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The structure of hemicalide from the marine sponge *Hemimycale* sp.

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Isolation of hemicalide

The fresh sponge (5 kg) was lyophilized to give 650 g of a wax, which was macerated for 6 hours with chloroform and then twice with a 9/1 mixture of methanol and water. The filtrates were pooled and concentrated to an aqueous syrup, which was partitioned with dichloromethane and ethyl acetate. After evaporation of the organic solutions (42 g), the extract was solubilized in a 9/1 methanol/water mixture and partitioned with hexane. The "defatted" methanolic extract was chromatographed on a column of Sigel with mixtures of ethyl acetate and methanol of increasing polarities. All these steps were monitored by a bio-assay and by LC/MS. The activity was located in the more polar fractions, which were pooled and subjected first to a LH20 exclusion chromatography, then to RP18 HPLC with a gradient of water in acetonitrile. It was thus obtained 0.5 mg of hemicalide. HRMS: m/z 1061.6780, calc. for $[\text{M-H}]^-$ 1061.6782 in the negative ESI mode, corresponding to $\text{C}_{59}\text{H}_{98}\text{O}_{16}$ (calc. 1061.6782). UV (DAD detection) 238, 306 nm.

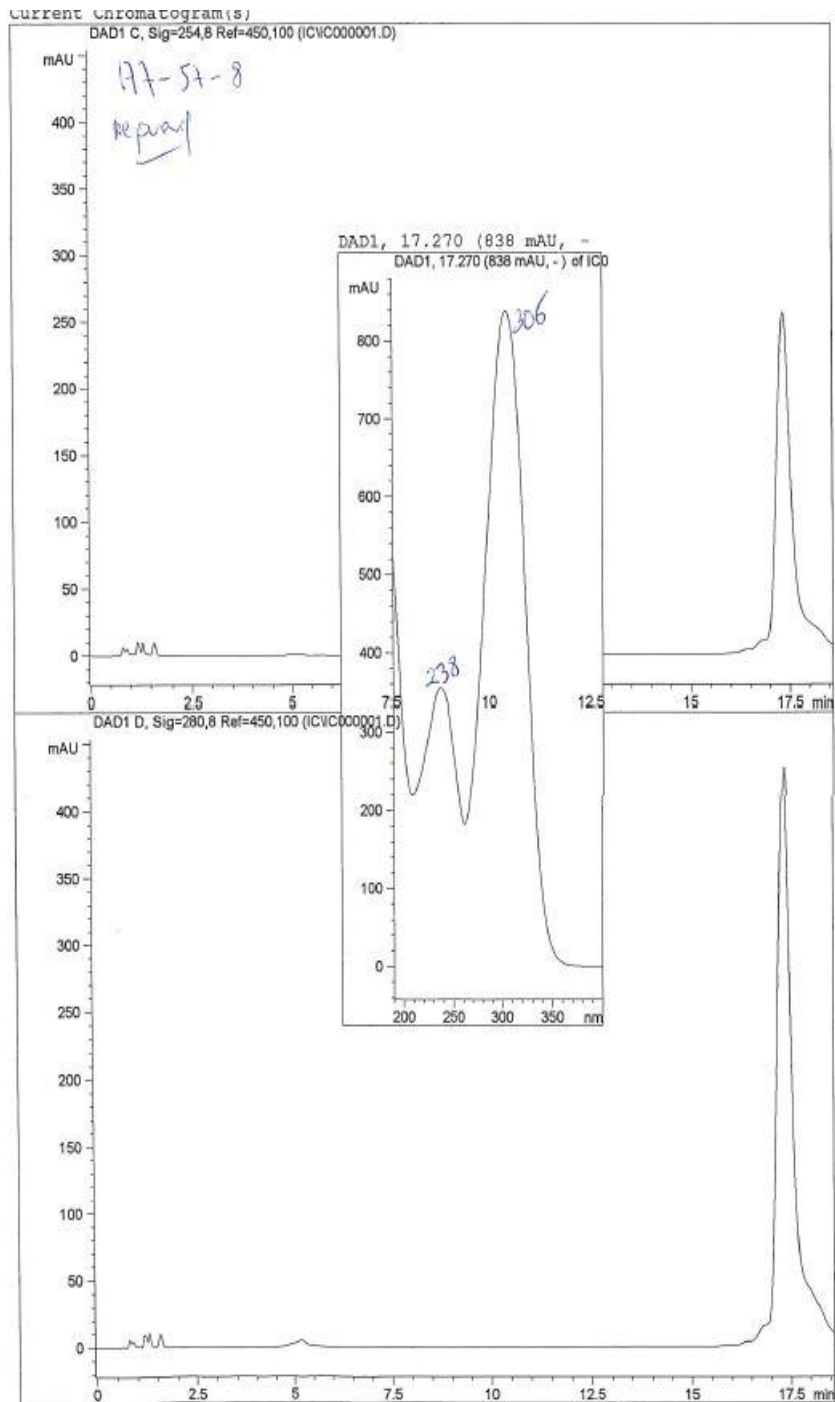


Figure S1: HPLC profile and UV of hemicalide

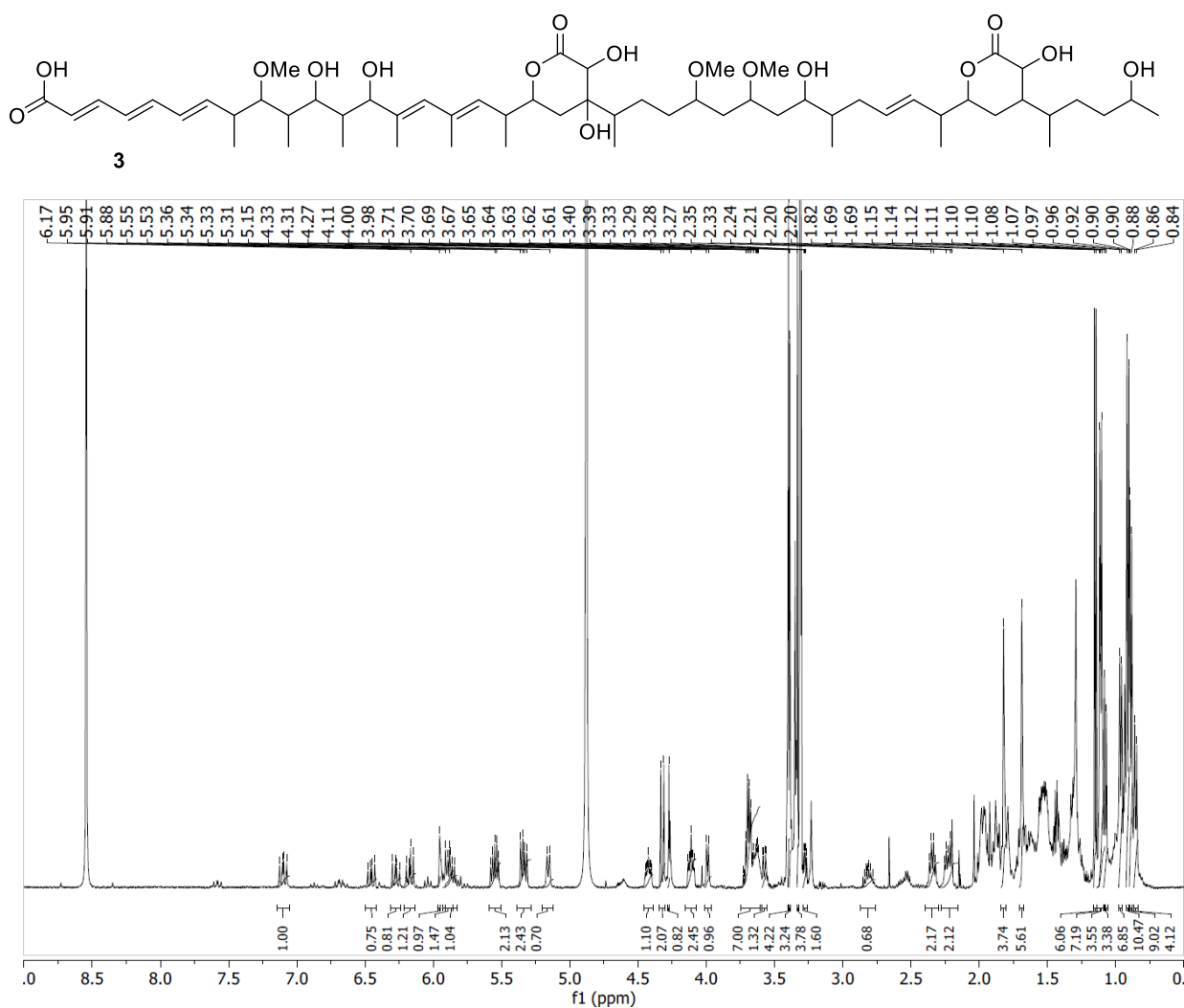


Figure S2: ^1H NMR spectrum of hemicalide in CD_3OD at 500 MHz

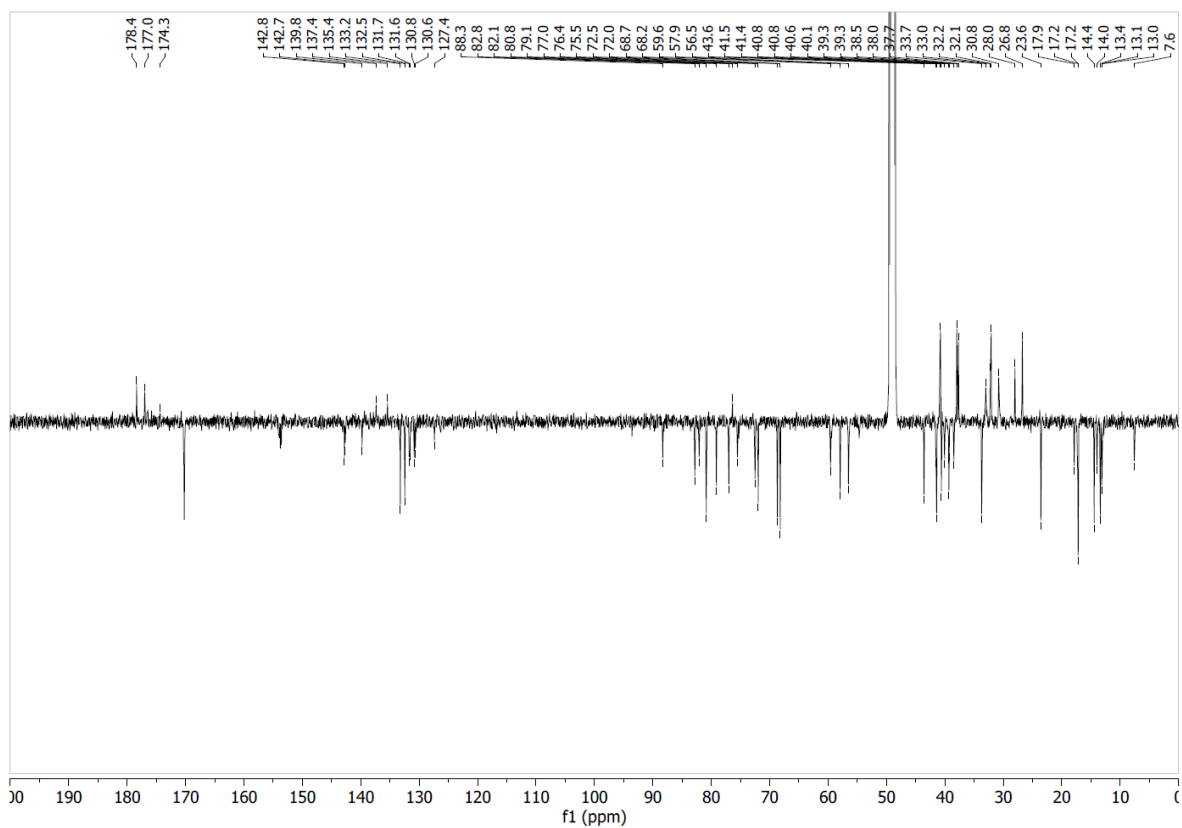
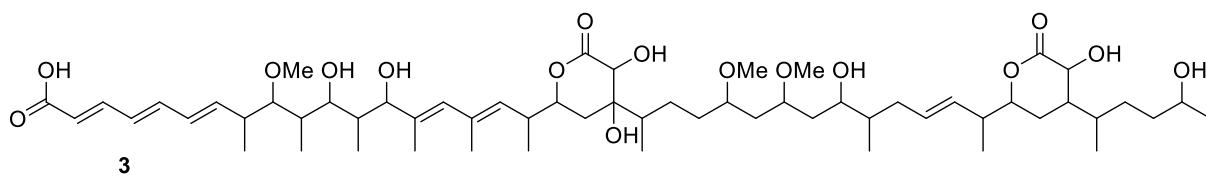


Figure S5: ^{13}C -DEPTQ NMR spectrum of hemicalide in CD_3OD at 126 MHz

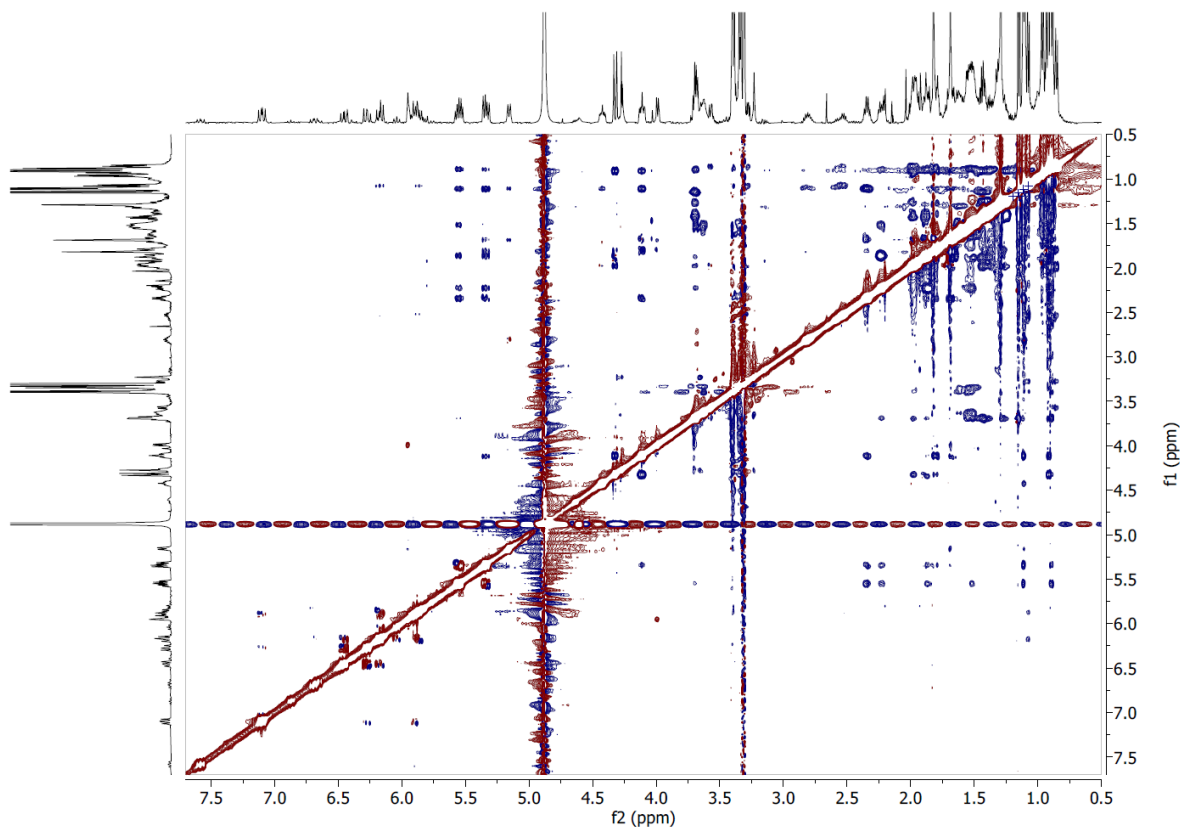
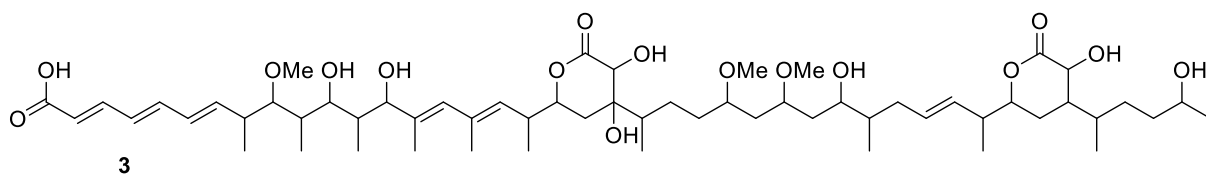


Figure S8: ROESY NMR spectrum of hemicialide in CD₃OD