

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

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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, ^1H 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 properties 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 Engineering melting point apparatus / LTD / and are uncorrected.

^1H -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).

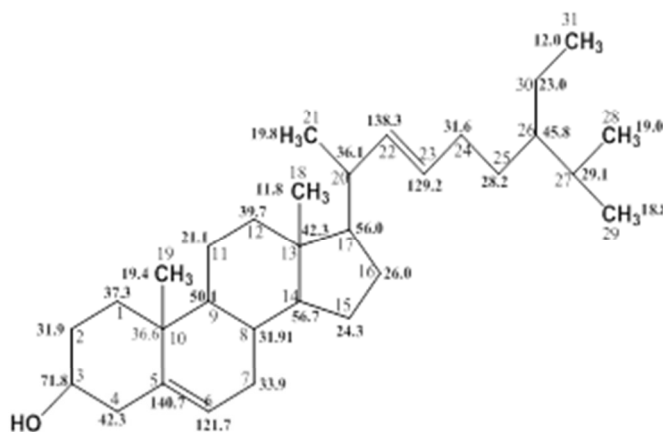
tetra decahydro cyclopenta phenanthren-3-ol:

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, $R_f=0.31$) 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 $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ (CDCl_3) 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.



Compound A

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 $2934\text{-}2846\text{ cm}^{-1}$ (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 $^{13}\text{C-NMR}$ of 1, exhibits 31 signals indicating the presence of at least 31 carbon atoms in the molecule. (Table 1, Figure 3) [4-12].

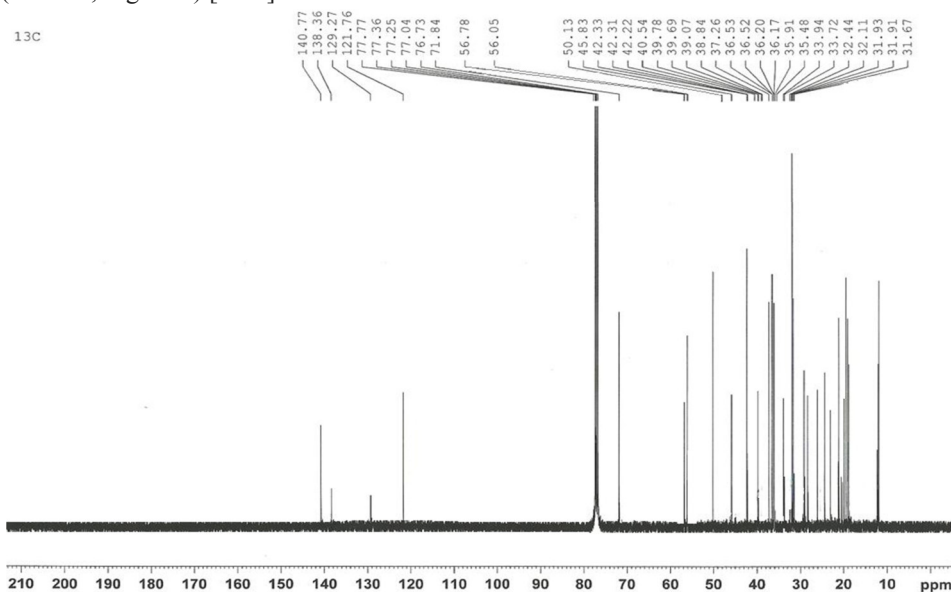


Figure 3: $^{13}\text{C-NMR}$ of compound A in CDCl_3

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

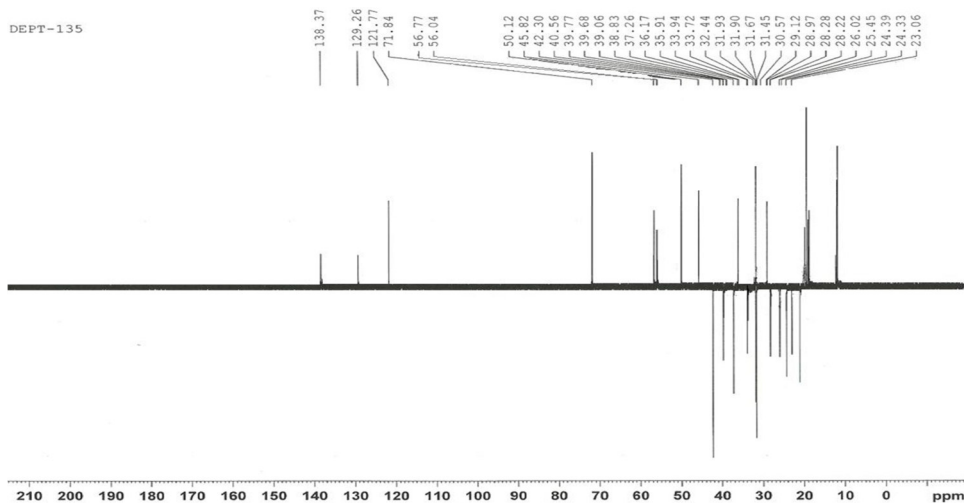


Figure 4: DEPT 135 of compound A in $CDCl_3$

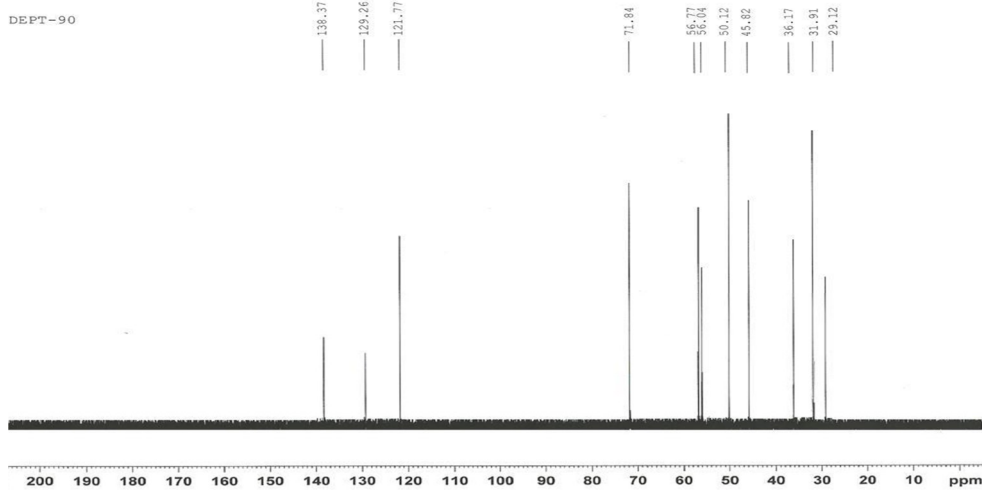


Figure 5: DEPT 90 of compound A in $CDCl_3$

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

Table 1 and 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).

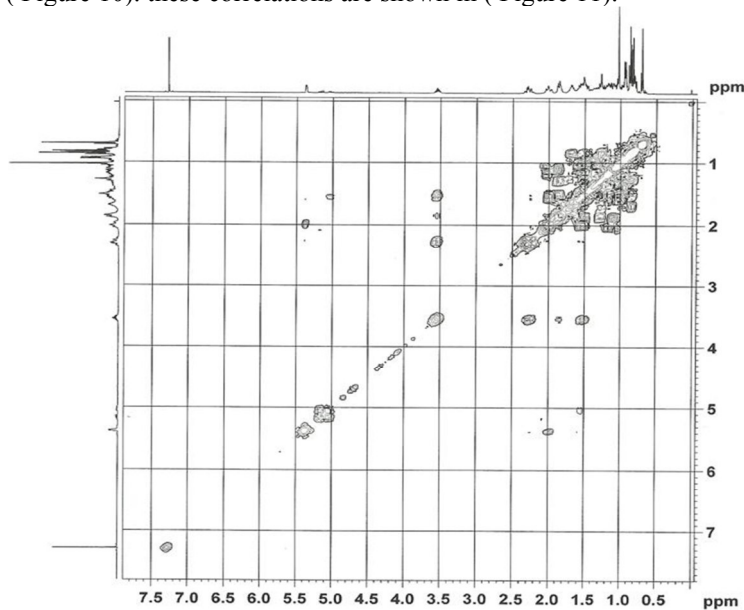


Figure 8: 1H - 1H COSY of compound A in $CDCl_3$

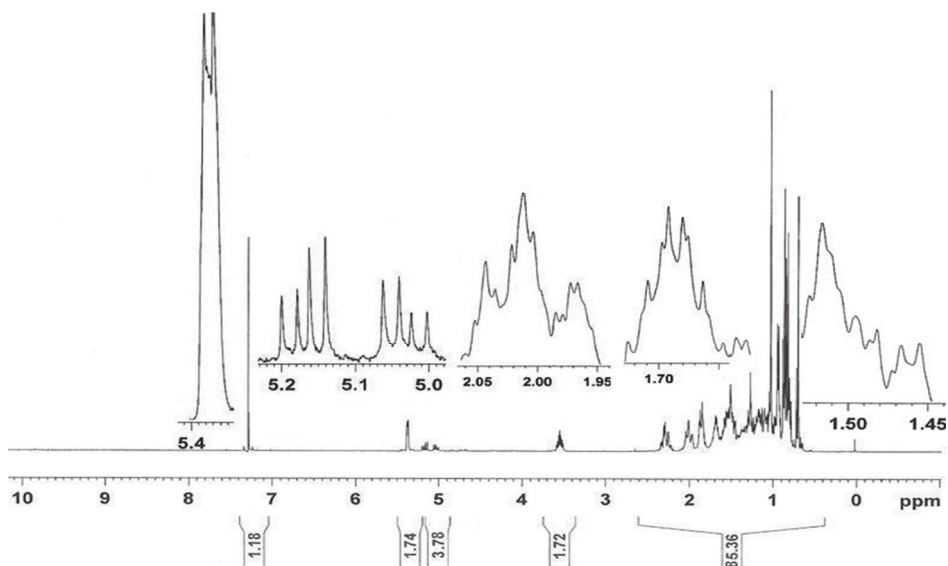


Figure 9: $^1\text{H-NMR}$ of compound A in CDCl_3

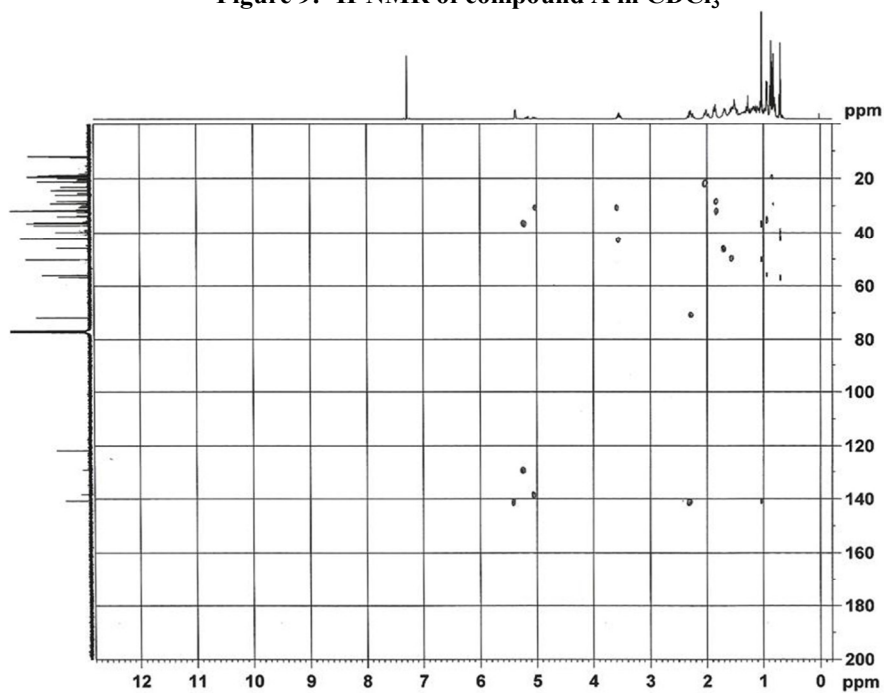


Figure 10: HMBC of compound A in CDCl_3

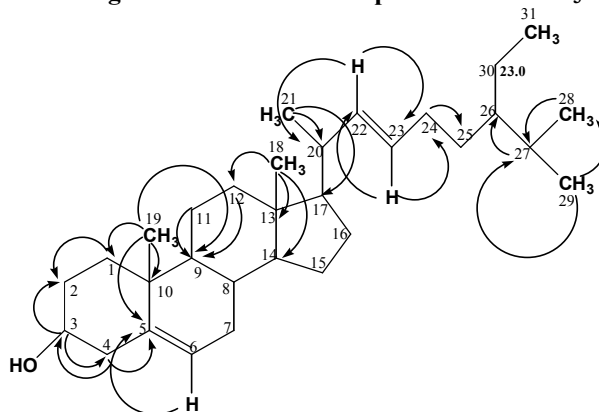


figure 11: HMBC spectroscopy for the compound A.

Table 1: ¹H-NMR, ¹³C-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 CHCl₃: MeOH (99.5 : 0.5, R_f=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., Lakus´ic ´,B. Z. Dos´lov-Kokorus , Kovac´evic , 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.; MORRILLI, T. , 2005-Spectrometric Identification of Organic Compounds . John Wiley & Sons Inc. , New York, 7th Edition , 502p.