



Research Article

ISSN : 0975-7384  
CODEN(USA) : JCPRC5

Structural determination of 1 fatty acid isolated from the *Cassis sieberiana*

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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 *Cassis Sieberiana* were collected in December 2002 at Dakar (Senegal). Fresh sea urchins were exhaustively treated with  $\text{CHCl}_3$  /  $\text{MeOH}$  1/1 (V/V). After evaporation of solvent the oily residue (4,3616g) was passed through an RP 18 column. The  $\text{CHCl}_3$  soluble fraction has been analyzed through spectroscopic means (NMR, Electro Spray Ionization mass spectrometry). It has been shown to contain the 1 triacylglycerol A.

**Keywords:** *Cassis Sieberiana*, reverse phase chromatography (R P), NMR, Electro Spray Ionization (ESI), triacylglycerol. [1-3].

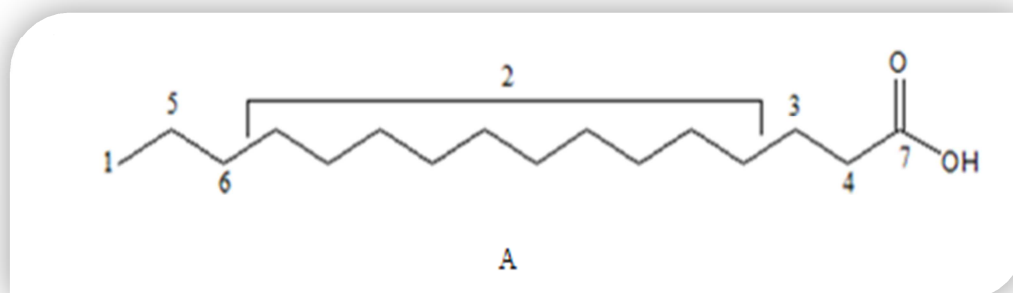
INTRODUCTION

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 *Cassis Sieberiana*.

After soaking in the mixture  $\text{CHCl}_3$  /  $\text{CH}_3\text{OH}$  (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 3CS6 eluted with methanol and chloroform contain sterol derivatives A. 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



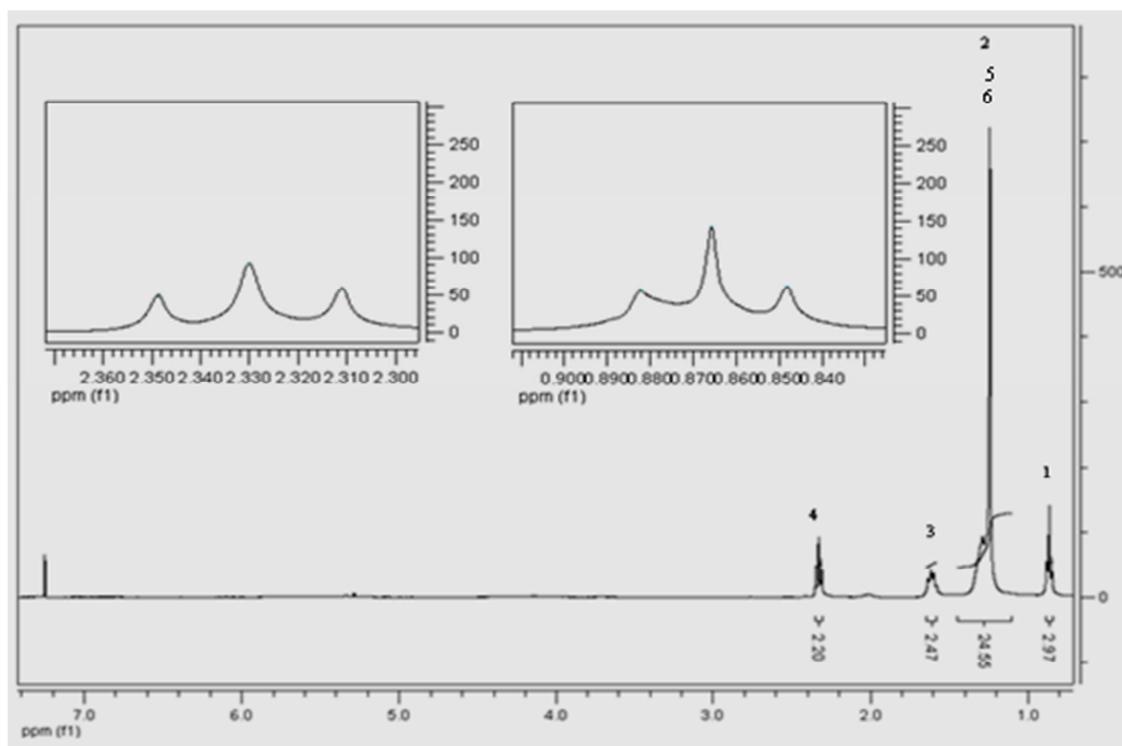


Figure 1:  $^1\text{H}$  NMR spectrum with spread region (0.90 to 0.84 and 2.33 to 2.29) of the compound A

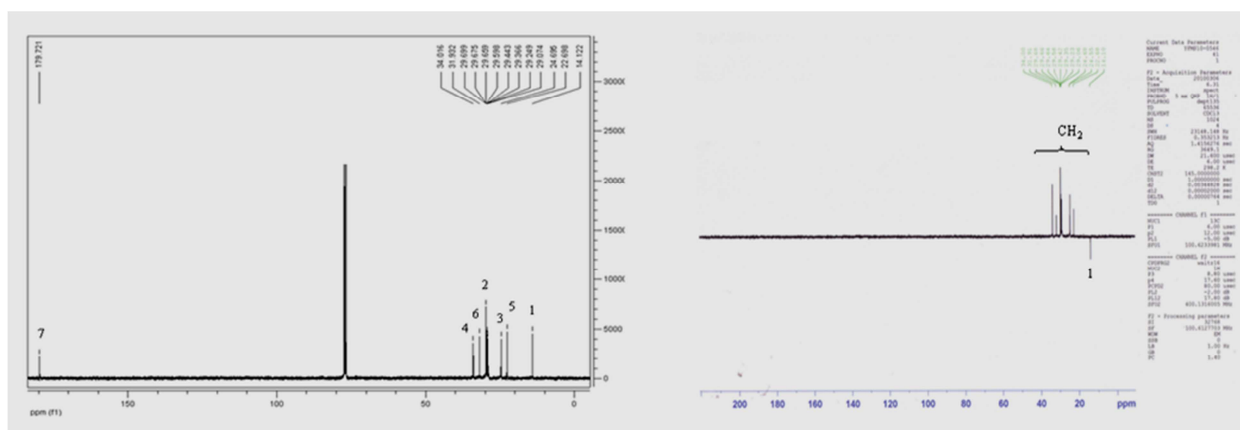


Figure 2:  $^{13}\text{C}$  NMR spectrum of compound A

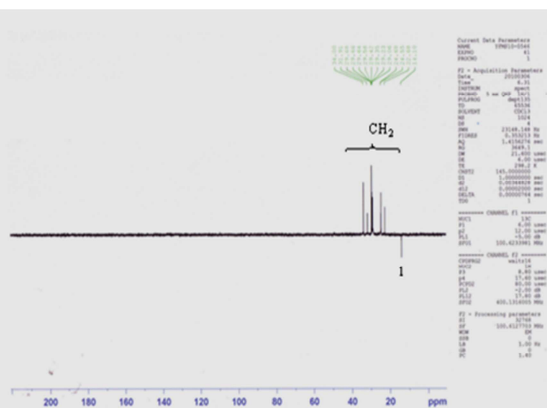


Figure 3: DEPT spectrum of compound A

## EXPERIMENTAL SECTION

### General Methods

The NMR spectra were measured on a Varian BRUKER ARX 400 MHz using deuterated chloroform ( $\text{CDCl}_3$ ) as an internal standard. Two-dimensional (2D) NMR was performed with  $^1\text{H}$ - $^1\text{H}$  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 *Cassis Sieberiana* collected in December 2002 at Dakar (Sénégal), were deposited at the Laboratory of Natural Products, Cheikh Anta Diop University.

### Extraction and Isolation

The *Cassis Sieberiana* (4,3616g g) was extracted with MeOH /Chloroform (1 L /1L) at room temperature. The concentrated MeOH /Chloroform fraction (1.42g) was subjected to silica gel ( $\text{SiO}_2$ ) 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 9 fractions (CS1 to CS9) were obtained. Fraction CS6 (402, 4 mg) was subjected to SiO<sub>2</sub> 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 15 subfractions (1CS6 to 15CS6). Subfraction 4CS6 ultimately furnished a new compound named A [7.3 mg, R<sub>f</sub> 0.84 in TLC (plate RP-18 F254) in n-hexane–EtOAc (7:3), v/v].

**Fatty acid (A) C<sub>16</sub>H<sub>32</sub>O<sub>2</sub>.** ESI-MS m/z: 211 [M – COOH]<sup>+</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, δ, ppm, J/Hz): 2.32 (2H, t, J = 7.44, H-4), 1.57-1.65 (2H, m, H-3), 1,24-1,29 (20H, bs, H-2), 0.86 (3H, t, J = 7.01, H-1), 1.57-1.65 (2H, m, H-5), 1.57-1.65 (2H, m, H-6).

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>, δ): 34.15 (C-4), 24.70 (C-3), 29.07-29.70 (C-2), 14.12 (C-1), 22,71 (C-5), 31.94 (C-6), 179.72 (C-7).

The count used is based on the chemical shifts of the NMR signals of the same nature in order to make interpretation more readable.

As a result of the superposition of some signals, the corresponding areas were plotted for better dispersion and easier allocation of signals considered.

Analysis of the <sup>13</sup>C NMR spectrum indicates the presence of a signal at δ = 179.72 ppm attributed to the carbon atom of the -COOH function (signal 7) [5[4 - 8].

The (signal 4) 34.01 ppm corresponds to the methylene at α of the acid function.

The signals between 22.70 to 31.93 ppm are attributed to all the methylene groups other) [9- 10]. DEPT spectrum on the absence of the (signal 7) to 179.72 ppm in the <sup>13</sup>C spectrum, confirming the existence of a quaternary carbon. The signal at δ = 14.12 ppm is attributed to methyl terminal. The signals are directed upward relative to groups CH<sub>2</sub> [11- 12].

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