Supporting Information

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A New Diketopiperazine from the Marine Sponge *Callyspongia* Species

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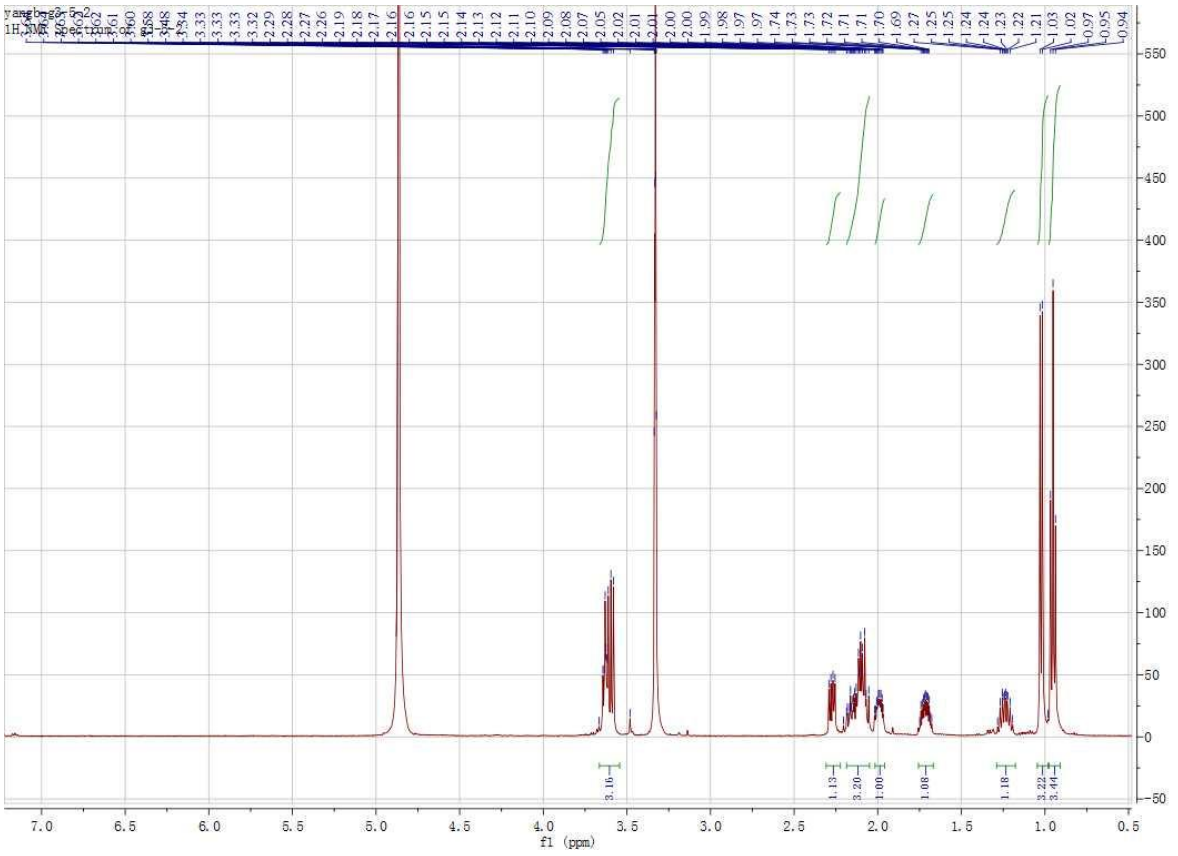
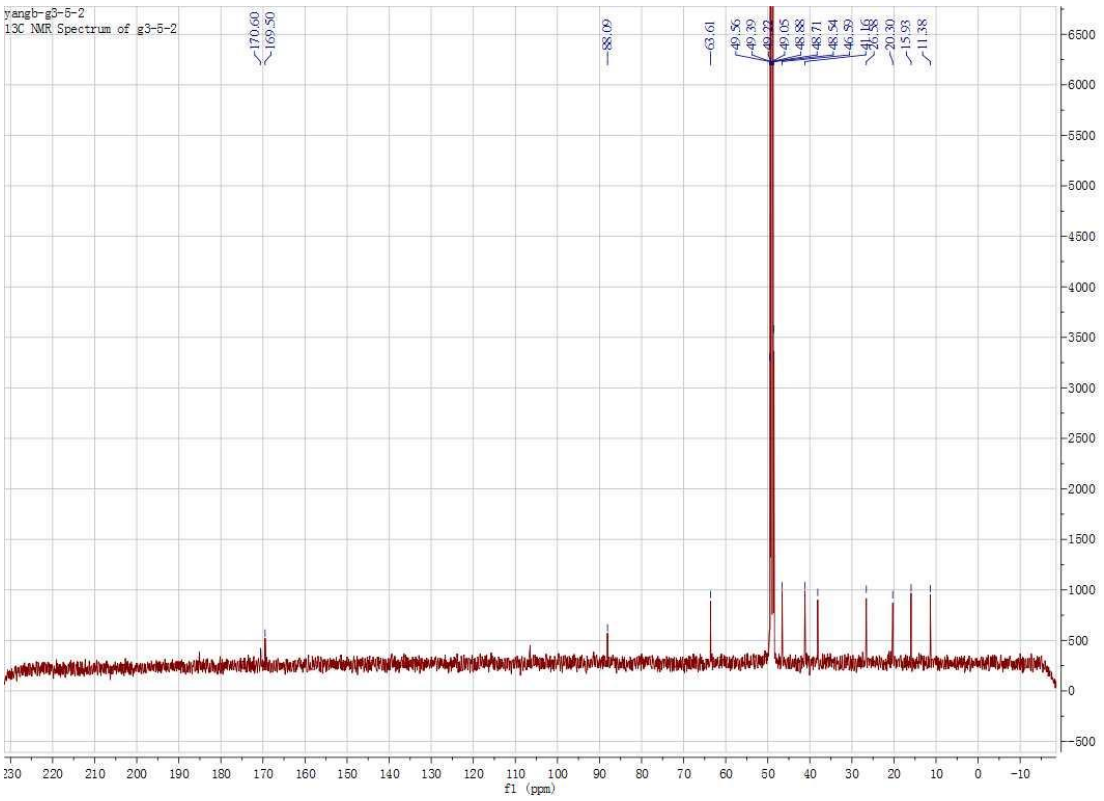
Extraction and Isolation

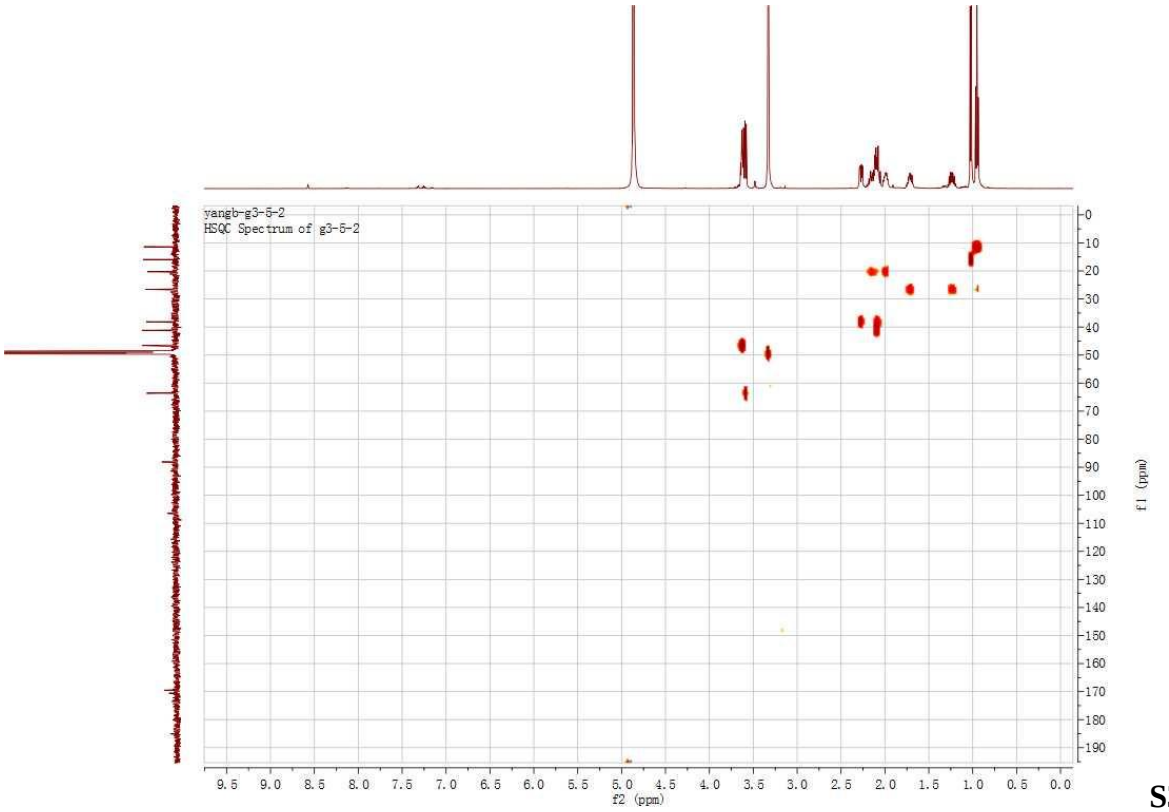
The 85% EtOH fraction was subjected to reversed-phase flash column chromatography, eluting with a solvent system of 40→100% MeOH, to afford 25 fractions. *Fr. 2* (2.2 g) was further separated by reverse phase flash column chromatography, eluting with 10→50% MeOH, to afford 12 subfractions (*Fr.g1–g12*). *Fr.g5* was separated by semi-preparative HPLC, eluting with MeOH-H_2O (12:88) at a flow rate of 2 ml min^{-1}, to provide compounds **1** (4.0 mg), **2** (5.2 mg). Fr.g7 were combined and was separated by semi-preparative HPLC, eluting with MeOH-H_2O (30:70) at a flow rate of 2 ml min^{-1}, to provide compounds **3** (5.2 mg) and **4** (7.6 mg). The *n*-BuOH layer was subjected to reversed-phase flash column chromatography, eluting with a solvent system of 0→50% MeOH, to afford 19 fractions (*Fr.n1–n19*). *Fr.n12* was further purified with silica gel column chromatography, eluting with PE-EtOAc (1: 2), to provide compounds **5** (2.2 mg) and **6** (3.7 mg). *Fr.n5* was subjected to Sephadex LH-20 (MeOH) to afford 5 subfractions (*Fr.n5-1–n5-5*). *Fr.n5-1* was further purified with silica gel column chromatography, eluting with MeOH-$CHCl_3$ (10:90), to provide compound **7** (2.2 mg). *Fr.n1* was further purified with semi-preparative HPLC, eluting with MeOH-H_2O (7:93) at a flow rate of 1.5 ml min^{-1}, to provide compound **8** (6.3 mg). *Fr.n2* was subjected to Sephadex LH-20 (MeOH) to afford 5 fractions (*Fr.n2-1–n2-5*). *Fr.n2-3* was further purified with semi-preparative HPLC eluting with MeOH-H_2O (22:78) at a flow rate of 1.5 ml min^{-1}, to provide compounds **9** (14.2 mg) and **10** (3.5 mg).



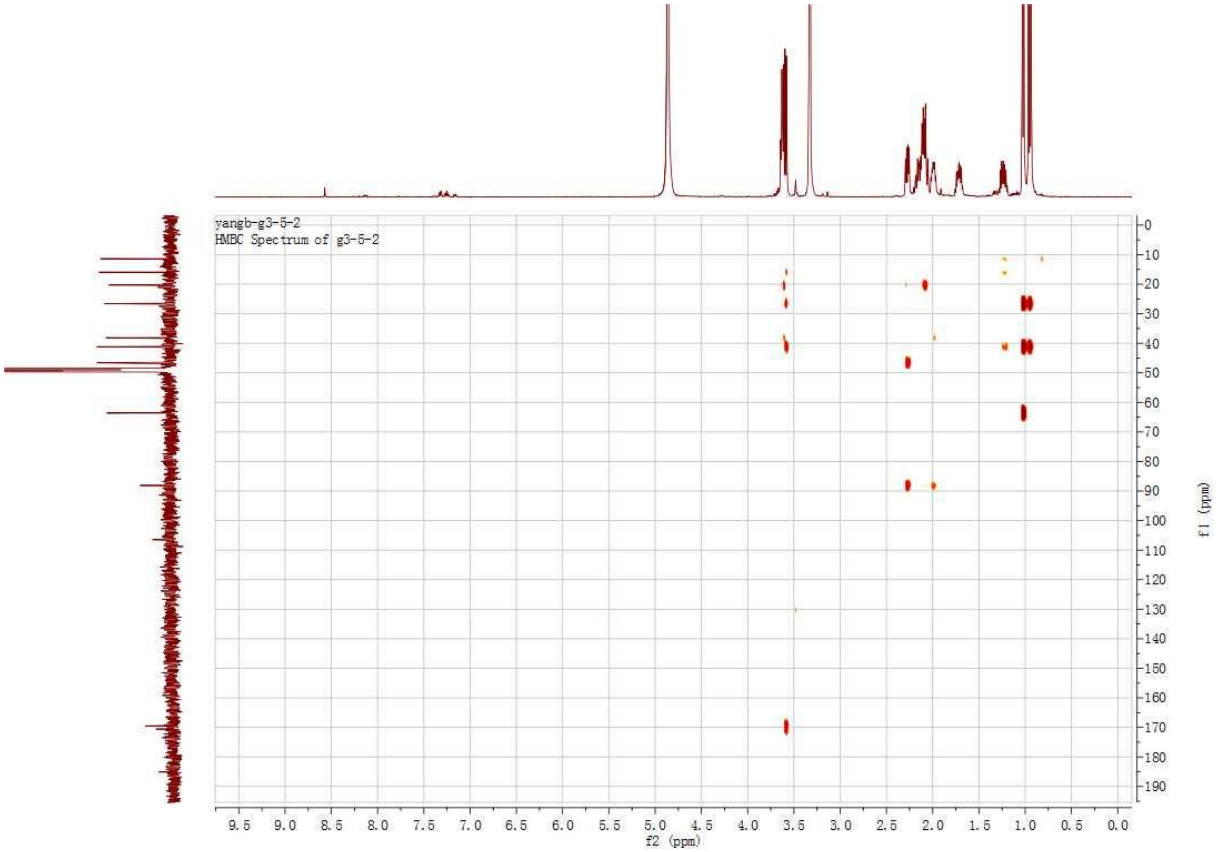
S1: ^{1}H-NMR (500 MHz, CD_3OD) Spectrum of Compound **1**



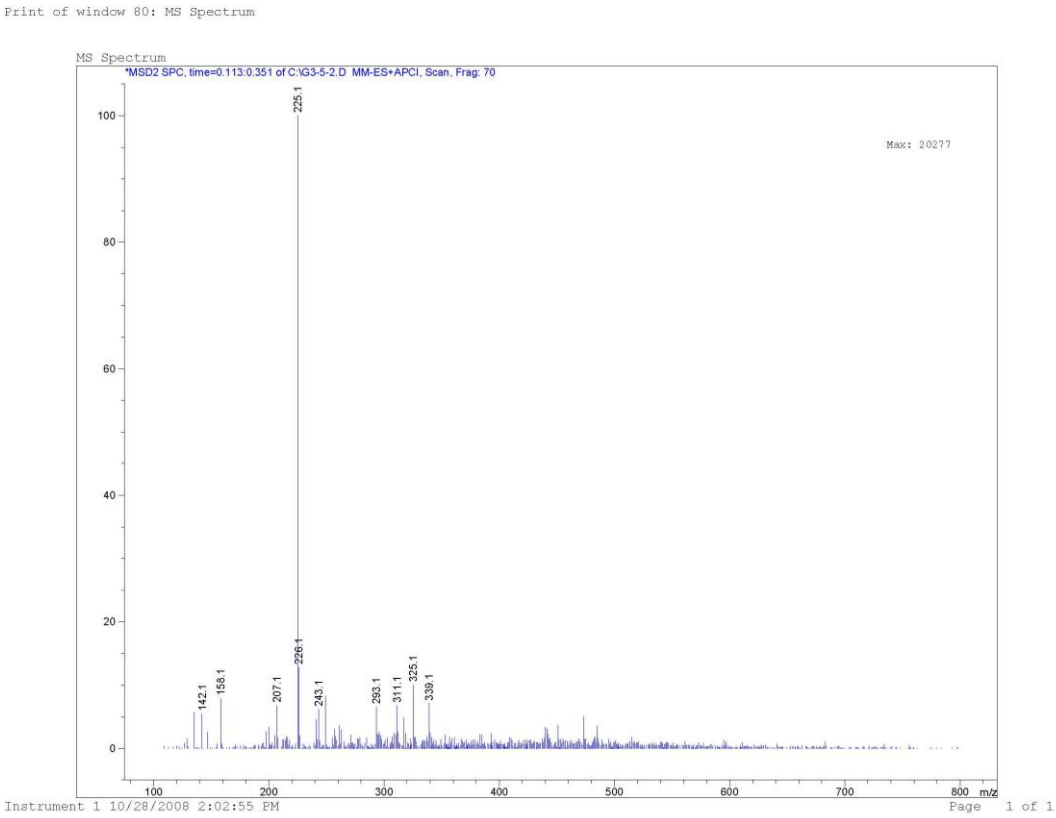
S2: ^{13}C-NMR (125 MHz, CD_3OD) Spectrum of Compound **1**



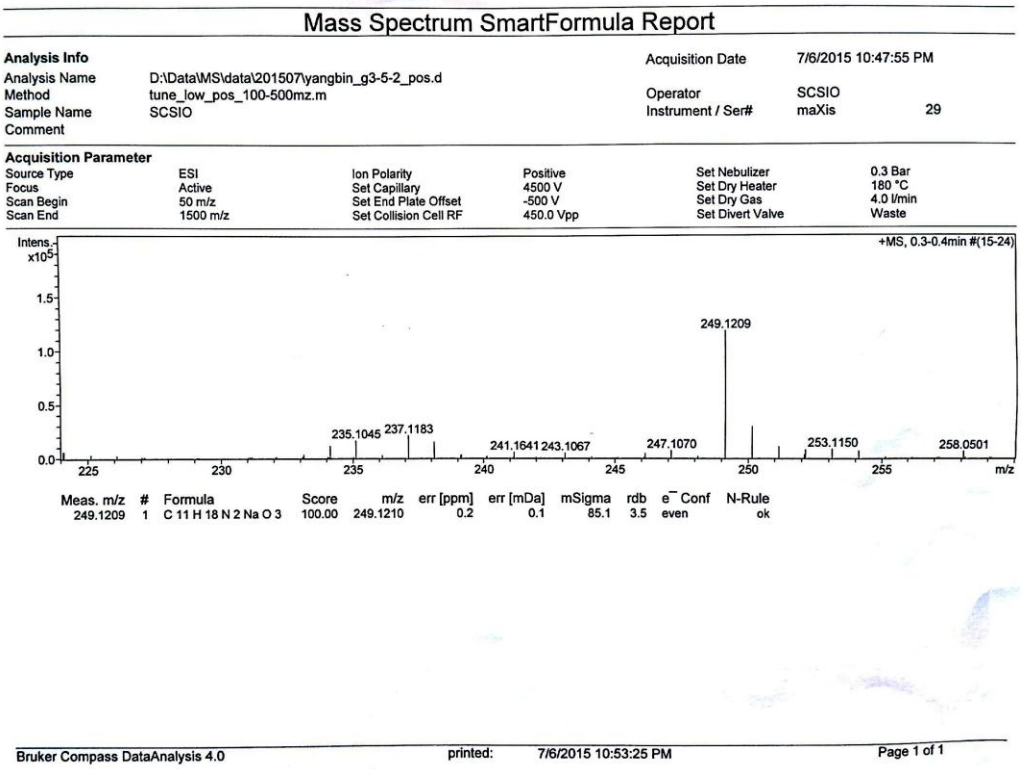
S3: **H**MQC (500 MHz) Spectrum of Compound **1**



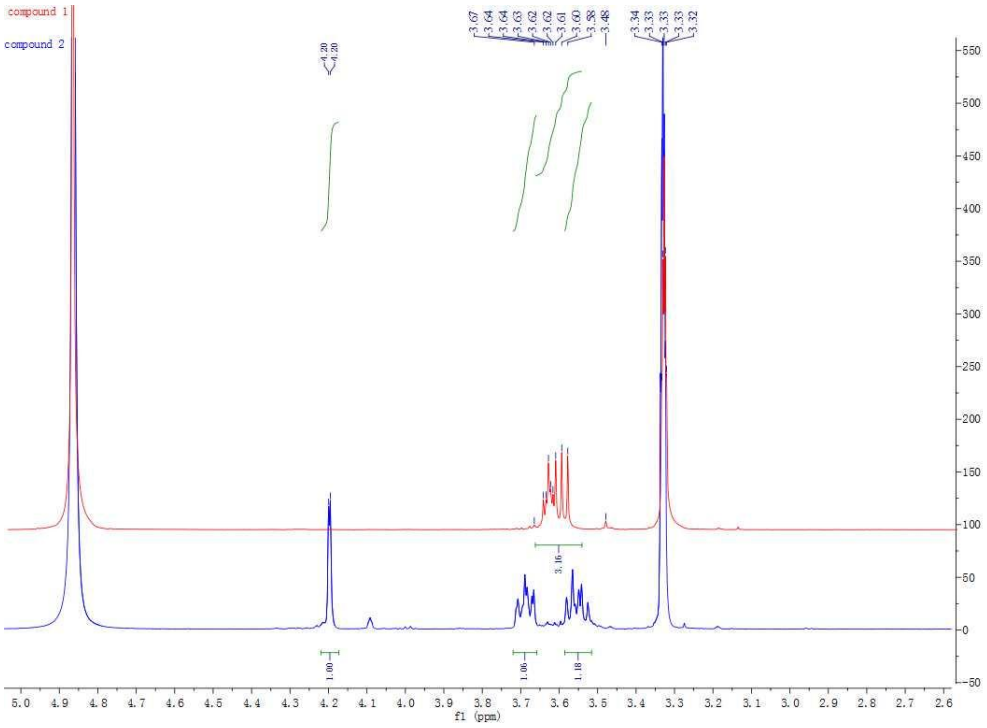
S4: HMBC (500 MHz) Spectrum of Compound **1**



S5: ESI-MS Spectrum of Compound **1**



S6: HR-ESI-MS Spectrum of Compound **1**



S7: ^{1}H-NMR Comparative Analysis of Compounds **1** and **2**