

Electronic supplementary Information for paper entitled:

**Saliramophenol, an unprecedented natural *t*-butylphenol derivative from
Salicornia ramosissima J. Woods**

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Figure Caption

Fig. S1: ^1H NMR (500.13 MHz, CDCl_3) spectrum of the compound **1**.

Fig. S2: Expansion of ^1H NMR spectrum of the compound **1**.

Fig. S3: ^{13}C NMR (75.13 MHz, CDCl_3) spectrum of the compound **1**.

Fig. S4: Expansion of ^{13}C NMR spectrum of the compound **1**.

Fig. S5: Other expansion of ^{13}C NMR spectrum of the compound **1**.

Fig. S6: HSQC NMR spectrum of the compound **1**.

Fig. S7: Expansion of HSQC NMR spectrum of the compound **1**.

Fig. S8: Other expansion of HSQC NMR spectrum of the compound **1**.

Fig. S9: HMBC NMR spectrum of the compound **1**.

Fig. S10: Expansion of HMBC NMR spectrum of the compound **1**.

Fig. S11: Other expansion of HMBC NMR spectrum of the compound **1**.

Fig. S12: MS-ESI (+) spectrum of the compound **1**.

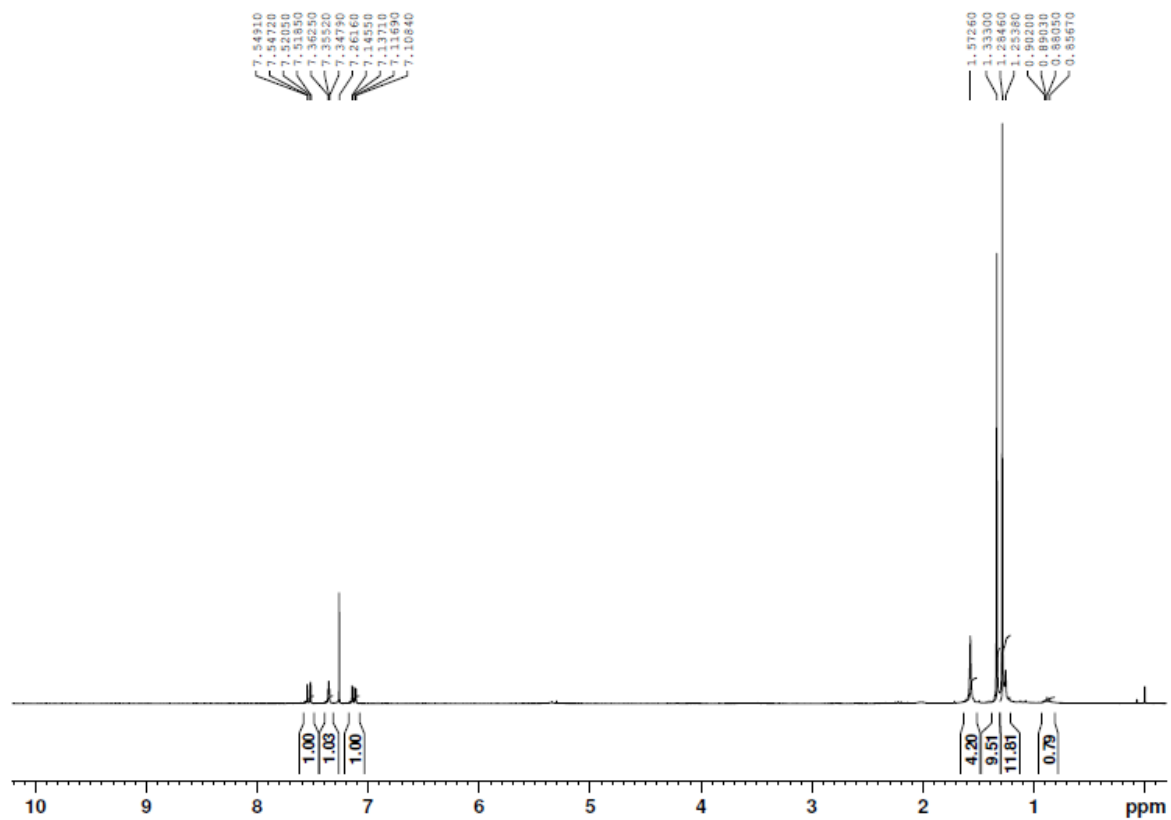
Fig. S13: MS-MS-ESI (+) spectrum of the peak m/z 663 of the MS-ESI spectrum of the compound **1**.

Fig. S14: MS-MS-ESI (+) spectrum of the peak m/z 685 of the MS-ESI spectrum of the compound **1**.

Fig. S15: HRMS spectrum of the compound **1**.

Fig. S16: FTIR spectrum of the compound **1**.

Fig. S17: Normalized UV-Vis spectrum of the compound **1** in DMSO



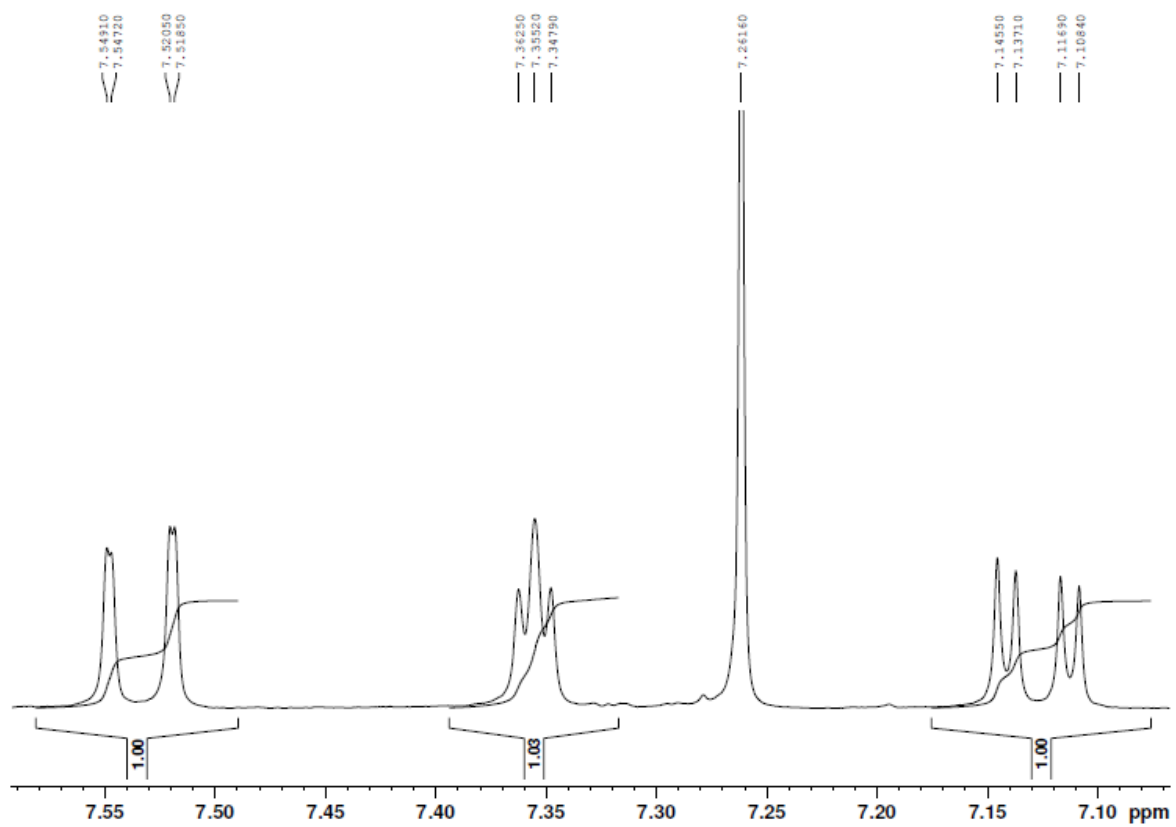


Fig. S2: Expansion of ^1H NMR spectrum of the compound **1**.

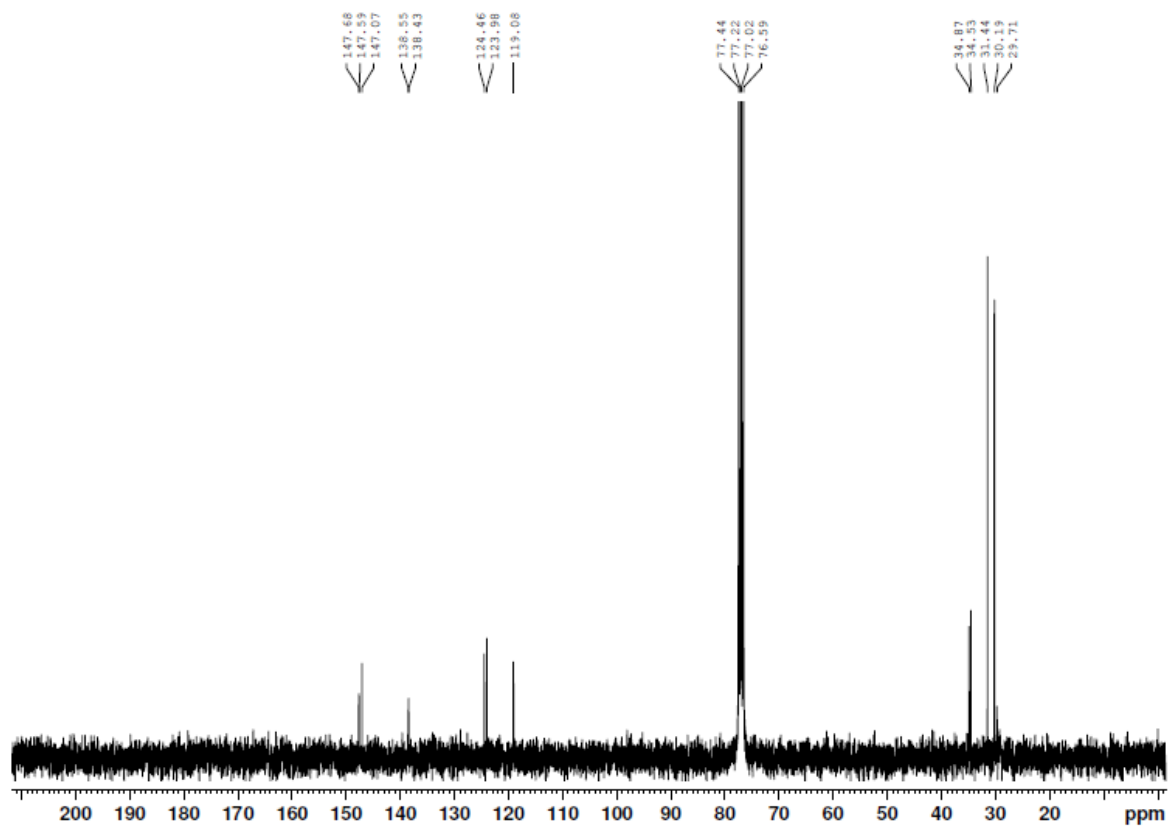


Fig. S3: ¹³C NMR spectrum of the compound 1.

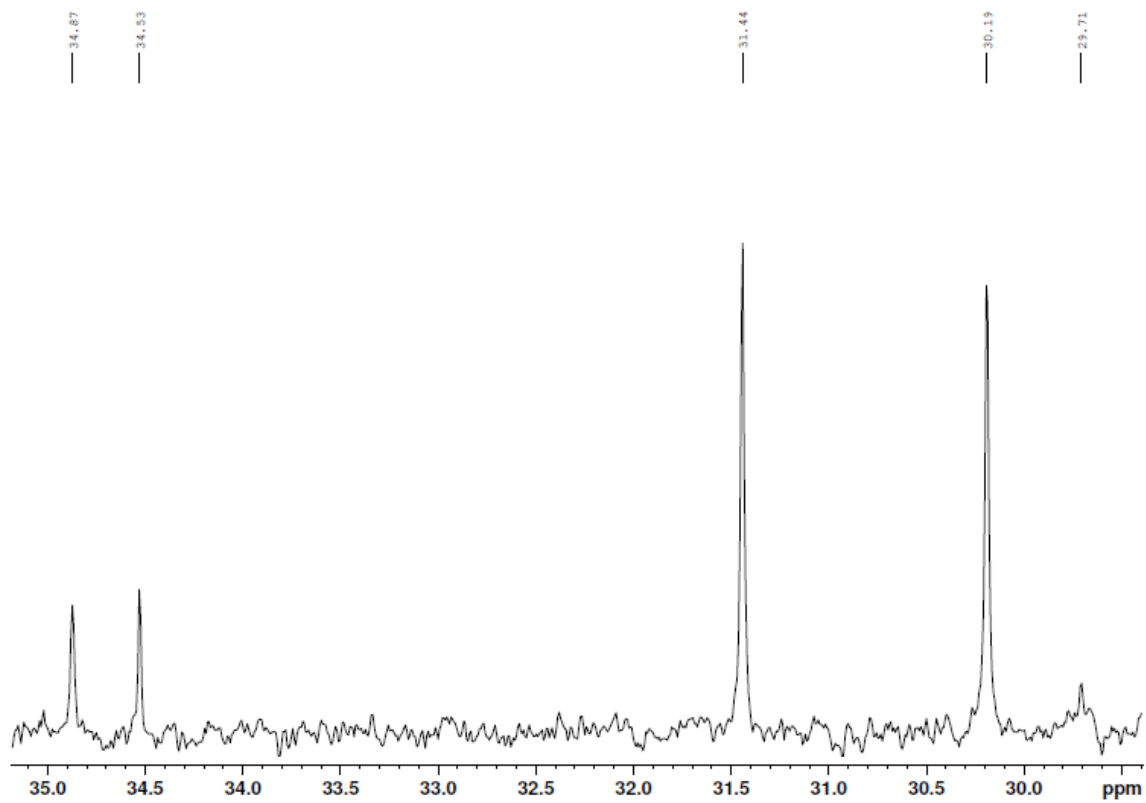


Fig. S4: Expansion of ^{13}C NMR spectrum of the compound 1.

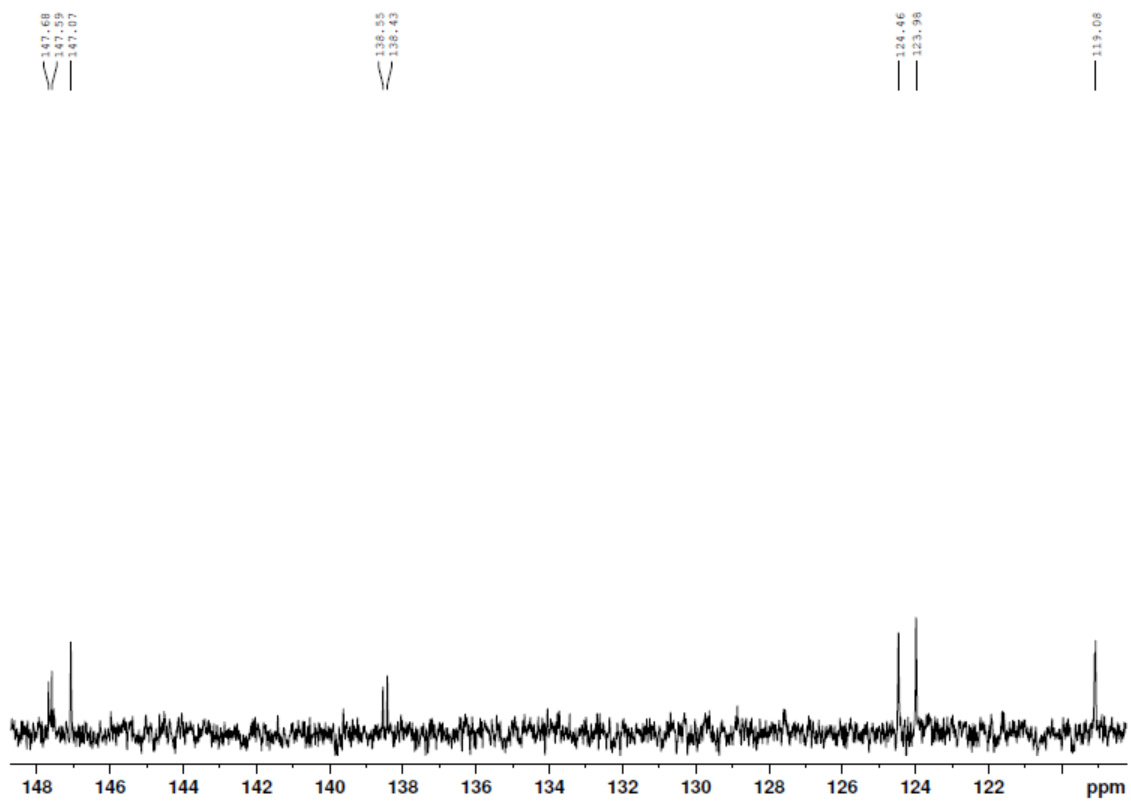


Fig. S5: Other expansion of ^{13}C NMR spectrum of the compound 1.

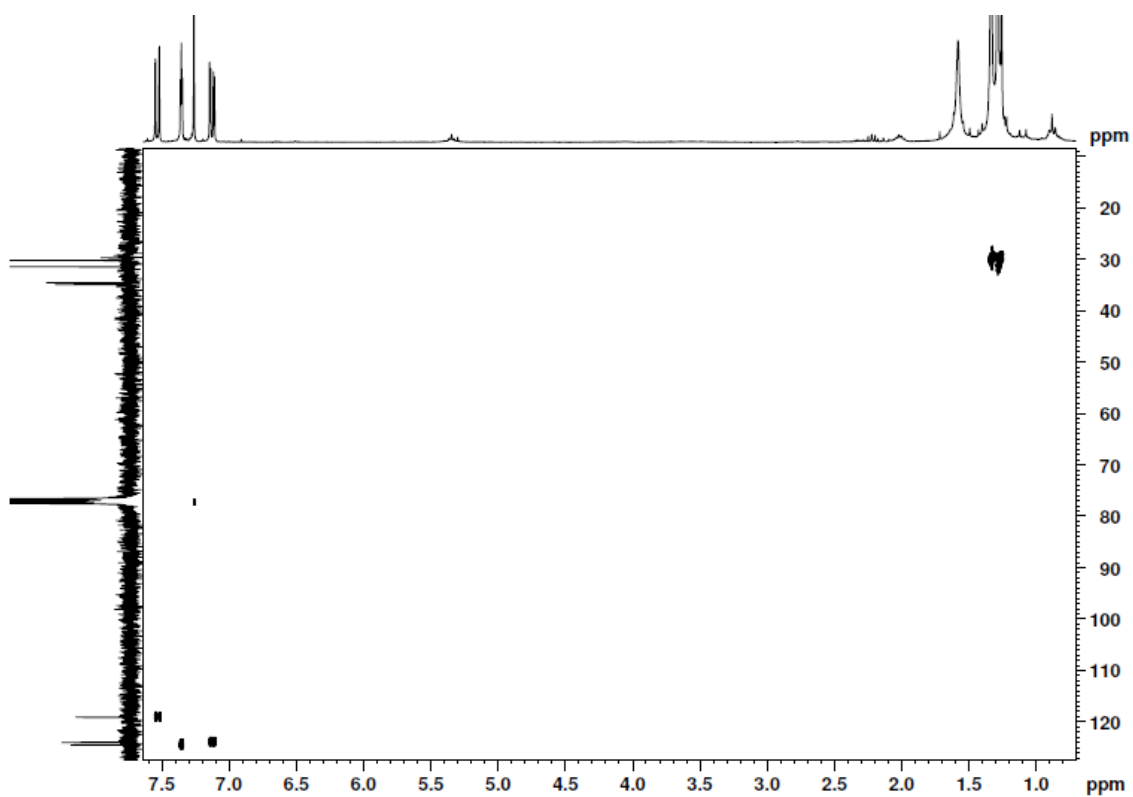


Fig. S6: HSQC NMR spectrum of the compound **1**.

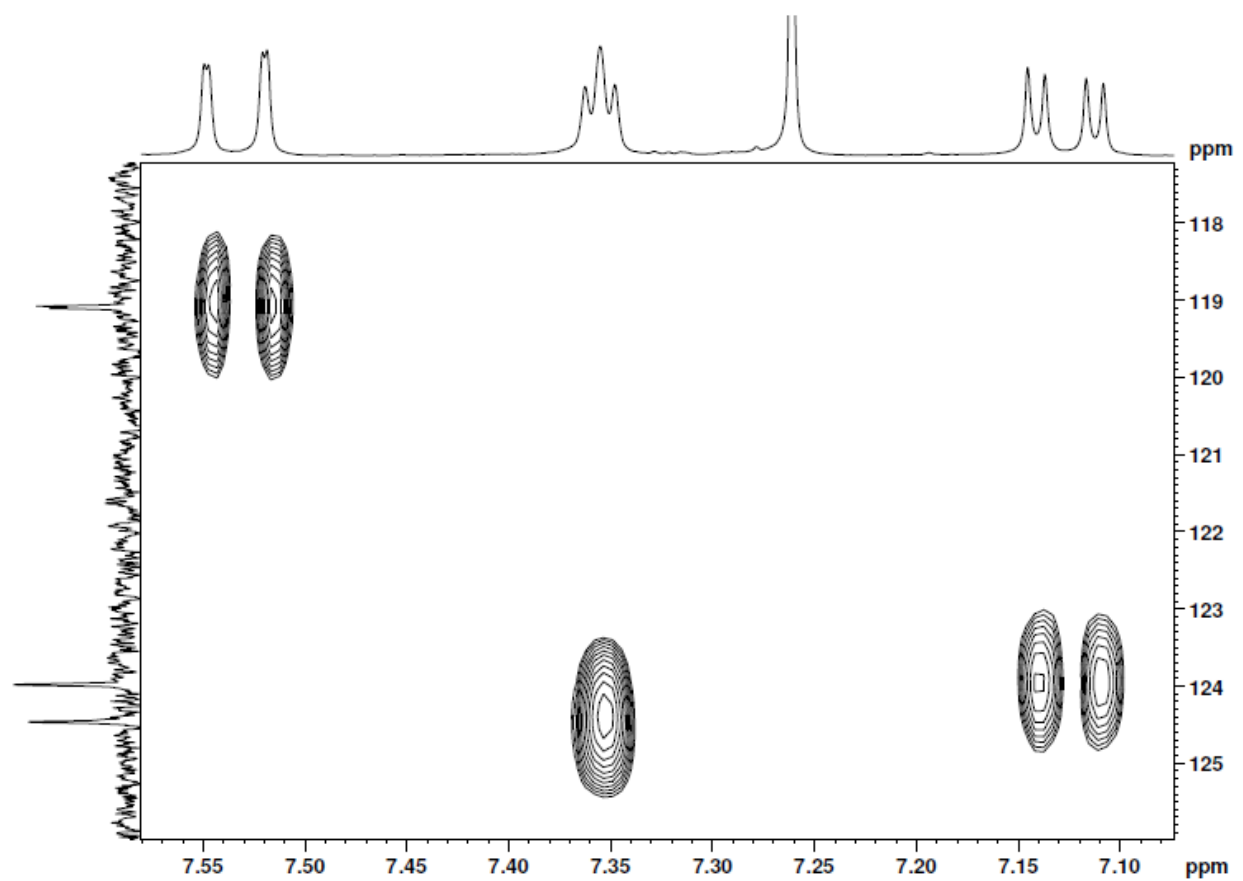


Fig. S7: Expansion of HSQC NMR spectrum of the compound **1**.

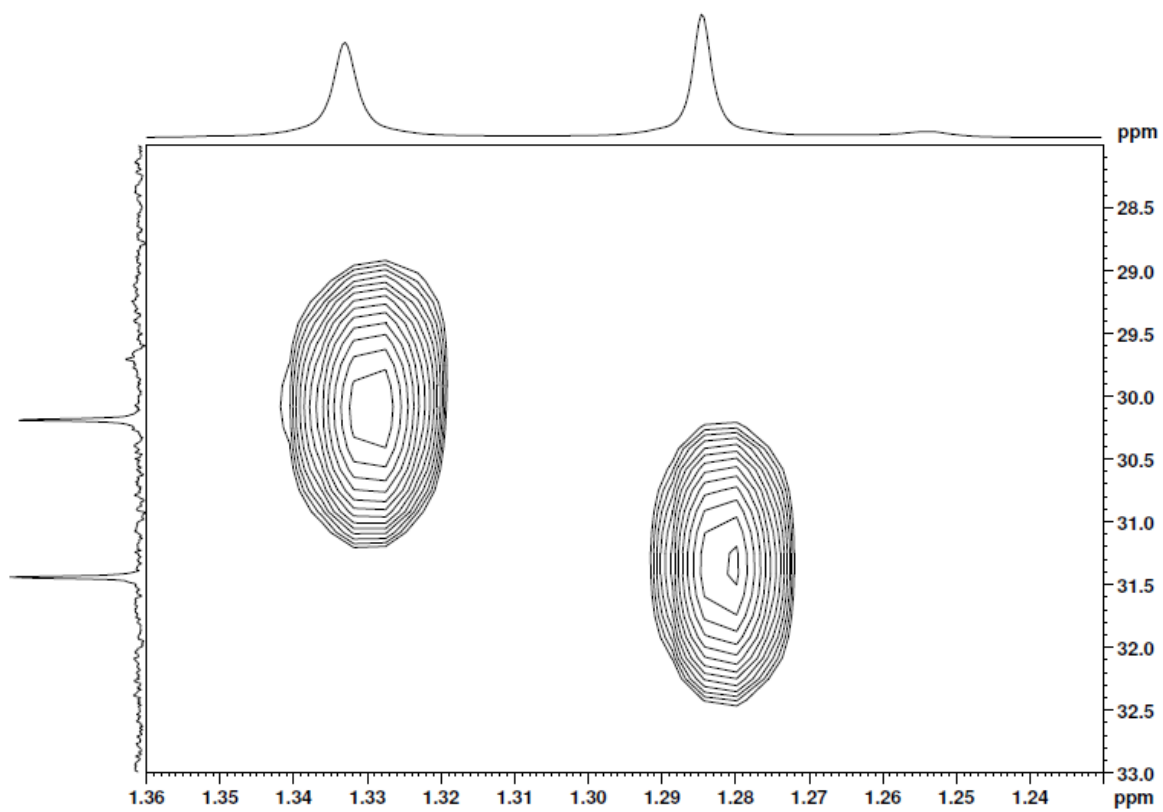


Fig. S8: Other expansion of HSQC NMR spectrum of the compound **1**.

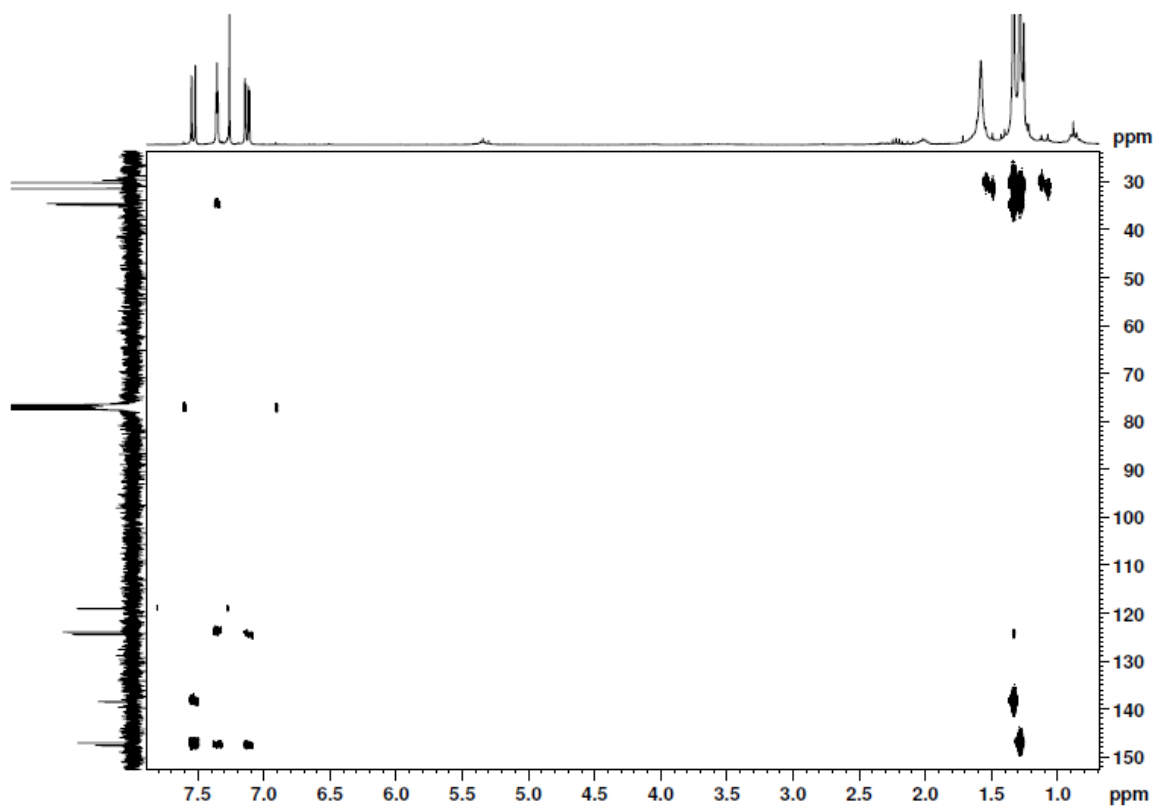


Fig. S9: HMBC NMR spectrum of the compound 1.

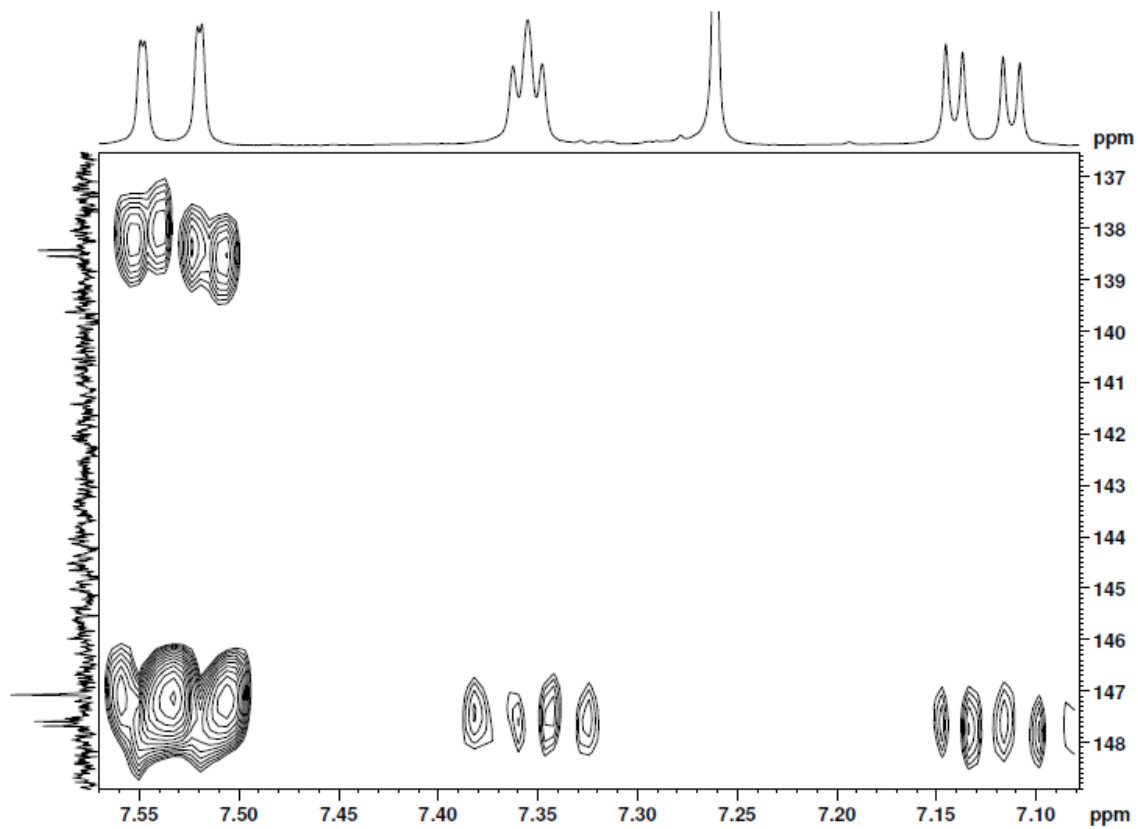


Fig. S10: Expansion of HMBC NMR spectrum of the compound **1**.

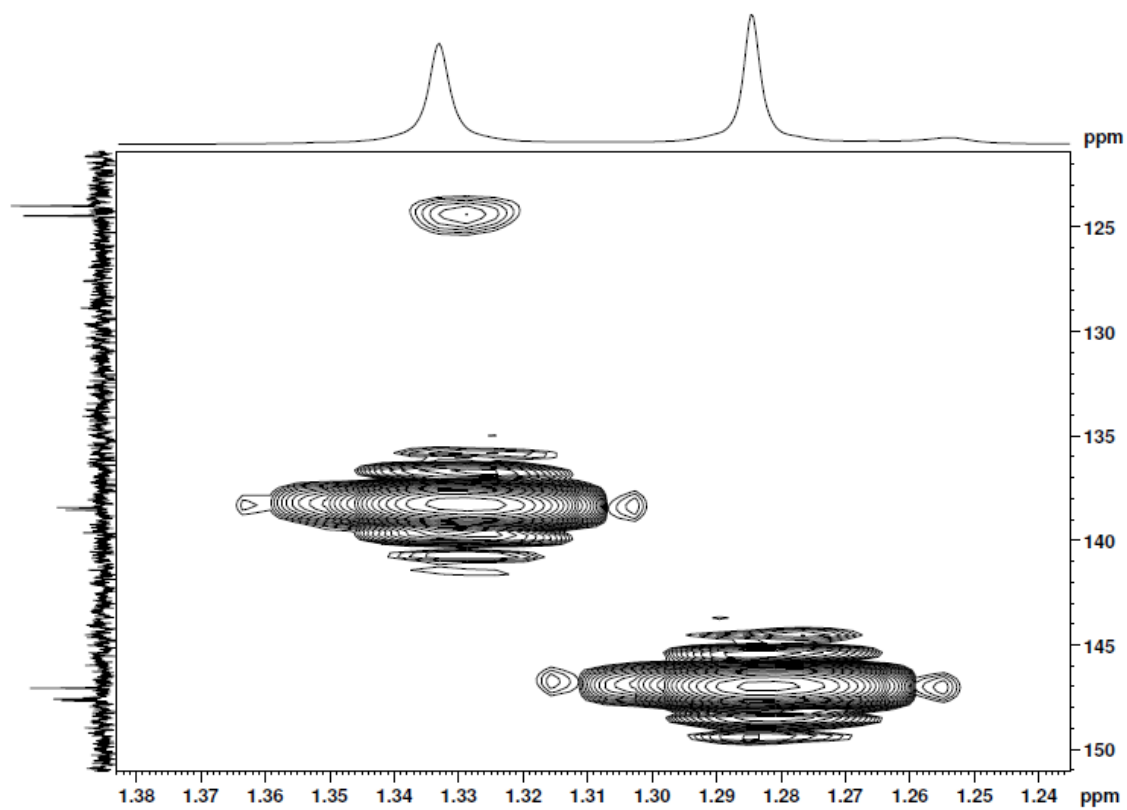


Fig. S11: Other expansion of HMBC NMR spectrum of the compound **1**.

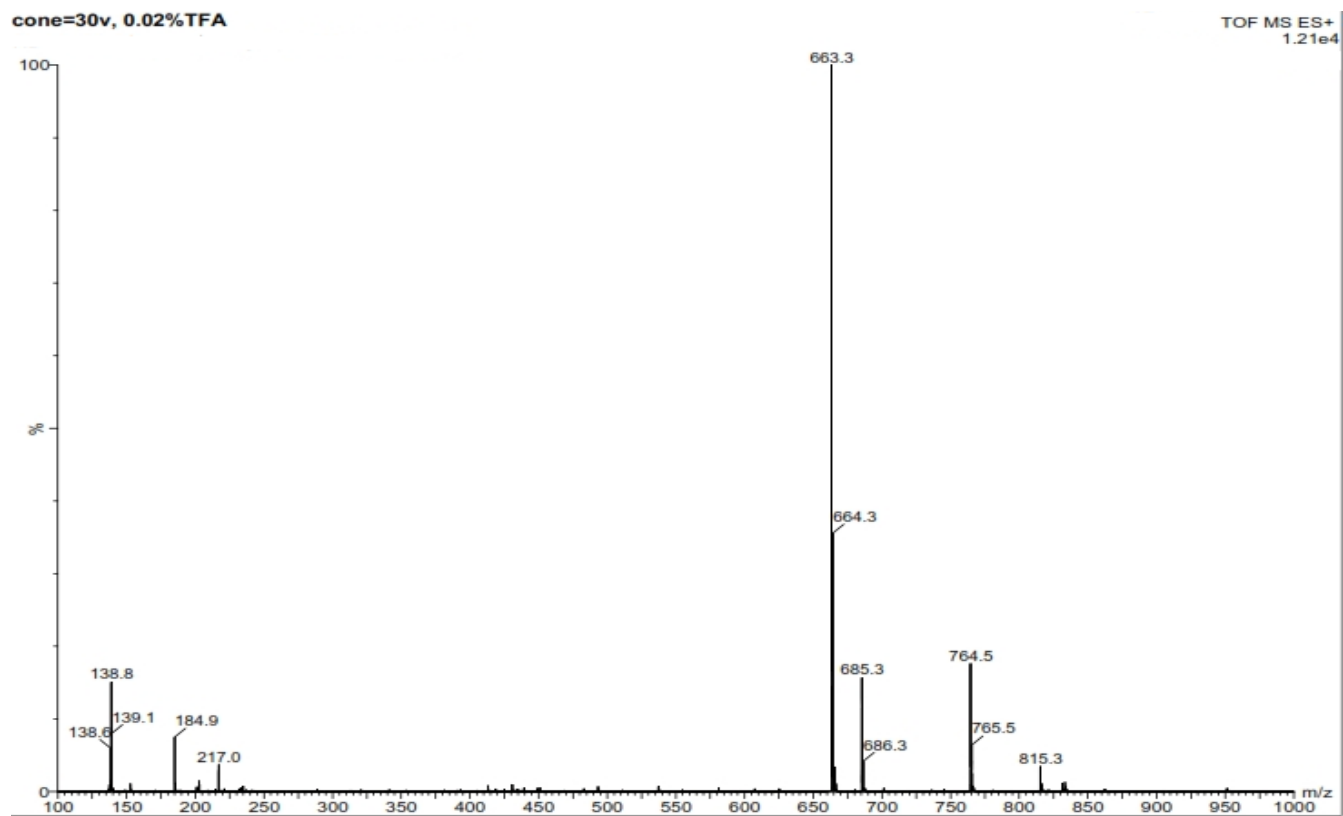


Fig. S12: MS-ESI (+) spectrum of the compound **1**.

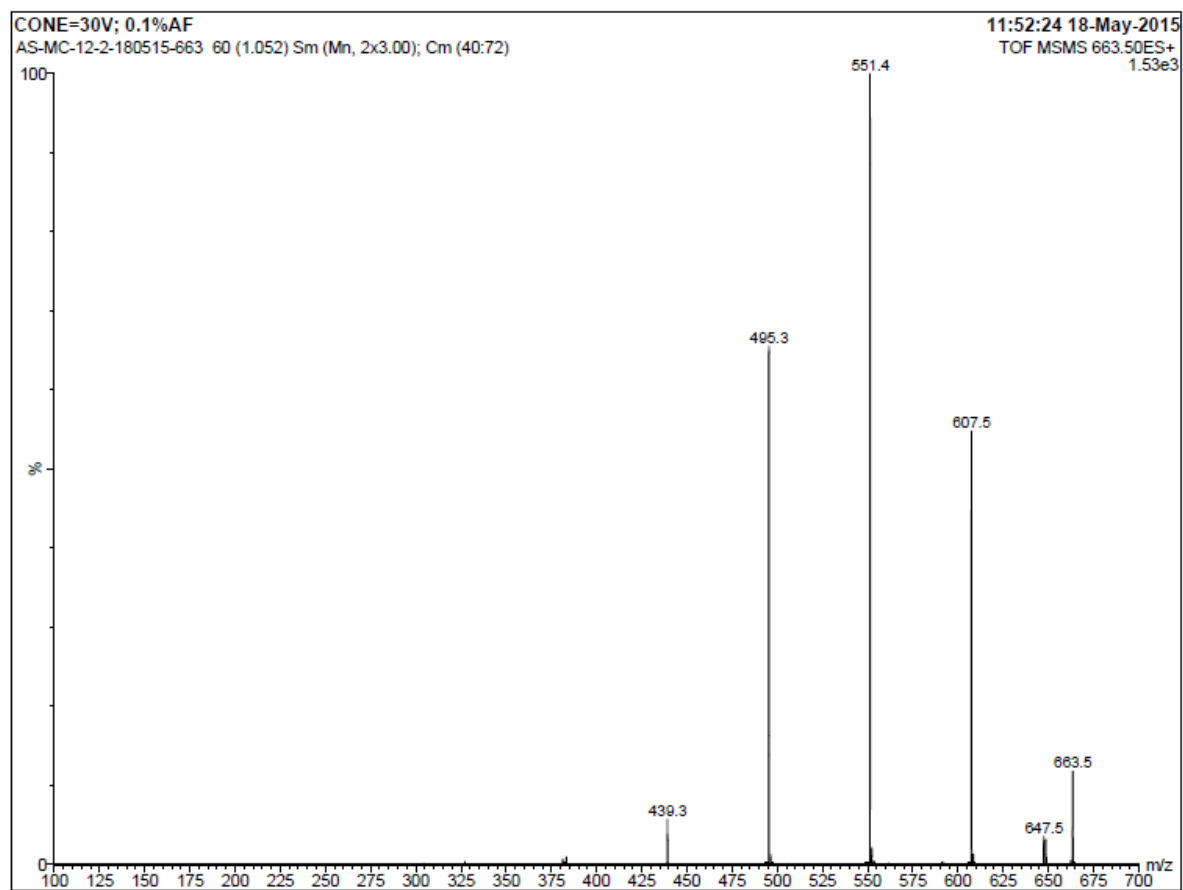


Fig. S13: MS-MS-ESI (+) spectrum of the peak m/z 663 of the MS-ESI spectrum of the compound **1**.

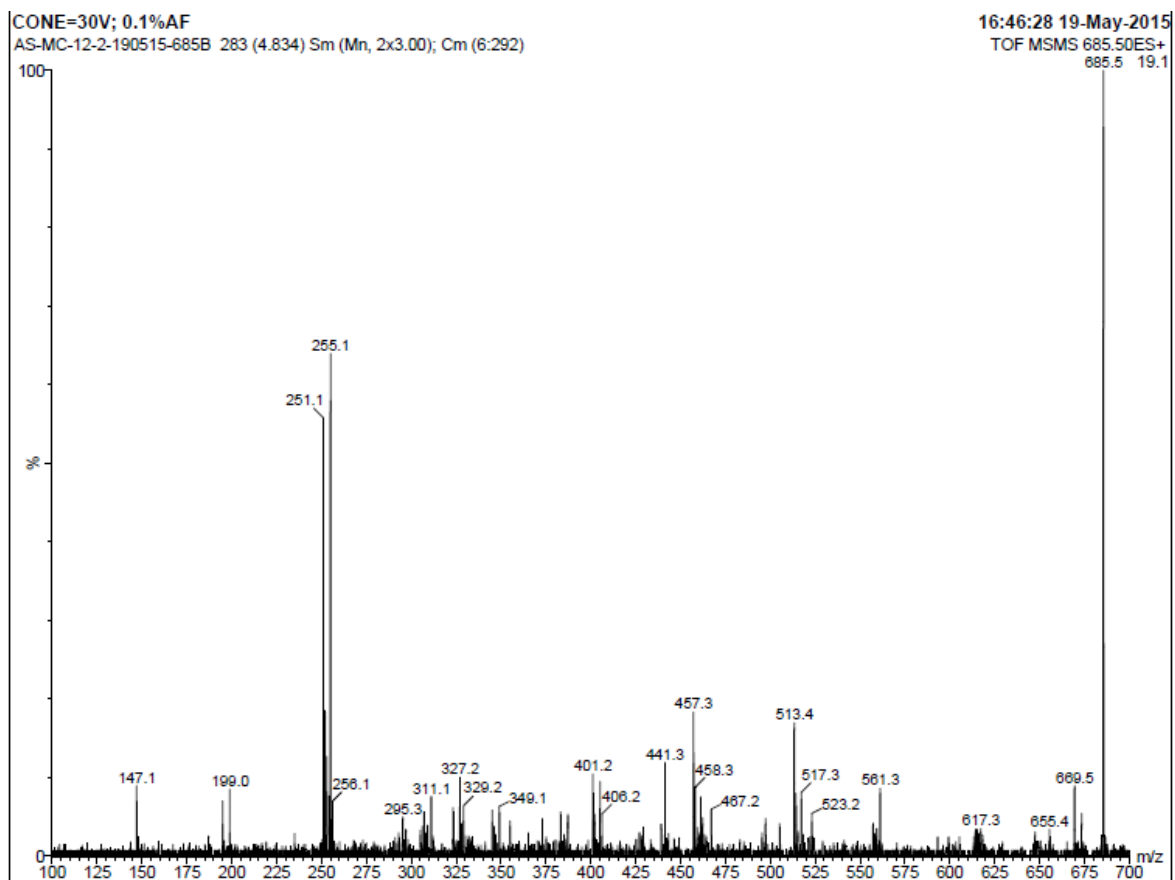


Fig. S14: MS-MS-ESI (+) spectrum of the peak m/z 685 of the MS-ESI spectrum of the compound **1**

Mass Spectrum Molecular Formula Report

Analysis Info
Analysis Name AS12299372000001.d
Sample Name VI-1

Electrospray (ESI)
Acquisition Date 12/3/2012 12:37:43 PM
Instrument micrOTOF

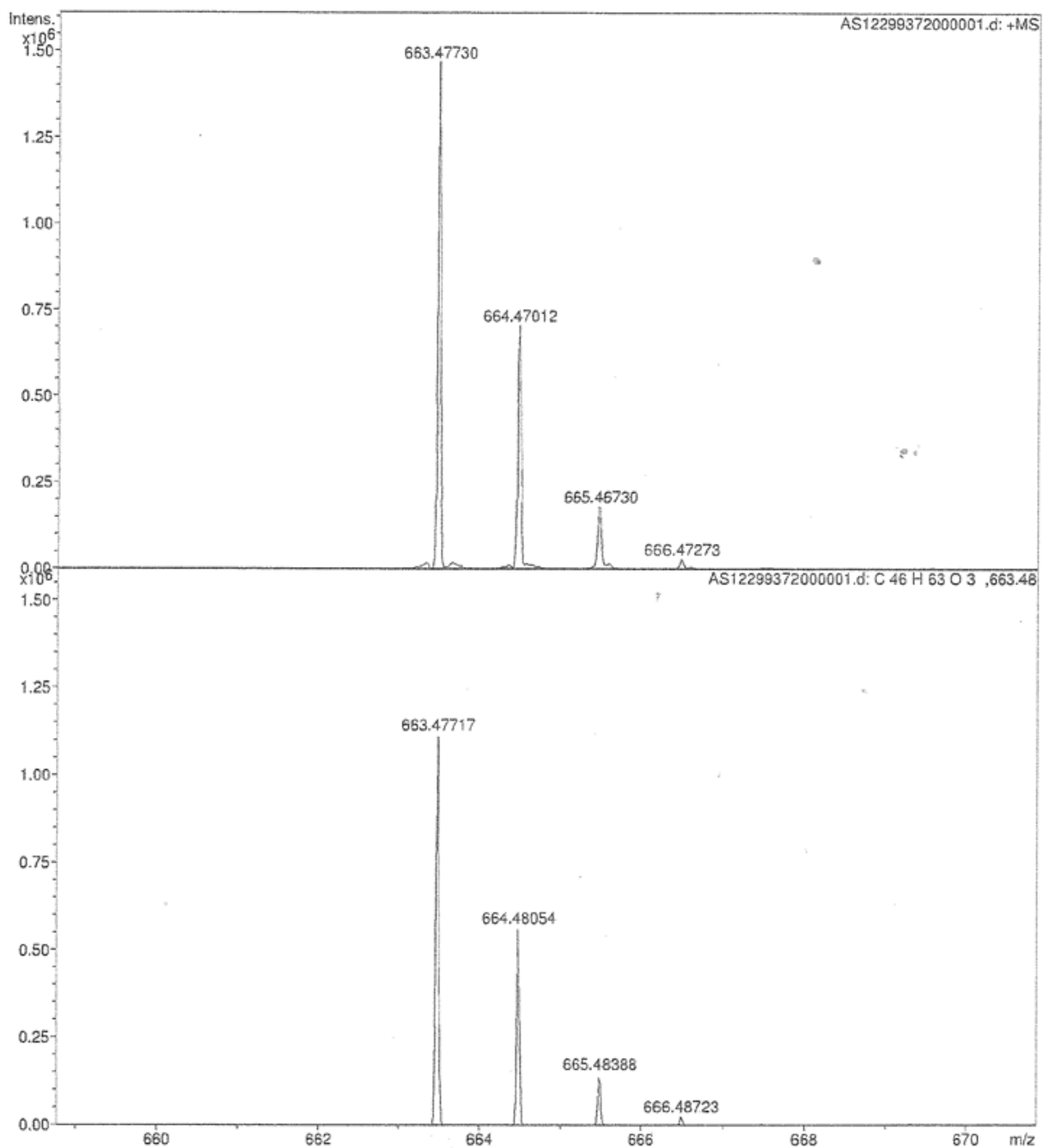


Fig. S15: HRMS spectrum of the compound 1.

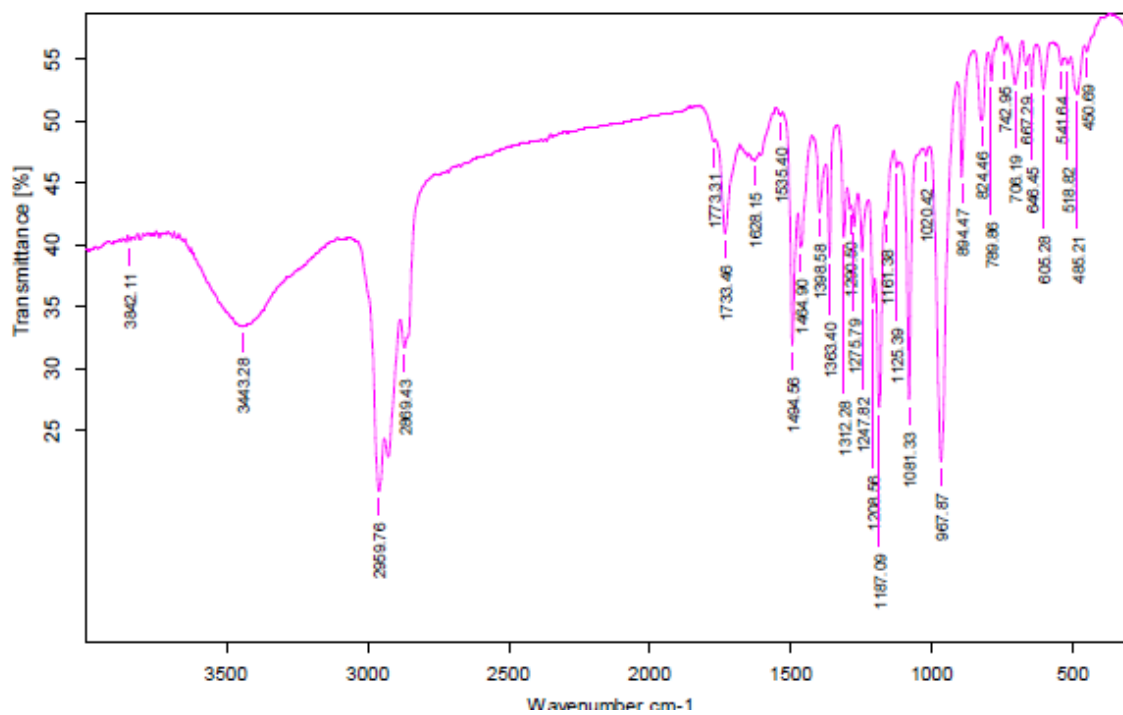


Fig. S16: FTIR spectrum of the compound 1.

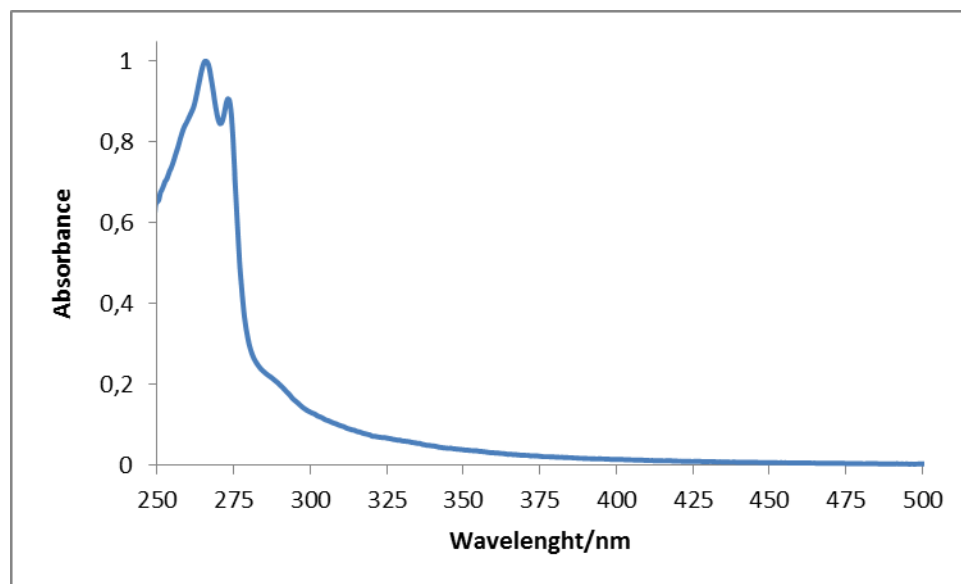


Fig. S17: Normalized UV-Vis spectrum of the compound1 in DMSO

EXPERIMENTAL PROCEDURES

General Experimental Procedures. NMR spectra (^1H , ^{13}C , HSQC, HMBC [71 ms (7 Hz)], COSY) were measured in CDCl_3 , on a Bruker Avance 500 with cryoprobe (Bruker, Wissembourg, France), operating at 500.13 MHz (^1H) and 75.47 MHz (^{13}C) and using TMS as internal standard. Chemical shifts were reported in δ units (ppm) and coupling constants (J) in Hz. The MS spectrum were obtained using ESI(+) with a Micromass Q-ToF 2TM mass spectrometer (Manchester, UK). The HRMS-ESI(+) were obtained using a MicroTof spectrometer with Apollo II (Bruker Daltonics, Bremen, Germany) with ESI interface, using a voltage of 4500 V applied to the needle, and a counter voltage between 100 and 150 V applied to the capillary. Column chromatography was performed with silica gel 60 (Aldrich Chemistry, 63-200 μm) and silica gel 60 (Acros Organics, 35-70 μm). Preparative thin layer chromatography was performed in glass plates (20 \times 20 cm) precoated with Merck silica gel 60 GF₂₅₄ (0.5 mm thickness) and activated in a kiln at 100-110 $^\circ\text{C}$ for 12 hours. Merck silica gel 60 GF₂₅₄ plates were used for TLC. Spots were detected in TLC under UV light (λ 254 and 366 nm).

Plant Material. The aerial parts of *Salicornia ramosissima* J. Woods were collected in Marina dos Puxadoiros (40 $^\circ$ 39' 23'' N, 8 $^\circ$ 40' 35'' W), Ria de Aveiro, Portugal. They were collected in vegetative state, from May to June, 2011 and identified by Helena Silva of the Department of Biology, University of Aveiro, Portugal. A voucher specimen (6606 AVE) was deposited in to the herbarium of the institution referred to above.

Extraction and Isolation. The air dried aerial parts of *Salicornia ramosissima* J. Wood (1087.6 g) were extracted with dichloromethane (2 \times 5 L \times 72h) at room temperature and filtered. The filtrate was evaporated *in vacuum* to obtain the CH_2Cl_2 extract (18.1 g) which was chromatographed on a silica gel 60 (63-200 μm) column eluted with a gradient system composed

by Hex:CH₂Cl₂ (1:9), CH₂Cl₂:EtOAc (1:0 and 3:7) and EtOAc:EtOH (1:1 and 0:1) yielding 10 fractions (A – J) some of them were chlorophylls. Fraction A (1.39 g) was applied to a silica gel 60 (35-70 μm) column eluted with Hex:CHCl₃ (from 100:0 to 0:100%) to yield 14 semi-purified fractions (A₁–A₁₄). Fraction A₁ (20 mg) was further purified by preparative thin layer chromatography [Hex:CH₂Cl₂ (6:4)] to give pure saliramophenol (**1**) (6 mg).

Saliramophenol (1): pale yellow oil; FTIR ν_{\max} (KBr) cm⁻¹: 3443 (O-H stretching), 2959 and 2869 (C–H stretching alkyl), 1398 and 1363 (C-H bending t-butyl) 1628 and 1535 (C=C stretching aromatic). ¹H NMR (CDCl₃, 500 MHz): Table 1; ¹³C NMR: (CDCl₃, 75.47 MHz): Table 1; MS-ESI *m/z* 663 [M+H]⁺, 685 [M+Na]⁺; HREIMS *m/z* 663.4773 [M+H]⁺ (calcd for C₄₆H₆₃O₃, 663.4772).