Chemistry and Materials Research
ISSN 2224- 3224 (Print) ISSN 2225- 0956 (Online)
Vol.9 No.2, 2017
www.iiste.org

Isolation and Identification of Tetra Decahydro Cyclopenta Phenanthren-3-ol from Erica verticillata

Dr. Madian Al-Salem, Suhair Ali
Scientific search in the department of organic - science college –Al-Baath University

Abstract
Phytochemical analysis of the $CHCl_3$ and methanol fractions of Erica verticillata. has led to the isolation and identification of a new tetra decahydro cyclopentaphenanthren-3-ol.The structure elucidation of the compounds was based on spectroscopy data IR, ^{1}H and ^{13}C – NMR, DEPT (90,135), HMQS, COSY and HMBC.
Keywords: Erica, spectroscopy data, phenanthren

1. Introduction

The genus Erica which belongs to the family verticillata, distributed throughout the world, in particular around the dead in the western mountains of the Levant-especially in Syria , Lebanon - on the limestone hills and rocks[1-4].

Erica australis L. (Ericaceae) is used in traditional medicine to treat many free-radical related ailments. In the present work, the stability and biological activity of the plant aqueous extracts submitted to an in vitro digestive process were investigated.

Chemical stability was monitored by HPLC-DAD and LC-MS/MS, while the bioactivities were evaluated through the inhibition of acetylcholinesterase (AChE) and DPPH radical scavenging activity [5].

Erica arborea L. and Erica carnea L.) were performed. Total polyphenols, tannins and flavonoids were determined spectrophotometrically and arbutin content was measured both spectrophotometrically and by HPLC coupled with DAD detection. Antioxidative properites of the ethanolic extracts were tested by means of FRAP (total antioxidant capacity), lipid peroxidation and DPPH free radical scavenging activity. A significant amount of arbutin was detected only in Arbutus unedo. All samples investigated showed excellent antioxidant activity[6].

Figure 1: Erica Verticillata

2. Experimental section

2.1. Materials and Methods:

Melting points were measured on an Electrothermal Entineering melting point apparatus / LTD / and are uncorrected.
^{1}H-NMR,^{13} C-NMR, and IR spectra were recorded on GC-MS-QP 2010 Shimadzu Bruker Ultra Shield 400MHz and Jasco FT-IR 410 respectively.
Rotational evaporator / Buchii /, analyzing preparative plates /TLC/ made of glass and aluminum, painted with Silica gel / Merck /, and solvents / Merck.

2.2. Plant collection and extraction procedure:

Green parts of Erica verticillata were collected from Hama in Syria, in 2016, air-dried (600 g) and extracted with $CHCl_3$. Obtained extracts were combined and concentrated under low pressure, yielding 19.50 g of extract. Fraction (5.0 g) was adsorbed onto silica gel (230 – 400 mesh, ASTM) and subjected to column chromatography (2×120 cm). The column was eluted successively with: n- hexane: benzene (70:30, 600 ml), benzene (400 ml), benzene :chloroform (50:50, 500 ml), and chloroform (600 ml).

tetra decahydro cyclopenta phenanthren-3-ol:

A white solid was obtained from fraction (5gr) of extract and purified on preparative TLC by using of $CHCl_3$: MeOH (99.5 : 0.5, Rf =0.31) mixture and recrystallized from hexane : chloroform mixture to give the compound A (39 mg). The compound is soluble in cold $CHCl_3$ and in hot n- Hexane and Benzene, m.p = 174-177 °C.

IR (KBr) cm-1 : 2431 , 2934 , 2846 , 1638 , 1465 , 1380 , 1056 ^{1}H-NMR and ^{13}C-NMR (CDCl3) δ (ppm) see Table 1.

3.Result and discussion:

Elucidation of structure of tetra decahydro cyclopenta phenanthren-3-ol [7-10]*:*

Tetra decahydro cyclopenta phenanthren-3-ol, compound A,was isolated from the concentrated chloroform extract of the air – dried leaves and flowers of the plant using silica gel column chromatography.

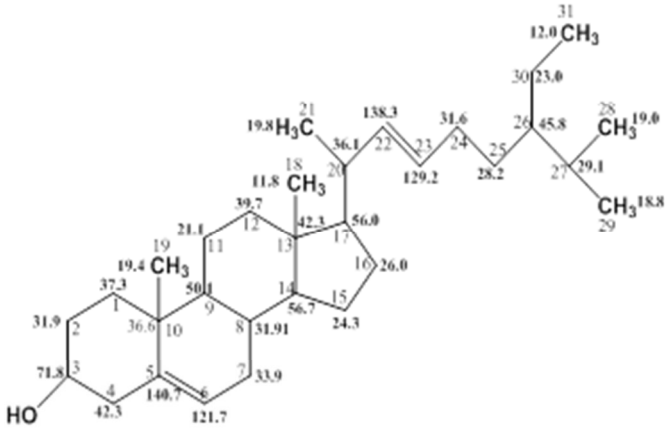

Figure 2: isolate compound A from Erica Verticillata.

The determination of the structure of compound A, was based on the usual spectral methods. Thus, the IR spectrum of 1 shows a broad band at 3431 cm^{-1} (O-H stretching), strong absorption band at 2934-2846 cm^{-1} (C-H stretching), a weak band at 1638 cm^{-1} (C=C stretching), and two medium bands at 1465 cm-1 and 1380 cm^{-1} (CH bending and CH_3 groups)

Moreover, the ^{13}C-NMR of 1, exhibits 31 signals indicating the presence of at least 31 carbon atoms in the molecule. (Table 1, Figure 3) [4-12].

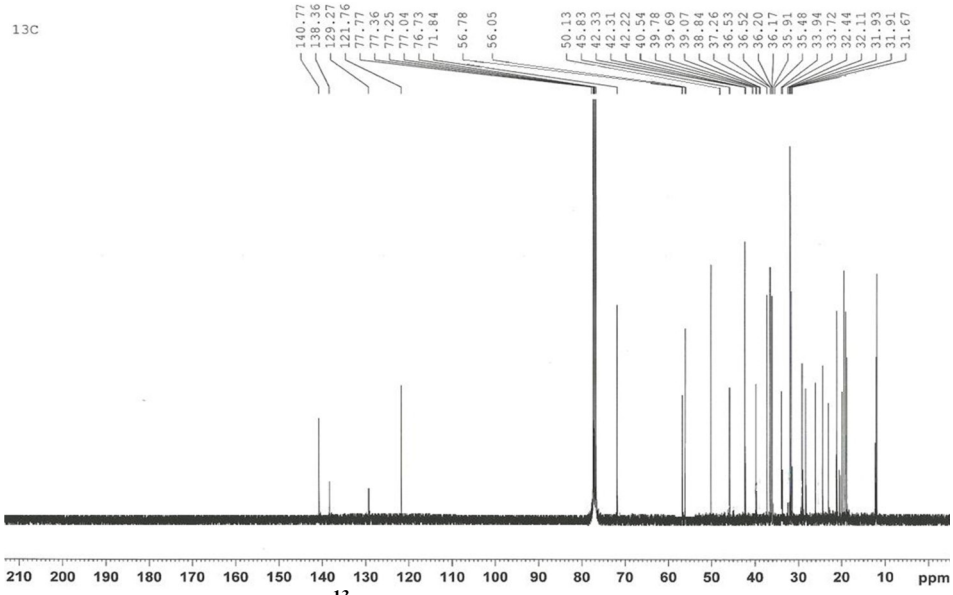

Figure 3: ^{13}C-NMR of compound A in $CDCl_3$

DEPT - 135, and DEPT- 90, however, show that these include 11 secondary, 11 tertiary, 3 quaternary and six primary carbons. (Figure 4,5).

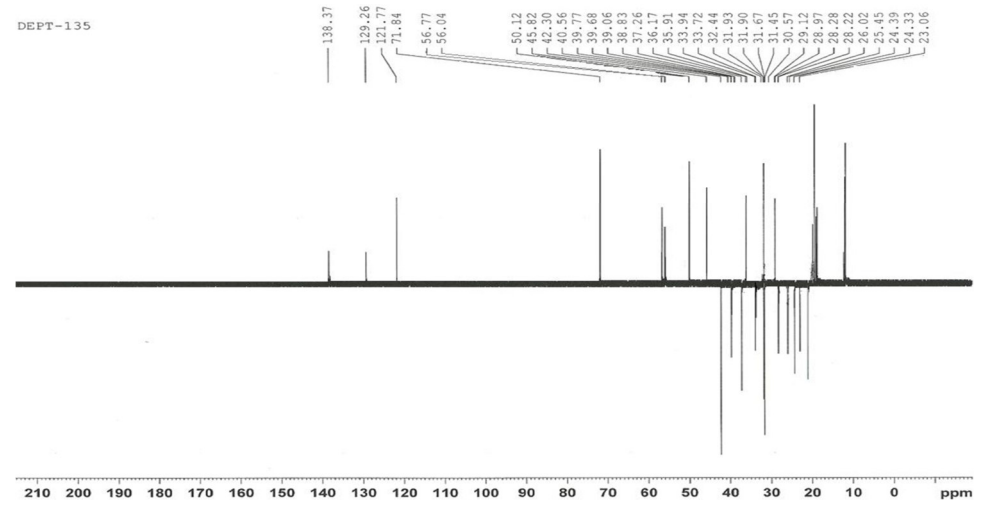

Figure 4: DEPT 135 of compound A in $CDCl_3$

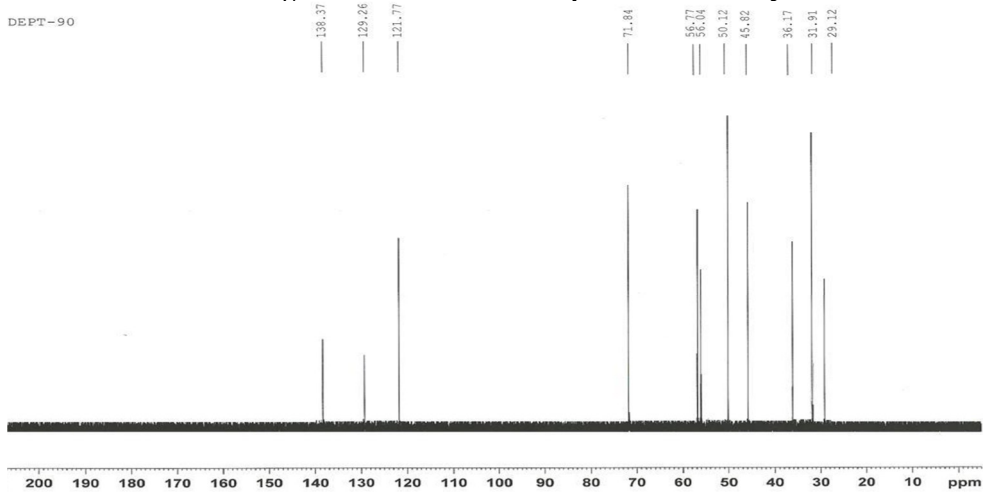

Figure 5: DEPT 90 of compound A in $CDCl_3$

Table 1, ^{1}H-^{1}H COSY and ^{1}H-NMR spectrums also display the spin – spin coupling between different protons (Figure 8,9).

Table 1and HMBC spectrum show the correlations between hydrogen and carbon atoms adjacent to them in compound A (Figure 10). these correlations are shown in (Figure 11).

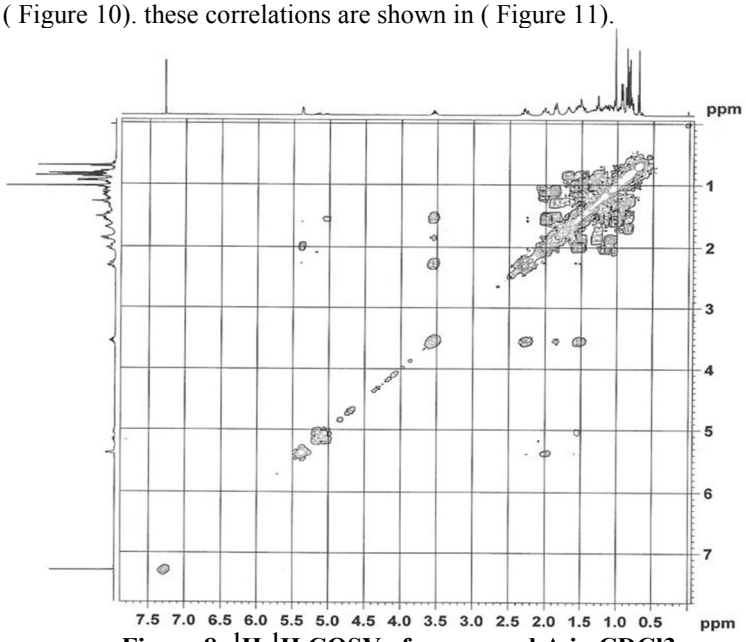

Figure 8: ^{1}H-^{1}H COSY of compound A in CDCl3

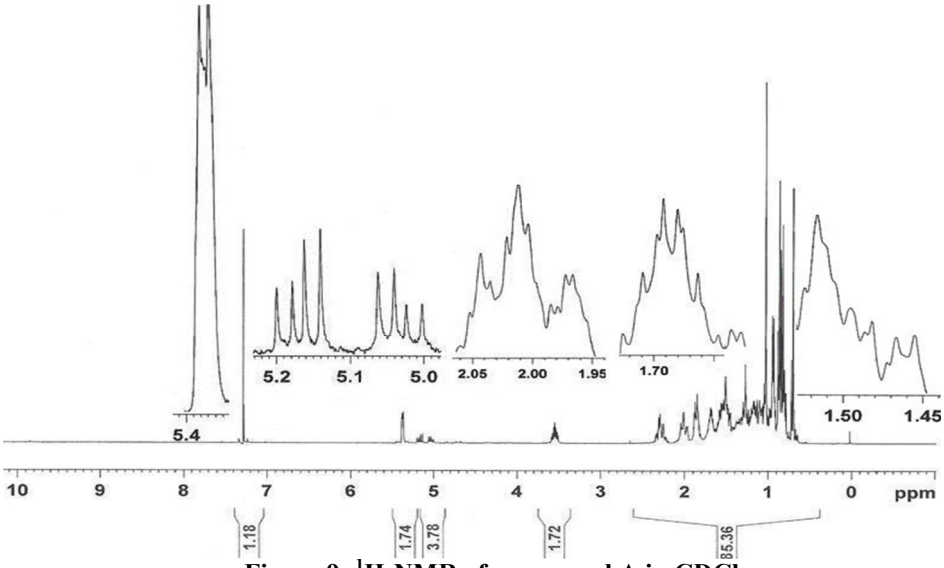

Figure 9: ^{1}H-NMR of compound A in $CDCl_3$

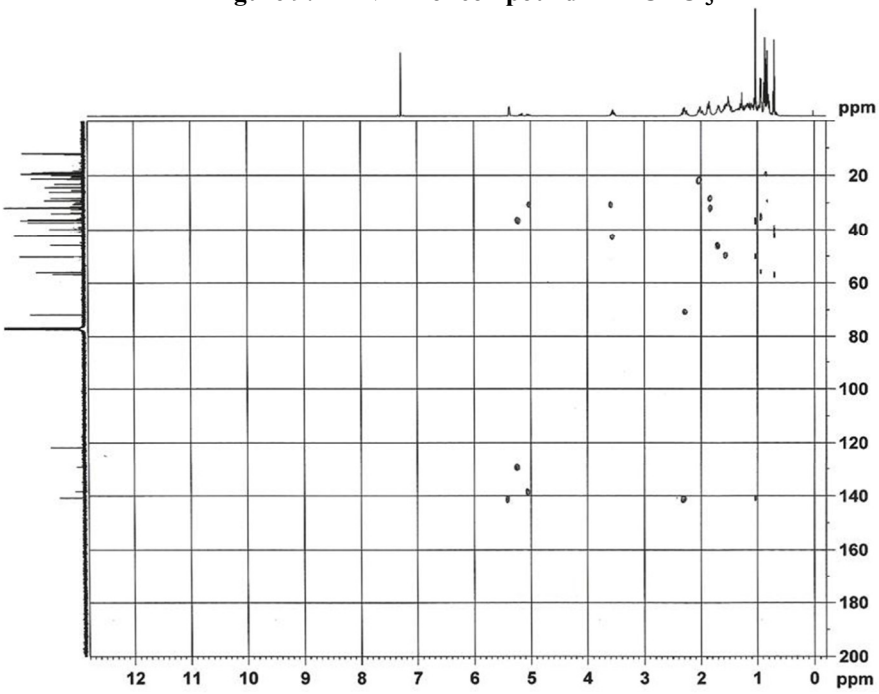

Figure 10: HMBC of compound A in $CDCl_3$

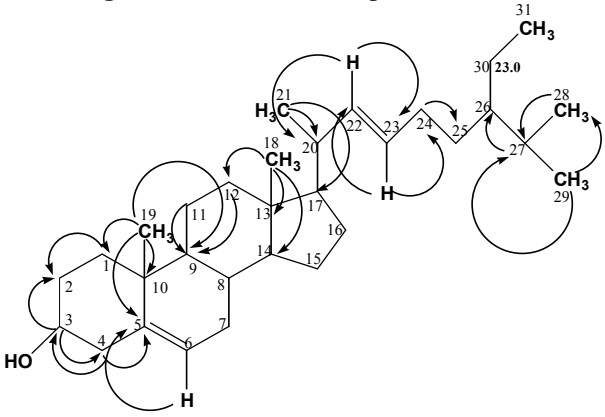

figure 11: HMBC spectroscopy for the compound A.

Table 1: ^{1}H-NMR, ^{13}C-NMR, DEPT, HMQC, COSY and HMBC data of compound A

C	^{13}C δ_C (ppm)	DEPT (135-90)	HMQC δ_H (j =H_Z)	^{1}H-^{1}HCOSY δ_H (ppm)	HMBC
1	37.26	CH2	1.84 -1.1 t j=6.3	3.55	C2
2	31.93	CH2	1.49 d j=6.1	3.55/1.84	
3	71.84	CH	3.55 m		C-4, C2
4	42.33	CH2	2.3 t j=6.2	3.55	C-3, C-5
5	140.77	C	----------	----------	
6	121.76	CH	5.37 d j=5.4		C-5
7	33.94	CH2	1.98 d j=4.3	5.37	
8	31.91	CH	1.96 d j=5.1	5.37	
9	50.13	CH	0.95 d j=6	1.55	
10	36.52	C	----------	----------	
11	21.10	CH2	1.55 m	2.05	C9
12	39.78	CH2	2.05-1.16 t j=4.4	1.55	C11
13	42.31	C	----------	----------	
14	56.78	CH	1.04 m	1.58	
15	24.33	CH2	1.58 m	1.96	
16	26.04	CH2	1.19 m	1.58/2.05	
17	56.05	CH	1.14 m	2.05	
18	11.89	CH3	0.7 s	----------	C-12, C-13, C14
19	19.43	CH3	1.03 s	----------	C-1, C-10, C-9, C5
20	36.17	CH	1.38 m		
21	19.86	CH3	0.94 d j=6.6	1.38	C-17, C-20
22	138.36	CH	5.17 dd j=16-5		C23, C20
23	129.27	CH	5.04 dd j=15-4.6	5.17	C-22, C24
24	31.67	CH2	1.88-1.48 d j=4	5.04	C-25
25	28.28	CH2	1.68 m		
26	45.83	CH	0.97 d j=5.3	1.28/1.48	
27	29.13	CH	1.69 qd j=7-2		C26
28	19.05	CH3	0.86 d j=6.1		C27
29	18.80	CH3	0.84 d j=4.5	1.28/1.69	C-27, C28
30	23.07	CH2	1.28 m	1.88	
31	12.01	CH3	0.82 t j=4.6		

Conclusions

In summary, We demonstrated in this article compound identity vertic tetra decahydro cyclopenta phenanthren-3-ol which is a new compound. the compound A seem a white solid, fully dissolved in chloroform, and purified on preparative TLC by using of $CHCl_3$: MeOH (99.5 : 0.5, Rf =0.31) mixture.

Acknowledgment

The author would like to thank Prof . Dr. Anwar Al- Khatib for his help in classifying the studied plant, and his gratitude to Dr. Thanaa shriteh for his useful remarks.

4. Reference

[1]. H.; Taher , 2008 , "Natural Products Chemistry." , Science Faculty , Al-Baath university , Syria , P. : 88, 304 .
[2]. Mouhamad ,A., the semitic plant in Syria, journal of living science,1982.
[3]. Fariha, A., herbal medicine,1991.
[4]. Jaber Al-Qahtani, forums center social forums.
[5]. Pilar,D.,Pedro,L.,Alice,M.,Amilia.p.,R., of Erica australis L. Aqueous Extracts and Their Therapeutic Potential as Acetylcholinesterase Inhibitors, Evidence-Based Complementary and Alternative Medicine,V.2015,P.1-7.
[6]. Pavlovic´,R. D., Lakusˇic .,B. Z. Dosˇlov-Kokorus , Kovacˇevic, N.,2009 Arbutin content and antioxidant activity of some Ericaceae species, Pharmazie,V. 64,N.10, P.656-659.
[7]. E.; Pretsch , P.;Bühlmann , C.; Affolter ," Structure Detrmination of Organic Compounds Table of Spectral Data" , Springer , Verlag , Berlin , 3rd English Edition , 2000 ,72, 250-259.
[8]. Silverstein R.M. , Webster E., "Spectrometric Identification of Organic Compounds." 6th Edition ,1996.
[9]. Breitmaier, Eberhard, Terpenes, Copyright © 2006 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim

ISBN: 3-527-31786-4
[10]. Abiodun, F.; Sajjad, A.; Irfan, M. Q.; Iqbal, M. C., Jou. Med. Plants Res., 2008, 2(12), 365-369.
[11]. Eman, M. E.; Huda, G., Taiwan Pharmaceutical Journal, 2007, 59, 113-132.
[12]. Kikuchi, T.; Ueda, S.; Kanazawa, J.; Naoe, H.; Yamada, T.; Tanaka, R . Three New Triterpene Esters from Pumpkin (*Cucurbita maxima*) Seeds . *Molecules,* 2014*,* , *19*(4), 4802-4813.SILVERSTEIN, R.; BASSLER, G.;MORRILLl,T. , 2005-Spectrometric Identification of Organic Compounds . John Wiley & Sons Inc. , New York, 7th Edition , 502p.