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Research Article

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Structural determination of 1 lipids isolated from the sea urchin *Echinometra lucunter*

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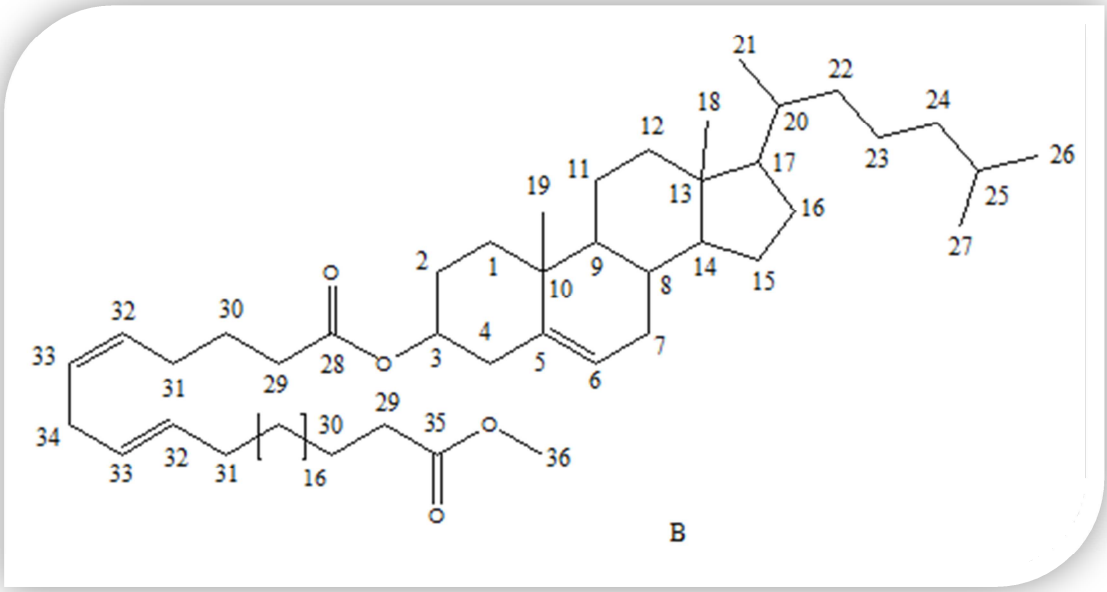
ABSTRACT

Usually we worked on marine organisms like algae and invertebrates of senegalese coasts but it's the first time we investigate sea urchin species. Specimens of the sea urchin Echinometra lucunter were collected in December 2002 at Dakar (Senegal). Fresh sea urchins were exhaustively treated with $CHCl_3$ /MeOH 1/1 (V/V). After evaporation of solvent the oily residue (1, 92 g) was passed through an RP 18 column. The $CHCl_3$ soluble fraction has been analyzed through spectroscopic means (NMR, Electro Spray Ionization mass spectrometry). It has been shown to contain the 2 sterol derivatives B.

Keywords: Sea urchin, reverse phase chromatography (R P), NMR, Electro Spray Ionization (ESI), sterol.

INTRODUCTION

Marine organisms from the coast of Senegal have been subjected to many studies on algae and invertebrates such as sponges and cnidarians [1-3]. The investigation, however broad it was, did not touch the echinoderms. In order to make our contribution to scientific research a bit more complete and balanced, we undertook the study of metabolites of the sea urchin *Echinometra lucunter*. After soaking in the mixture $CHCl_3$ /CH_3OH (1 / 1, V / V) phase obtained is evaporated to dryness to give a residue. The crude extract was subjected to reverse phase chromatography and RP 18: fractions 6A5 and 10A5 respectively eluted with methanol and chloroform contain sterol derivatives B. With the exception of cycles, the numbering is unconventional. It is based on the chemical shifts of NMR signals of the same nature in order to make spectroscopic interpretation easier.



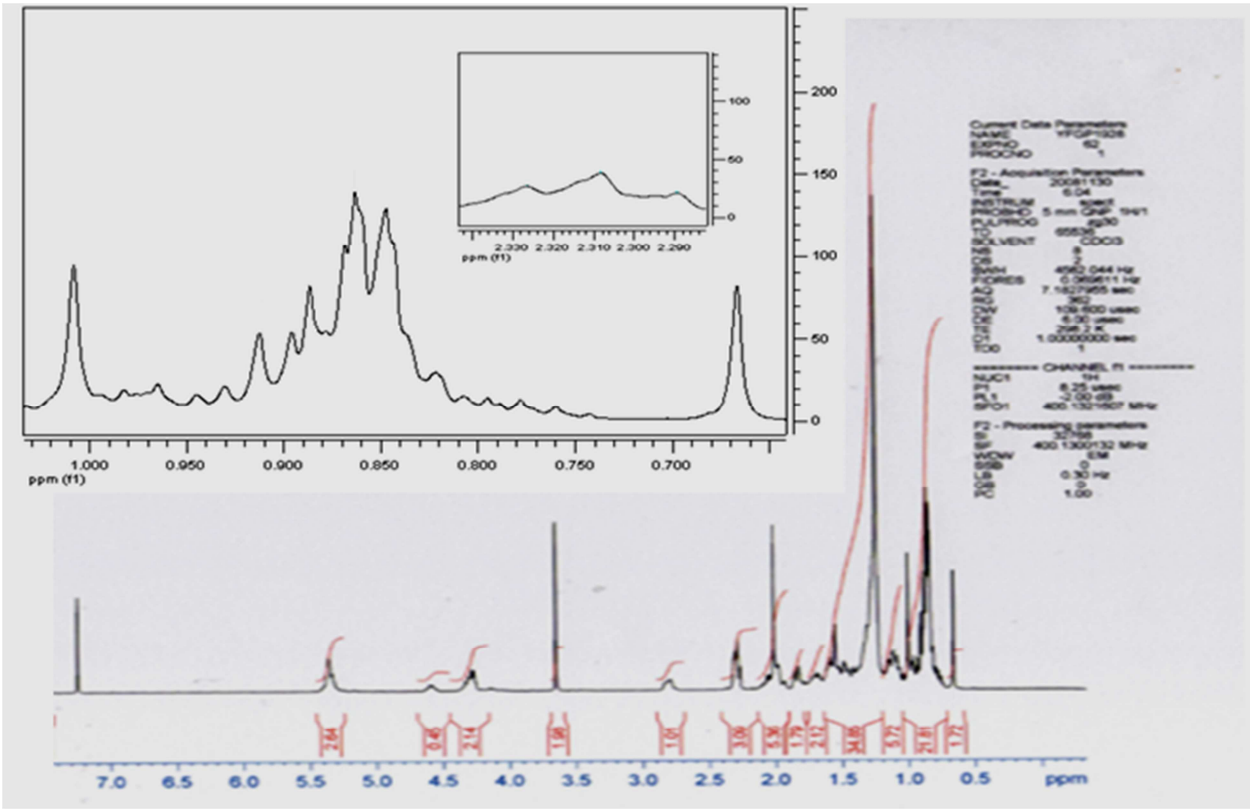


Figure 1: [1] HNMR spectrum with spread region (1,008 to 0,667 and 2.30 to 2.29) of the compound B

EXPERIMENTAL SECTION

General Methods

The NMR spectra were measured on a Varian BRUKER ARX 400 MHz using deuterated chloroform ($CDCl_3$) as an internal standard. Two-dimensional (2D) NMR was performed with ^1H–^1H COSY. ESI-MS spectra were obtained using a FISONG VG Autospec M. Thin-layer chromatography (T.L.C.) was performed using silica gel 60 F254 and silica gel 60 RP-18 F254.

Urchin Material

Specimens of the sea urchin *Echinometra lucunter* collected in December 2002 at Dakar (Sénégal), were deposited at the Laboratory of Natural Products, Cheikh Anta Diop University.

Extraction and Isolation

The sea urchin *Echinometra lucunter* (393g) was extracted with MeOH /Chloroform (1 L /1L) at room temperature. The concentrated MeOH /Chloroform fraction (1.92g) was subjected to silica gel (SiO_2) column chromatography and eluted with mixtures water-methanol (9/1, 7/3, 4/6, 2/8), methanol 100% , methanol/chloroform (9/1) and finally chloroform 100%. Each eluant was monitored by thin layer chromatography (TLC), and 11 fractions (A1 to A4, A5, B and C) were obtained. Fraction 10A5 (34 mg) was subjected to SiO_2 column chromatography and was eluted with n-hexane –EtOAc (9:1→8:2→7:3→6:4→5:5→4:6→2:8→1:9, v/v) and MeOH (100%), to give 20 subfractions (10A5 to 19A5). Subfraction 11A5 ultimately produced a new compound named B [2.3 mg, Rf 0.62 in TLC (plate RP-18 F254) in n-hexane–EtOAc (8:2), v/v.

Sterol derivative (B). $C_{58}H_{97}O_4$. ESI-MS m/z: 859 $[M + 2H]^+$; ^{1}HNMR (400 MHz, $CDCl_3$, δ, ppm, J/Hz): 5.34 (1H, m, H-6), 2.30 (2H, t,H-29, J = 7.58), 3.52 (1H, m, H-3), 2.24 (2H, m, H-4), 2.02, 1.10 (2H, m, H-12a, 12e), 1.92 (2H, m, H-2), 1.80, 1.22 (2H, m, H-16a, 16e), 3.64 (3H, s, H-36), 1.62, 1.36 (2H, m, H-11a, 11e), 1.58 (1H, m, H-25), 1.57, 1.49 (2H, m, H-15a, 15e), 1.43, (1H, m, H-9), 1.33- 1.25 ($(CH_2)_{16}$, m, 1.26 (1H, m, H-20), 1.26 - 1.02 (2H, m, H-22a, 22e), 2.81 (2H, t, H-34), 1.57-1.60 (2H, m, H-30), 1.19, 1.08 (2H, m, H-23), 1.08 (1H, m, H-17), 1.01 (1H, m, H-14), 0.92 (3H, d, J = 6.53,H-21), 0.87 (3H, s, H-19), 0.85 (6H, d, H-27, 26), 0.84, 0.81 (2H, m, H-24a, 24e), 0.67 (3H, s, H-18); 5,33 - 5,36 (4H, m, H-32, 33), 1,98 – 2,09 (4H, m, H-31).

^{13}C NMR (100 MHz, $CDCl_3$, δ) : 170.11 (C-28), 172.6 (C-35), 122.64 (C-5), 139.66 (C-6), 74 (C-3), 128,80 - 130,88 (C-32, 33), 56.40 (C-17), 51.50 (C-14), 34.14 (C-29), 51.01 (C-9), 44.73 (C-13), 39.40 (C-12, C-24), 36.95 (C-10), 25.64 (C-31), 35.94 (C-22), 35.23 (C-20), 34.31 (C-1), 33.20 (C-4), 31.94 (C-34), 29.71 (C-2), 29.71- 29.13 ($(\underline{C}H_2)_{16}$), 28.24 (C-16), 27.99 (C-25), 26.28 (C-15), 51,43 (C-36), 23.40 (C-23), 22.81 (C-27), 24.98 (C-30), 25.64 (C-34), 22.55 (C-26), 20.61 (C-11), 18.58 (C-19), 18.07 (C-21), 12.64 (C-18).

The resonant multiplet δ= 3.52 ppm is typical of proton of 3ß-hydroxysterol. The multiplet at δ= 0.67 ppm is typical of tertiary methyl C-18 of sterol skeletons [4, 5]. The two triplets at 0, 88 ppm and 2.33 ppm, associated with broadened singlet between 1.24 to 1.29 ppm indicate a set of signals characteristic of fatty acids spectra [6, 7, 8].

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