Available online www.jocpr.com

Journal of Chemical and Pharmaceutical Research, 2016, 8(8):139-141

Research Article

ISSN : 0975-7384
CODEN(USA) : JCPRC5

Structural determination of 1 triacylglycerol isolated from the *Cassis sieberiana*

A. Ould Kaihil[1,2], M. S. Diop[1] and M. Said Minnih[1,2]

[1]*Université Cheikh Anta Diop Faculté des Sciences et Techniques, Laboratoire des Produits Naturels, Département de Chimie, Dakar, Sénégal*
[2]*Unité de Chimie Moléculaire et Environnement, Université de Sciences, de Technologie et de Médecine, Nouakchott, Mauritanie*

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 $CHCl_3$ /MeOH 1/1 (V/V). After evaporation of solvent the oily residue (4,3616g) 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 1 triacylglycerol A.

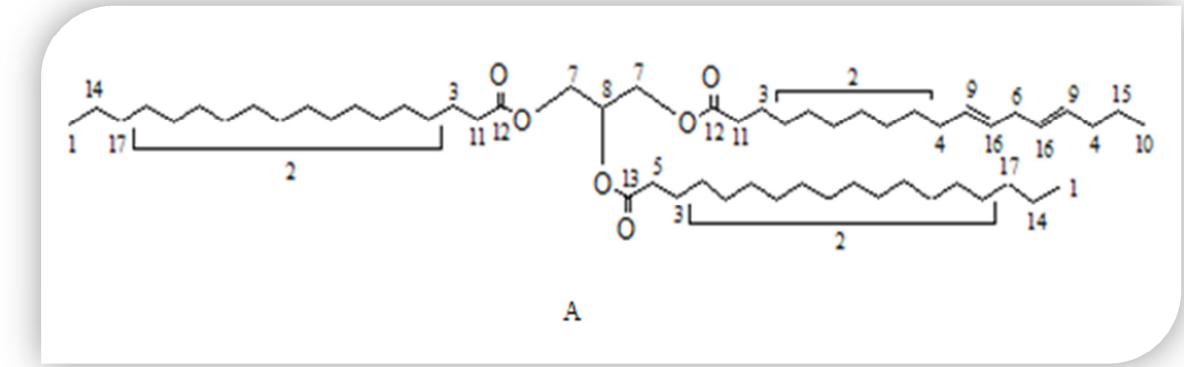
Keywords: *Cassis Sieberiana*, reverse phase chromatography (R P), NMR, Electro Spray Ionization (ESI), triacylglycerol. [1-3].

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 $CHCl_3$ /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 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.



A

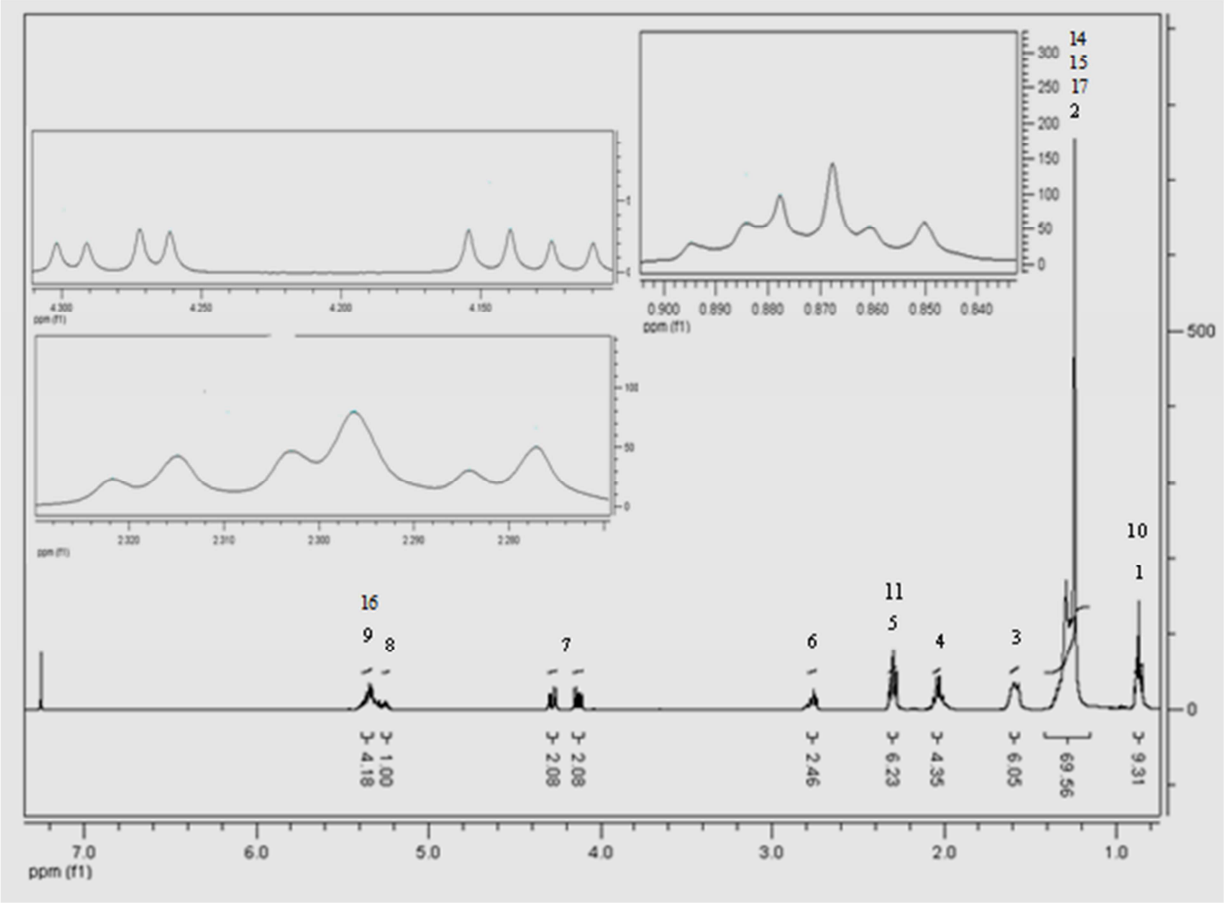


Figure 1: ^{1} HNMR spectrum with spread region (0.90 to 0.84; 2.33 to 2.29 and 4.30 to 4.15) of the compound A

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 ^{1}H–^{1}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 (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_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 15 subfractions (1CS6 to 15CS6). Subfraction 3CS6 ultimately furnished a new compound named A [9.4 mg, Rf 0.90 in TLC (plate RP-18 F254) in n-hexane–EtOAc (8:2), v/v].

Triacylglycerol (A) $C_{57}H_{106}O_6$. ESI-MS m/z: 551 [M - $C_{35}H_{66}O_4$]$^+$; ^{1}HNMR (400 MHz, $CDCl_3$, δ, ppm, J/Hz): 4.28 (2H, dd, J = 5.94 ; 11.88 H-7a), 4.13 (2H, dd, J = 4.32 ; 11.89 H-7b), 5.26 (1H, m, H-8), 2.31 (2H, t, J = 7.54, H-5), 1.57-1.61 (6H, m, H-3), 2.01-2,06 (4H, m, H-4), 5.33-5.36 (4H, m, H-9, 16), 2.81 (2H, m, H-6), 0.87 (6H, t, J = 7.30, H-1), 2.03 (4H, t, J = 7.44, H-11), 1.24-1.29 (66H, bs, H-2, 14, 15, 17), 0.88 (3H, t, J = 6.87, H-10).

^{13}C NMR (100 MHz, $CDCl_3$, δ): 62.09 (C-7), 68.89 (C-8), 34.06 (C-5), 24.87 (C-3), 27.21 (C 4), 127.88-128.08 (C-9), 25.63 (C-6), 14.12 (C-1), 34.20 (C-11), 29.06- 29.71 (C-2), 14.07 (C-10), 172.82 (C-12), 173.28 (C-13), 22.70 (C-14), 22.58 (C-15), 129.98-130.22 (C-16), 31.39 (C-17).

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 plated for better dispersion and easier allocation of signals considered.

The multiplet at δ = 5.26 ppm and the doublets split at δ = 4.31 ppm and 4.15 ppm are characteristic of a glycerol rest [4 - 8].
Analysis of the ^{13}C NMR spectrum indicates the presence of two signals at δ = 173.29 ppm attributed to two carbonyl (C-13) and δ = 172.88 ppm to the carbonyl (C-12) [9- 10].

Signals 14.12 and 14.07 ppm respectively are assigned to the two methyl groups of saturated acyl chains and that of the unsaturated acyl chain triacylglycerol [11- 12].

REFERENCES

[1]J. Mirallès, M. Diop, A. Ferrer and J. M. Kornprobst, Comp. *Biochem. Physiol*., 94 B, 91 **(1989)**.
[2]M. Diop, A. Samb, V. Costantino, E. Fattorusso and A. Mangoni, *J. Nat. Prod.*, 59,271 **(1996)**.
[3]M. S. Diop and A. Samb. C. R. *Chimie* 7, 965 **(2004)**.
[4]D. R. Burdi, M. Hasan V, Uddin, *Pakistan Journal of Pharmaceutical Sciences.*, 4(2) (1991).
[5]M . D i o p , S a m b A , Costantino V, E. Fattorusso and A. Mangoni, *J. Nat. Prod*., 59 (**1996**).
[6]Diaz M. F, Gavin J. A. and Andrade J. B.Grasa., *Yaceities*., 59(3) (**2008**).
[7]G. Knothe, Quantitative analysis of mixtures of fatty compounds by ^{1}H-NMR, *Lipid Technology* **2003**.
[8]Maurizio R., Soma, Martha P. Mims, Mohan V, Chari, Deborah Rees and Joel D. Morrisett, *The journal of biological chemistry*., 267 (1992).
[9]M. B. Luisa, S. Annalaura, High Resolution Nuclear Magnetic Resonance: From Chemical Structure to Food Authenticity, Grasas y Aceites., 53 (**2002**).
[10]Maritza F. Díaz, and José A. Gavín, *J. Braz. Chem. Soc*., 18 (**2007**).
[11]Canonica L, B. Danieli, G. Ferrari, J. Krepinsky, M .Haimova, *Gazetta Chimica Italiana*., 107 (**1977**).
[12]Mantsch H .H, I.C.P. Smith., *Can., J. Chem*., 51 (**1973**).