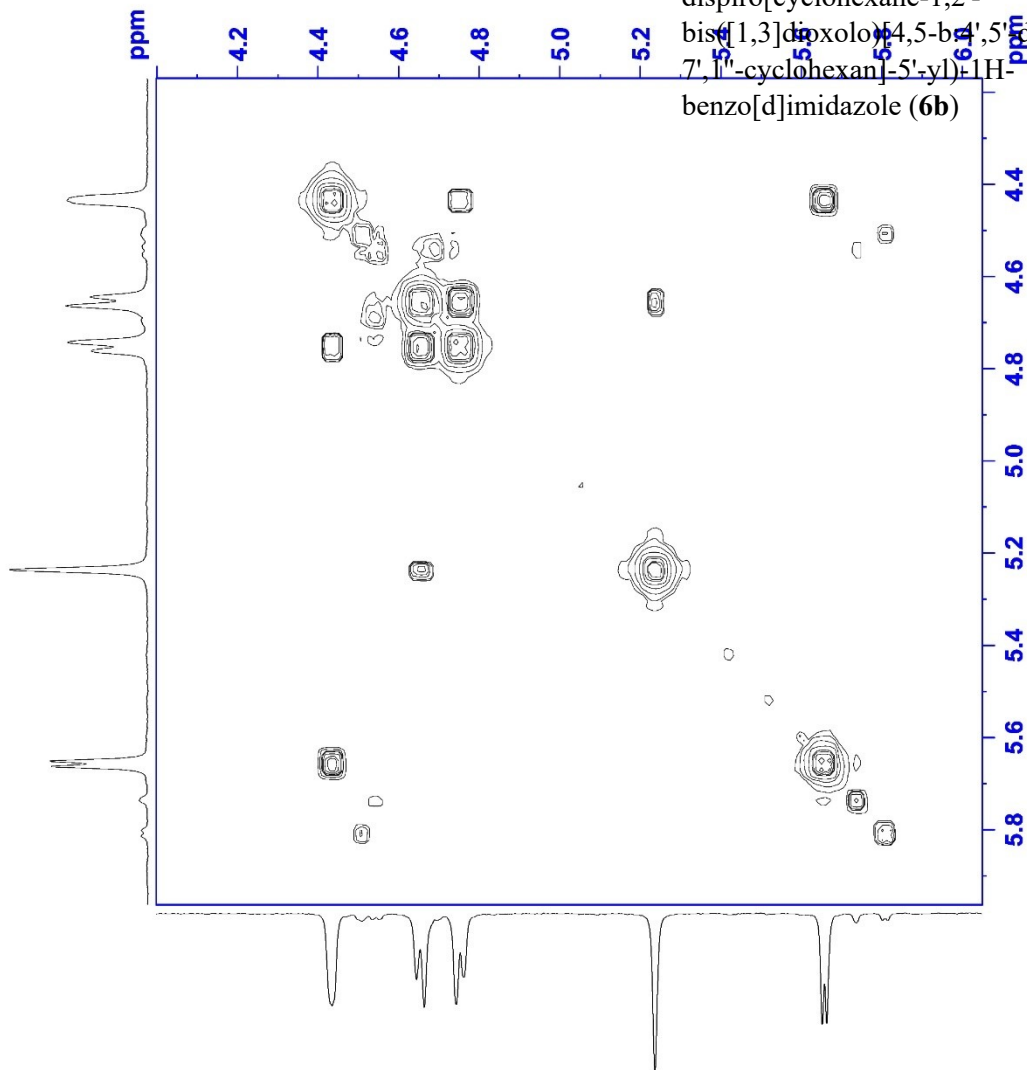


Figure S37: COSY-NMR at 500MHz, in CDCL₃ of compound 5, 6-dibromo-2-((2*a*,*b*,5*R*,5*a*,*S*,8*a*,*S*,8*b*,*R*)-tetrahydro-5*H*-dispiro[cyclohexane-1,2'-bis(1,3)dioxolo[4,5-*b*']pyran-7,1'-cyclohexan]-5'-yl)-1*H*-benzo[*d*]imidazole (6b)

Umair / Dr. Sammer / UK-I2A2R / CDCl3
BB



23.56
23.81
23.93
24.88
24.94
23.38
33.38
34.18
35.68
35.82

66.15
70.21
70.44
72.26

96.04
110.34
110.19

153.17

Current Data Parameters
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EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
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Time_ 22.07 h
INSTRUM Avance NEO 400MHz
PROBHD Z114854_0013 (zpg)
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 16384
DS 8
SWH 23809.523 Hz
FIDRES 1.453218 Hz
AQ 0.6881280 sec
RG 101
DW 21.000 usec
DE 6.50 usec
TE 298.5 K
D1 2.0000000 sec
D11 0.0300000 sec
D10 16
SFO1 100.6243395 MHz
NUC1 13C
P1 10.00 usec
PLW1 56.4300031 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG2 waltz65
PCPD2 90.00 usec
PLW2 13.21300030 W
PLW12 0.31972000 W
PLW13 0.16080999 W

Processing parameters
SI 32768
SF 100.6127703 MHz
WDW EM
SS 0
LB 1.00 Hz
GB 0
PC 1.40

Figure-S38: C^{13} -NMR BB at 500 MHz, CDCl₃, of compound 5, 6-dibromo-((3*a*,*R*,5'*R*,5*a*'*S*,8*a*'*S*,8*b*'*R*)-tetradiSpiro[cyclohexane-1,2'-bis([1,3]dioxolo)[4,5-*b*:4',5'-cyclohexan]-5'-yl)]-1*H*-benzo

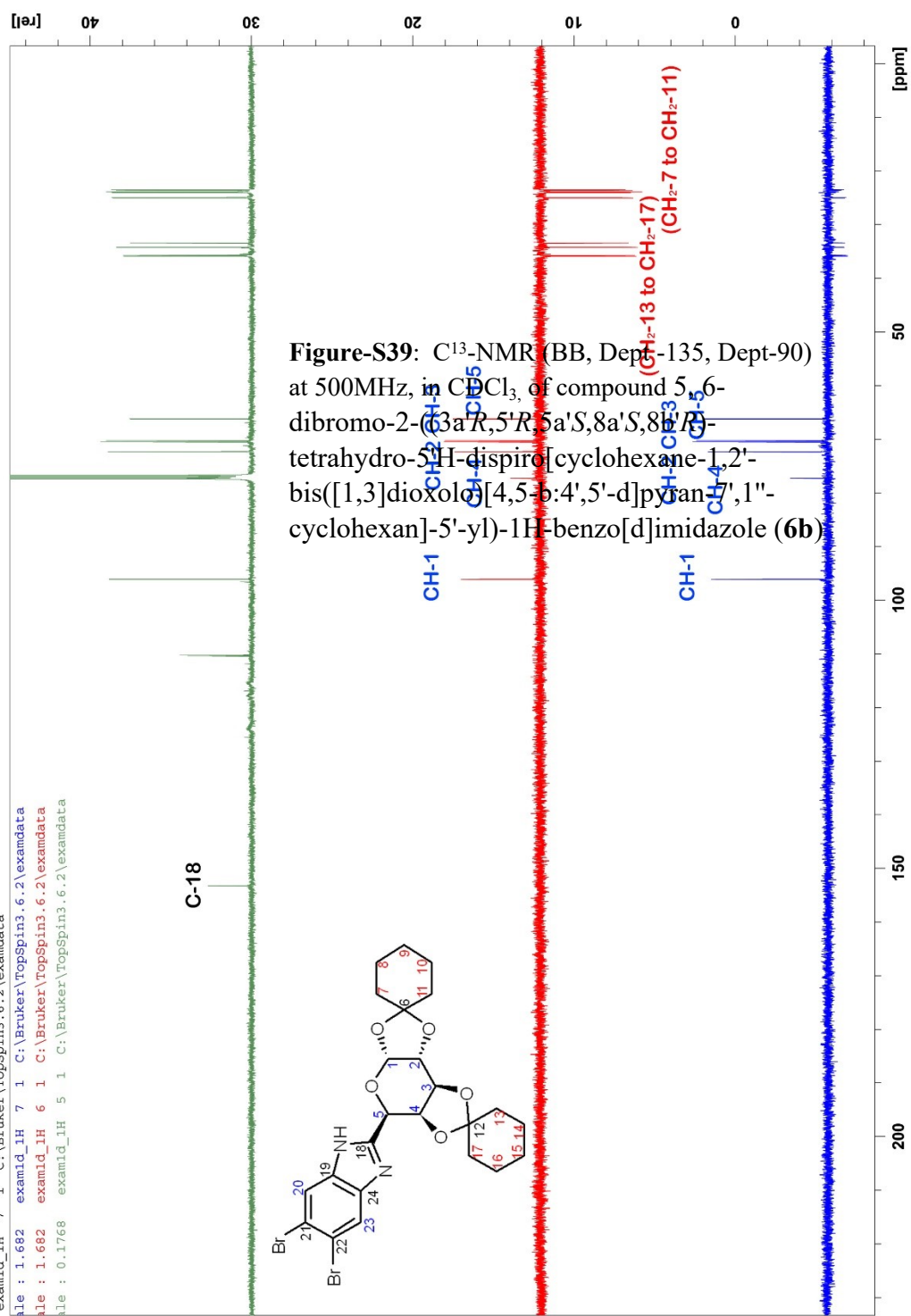
(6b)

150 140 130 120 110 100 90 80 70 60 50 ppm 40 30

```

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file : 1.682 examid_IH 6 1 C:\Bruker\TopSpin3.6.2\examdata
file : 0.1768 examid_IH 5 1 C:\Bruker\TopSpin3.6.2\examdata

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Current Data Parameters
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EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
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Time_ 23:06 h
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PULPROG zgpg30
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SOLVENT CDCl3
NS 32
DS 8
SWH 4424.779 Hz
FIDRES 8.642146 Hz
AQ 0.1157120 sec
RG 327.5
DM 113.000 usec
DE 6.50 usec
TE 298.0 K
CNST2 145.0000000
D0 0.0000300 sec
D1 0.0000300 sec
D2 0.0000300 sec
D3 0.00172414 sec
D4 0.00172414 sec
D11 0.03000000 sec
D13 0.03000000 sec
D16 0.0000400 sec
D21 0.0002000 sec
DZ1 0.00345000 sec
DZ2 0.00002760 sec
TD0 1
ZGPGTNS
SF01 400.1322007 MHz
NUC1 1H
F1 14.00 usec
PC 1.00000000
P28 1000.00 usec
PLW1 13.21390030 W
SF02 100.6215223 MHz
NUC2 13C
CPDPRG2 garp
P4 14.00 usec
PCPD2 20.00 usec
PCPD1 80.00 usec
PLM2 56.4300031 W
PLM1 0.88172001 W
GPNAM[1] SMSQ10.100
GPNAM[2] SMSQ10.100
GPZ2 20.10 usec
PL6 1000.00 usec

F1 - Acquisition parameters
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FIDRES 141.530792 Hz
SW 180.040 ppm
F1MODE Echo-Antiecho
F2 - Processing parameters
SI 1024
SF 400.1300175 MHz
WDW EM
SSB 2
LB 0 Hz
GB 0
PC 1.00
F1 - Processing parameters
SI 1024
SF echo-antiecho
SF 100.6127703 MHz
WDW EM
SSB 2
LB 0 Hz
GB 0

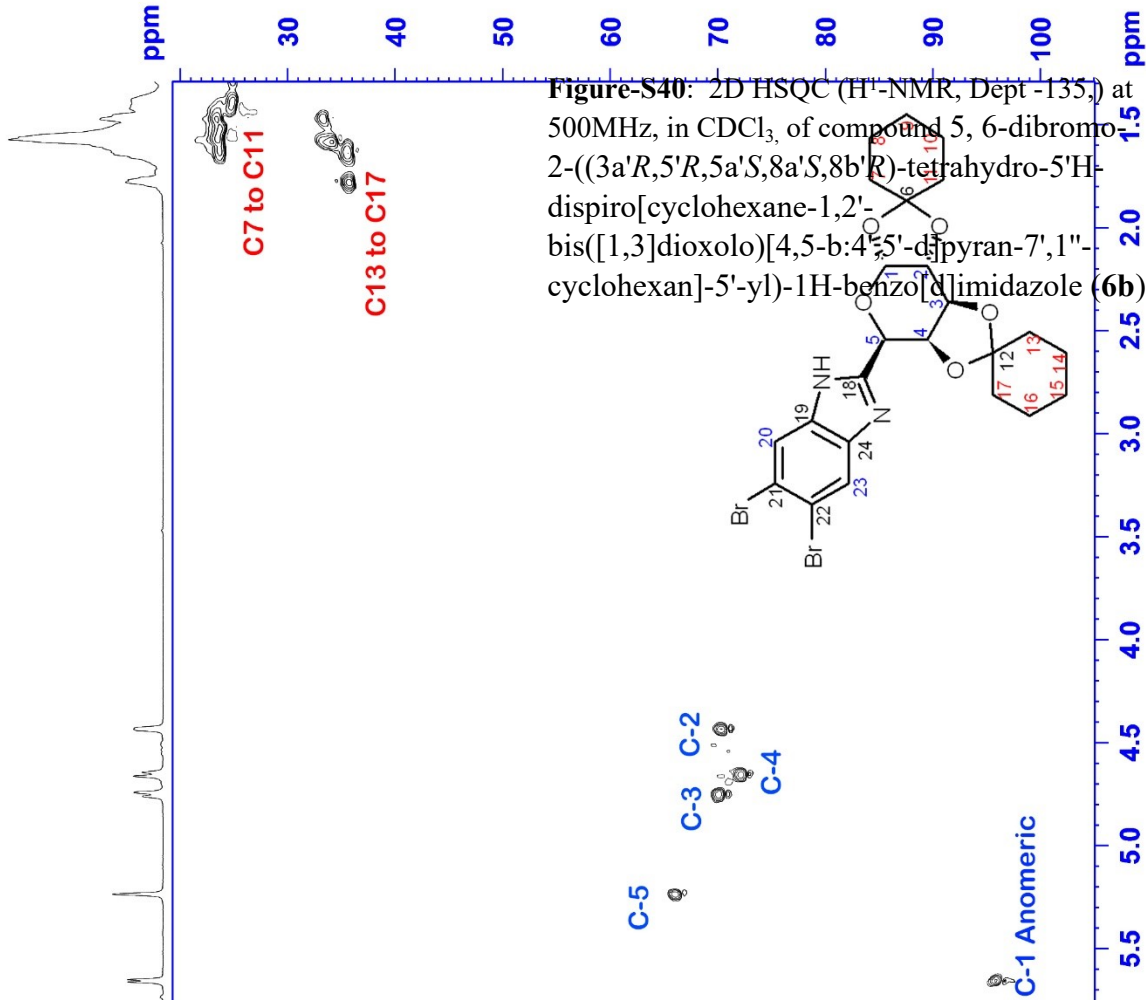


Figure-S40: 2D HSQC (^1H -NMR, Dept -135 $^\circ$) at 500MHz, in CDCl_3 , of compound 5, 6-dibromo 2-((3a'R,5'R,5a'S,8a'S,8b'R)-tetrahydro-5'H-dispiro[cyclohexane-1,2'-bis([1,3]dioxolo)[4,5-b:4'5'-d]pyran-7',1''-cyclohexan]-5'-yl)-1H-benzod[imidazole (6b)

- Single Crystal XRD analysis of compound 6b:

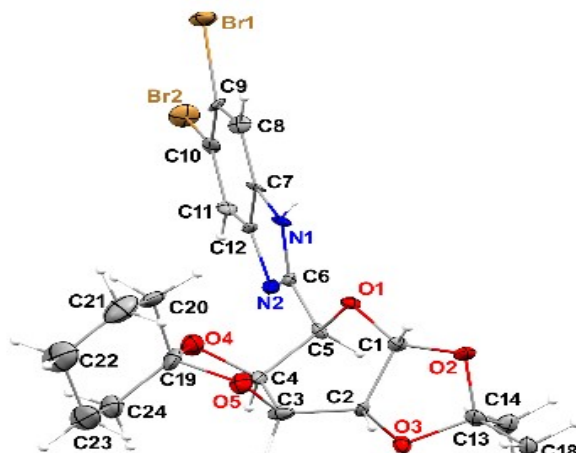


Figure-S41: ORTEP view of compound **6b**.

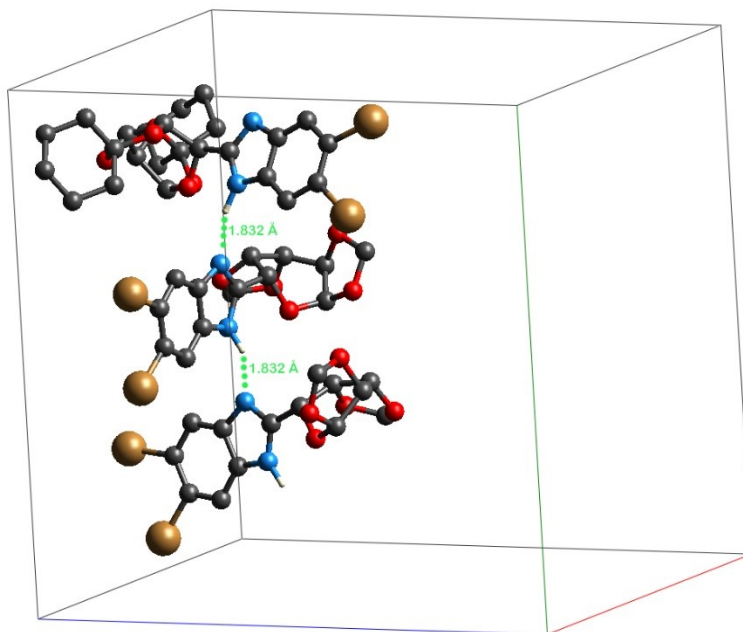


Figure-S42: Compound **6b** hydrogen bonded with a bond length of 1.832 Å in a unit cell.

Identification code	UK12A2	
Empirical formula	C ₄₈ H ₅₃ Br ₄ N ₄ O ₁₀	
Formula weight	1165.58	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	P 21 21 21	
Unit cell dimensions	a = 15.5001(5) Å	α = 90°.
	b = 18.3458(6) Å	β = 90°.
	c = 19.5228(6) Å	γ = 90°.
Volume	5551.5(3) Å ³	
Z	4	
Density (calculated)	1.395 Mg/m ³	
Absorption coefficient	3.983 mm ⁻¹	
F(000)	2356	
Crystal size	0.140 x 0.050 x 0.010 mm ³	
Theta range for data collection	3.306 to 68.230°.	
Index ranges	-18<=h<=18, -22<=k<=22, -23<=l<=23	
Reflections collected	93810	
Independent reflections	10156 [R(int) = 0.2207]	
Completeness to theta = 67.679°	100.0 %	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	10156 / 0 / 599	
Goodness-of-fit on F ²	1.077	
Final R indices [I>2sigma(I)]	R1 = 0.0713, wR2 = 0.1882	
R indices (all data)	R1 = 0.0941, wR2 = 0.2042	
Absolute structure parameter	0.000(15)	
Extinction coefficient	n/a	
Largest diff. peak and hole	3.353 and -0.589 e.Å ⁻³	
CCDC	2296464	

Table S5: Crystal data and structure refinement for **6b**

10. 5-bromo-2-((3a'R,5'R,5a'S,8a'S,8b'R)-tetrahydro-5'H-dispiro[cyclohexane-1,2'-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-7',1''-cyclohexan]-5'-yl)-1H-benzo[d]imidazole (7b) :

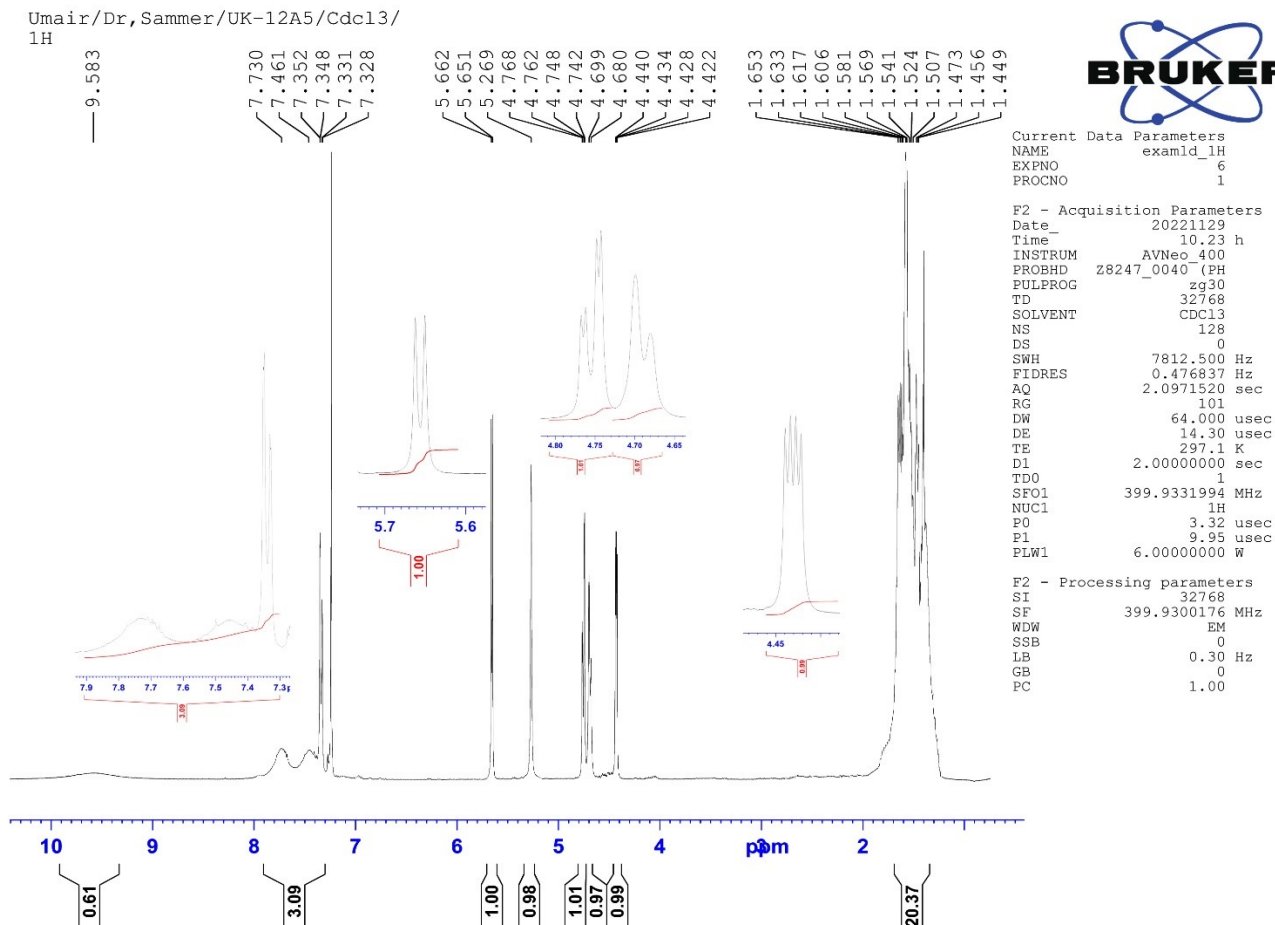
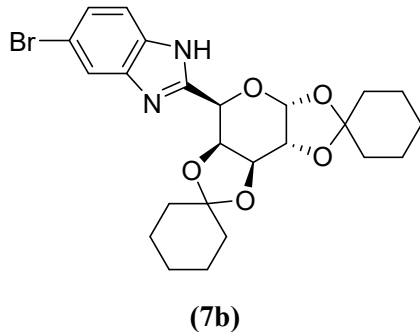


Figure-S36: $^1\text{H-NMR}$, in CDCl_3 , of compound 5-bromo-2-((3a'R,5'R,5a'S,8a'S,8b'R)-tetrahydro-5'H-dispiro[cyclohexane-1,2'-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran-7',1''-cyclohexan]-5'-yl)-1H-benzo[d]imidazole (7b)

- FAB +ve mood low and high resolution of compound 7b

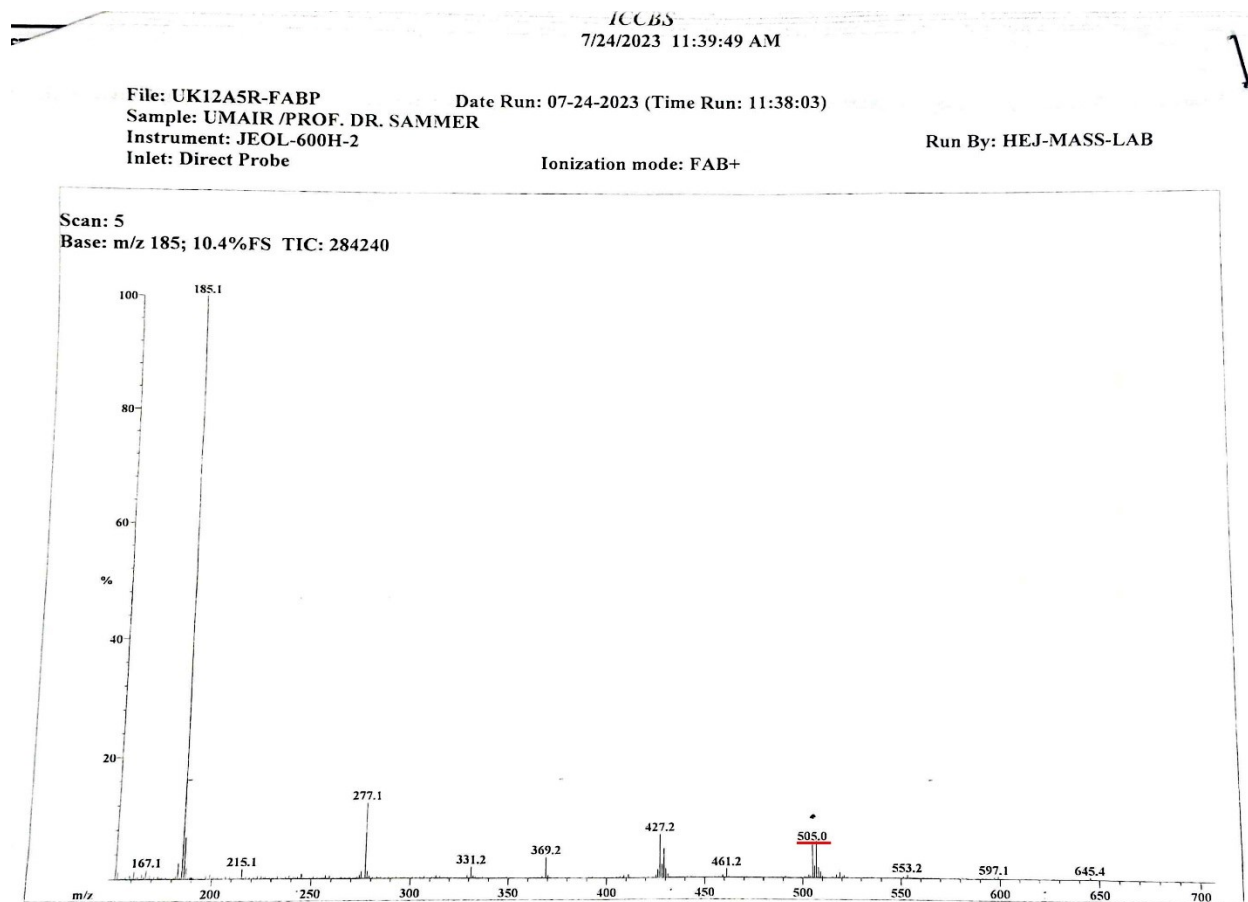


Figure S37: LR-FAB (+ve) shows the m/z value of 505.0 of compound (7b)

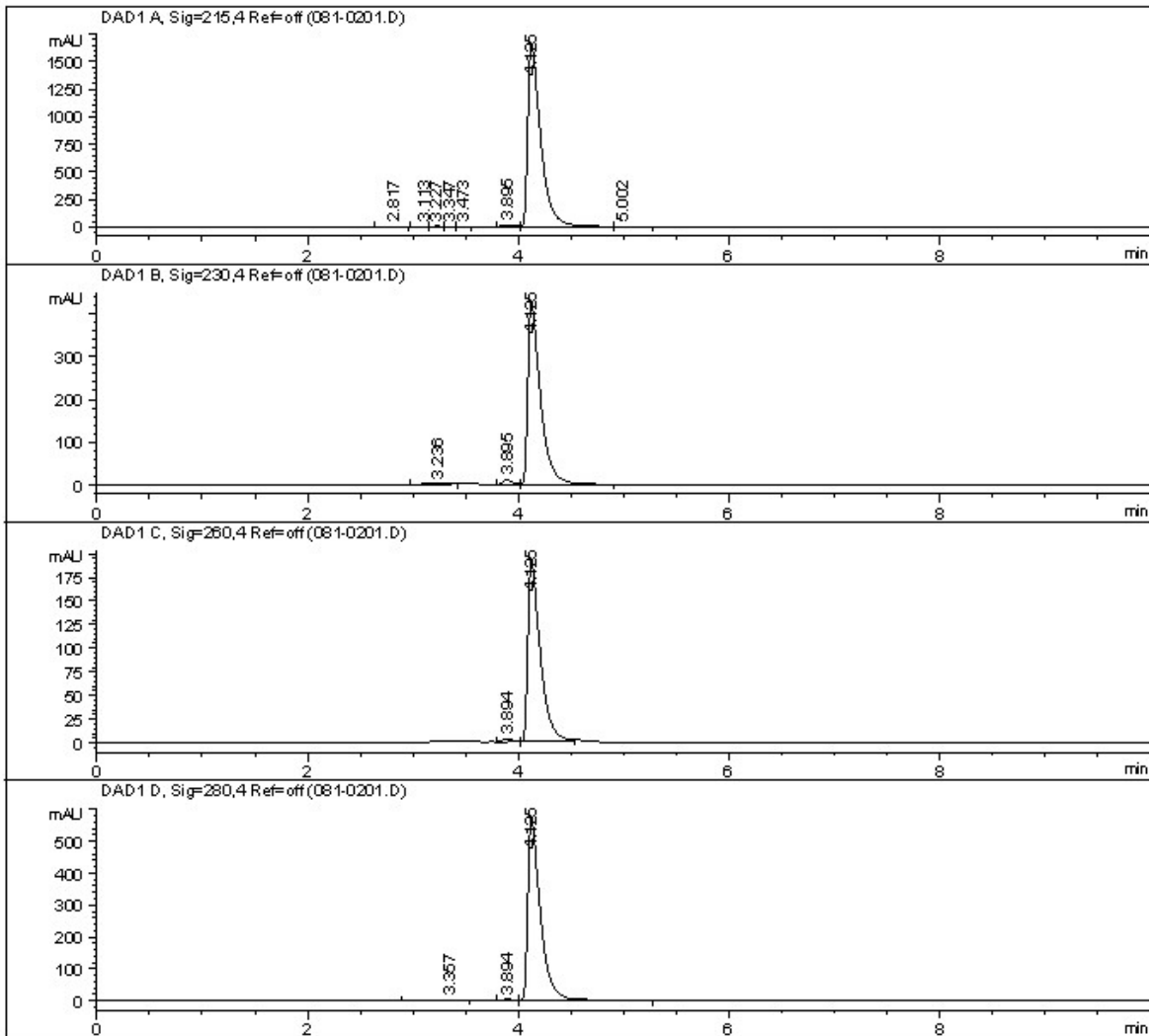
High resolution FAB positive mode at JEOL HX 110 Mass spectrometer of compound **7b** is **505.1338**, while theoretical mass was 505.1362 which corresponds to the composition of **C₂₄H₃₀N₂O₅Br**.

UPLC chromatogram of compound 7b

Data File C:\CHEM32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\081-0201.D
 Sample Name: UK-12A-5

```

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Acq. Operator   : junaid                      Seq. Line :    2
Acq. Instrument : UPLC 1                      Location  : Vial 81
Injection Date  : 13/02/2024 3:19:50 pm      Inj       :    1
                                           Inj Volume: 5.000 µl
Sequence File   : C:\Chem32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\UMAIR.S
Method          : C:\CHEM32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\UMAIR.M (Sequence
                  Method)
Last changed    : 13/02/2024 2:56:19 pm by junaid
Method Info     : column: C-4 (4.6 X 250mm) 5 µm 300A MN
  
```



Area Percent Report with Performance

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Multiplier:           :      1.0000
Dilution:             :      1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 A, Sig=215,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
2.817	-	27.24686	3.57714	0.81	0.1076	3786	-	-
3.113	-	22.65406	3.09987	2.35	0.1061	4767	1.63	1.11
3.227	-	23.71906	3.42440	1.23	-	-	-	1.04
3.347	-	12.06933	2.06440	1.15	-	-	-	1.04
3.473	-	6.58936	1.11123	1.70	-	-	-	1.04
3.895	-	106.71966	17.55243	0.67	0.0926	9795	-	1.12
4.125	-	1.41496e4	1666.28442	0.35	0.1235	6190	1.25	1.06
5.002	-	15.68651	1.63637	0.70	0.1551	5759	3.70	1.21

Signal 2: DAD1 B, Sig=230,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
3.236	-	26.43633	1.49282	2.00	0.3322	525	-	-
3.895	-	61.07037	10.56910	0.72	0.0889	10628	1.84	1.20
4.125	-	3547.29810	429.25229	0.36	0.1202	6531	1.29	1.06

Signal 3: DAD1 C, Sig=260,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
3.894	-	16.64886	3.13515	0.83	0.0852	11572	-	-
4.125	-	1547.11841	192.96204	0.37	0.1193	6624	1.32	1.06

Signal 4: DAD1 D, Sig=280,4 Ref=off

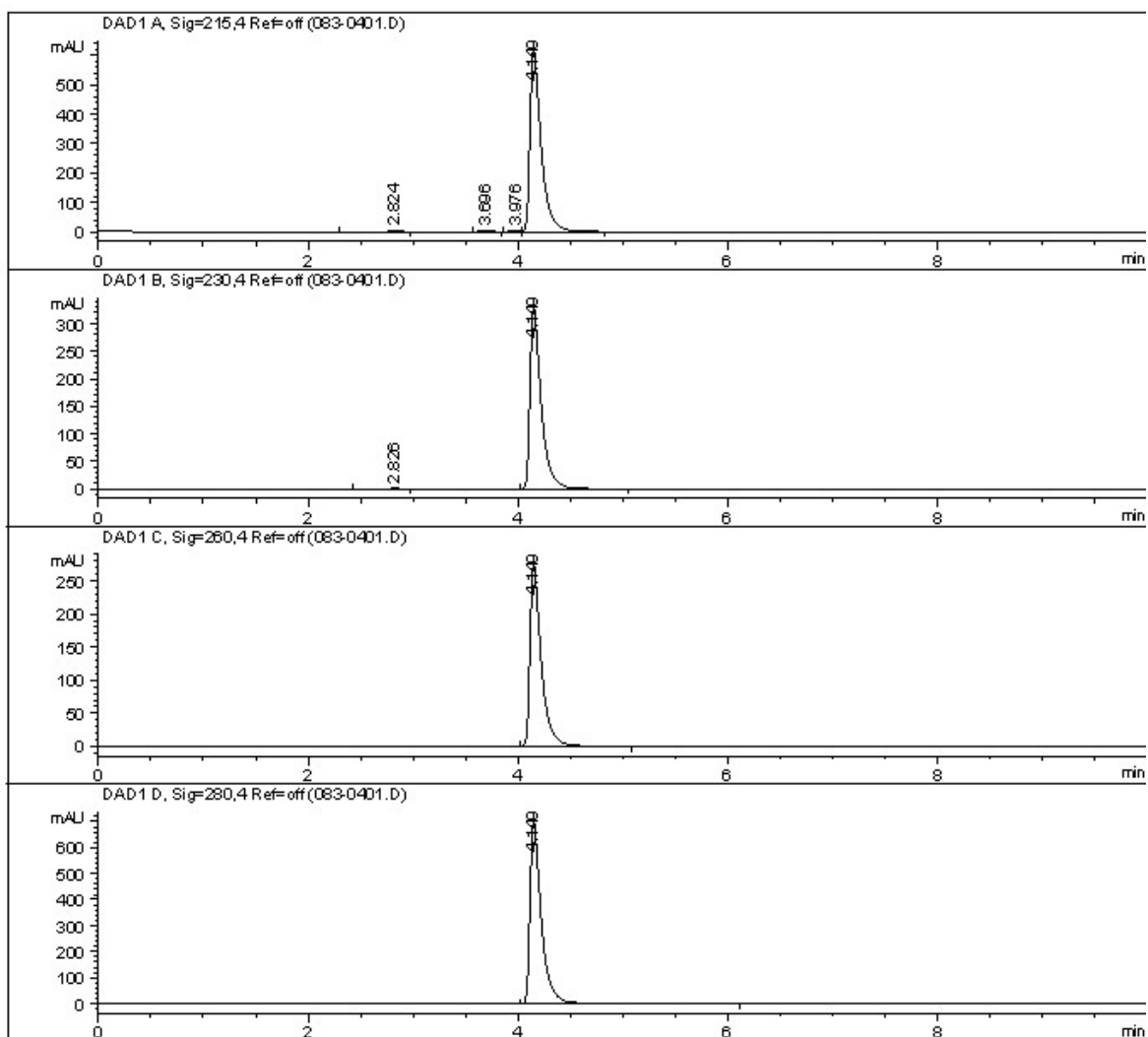
RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
3.357	-	19.16838	1.02615	2.36	0.3100	648	-	-
3.894	-	20.14855	3.67532	0.74	0.0852	11572	1.60	1.16
4.125	-	4737.69678	575.70844	0.36	0.1189	6669	1.33	1.06

=====
 *** End of Report ***

UPLC chromatogram of compound 4b

Data File C:\CHEM32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\083-0401.D
Sample Name: UK-12A-4

```
=====
Acq. Operator   : junaid                      Seq. Line :    4
Acq. Instrument : UPLC 1                     Location  : Vial 83
Injection Date  : 13/02/2024 3:43:32 pm      Inj       :    1
                                           Inj Volume: 5.000 µl
Sequence File   : C:\Chem32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\UMAIR.S
Method          : C:\CHEM32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\UMAIR.M (Sequence
Method)
Last changed    : 13/02/2024 2:56:19 pm by junaid
Method Info     : column: C-4 (4.6 X 250mm) 5 µm 300A MN
=====
```



Area Percent Report with Performance

```
=====
Multiplier:           :      1.0000
Dilution:             :      1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```


Data File C:\CHEM32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\083-0401.D
Sample Name: UK-12A-4

Signal 1: DAD1 A, Sig=215,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
2.824	-	42.40036	4.24094	1.45	0.1133	3446	-	-
3.696	-	21.82827	3.42733	0.89	0.0983	7815	4.84	1.31
3.976	-	21.88741	3.79497	1.12	0.0995	8837	1.66	1.08
4.149	-	4616.23145	623.58173	0.44	0.1059	8494	0.99	1.04

Signal 2: DAD1 B, Sig=230,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
2.826	-	9.74197	1.08728	1.19	0.1133	3446	-	-
4.149	-	2469.94141	333.21481	0.43	0.1057	8529	7.10	1.47

Signal 3: DAD1 C, Sig=260,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
4.149	-	2058.89551	277.60541	0.43	0.1058	8507	-	-

Signal 4: DAD1 D, Sig=280,4 Ref=off

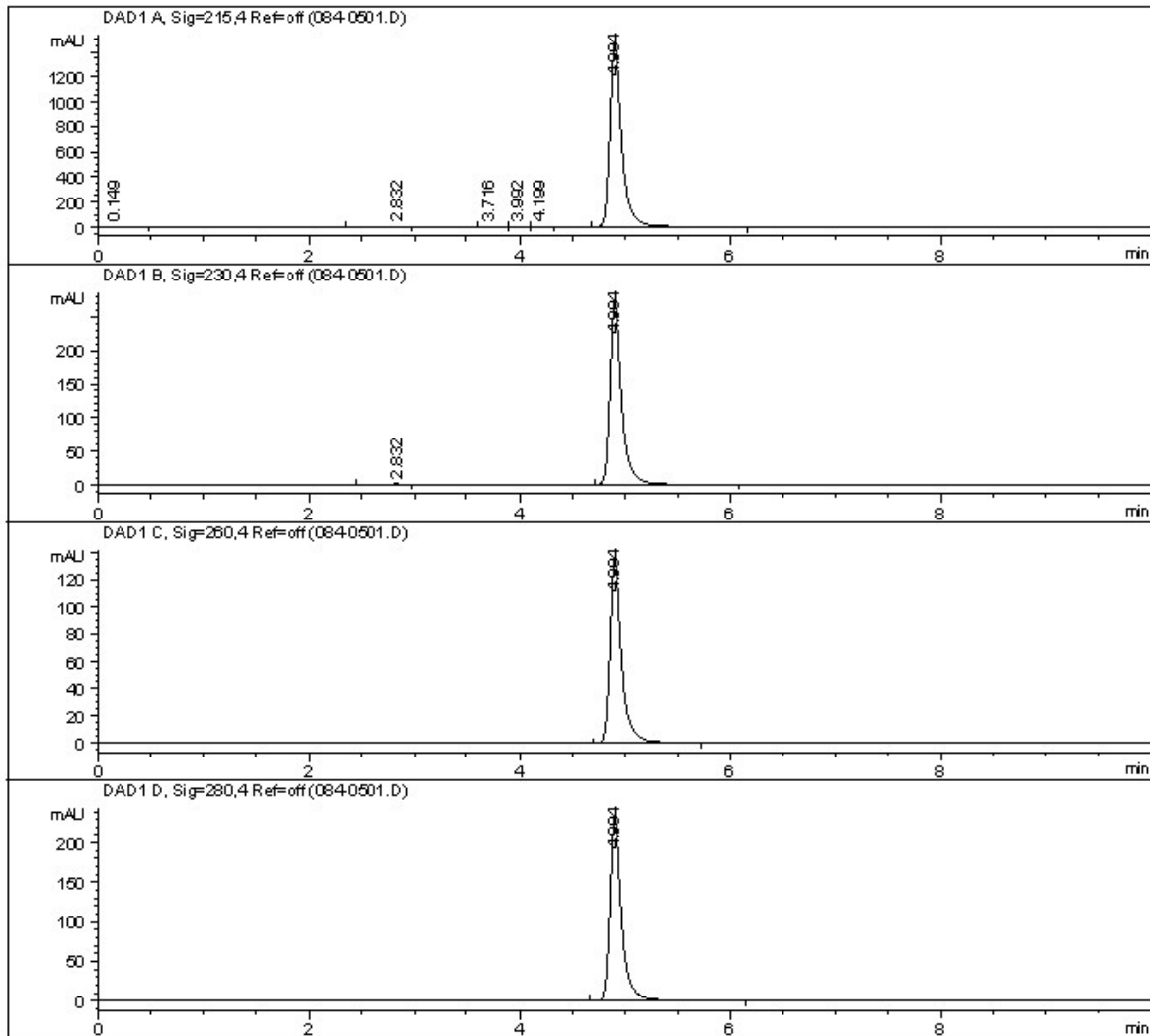
RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
4.149	-	5274.24707	706.34021	0.43	0.1061	8468	-	-

=====
*** End of Report ***

UPLC chromatogram of compound 5b

Data File C:\CHEM32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\084-0501.D
Sample Name: UK-12A-3

```
=====
Acq. Operator   : junaid                      Seq. Line :    5
Acq. Instrument : UPLC 1                      Location  : Vial 84
Injection Date  : 13/02/2024 3:55:25 pm      Inj       :    1
                                           Inj Volume: 5.000 µl
Sequence File   : C:\Chem32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\UMAIR.S
Method          : C:\CHEM32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\UMAIR.M (Sequence
                  Method)
Last changed    : 13/02/2024 2:56:19 pm by junaid
Method Info     : column: C-4 (4.6 X 250mm) 5 µm 300A MN
=====
```



Area Percent Report with Performance

```
=====
Multiplier:      :      1.0000
Dilution:        :      1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: DAD1 A, Sig=215,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
0.149	-	16.68434	1.14176	0.67	0.2367	2	-	-
2.832	-	35.10935	4.42344	1.25	0.0983	4599	9.41	19.07
3.716	-	16.69746	2.59213	0.63	0.0914	9138	5.48	1.31
3.992	-	11.49987	1.79916	0.76	0.0944	9904	1.74	1.07
4.199	-	10.46346	1.53487	1.10	0.1057	8745	1.21	1.05
4.904	-	1.18122e4	1472.91748	0.61	0.1140	10255	3.77	1.17

Signal 2: DAD1 B, Sig=230,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
2.832	-	8.44363	1.08245	1.20	0.1975	1140	-	-
4.904	-	2162.31494	273.87820	0.62	0.1120	10640	7.87	1.73

Signal 3: DAD1 C, Sig=260,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
4.904	-	1057.20068	135.24602	0.62	0.1109	10838	-	-

Signal 4: DAD1 D, Sig=280,4 Ref=off

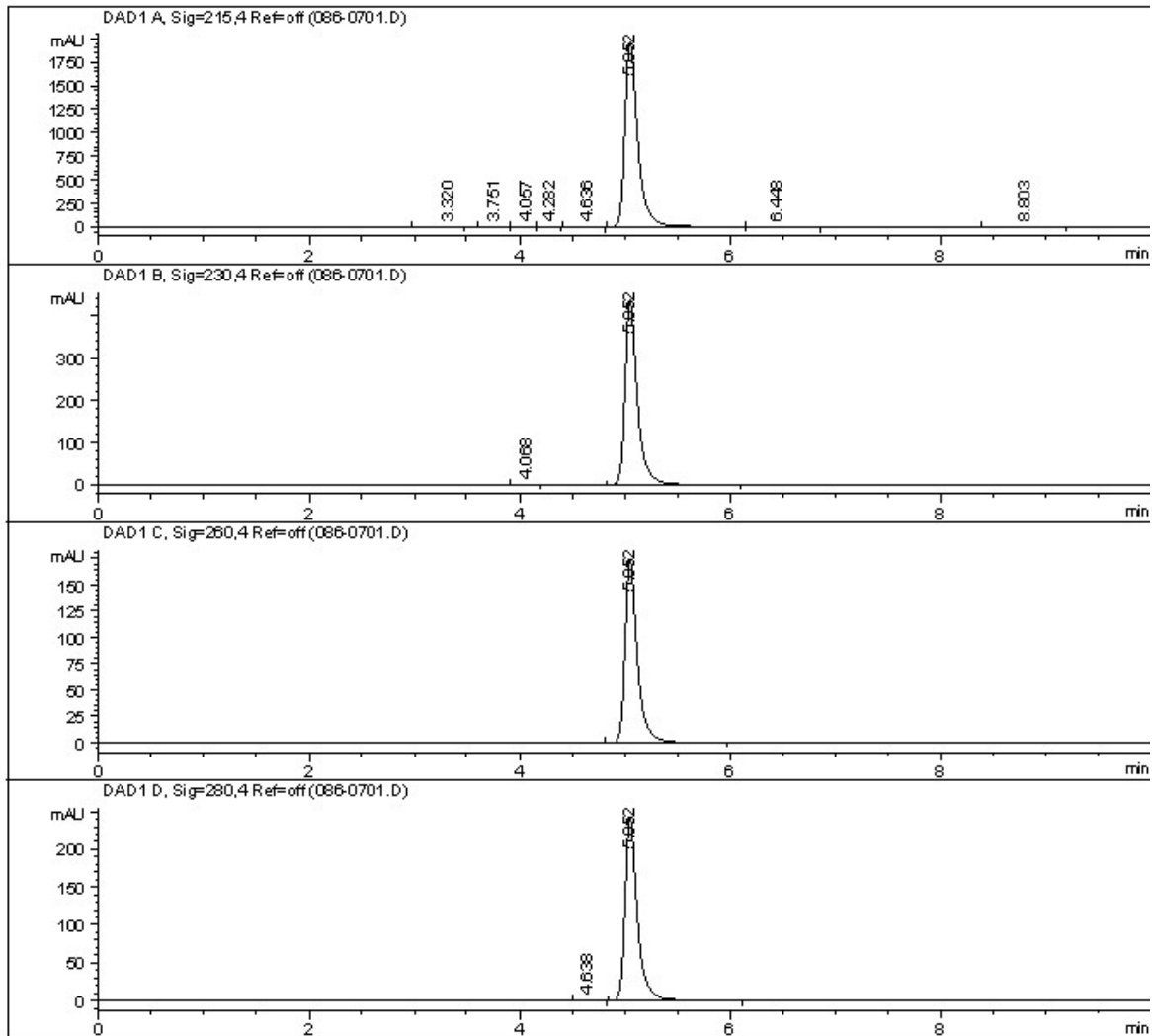
RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
4.904	-	1829.39355	233.40300	0.62	0.1109	10844	-	-

=====
*** End of Report ***

UPLC chromatogram of compound 6b

Data File C:\CHEM32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\086-0701.D
Sample Name: UK-GACDB

```
=====
Acq. Operator   : junaid                      Seq. Line :    7
Acq. Instrument : UPLC 1                      Location  : Vial 86
Injection Date  : 13/02/2024 4:19:10 pm      Inj       :    1
                                           Inj Volume: 5.000 µl
Sequence File   : C:\Chem32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\UMAIR.S
Method          : C:\CHEM32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\UMAIR.M (Sequence
Method)
Last changed   : 13/02/2024 2:56:19 pm by junaid
Method Info    : column: C-4 (4.6 X 250mm) 5 µm 300A MN
=====
```



Area Percent Report with Performance

```
=====
Multiplier:      :      1.0000
Dilution:        :      1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: DAD1 A, Sig=215,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
3.320	-	31.57682	1.51030	4.45	0.3017	671	-	-
3.751	-	13.58976	1.83765	0.64	0.1057	6984	1.24	1.13
4.057	-	21.92994	2.33251	1.54	0.1527	3905	1.39	1.08
4.282	-	8.10556	1.08677	1.40	0.1297	6033	0.94	1.06
4.636	-	24.56827	3.42490	0.94	0.1029	11242	1.79	1.08
5.052	-	1.67492e4	1956.52368	0.62	0.1226	9408	2.17	1.09
6.448	-	21.08078	1.73713	0.87	0.1600	8994	5.80	1.28
8.803	-	26.10264	1.51557	1.07	0.2622	6239	6.55	1.37

Signal 2: DAD1 B, Sig=230,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
4.068	-	7.43790	1.04434	1.46	0.1167	6731	-	-
5.052	-	3525.97095	433.83145	0.62	0.1152	10660	4.99	1.24

Signal 3: DAD1 C, Sig=260,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
5.052	-	1407.99158	174.68004	0.63	0.1142	10845	-	-

Signal 4: DAD1 D, Sig=280,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
4.638	-	6.87752	1.05156	0.79	0.0990	12158	-	-
5.052	-	1962.68347	243.15976	0.63	0.1144	10815	2.28	1.09

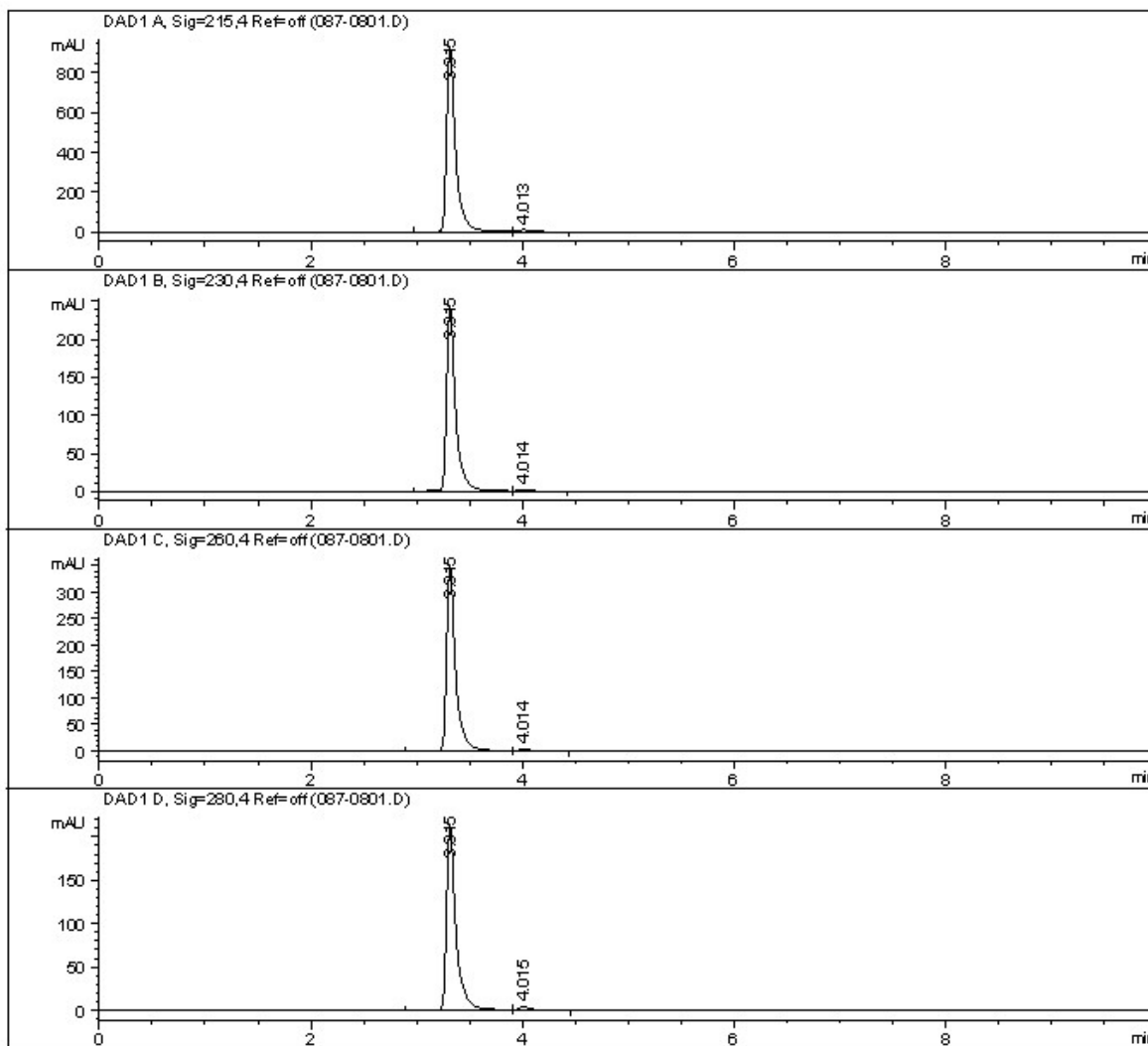
*** End of Report ***

UPLC chromatogram of compound 3a

Data File C:\CHEM32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\087-0801.D
 Sample Name: UK-GALH

```

=====
Acq. Operator   : junaid                      Seq. Line :    8
Acq. Instrument : UPLC 1                      Location  : Vial 87
Injection Date  : 13/02/2024 4:31:03 pm      Inj       :    1
                                           Inj Volume: 5.000 µl
Sequence File   : C:\Chem32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\UMAIR.S
Method          : C:\CHEM32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\UMAIR.M (Sequence
                  Method)
Last changed    : 13/02/2024 2:56:19 pm by junaid
Method Info     : column: C-4 (4.6 X 250mm) 5 µm 300A MN
  
```



Area Percent Report with Performance

```

=====
Multiplier:      :      1.0000
Dilution:        :      1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 A, Sig=215,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
3.315	-	5585.14697	931.62054	0.53	0.0818	9093	-	-
4.013	-	97.37994	11.36841	0.60	0.1073	7752	4.34	1.21

Signal 2: DAD1 B, Sig=230,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
3.315	-	1463.15112	243.15395	0.54	0.0816	9124	-	-
4.014	-	24.47080	2.81728	0.62	0.1111	7228	4.26	1.21

Signal 3: DAD1 C, Sig=260,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
3.315	-	2071.30249	350.07733	0.53	0.0814	9182	-	-
4.014	-	29.21347	3.90823	0.68	0.1000	8923	4.53	1.21

Signal 4: DAD1 D, Sig=280,4 Ref=off

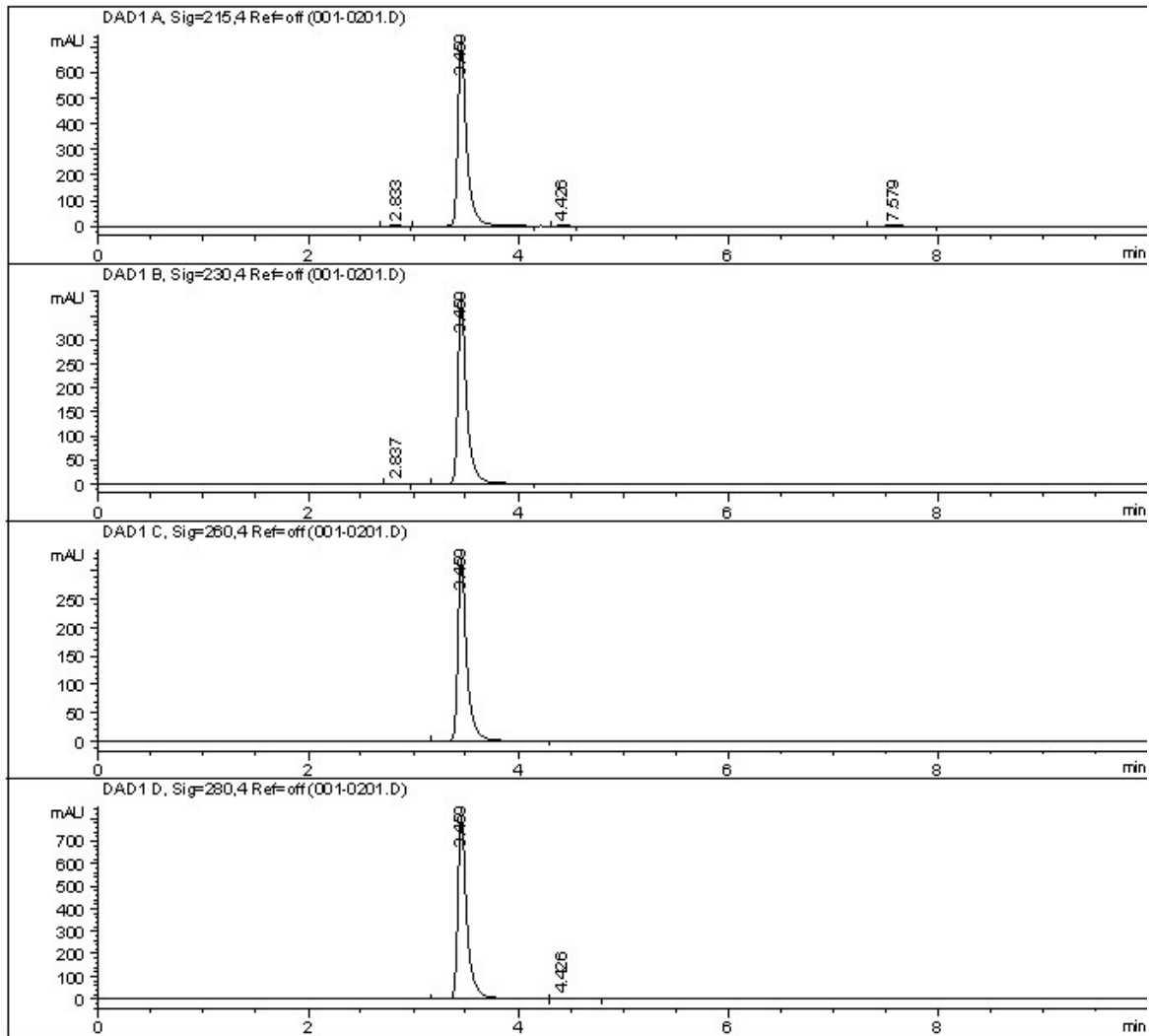
RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
3.315	-	1324.80798	213.58643	0.51	0.0840	8626	-	-
4.015	-	30.22739	4.01984	0.65	0.0987	9169	4.50	1.21

=====
*** End of Report ***

UPLC chromatogram of compound 4a

Data File C:\CHEM32\1\DATA\UMAIR\UMAIR 2024-02-12 15-47-36\001-0201.D
Sample Name: UKG-ALF2

```
=====
Acq. Operator   : junaid                      Seq. Line :    2
Acq. Instrument : UPLC 1                     Location  : Vial 1
Injection Date  : 12/02/2024 3:59:50 pm      Inj       :    1
                                                Inj Volume: 5.000 µl
Sequence File   : C:\Chem32\1\DATA\UMAIR\UMAIR 2024-02-12 15-47-36\UMAIR.S
Method          : C:\CHEM32\1\DATA\UMAIR\UMAIR 2024-02-12 15-47-36\UMAIR.M (Sequence
                  Method)
Last changed    : 12/02/2024 3:47:29 pm by junaid
Method Info     : column: C-4 (4.6 X 250mm) 5 µm 300A MN
=====
```



Area Percent Report with Performance

```
=====
Multiplier:           :      1.0000
Dilution:             :      1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```


Signal 1: DAD1 A, Sig=215,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
2.833	-	27.59914	3.92927	0.73	0.1017	4303	-	-
3.459	-	4310.45410	716.11633	0.57	0.0828	9668	3.98	1.22
4.426	-	11.62718	1.82169	1.01	0.0989	11101	6.25	1.28
7.579	-	19.86694	1.71173	0.88	0.1778	10071	13.39	1.71

Signal 2: DAD1 B, Sig=230,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
2.837	-	6.65760	1.00267	0.73	0.1011	4350	-	-
3.459	-	2252.34741	381.79037	0.57	0.0829	9655	3.97	1.22

Signal 3: DAD1 C, Sig=260,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
3.459	-	1904.16602	321.97821	0.56	0.0831	9610	-	-

Signal 4: DAD1 D, Sig=280,4 Ref=off

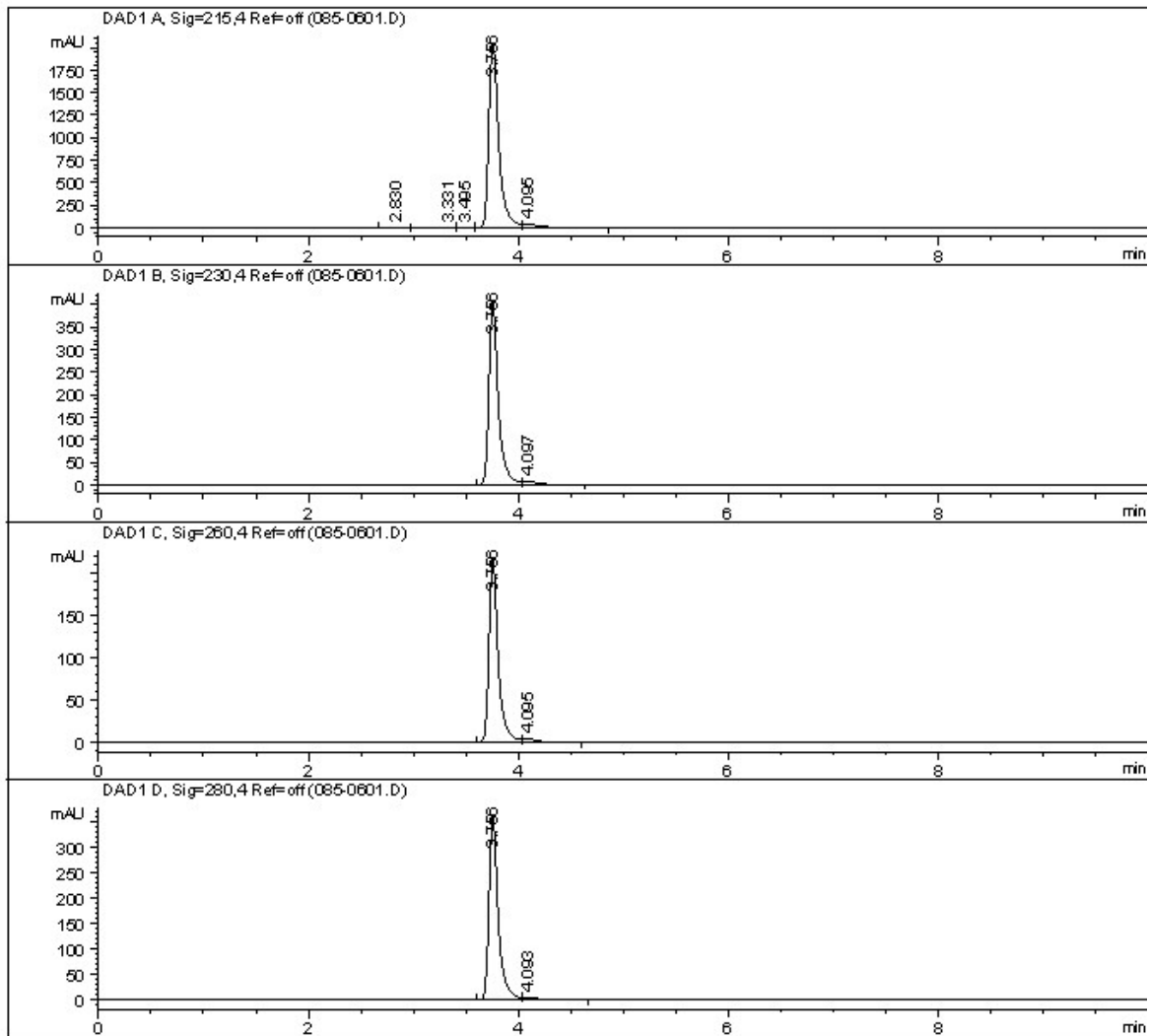
RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
3.459	-	4816.05811	813.93890	0.56	0.0830	9631	-	-
4.426	-	14.36475	1.91712	0.74	0.1044	9952	6.06	1.28

=====
*** End of Report ***

UPLC chromatogram of compound 5a

Data File C:\CHEM32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\085-0601.D
Sample Name: UK-GACL-2

```
=====
Acq. Operator   : junaid                      Seq. Line :    6
Acq. Instrument : UPLC 1                     Location  : Vial 85
Injection Date  : 13/02/2024 4:07:17 pm      Inj       :    1
                                           Inj Volume: 5.000 µl
Sequence File   : C:\Chem32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\UMAIR.S
Method          : C:\CHEM32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\UMAIR.M (Sequence
Method)
Last changed   : 13/02/2024 2:56:19 pm by junaid
Method Info    : column: C-4 (4.6 X 250mm) 5 µm 300A MN
=====
```



Area Percent Report with Performance

```
=====
Multiplier:      :      1.0000
Dilution:        :      1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Data File C:\CHEM32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\085-0601.D
Sample Name: UK-GACL-2

Signal 1: DAD1 A, Sig=215,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
2.830	-	16.91151	2.55702	0.81	0.0950	4928	-	-
3.331	-	48.86526	2.40011	4.06	0.3520	497	1.32	1.18
3.495	-	23.30044	2.38794	0.93	-	-	-	1.05
3.756	-	1.35003e4	2047.88220	0.61	0.0946	8714	-	1.07
4.095	-	400.53870	42.69287	0.44	0.1251	5931	1.81	1.09

Signal 2: DAD1 B, Sig=230,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
3.756	-	2491.60254	407.10132	0.62	0.0872	10265	-	-
4.097	-	67.26482	7.98565	0.55	0.1269	5779	1.87	1.09

Signal 3: DAD1 C, Sig=260,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
3.756	-	1316.99841	217.63165	0.63	0.0860	10552	-	-
4.095	-	29.52357	3.45678	0.50	0.1225	6186	1.91	1.09

Signal 4: DAD1 D, Sig=280,4 Ref=off

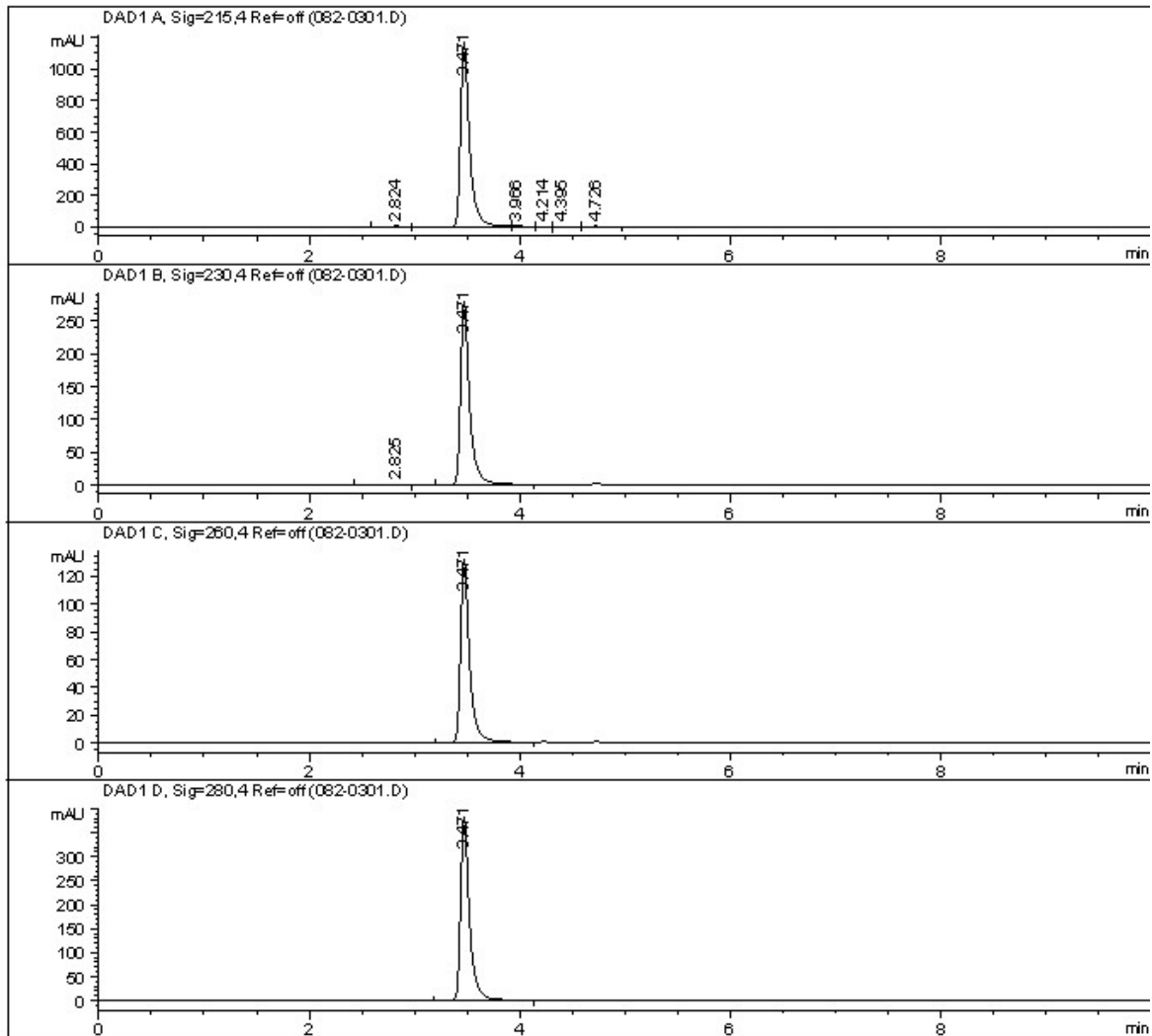
RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
3.756	-	2188.78882	361.14264	0.63	0.0862	10500	-	-
4.093	-	42.85130	4.84483	0.42	0.1204	6406	1.92	1.09

=====
*** End of Report ***

UPLC chromatogram of compound 7a

Data File C:\CHEM32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\082-0301.D
Sample Name: UK-GALBR

```
=====
Acq. Operator   : junaid                      Seq. Line :    3
Acq. Instrument : UPLC 1                      Location  : Vial 82
Injection Date  : 13/02/2024 3:31:42 pm      Inj       :    1
                                           Inj Volume: 5.000 µl
Sequence File   : C:\Chem32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\UMAIR.S
Method          : C:\CHEM32\1\DATA\UMAIR\UMAIR 2024-02-13 14-56-21\UMAIR.M (Sequence
                  Method)
Last changed    : 13/02/2024 2:56:19 pm by junaid
Method Info     : column: C-4 (4.6 X 250mm) 5 µm 300A MN
=====
```



Area Percent Report with Performance

```
=====
Multiplier:      :      1.0000
Dilution:        :      1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: DAD1 A, Sig=215,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
2.824	-	30.33011	4.30497	0.88	0.1000	4426	-	-
3.471	-	7008.73242	1169.25415	0.57	0.0836	9564	4.14	1.23
3.966	-	35.88287	4.02049	0.33	0.1546	3648	2.45	1.14
4.214	-	14.61916	1.82261	0.83	-	-	-	1.06
4.395	-	8.54136	1.04321	1.00	0.1440	5157	-	1.04
4.726	-	20.46066	2.68093	0.81	0.1167	9093	1.49	1.08

Signal 2: DAD1 B, Sig=230,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
2.825	-	7.67525	1.04858	0.97	0.1963	1149	-	-
3.471	-	1663.11353	280.86484	0.56	0.0831	9668	2.71	1.23

Signal 3: DAD1 C, Sig=260,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
3.471	-	778.96527	132.36870	0.57	0.0829	9725	-	-

Signal 4: DAD1 D, Sig=280,4 Ref=off

RetTime [min]	k'	Area [mAU*s]	Height [mAU]	Symm.	Width [min]	Plates	Resol ution	Select ivity
3.471	-	2252.70557	383.80142	0.57	0.0826	9799	-	-

=====
*** End of Report ***