NMR SPECTROSCOPY AND TANDEM MASS SPECTROMETRIC FINGERPRINTS OF A TRIGLYCOSYL LIPID FROM THE HALOPHILIC ARCHAEA *Haloarcula marismortui*

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Lipids from the extremely halophilic Archaea, *Haloarcula marismortui*, contain abundant phytanyl diether lipids, including several phospholipids and a triglycosyl archaeol (TGA). In order to identify the TGA structure, NMR and tandem-mass spectrometry were employed to provide a fingerprint analysis. The positive ESI-MS analysis showed a main ion at *m/z* 1162 $[M+Na]^+$, and the tandem-MS showed the fragments at *m/z* 1000 $[M-162]^+$, 838 $[M-324]^+$ and 676 $[M-486]^+$, which represent successive removal of hexosyl units, and the fragments at *m/z* 509, 347 and 185 arising from tri-, di-, and monosaccharide units, were consistent with TGA structure. The core ether lipid was obtained by acid hydrolysis (1 M MeOH-HCl, 100 ºC, 14 h) and confirmed by ESI-MS/MS as 2,3-di-*O*-phytanyl-*sn*-glycerol (C20, C20), which gave rise to a precursor-ion at *m/z* 660 $[M+Li]^+$, and its fragment-ion at *m/z* 379 $[M+Li]^+$. The monosaccharides were identified by GC-MS, as glucose and mannose. The glycosidic linkages were achieved by methylation-GC-MS, which gave rise to 2,3,4,6-Me_4-Glc, 3,4,6-Me_3-Glc, and 2,3,4-Me_3-Man, in a 1:1:1 molar ratio. In the ^{13}C-NMR spectroscopy, signals from anomeric region of carbohydrates were found from β-Glc at δ104.25 ($J_{C\text{-}1/H\text{-}1}$ = 158.5 Hz), α-Glc at δ 99.27 ($J_{C\text{-}1/H\text{-}1}$ = 168.8 Hz), and α-Man at δ 97.39 ($J_{C\text{-}232\ 1/H\text{-}1}$ = 168.1 Hz). Other carbohydrate signals were distributed between δ 62.0 and 78.7. *O*-Linked glycosidic signals were at δ 78.66 and 68.18, consistent with *O*-substituted C-2 of Glc*p* and *O*-substituted C-6 of Man*p* units, respectively

Keywords: *Haloarcula marismortui*, glycolipid, fingerprint analysis.

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