

Bis-Triazologlycolipid mimetics - Low molecular weight organogelator

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General procedure for *bis*-propargylated derivatives and its corresponding NMR data are provided in ESI.

¹H NMR, ¹³C NMR, DEPT-135, 2D NMR and Mass spectrum are available in the ESI

Figure 6: ¹H NMR spectrum (300 MHz, CDCl₃) of compound, 3.

Figure 7: ¹³C NMR spectrum (75 MHz, CDCl₃) of compound, 3.

Figure 8: ¹H NMR spectrum (300 MHz, CDCl₃) of compound, 5a.

Figure 9: ¹³C NMR spectrum (75 MHz, CDCl₃) of compound, 5a.

Figure 10: ¹H NMR spectrum (300 MHz, CDCl₃) of compound, 7a.

Figure 11: ¹³C NMR spectrum (75 MHz, CDCl₃) of compound, 7a.

Figure 12: DEPT-135 spectrum (75 MHz, CDCl₃) of compound, 7a.

Figure 13: ¹H-¹³C correlation spectrum (300 MHz, CDCl₃) of compound, 7a.

Figure 14: Mass spectrum of compound, 7a.

Figure 15: ¹H NMR spectrum (300 MHz, CDCl₃) of compound, 4.

Figure 16: ¹³C NMR spectrum (75 MHz, CDCl₃) of compound, 4.

Figure 17: ¹H NMR spectrum (300 MHz, CDCl₃) of compound, 6b.

Figure 18: ¹³C NMR spectrum (75 MHz, CDCl₃) of compound, 6b.

Figure 19: ¹H NMR spectrum (300 MHz, CDCl₃) of compound, 8b.

Figure 20: ¹³C NMR spectrum (75 MHz, CDCl₃) of compound, 8b.

Figure 21: DEPT-135 spectrum (75 MHz, CDCl₃) of compound, 8b.

Figure 22 Mass spectrum of compound, 8b.

Figure 23 ¹H NMR expansion spectrum (300 MHz, CDCl₃) of compound, 5a.

Figure 24 ¹H NMR expansion spectrum (300 MHz, CDCl₃) of compound, 6b.

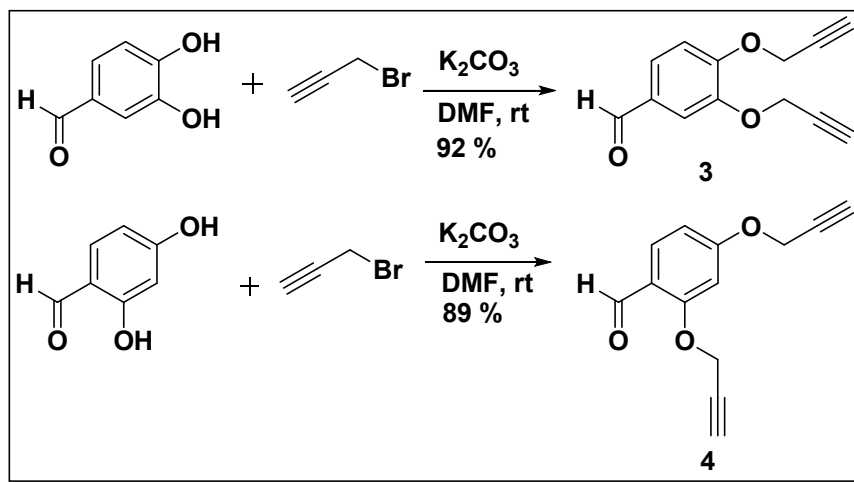
Figure 25 ¹H NMR expansion spectrum (300 MHz, CDCl₃) of compound, 7a.

Figure 26 ¹H NMR expansion spectrum (300 MHz, CDCl₃) of compound, 8b.

Table: 1 Optimisation of solvents

Entry No	Solvent	Yield (%)
1	Tertiary butanol	-
2	Acetonitrile	-
3	Tetrahydrofuran	89

While assigning the spectral data, several abbreviations were used and these include ‘Ar’ for aromatic, ‘Sac’ for saccharide ‘Alk’ for alkene and “trz” for triazole respectively.



Synthesis of *bis*-propargylated aldehydes (3) and (4).

1. General procedure for the synthesis of *bis* alkylated aldehyde, 3 & 4:

To a solution of dihydroxy benzaldehyde, (1 mmol) in dry DMF (15 ml) were added potassium carbonate (5 equiv.) and after stirring the solution for 5 minutes vigorously, propargyl bromide (2.2 equiv.) was added and the reaction mixture kept for stirring for 24 hrs. After careful work up, the product was further purified by column chromatography.

Spectroscopic data for compounds 3 and 4 are as follows.

1.1 Physicochemical and spectrochemical data of 3,4-bis(prop-2-ynyloxy)benzaldehyde (3):

Colourless crystal, Yield: 89 % (0.19 g), mp 96-98 °C, ¹H NMR (300 MHz, CDCl₃): δ 9.82 (s, 1H, -CHO), 7.51-7.45 (m, 2H, Ar-H), 7.11 (d, J = 8.1 Hz, 1H, Ar-H), 4.79 (d, J = 2.4 Hz, 2H, -CH₂), 4.76 (d, J = 2.4 Hz, 2H, -CH₂), 2.50 (m, 2H, -C≡H), ¹³C NMR (75 MHz, CDCl₃) : δ 190.7, 152.7, 147.8, 130.8, 126.7, 113.2, 112.6, 77.7, 77.5, 76.7, 76.5, 56.8, 56.7. Elemental Analysis: C, 72.89; H, 4.71, Found: C, 72.82; H, 4.69.

1.2 Physicochemical and spectrochemical data of 2,4-bis(prop-2-ynyloxy)benzaldehyde (4):

Colourless crystal, Yield: 77 % (0.72 g), mp 104-106 °C, ¹H NMR (300 MHz, CDCl₃): δ 10.32 (s, 1H, -CHO), 7.85 (d, J = 9 Hz, 1H, Ar-H), 6.70 (s, 1H, Ar-H), 6.67 (d, J = 1.8 Hz, 1H, Ar-H), 4.81 (d, J = 2.4 Hz, 2H, -CH₂), 4.77 (d, J = 2.4 Hz, 2H, -CH₂), 2.60 (s, 2H, -C≡H), ¹³C NMR (75 MHz, CDCl₃): δ:188.1, 163.6, 161.4, 130.6, 120.1, 107.5, 100.4, 77.5, 77.0, 76.7, 76.5, 56.5, 56.1, Elemental Analysis: C, 72.89; H, 4.71, Found: C, 72.85 ; H, 4.67.

2. General procedure for the synthesis of α, β-unsaturated β-C-glycosidic ketones, 5a, 5b, 6a, 6b:

To a solution of β-C-glycosidic ketone, **1**, **2** (1 mmol) in dry DCM (5 ml) were added pyrrolidine (30% mol) and bis-propargylated-benzaldehyde **3**, **4** (1.2 mmol). After stirring at room temperature for given period of time, the reaction mixture was evaporated under reduced pressure and extracted by EtOAc-water. The ethylacetate layer was dried over anhyd. Na₂SO₄ and concentrated to dryness. The product was

further purified by column chromatography.

Spectroscopic data for compounds **5a**, **5b**, **6a** and **6b** are as follows.

2.1 Physicochemical and spectral data for (E)-1-(4,6-*O*-butylidene- β -D-glucopyranosyl)-4-(3,4-bis prop-1'-oxo-2'-yne phenyl)-but-3-ene-2-one (5a)

White solid, Yield: 91 % (0.43 g), mp 156-158 °C, ^1H NMR (300 MHz, CDCl_3): δ 7.55 (d, J = 15.9 Hz, 1H, Alk-H), 7.28 (s, 1H, Ar-H), 7.22 (d, J = 8.4 Hz, 1H, Ar-H), 7.08 (d, J = 8.1 Hz, 1H, Ar-H), 6.68 (d, J = 16.2 Hz, 1H, Alk-H), 4.82 (s, 4H, $-\text{CH}_2$), 4.55 (t, J = 5Hz, 1H, Sac-H), 4.15 (dd, J = 3.9 Hz, J = 9.6 Hz, 1H, Sac-H), 3.98-3.92 (m, 1H, Sac-H), 3.74 (t, J = 8.7 Hz, 1H, Sac-H), 3.47-3.35 (m, 3H, Sac-H), 3.26 (t, J = 9.0 Hz, 1H, Sac-H), 3.17-3.11 (m, 2H, Sac-H, $-\text{CH}_2$), 2.97 (dd, J = 6.9 Hz, J = 16.1 Hz, $-\text{CH}_2$), 1.66-1.62 (m, 2H, $-\text{CH}_2$), 1.44 (q, J = 7.5 Hz, 2H, $-\text{CH}_2$), 0.94 (t, J = 7.2 Hz, 3H, Sac-H), ^{13}C NMR (75 MHz, CDCl_3) : δ :198.0, 149.9, 147.6, 143.5, 128.3, 125.1, 123.8, 114.2, 113.9, 102.5, 80.5, 78.0, 77.9, 76.4, 76.4, 76.2, 75.4, 74.5, 70.6, 68.3, 57.0, 56.7, 43.4, 36.2, 30.9, 17.5, 13.9, Elemental Analysis: C, 66.37; H, 6.43, Found: C, 66.35; H, 6.41.

2.2 Physicochemical and spectral data for (E)-1-(2,3,4,6-tetra-*O*-acetyl- β -D-glucopyranosyl)-4-(3,4-bis prop-1'-oxo-2'-yne phenyl)-but-3-ene-2-one (5b)

Syrupy liquid, Yield: 89 % (0.52 g), ^1H NMR (300 MHz, CDCl_3): δ 7.50 (d, J = 16.2 Hz, 1H, Alk-H), 7.28 (m, 2H, Ar-H), 7.27 (d, J = 8.4 Hz, Ar-H), 7.07 (d, J = 8.4 Hz, 1H, Ar-H), 6.63 (d, J = 16.2 Hz, 1H, Alk-H), 5.24 (t, J = 9.3 Hz, 1H, Sac-H), 5.08 (t, J = 9.8 Hz, 1H, Sac-H), 4.99 (t, J = 9.6 Hz, 1H, Sac-H), 4.81 (d, J = 2.4 Hz, 4H, $-\text{CH}_2$), 4.26 (dd, J = 4.8 Hz, J = 12.6 Hz, 1H, Sac-H), 4.15-4.12 (m, 1H, Sac-H), 4.03 (dd, J = 2.1 Hz, J = 12.3 Hz, 1H, Sac-H), 3.74-3.70 (m, 1H, Sac-H), 3.02 (dd, J = 8.4 Hz, J = 16.2 Hz, 1H, $-\text{CH}_2$), 2.67 (dd, J = 3.2 Hz, J = 16.2 Hz, 1H, $-\text{CH}_2$), 2.58-2.55 (m, 2H, $-\text{C}\equiv\text{C}$), 2.02 (t, J = 2.9 Hz, 12H, $-\text{OCOCH}_3$), ^{13}C NMR (75 MHz, CDCl_3) : δ 196.0, 170.6, 170.2, 170.0, 169.6,

149.9, 147.6, 143.4, 128.2, 125.0, 123.8, 114.2, 113.7, 78.0, 77.9, 76.6, 76.4, 75.7, 74.2, 71.7, 68.5, 62.1, 56.9, 56.7, 42.5, 29.7, 20.7 (2C), 20.6, Elemental Analysis: C, 61.64; H, 5.52, Found: C, 61.60; H, 5.49; N, 32.81.

2.3 Physicochemical and spectral data for (E)-1-(4,6-*O*-butylidene- β -D-glucopyranosyl)-4-(2,4-bis prop-1'-oxo-2'-yne phenyl)-but-3-ene-2-one (6a)

Syrupy liquid, Yield: 79 % (0.89 g), ^1H NMR (300 MHz, CDCl_3): δ 7.87 (d, J = 16.2 Hz, 1H, Alk-H), 7.52 (d, J = 8.7 Hz, 1H, Ar-H), 6.77 (s, 1H, Ar-H), 6.72-6.62 (m, 2H, Ar-H, Alk-H), 4.77 (d, J = 2.4 Hz, 2H, $-\text{CH}_2$), 4.74 (d, J = 2.4 Hz, 2H, $-\text{CH}_2$), 4.53 (t, J = 5.1 Hz, 1H, Ace-H), 4.16-4.11 (m, 2H, Sac-H), 3.96-3.89 (m, 1H, Sac-H), 3.73 (t, J = 8.7 Hz, 1H, Sac-H), 3.43 (t, J = 8.9 Hz, 2H, Sac-H), 3.25 (t, J = 9.0 Hz, 1H, Sac-H), 3.11 (dd, J = 4.3 Hz, J = 16.2 Hz, 1H, $-\text{CH}_2$), 2.99 (dd, J = 6.9 Hz, J = 16.1 Hz, 1H, $-\text{CH}_2$), 2.57 (t, 2.1 Hz, 2H, $-\text{C}\equiv\text{CH}$), 1.65-1.60 (m, 2H, $-\text{CH}_2$), 1.42 (q, J = 7.8 Hz, 2H, $-\text{CH}_2$), 0.92 (t, J = 7.4 Hz, 3H, $-\text{CH}_3$), ^{13}C NMR (75 MHz, CDCl_3) : δ 198.6, 160.7, 157.8, 138.7, 130.0, 125.3, 117.7, 107.4, 102.5, 100.8, 80.4, 77.9, 75.4, 74.8, 70.6, 68.4, 56.3, 56.0, 43.3, 36.2, 17.5, 13.9, Elemental Analysis: C, 66.37; H, 6.43, Found: C, 66.33; H, 6.38.

2.4 Physicochemical and spectral data for (E)-1-(2,3,4,6-tetra-*O*-acetyl- β -D-glucopyranosyl)-4-(2,4-bis prop-1'-oxo-2'-yne phenyl)-but-3-ene-2-one (6b)

Syrupy liquid, Yield: 79 % (0.89 g), ^1H NMR (300 MHz, CDCl_3): δ 7.82 (d, J = 16.2 Hz, 1H, Alk-H), 7.52 (d, J = 8.7 Hz, 1H, Ar-H), 6.75-6.63 (m, 3H, Alk-H, Ar-H), 5.23 (t, J = 9.3 Hz, 1H, Sac-H), 5.08 (t, J = 9.8 Hz, 1H, Sac-H), 4.99 (t, J = 9.8 Hz, 1H, Sac-H), 4.79 (s, 2H, $-\text{CH}_2$), 4.74 (s, 2H, $-\text{CH}_2$), 4.27 (dd, J = 4.8 Hz, J = 12.3 Hz, 1H, Sac-H), 4.16-4.00 (m, 4H, Sac-H), 3.72 (dd, J = 3H, J = 9.8 Hz, 1H, Sac-H), 3.02 (dd, J = 8.4 Hz, J = 16.2 Hz, 1H, Sac-H), 2.67 (dd, J = 3 Hz, J = 16.2 Hz, 1H, Sac-H), 2.59 (d, J = 2.1 Hz,

2H, -C≡H), 2.05-2.01 (m, 12H, -COCH₃), ¹³C NMR (75 MHz, CDCl₃) : δ 196.5, 170.7, 170.3, 170.0, 169.6, 160.7, 157.8, 138.5, 130.1, 125.2, 117.6, 107.4, 100.8, 77.9, 77.8, 76.5, 76.2, 75.7, 74.3 (2C), 71.8, 68.6, 62.1, 60.4, 56.3, 56.0, 42.3, 30.9, 21.0, 20.7 (2C), 20.6 (2C), 14.2, Elemental Analysis: C, 61.64; H, 5.52, Found: C, 61.61 ; H, 5.47.

Synthesis of Gels:

The gelation ability of the bis-triazologlycolipids was determined by the “inversion tube method.” All the synthesized triazologlycolipids was tested for gelation by the method where 1 mg of the compound was dissolved in 1 mL of the solvent in a close capped vial to result in 1% CGC. The mixture was heated till all the solid gets dissolved and the vial was cooled to 25 °C and left for 12h at this temperature and then turned upside down. When the gelator formed is immobilized at this stage, it is said to be a Gel and it is denoted by “G”. Some of them forms partial gel is denoted as “PG”. Some of them which are insoluble is represented as “I” and those which got precipitated is denoted as “P”.



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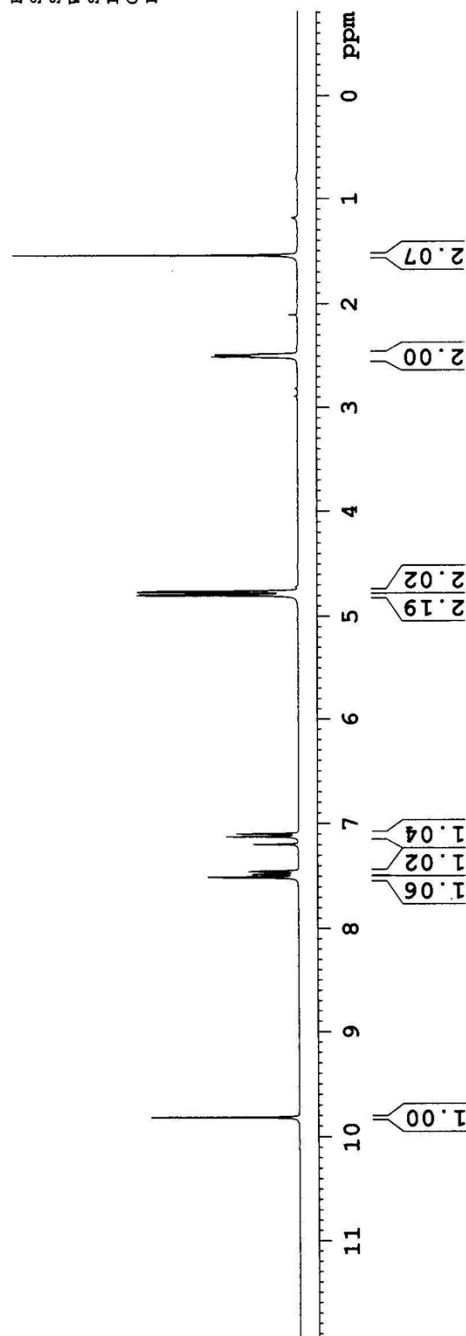
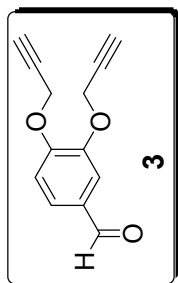
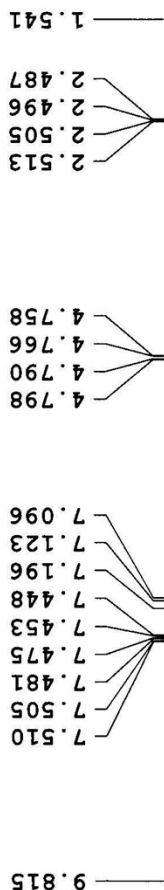


Figure 6 ¹H NMR spectrum (300 MHz, CDCl₃) of compound, 3.

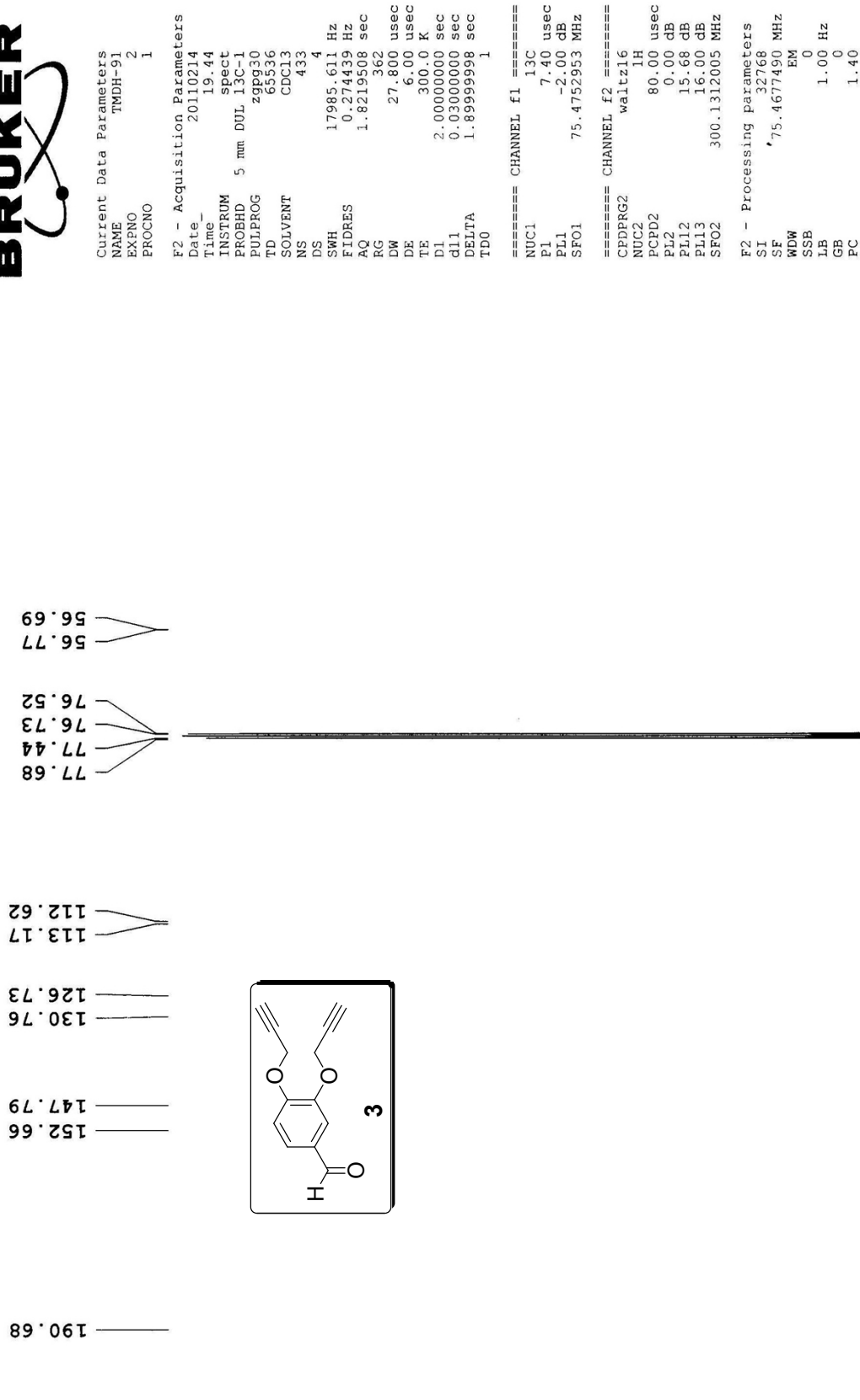
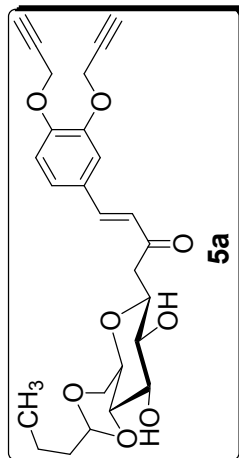
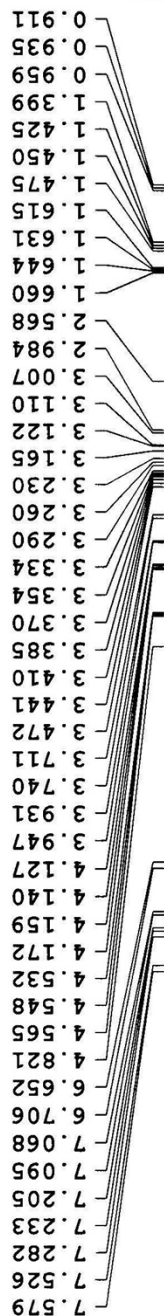


Figure 7 ¹³C NMR spectrum (75 MHz, CDCl₃) of compound, 3.



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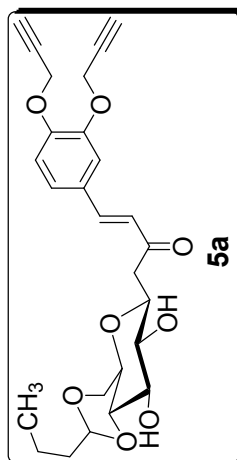
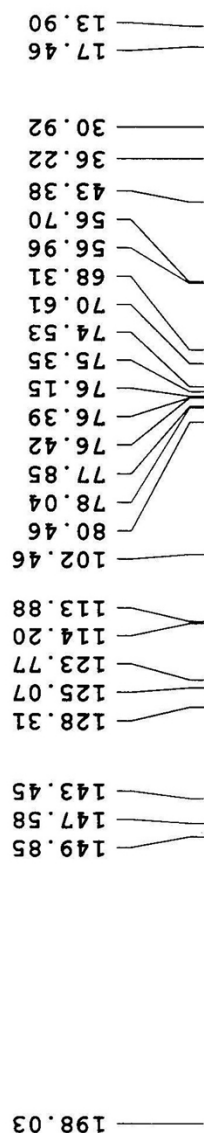
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FIDRES 0.094190 Hz
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RG 456.1
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

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F2 - Processing parameters
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SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure 8 ¹H NMR spectrum (300 MHz, CDCl₃) of compound, 5a.



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PROCNO 1

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F2 - Processing parameters
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Figure 9 ¹³C NMR spectrum (75 MHz, CDCl₃) of compound, 5a.



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PROCNO	1

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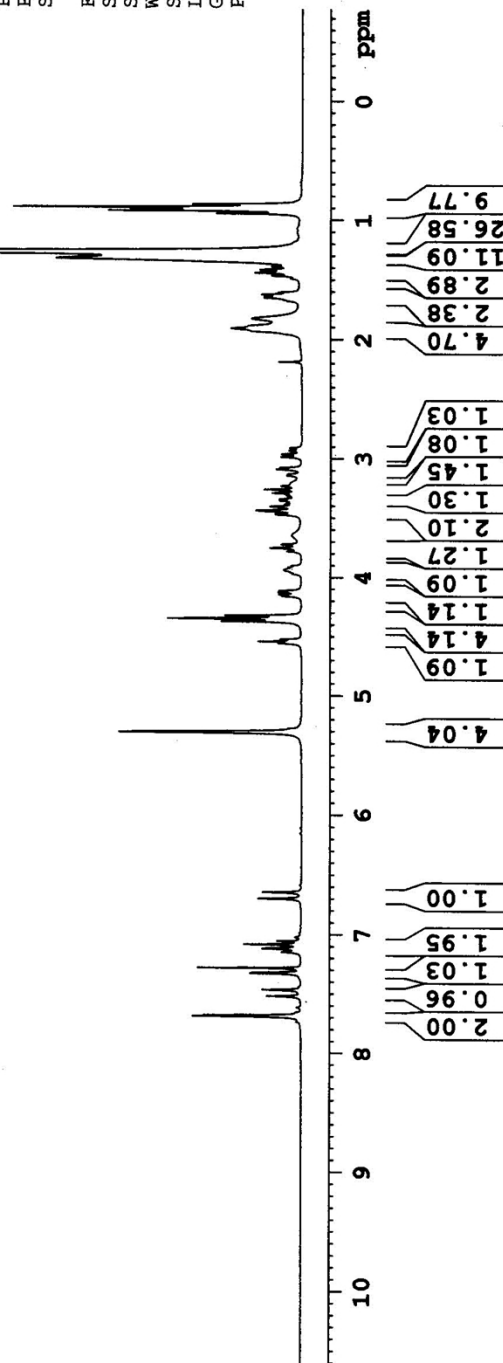
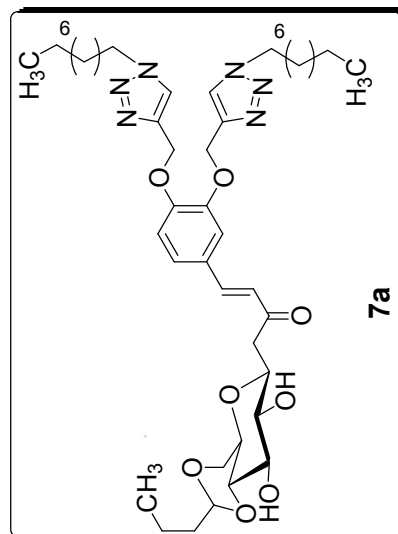
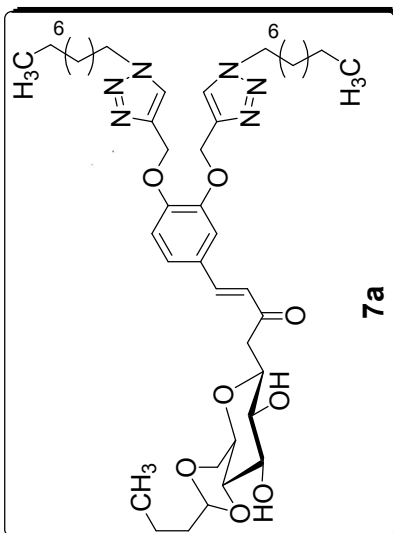
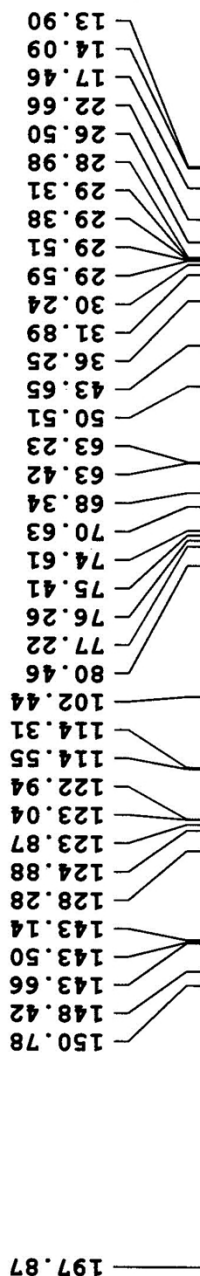


Figure 10 ^1H NMR spectrum (300 MHz, CDCl_3) of compound, 7a.



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 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 812.7
 DW 27.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
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F2 - Processing parameters
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 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
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Figure 11 ¹³C NMR spectrum (75 MHz, CDCl₃) of compound, 7a.



Current Data Parameters
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 PROCNO 1

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 Time_ 7.32

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 SF01 75.4752953 MHz

===== CHANNEL f2 =====
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 PL12 15.68 dB
 SF02 300.1312005 MHz

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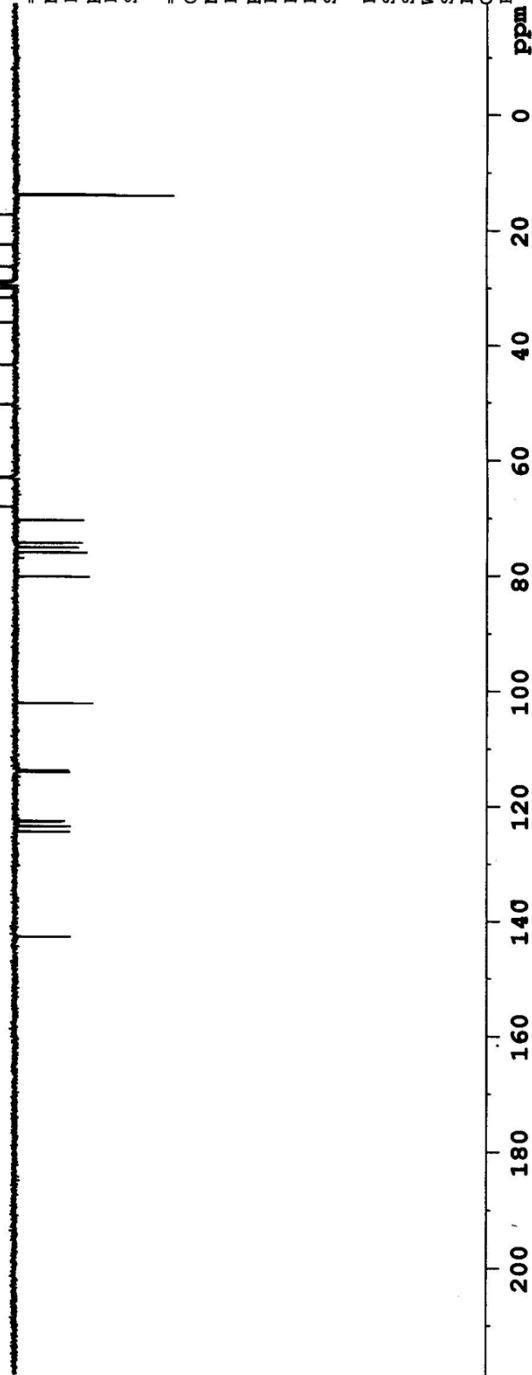
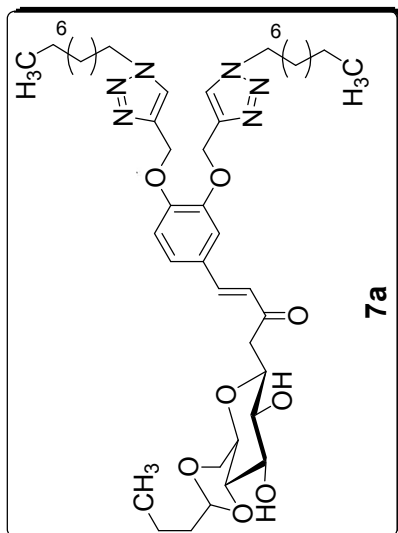


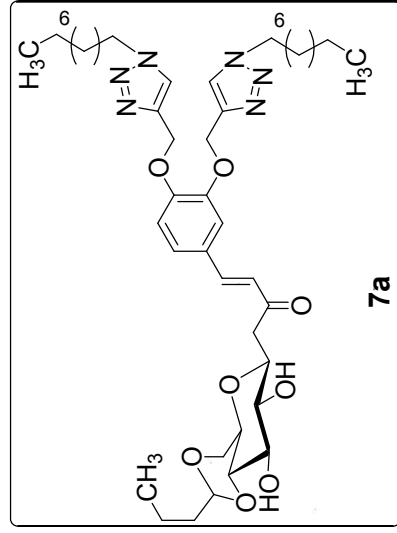
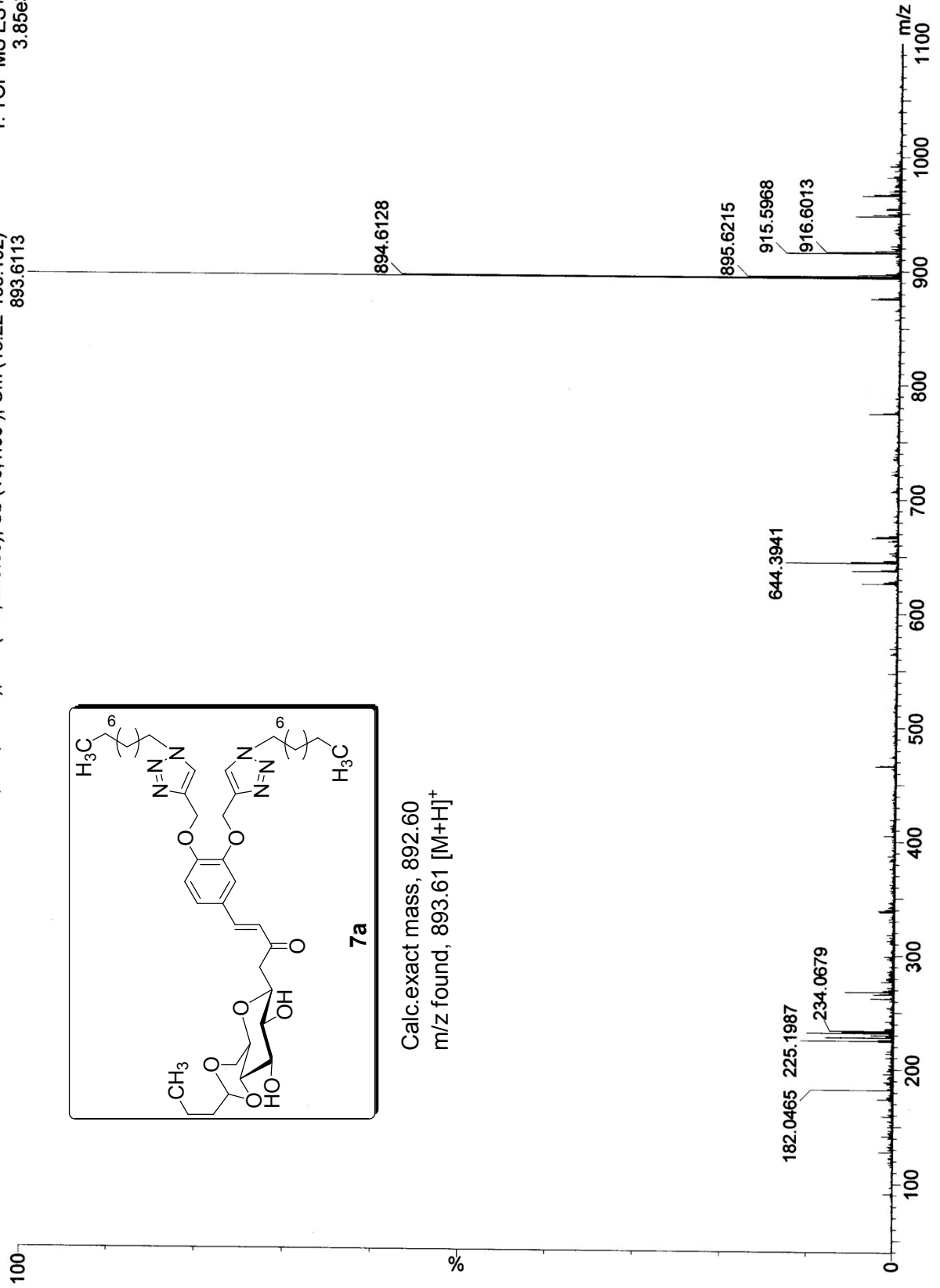
Figure 12 DEPT-135 spectrum (75 MHz, CDCl₃) of compound, 7a.

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1: TOF MS ES+
3.85e3

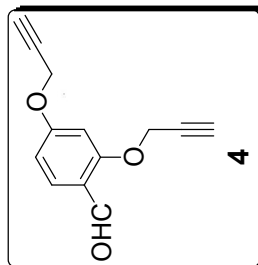
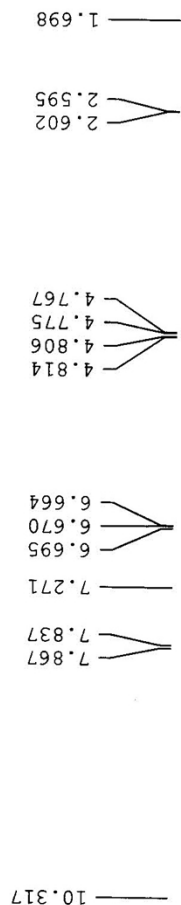
WATERS-Q-ToF Premier-HAB213

12:02:5326-Jul-2013



Calc. exact mass, 892.60
m/z found, 893.61 [M+H]⁺

Figure 14 Mass spectrum of compound, 7a.



Current Data Parameters
NAME TMDH-133
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110609
Time_ 9.16
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 181
DE 81.000 usec
TE 6.00 usec
D1 300.0 K
D1 1.00000000 sec
TD0 1

CHANNEL f1
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SF01 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300031 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

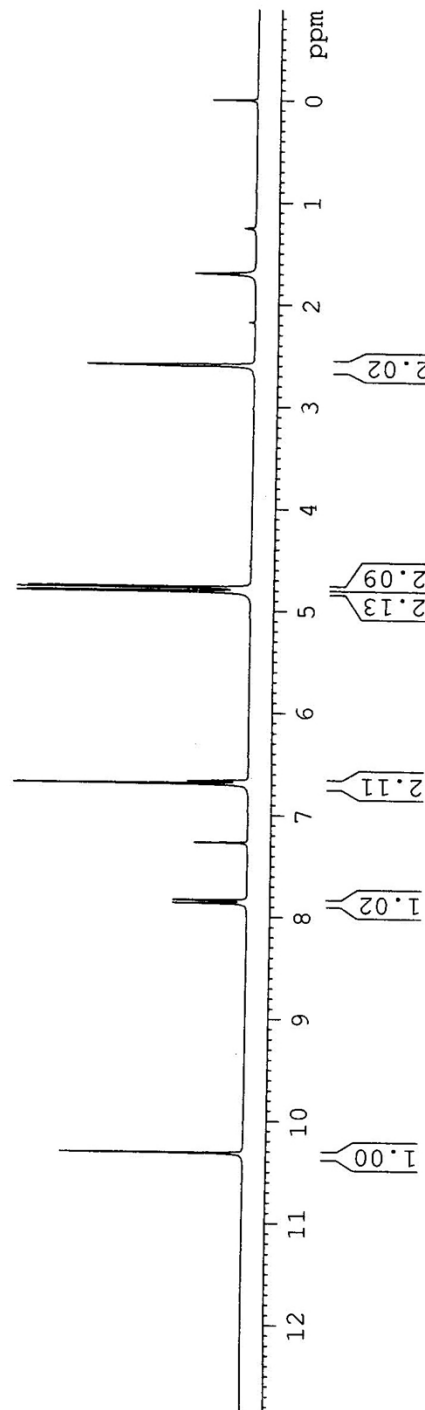
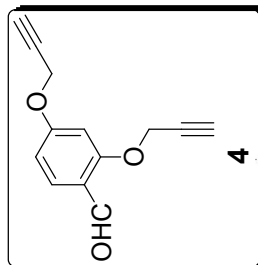


Figure 15 ¹H NMR spectrum (300 MHz, CDCl₃) of compound, 4.



188.09
163.64
161.37
130.57
120.11
107.50
100.42
77.46
77.03
76.69
76.46
56.45
56.10



Current Data Parameters
NAME TMDH-133
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110609
Time_ 9.24
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 810
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 512
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 9.30 usec
PL1 0.00 dB
SFO1 75.4752953 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 15.68 dB
PL13 16.00 dB
SFO2 300.1312005 MHz

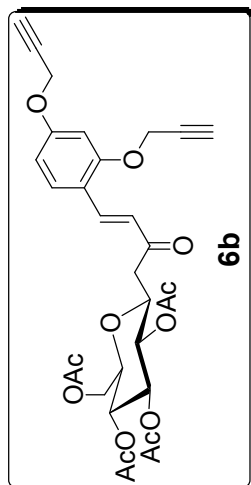
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

200 180 160 140 120 100 80 60 40 20 0 ppm

Figure 16 ¹³C NMR spectrum (75 MHz, CDCl₃) of compound, 4.



7.847
7.793
7.532
7.503
6.745
6.691
6.663
6.634
5.259
5.228
5.197
5.110
5.078
5.045
5.024
4.991
4.959
4.787
4.743
4.737
4.294
4.278
4.253
4.237
4.157
4.134
4.110
4.086
4.037
3.997
3.741
3.731
3.708
3.699
3.663
3.035
3.009
2.981
2.699
2.689
2.645
2.635
2.582
2.048



Current Data Parameters
NAME TMDH-139
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110621
Time 11.47
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 101.6
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.15 usec
PL1 0.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300007 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

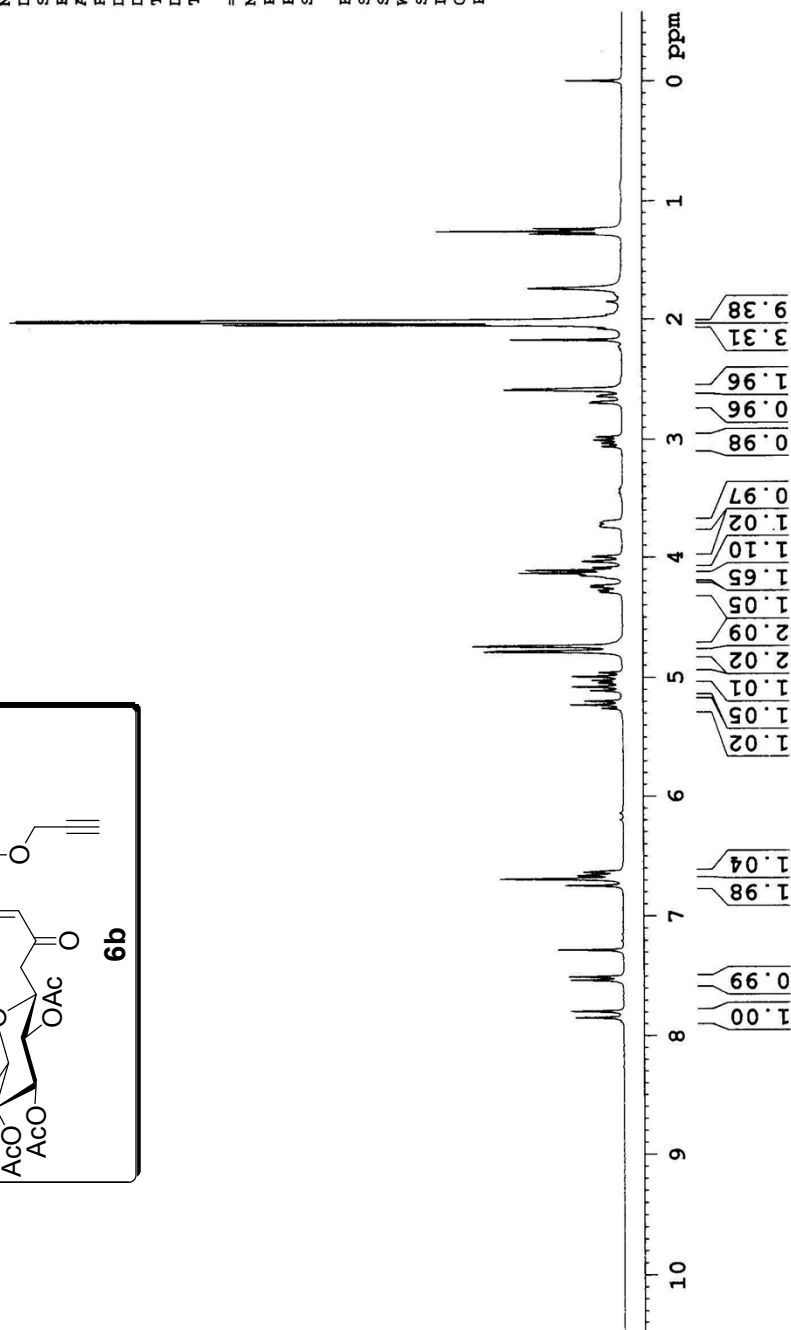
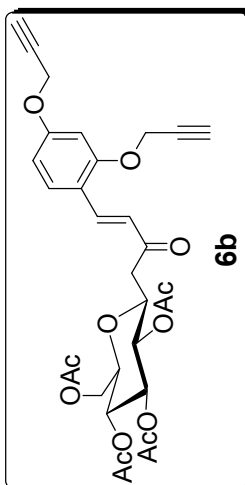
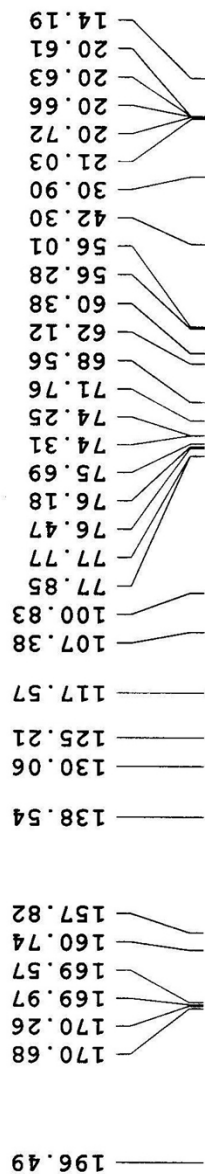


Figure 17 ¹H NMR spectrum (300 MHz, CDCl₃) of compound, 6b.



Current Data Parameters
 NAME TMDH-139
 EXPNO 2
 PROCNO 1

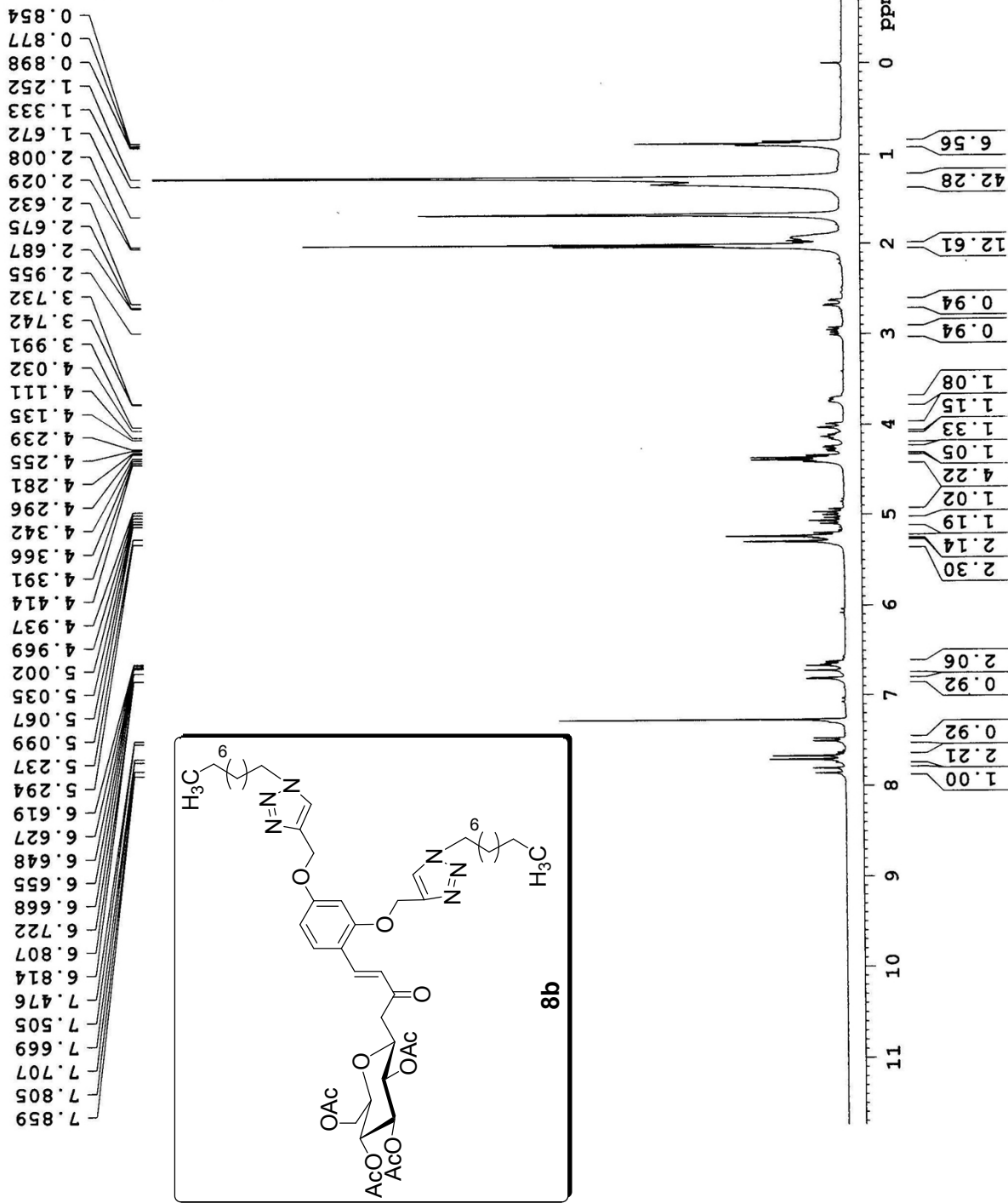
F2 - Acquisition Parameters
 Date_ 20110621
 Time_ 11.56
 INSTRUM spect
 PROBD 5 mm DUL 13C-1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 880
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 812.7
 DW 27.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TDO 1

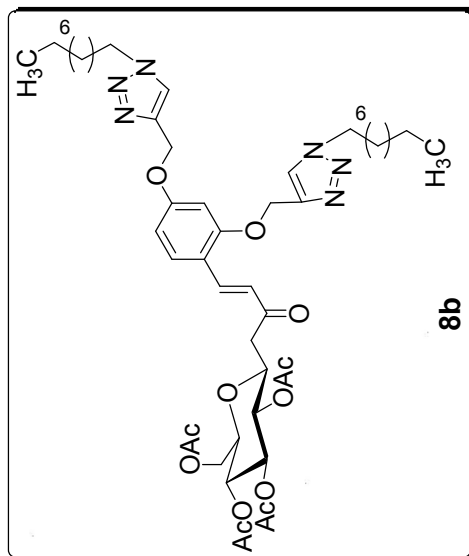
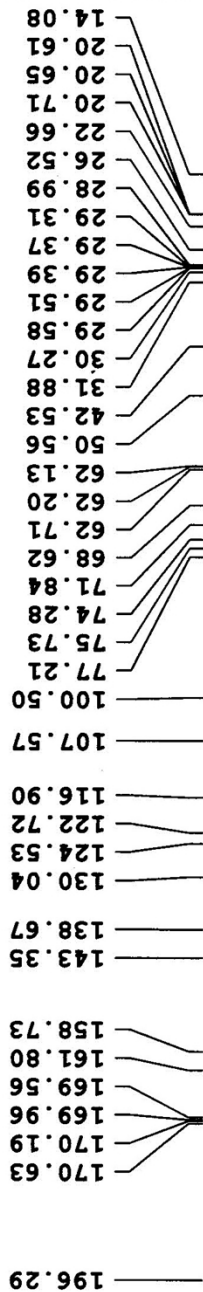
===== CHANNEL f1 =====
 NUC1 13C
 P1 9.30 usec
 PL1 0.00 dB
 SFO1 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P2 80.00 usec
 PL2 0.00 dB
 PL12 15.68 dB
 PL13 16.00 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677490 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure 18 ¹³C NMR spectrum (75 MHz, CDCl₃) of compound, 6b.

Figure 19 ¹H NMR spectrum (300 MHz, CDCl₃) of compound, 8b.



Current Data Parameters
 NAME TMDH-DDT
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120313
 Time_ 6.18
 INSTRUM spect
 PROBHD 5 mm DUL 13C-1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 6660
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 32768
 DW 27.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.30 usec
 PL1 0.00 dB
 SFO1 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 15.68 dB
 PL13 16.00 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677490 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure 20 ¹³C NMR spectrum (75 MHz, CDCl₃) of compound, 8b.



Current Data Parameters
 NAME TMDH-DDT-DEPT
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20120406
 Time 9.59
 INSTRUM spect
 PROBD 5 mm DUL 13C-1
 PULPROG dept135
 TD 65536
 SOLVENT CDC13
 NS 2000
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 16384
 DW 27.800 usec
 DE 6.00 usec
 TE 300.0 K
 CNST2 145.0000000
 D1 2.0000000 sec
 d2 0.00344828 sec
 d12 0.00002000 sec
 DELTA 0.00001184 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.30 usec
 P2 18.60 usec
 PL1 0.00 dB
 SFO1 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P3 13.15 usec
 P4 26.30 usec
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 15.68 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677490 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

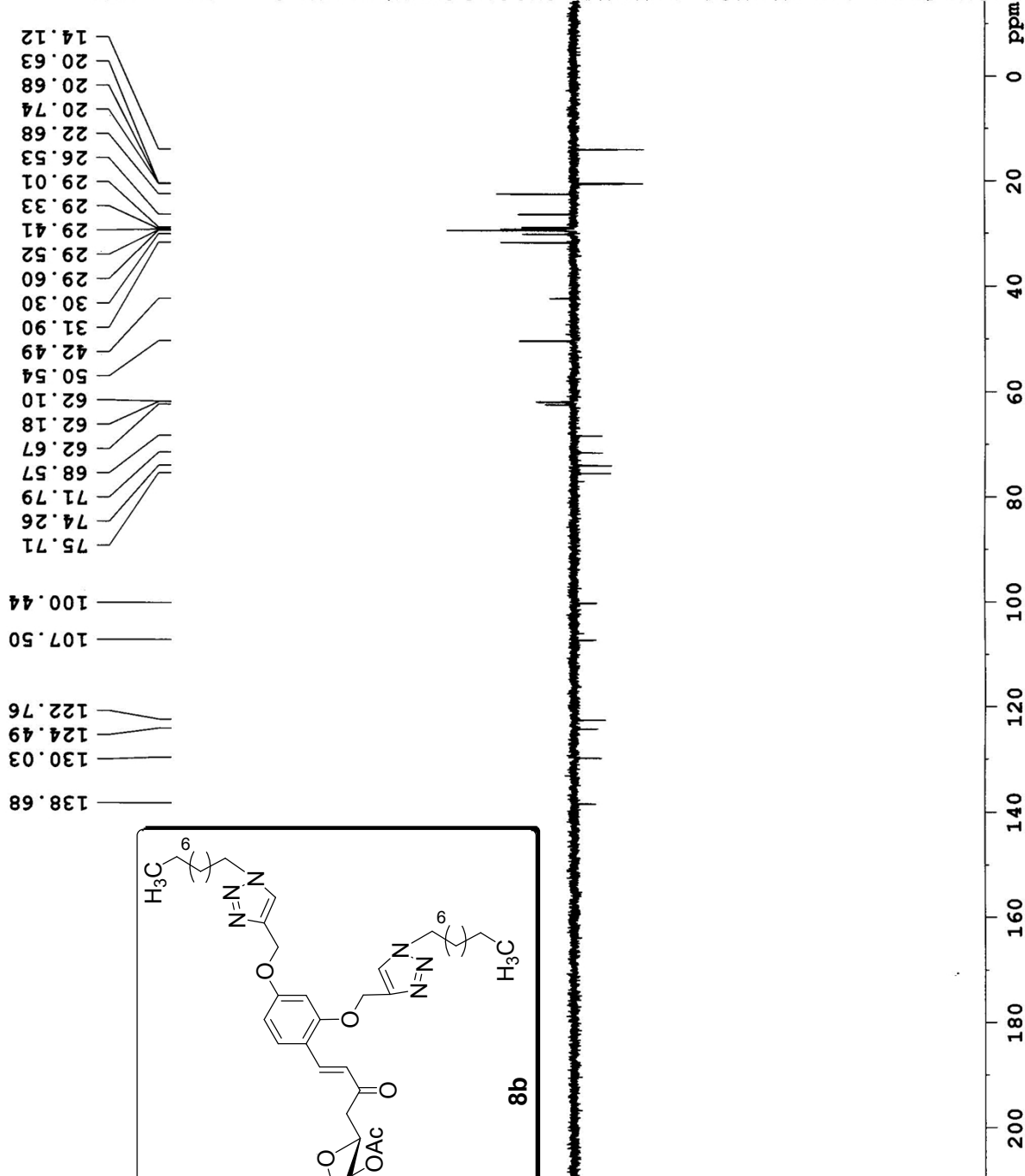


Figure 21 DEPT-135 spectrum (75 MHz, CDCl₃) of compound, 8b.

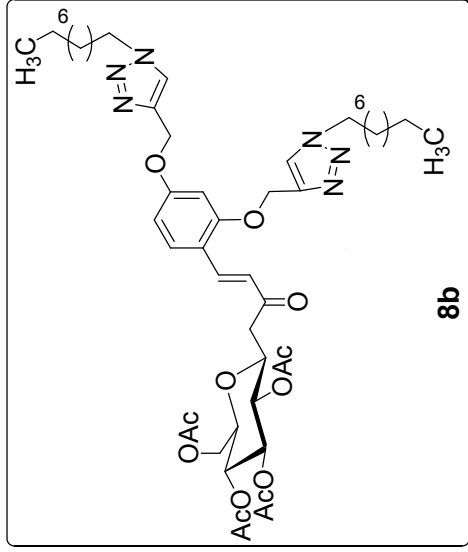
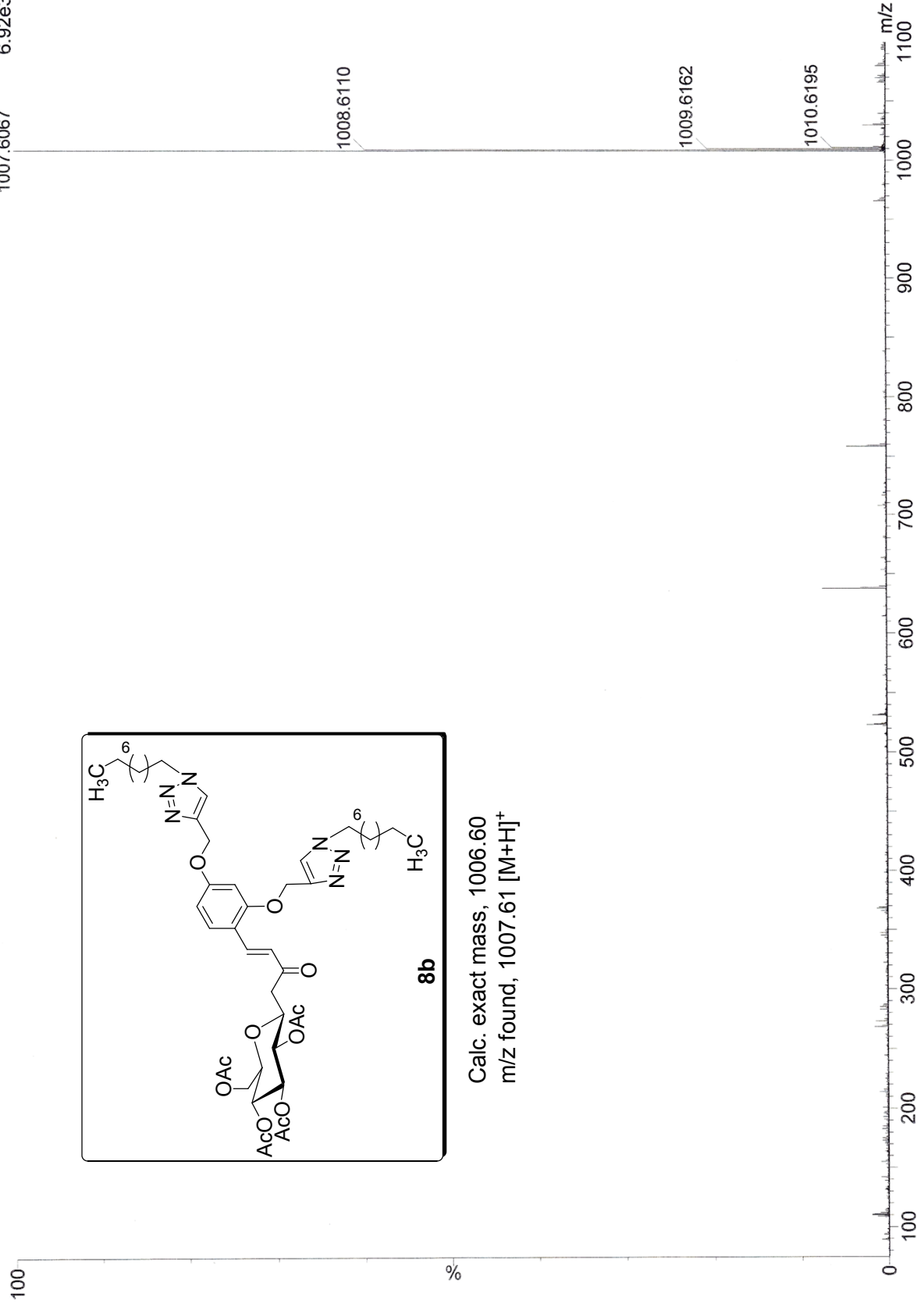
Electrospray ionisation-MS

402A 15 (0.357) AM (Cen,4, 100.00, Ar,8500.0,556.28,0.31,LS 10); Sm (SG, 2x5.00); Sb (10,1.00); Cm (15:19-48:54)

WATERS-Q-ToF Premier-HAB213

12:20:4127-Sep-2013

1: TOF MS ES+
6.92e3



Calc. exact mass, 1006.60
m/z found, 1007.61 [M+H]⁺

Figure 22 Mass spectrum of compound, 8b.

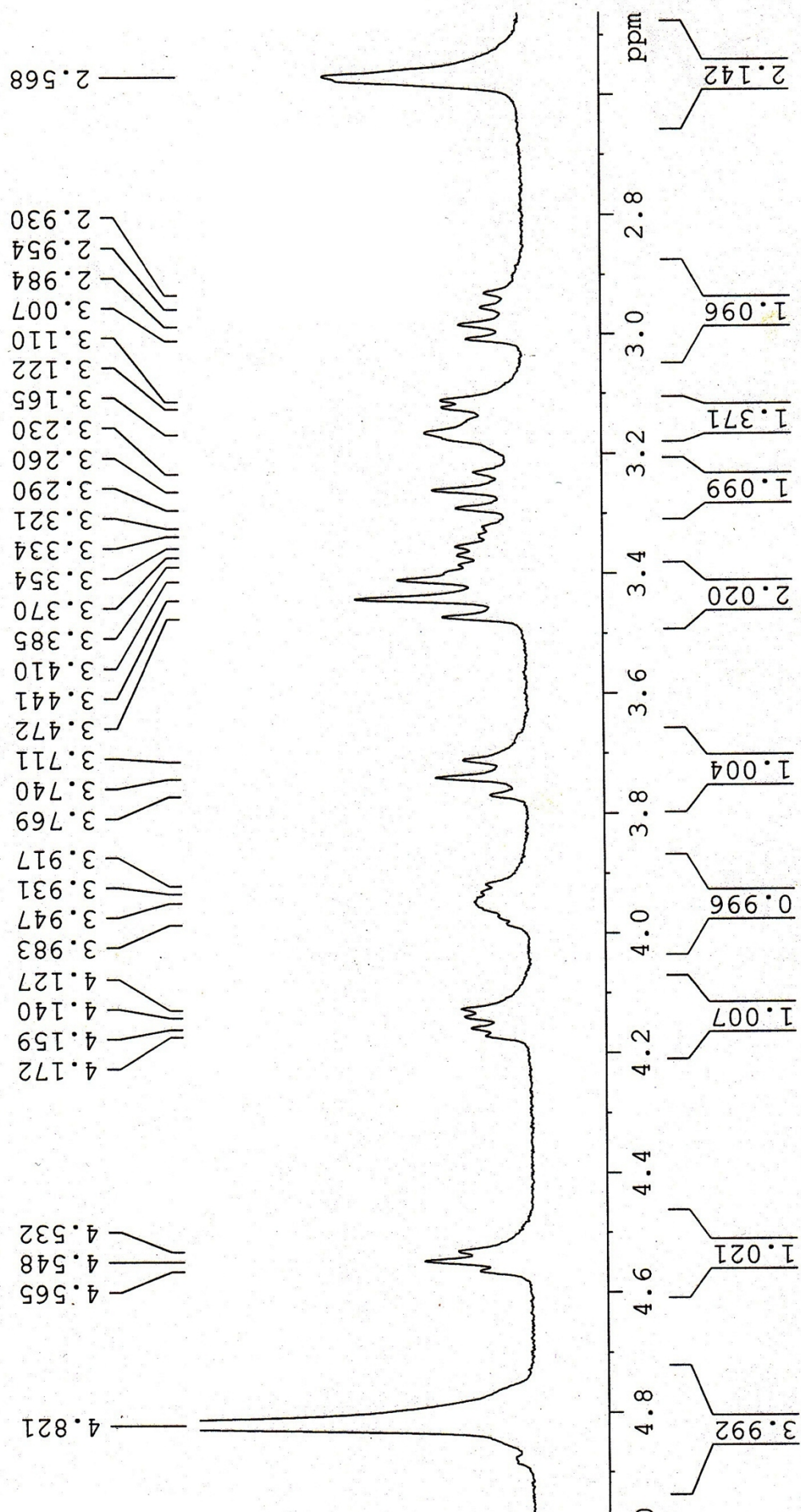


Figure 23 ¹H NMR spectrum (300 MHz, CDCl₃) expansion of compound, 5a.

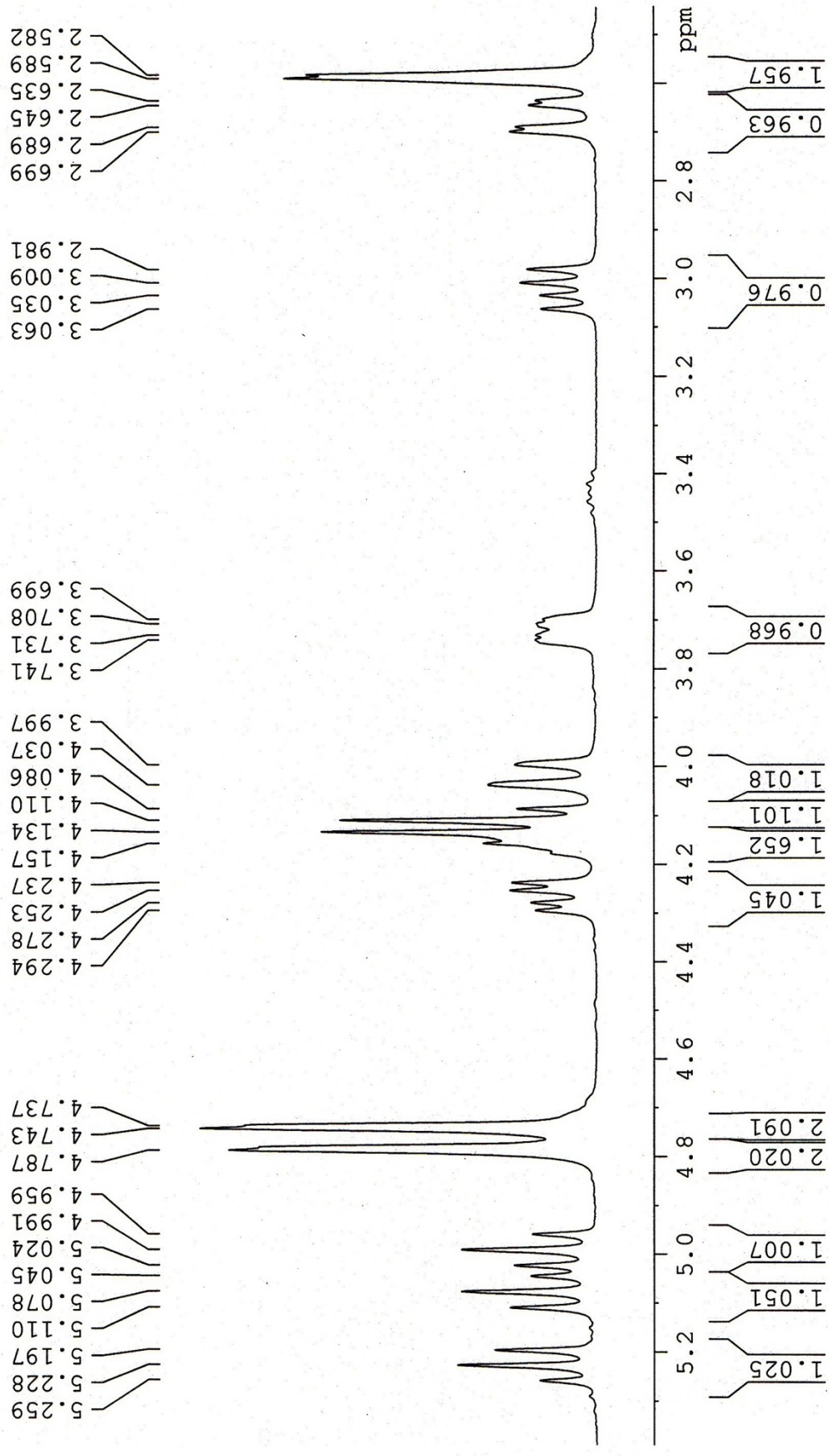


Figure 24 ¹H NMR spectrum (300 MHz, CDCl₃) expansion of compound, 6b.

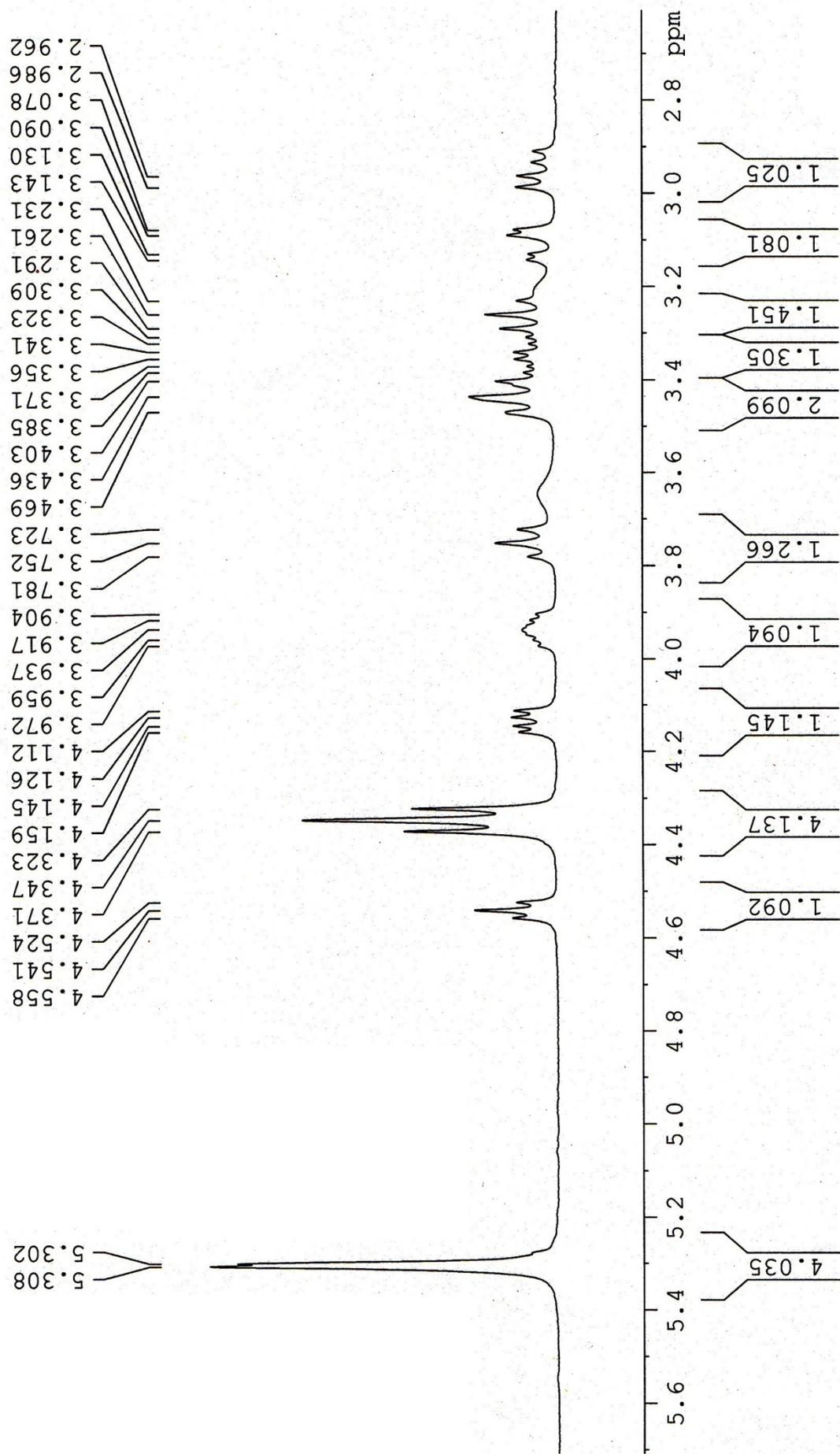


Figure 25 ^1H NMR spectrum (300 MHz, CDCl_3) expansion of compound, 6b.

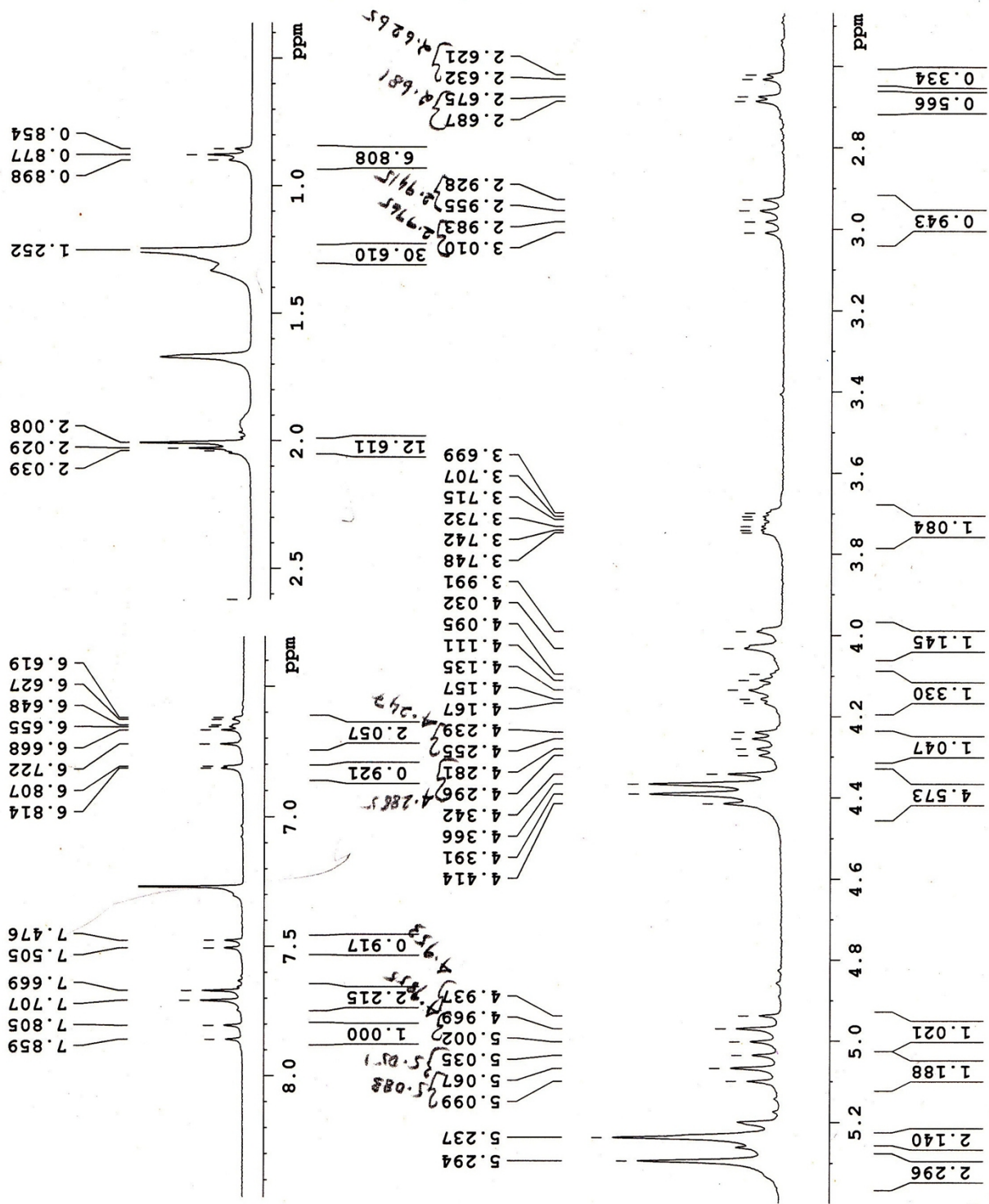


Figure 26 ^1H NMR spectrum (300 MHz, CDCl_3) expansion of compound, 8b.