Supplemental data for:

Positive- and Negative-Tandem Mass Spectrometric Fingerprints of Lipids from the Halophilic Archaea *Haloarcula marismortui*

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Running title: Mass Spectrometric Fingerprints of Lipids from *Haloarcula marismortui*

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Figure Legends

Figure S-1 – (**A**) ^{13}C-NMR spectrum of total polar lipid fraction from *H. marismortui*. Carbohydrates appear as their anomeric region at δ 97 – 104. Signals of the phytanyl chain were present at δ 20.3 and δ 23.1 (CH_3), and tertiary carbons (CH) between δ 28.78 and 33.64. Small signals arising from double bonds are present between δ 120 – 140, and CH_3 groups from the unsaturated chain are between δ 15 – 17. (**B**) ^{13}C-DEPT spectrum showing the chemical shifts from CH_2, occurring between δ 25.2 – 25.6 and δ 37.6 – 40.2, with CH_2-O between δ 68.1-71.0.

Figure S-2 – ^{1}H-NMR spectrum of total polar lipid fraction from *H. marismortui*. The amplified region between δ 5.0 and 5.5 indicates the presence of olefinic protons of double bonds.

Figure S-3 – (**A**) Unsaturated archaeol structure showing connectivities of CH_3 groups and olefinic protons, as detected by HMQC and COSY. (**B**) ^{1}H,^{13}C HMQC spectrum showing, in amplified squares, the connectivities of ^{1}H/^{13}C of methylic side-chain carbons in the unsaturated phytanyl chain at δ 1.57/16.45 (H/C 18/18' and 19,19') and 1.65/16.9 (H/C 17,17'), as well as those of olefinic protons with carbons in double bonds, at δ 5.31/121.6 (H/C 2,2'), 5.08/124.9 (H/C 6,6') and 5.10/126.0 (H/C-10,10'). (**C**) Partial ^{1}H/^{1}H COSY spectrum of total polar lipid fraction, showing connectivities between methylic and olefinic protons at δ 1.57/5.08 (H6,6'/H18,18' and H10,10'/H19,19') and 1.65/5.31 (H2,2'/H17,17')..

Figure S-4 – (**A**) ^{13}C and (**B**) ^{13}C-DEPT NMR spectra of purified triglycosyl archaeol. The anomeric region from carbohydrates is consistent with the presence of units of β-Glc*p* at δ 104.2 ($J_{C\text{-}1/H\text{-}1}$ = 158.5 Hz), α-Glc*p* at δ 99.3 ($J_{C\text{-}1/H\text{-}1}$ = 168.8 Hz) and α-Man*p* at δ 97.4 ($J_{C\text{-}1/H\text{-}1}$ = 168.1 Hz). Signals of the phytanyl chain are distributed between δ 20 and 71, as those in Figure S-2.

Figure S-1

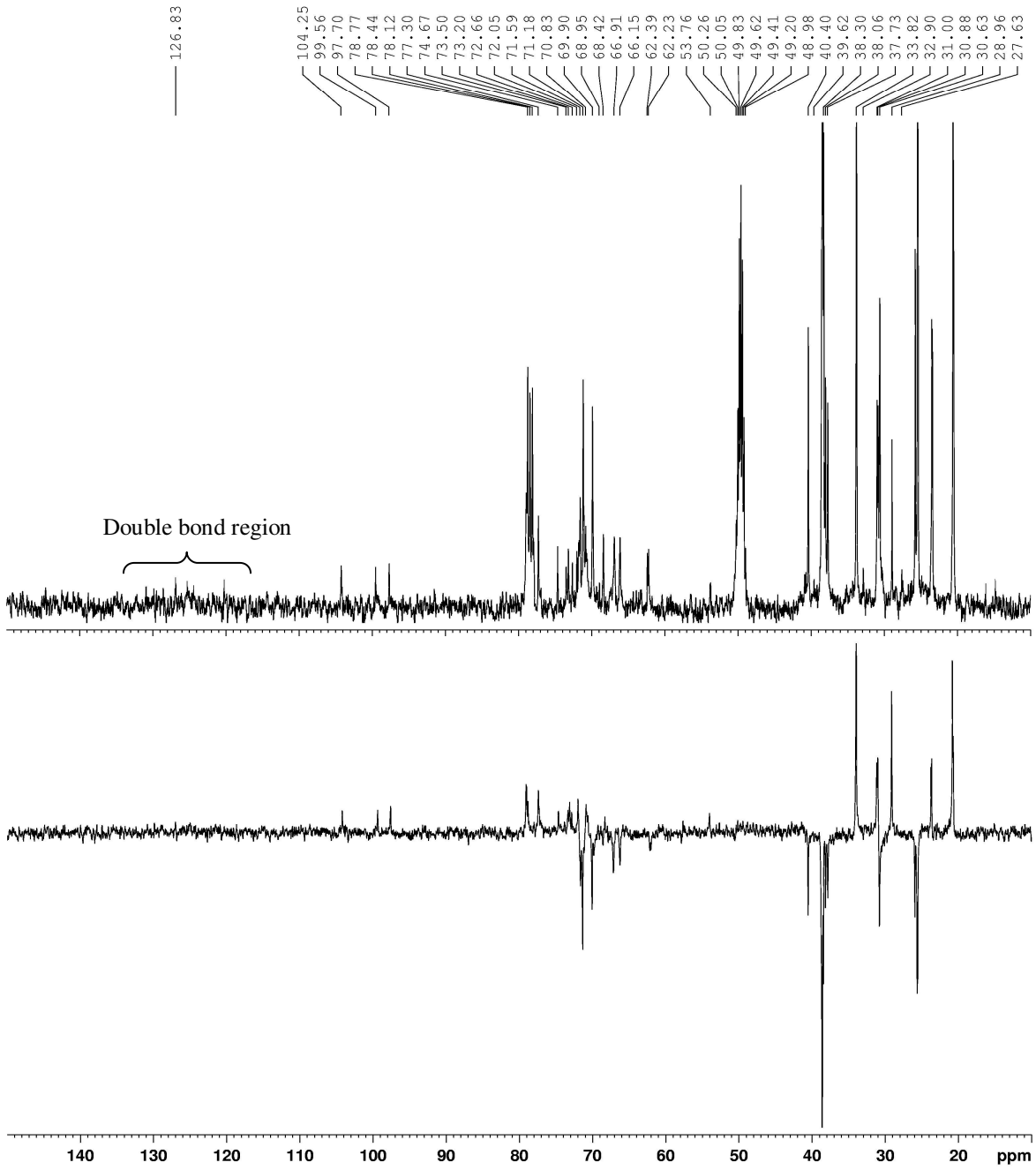

126.83
104.25
99.56
97.70
78.77
78.44
78.12
77.30
74.67
73.50
73.20
72.66
72.05
71.59
71.18
70.83
69.90
68.95
68.42
66.91
66.15
62.39
62.23
53.76
50.26
50.05
49.83
49.62
49.41
49.20
48.98
40.40
39.62
38.30
38.06
37.73
33.82
32.90
31.00
30.88
30.63
28.96
27.63
Double bond region
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

Figure S-2

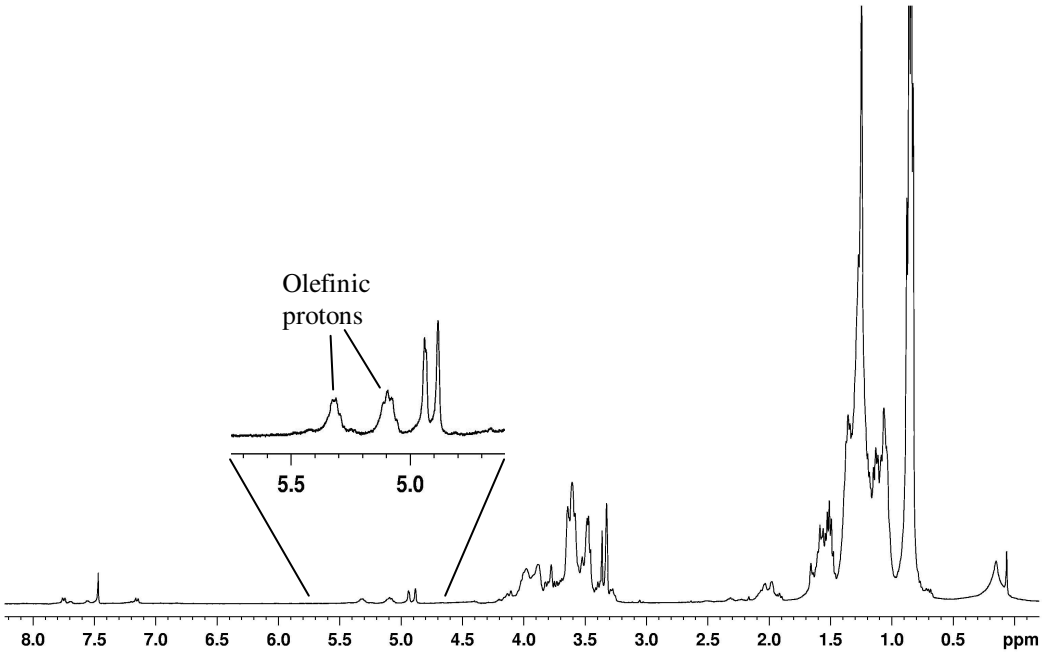

Olefinic
protons
5.5
5.0
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

Figure S-3

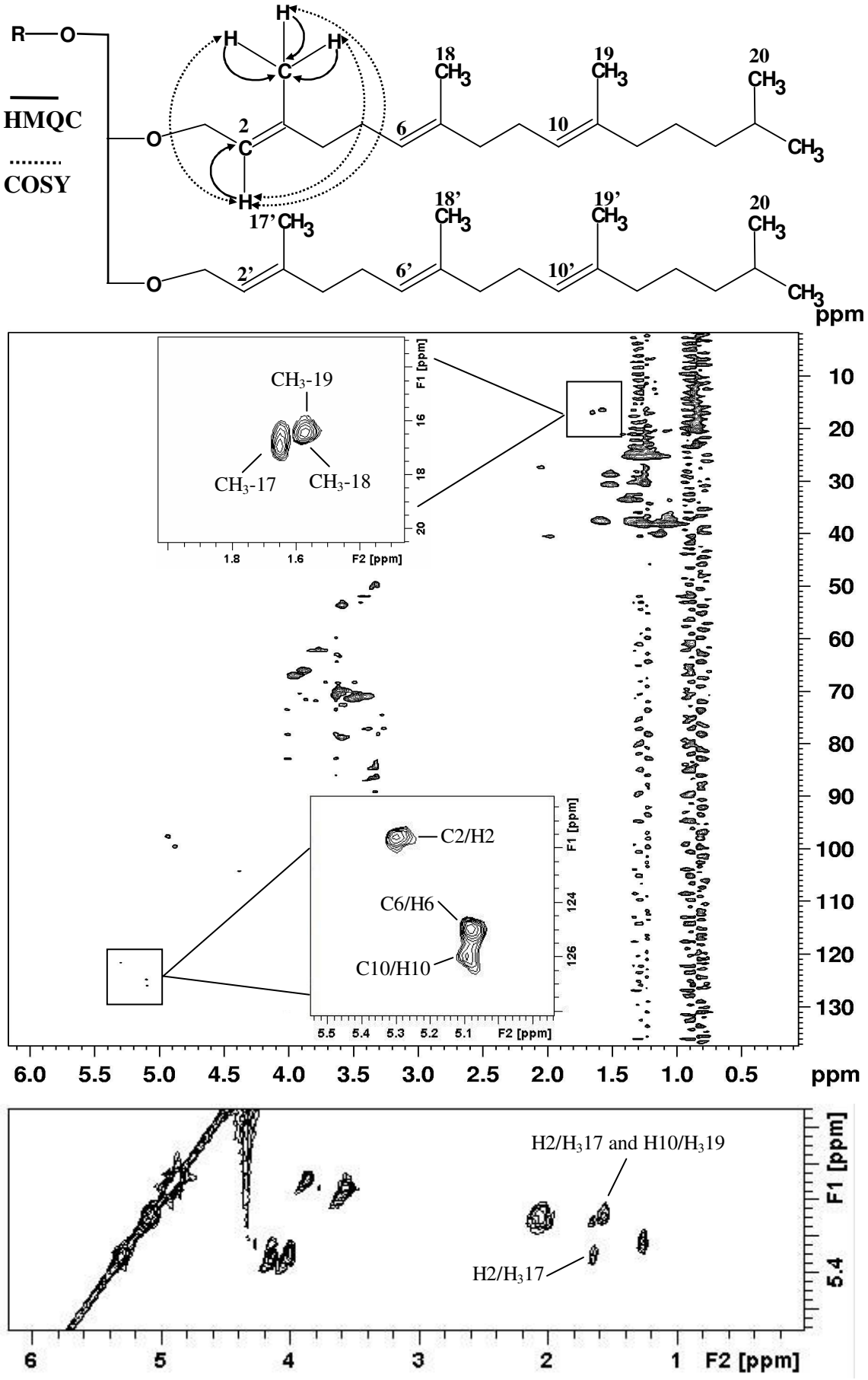
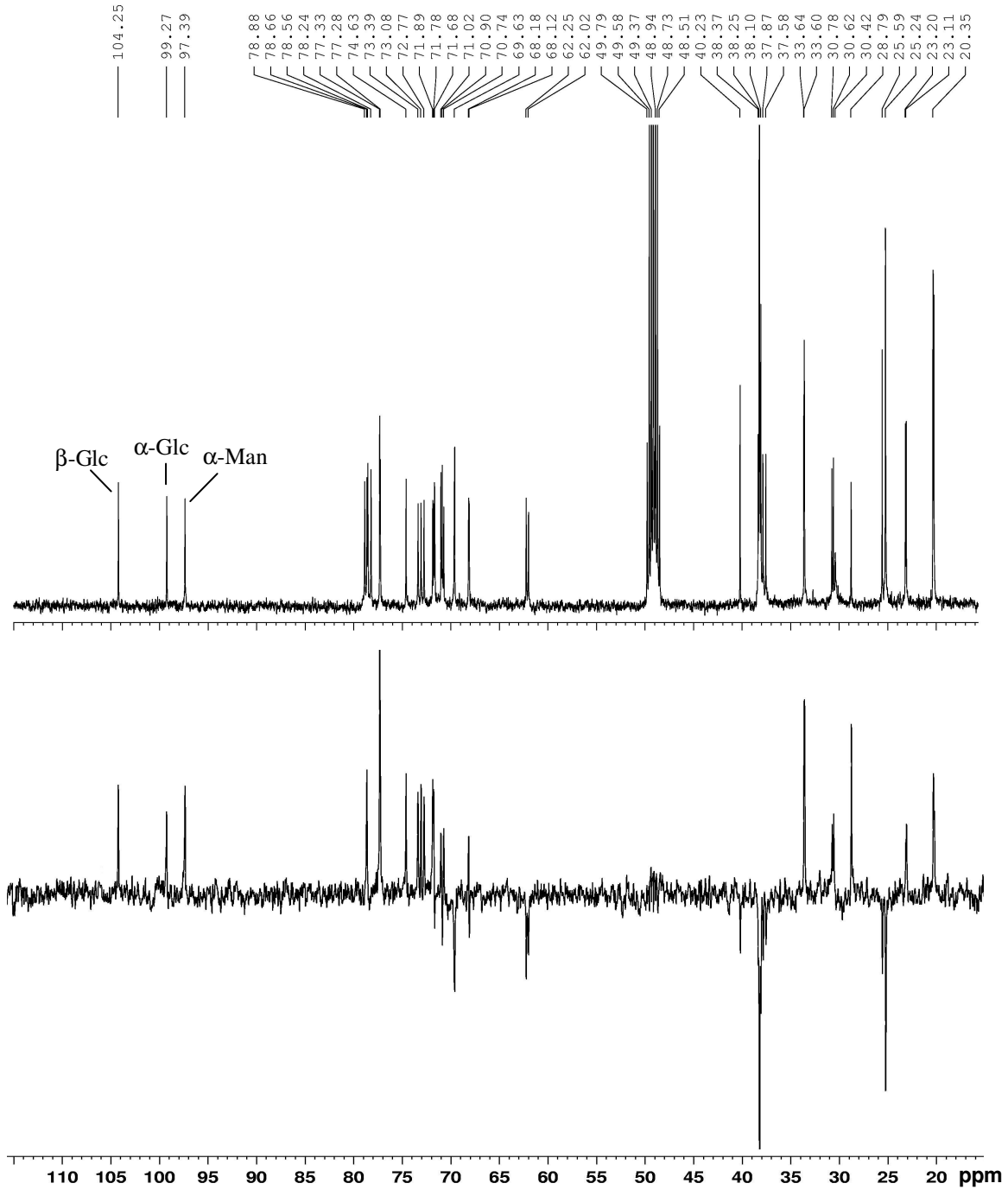

HMQC
COSY
CH3-19
CH3-17
CH3-18
F1 [ppm]
F2 [ppm]
C2/H2
C6/H6
C10/H10
ppm
H2/H317 and H10/H319
H2/H317

Figure S-4


104.25
99.27
97.39
78.88
78.66
78.56
78.24
77.33
77.28
74.63
73.39
73.08
72.77
71.89
71.78
71.68
71.02
70.90
70.74
69.63
68.18
68.12
62.25
62.02
49.79
49.58
49.37
48.94
48.73
48.51
40.23
38.37
38.25
38.10
37.87
37.58
33.64
33.60
30.78
30.62
30.42
28.79
25.59
25.24
23.20
23.11
20.35
β-Glc
α-Glc
α-Man
110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 ppm