

# Isolation and Identification of Benzyl Hepta Methyl Docosahydropicene Carboxylic Acid from Erica Verticillata

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## Abstract

Phytochemical analysis of the  $\text{CH}_2\text{Cl}_2$  fractions of Erica verticillata. has led to the isolation and identification of a new Benzyl hepta methyl docosahydropicene carboxylic acid. The structure elucidation of compound was based on spectroscopy data IR,  $^1\text{H}$  and  $^{13}\text{C}$  – NMR, DEPT (90,135),  $^1\text{H}$ - $^1\text{H}$  COSY and HETCOR.

**Keywords:** Erica, docosahydropicene , hepta methyl

## 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.

$^1\text{H}$ -NMR,  $^{13}\text{C}$ -NMR, and IR spectra were recorded on 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 2015, and air-dried (500 g) were extracted three times with  $\text{CH}_2\text{Cl}_2$ . The extracts were combined and concentrated under low pressure to give 48.28g of extract. It was taken (22.05) gr of extract to treat with  $\text{Et}_2\text{O}$  to give soluble part and another part is insoluble.

The part soluble in  $\text{Et}_2\text{O}$  concentrated under vacuum to give 9.02 g of its 3.02 g from the part soluble in  $\text{Et}_2\text{O}$  were loaded on chromatographic column (2 cm. diameter, 120 cm. long) over silica gel ( 230 – 400 mesh, ASTM ).

The column was eluted successively with N- hexane: chloroform (50 : 50, 600 ml), N- hexane (400 ml.) and chloroform (600 ml.).

**Benzyl hepta methyl docosahydronic acid**: was obtained from the second fraction, purified by preparative TLC (N- hexane: chloroform, 60 : 40,  $R_f = 0.83$ ) and white crystalline solid, soluble in cold  $\text{CHCl}_3$ , and in hot N-Hexane and chloroform, m.p=134-135°C, 26 mg.

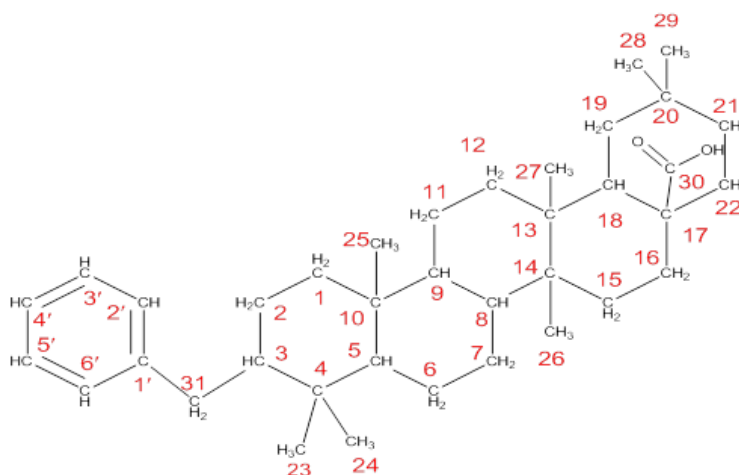
IR ( KBr )  $\text{cm}^{-1}$  : 3442 , 2920 , 2850 , 1737,, 1636 1466 , 1384.

$^1\text{H}$ NMR ,  $^{13}\text{C}$ NMR ( $\text{CDCl}_3$ )  $\delta$  (ppm) see Table (1).

### 3.Result and discussion:

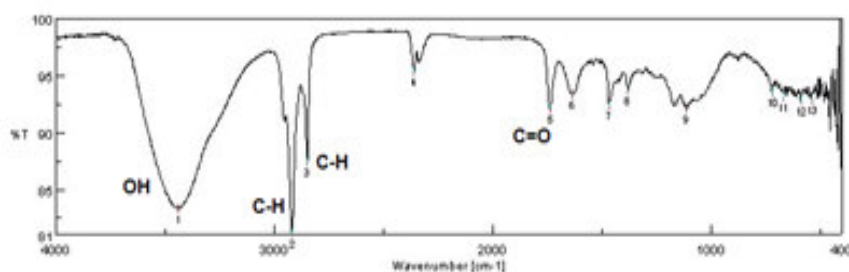
#### Elucidation of structure of Benzyl hepta methyl docosahydronic acid [7-9]:

Benzyl hepta methyl docosahydronic acid, was isolated from the concentrated dichloromethane extract of the air – dried leaves and flowers of the plant using silica gel column chromatography.



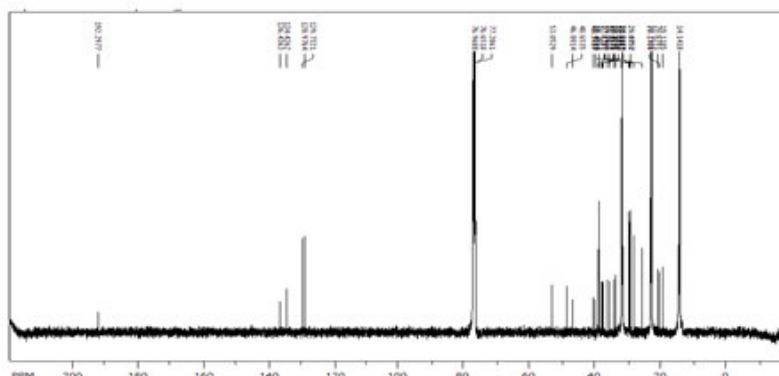
**Figure 2:** Benzyl hepta methyl docosahydronic acid extracted from Erica.

The determination of the structure of Benzyl hepta methyl docosahydronic acid, was based on the usual spectral methods. Thus, the IR spectrum of **3** shows a broad band at  $3442 \text{ cm}^{-1}$  (O-H stretching), strong absorption band at  $2850\text{-}2920 \text{ cm}^{-1}$  (C-H stretching), a weak band at  $1634 \text{ cm}^{-1}$  (C=C stretching), and two medium bands at  $1466 \text{ cm}^{-1}$  and  $1384 \text{ cm}^{-1}$  (CH bending and  $\text{CH}_3$  groups) and C=O stretching  $1737 \text{ cm}^{-1}$ .



**Figure 3:** IR of Benzyl hepta methyl docosahydronic acid in KBr

Moreover, the  $^{13}\text{C}$ -NMR of the isolated compound, exhibits 32 signals indicating the presence of at least 32 carbon atoms in the molecule . (Table 1, Figure 4).

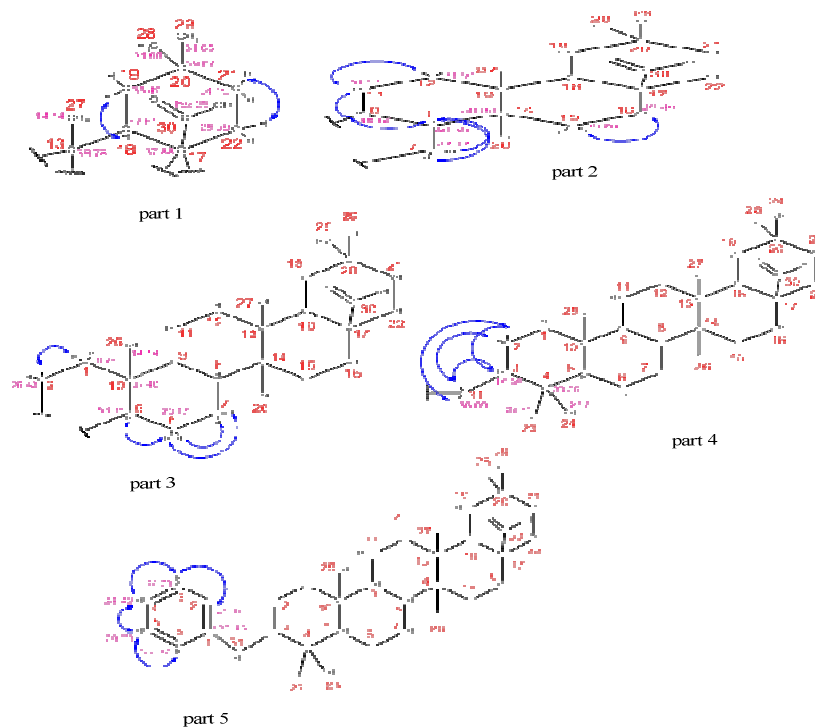


**Figure 4:**  $^{13}\text{C}$ -NMR of Benzyl hepta methyl docosahydronic acid in  $\text{CDCl}_3$

DEPT - 135, and DEPT- 90, however, show that these include 12 secondary, 8 tertiary, 8 quaternary and quater primary carbons (Figure 5,6).







**figure 10:** HETCOR spectroscopy for Benzyl hepta methyl docosahydric acid.  
**Tab(1):** <sup>1</sup>H-NMR, <sup>13</sup>C-NMR, DEPT, , COSY and HETCOR data of Benzyl hepta methyl docosahydric acid

COSY	HETCOR	<sup>13</sup> C NMR	Dept 90-135	No
H <sub>2a</sub> = 0.9867	H <sub>1a</sub> =1.3602	39.75	CH <sub>2</sub>	1
-----	H <sub>1b</sub> =1.8040			
H <sub>1a</sub> =1.3602 H <sub>3</sub> = 0.9062 H <sub>31b</sub> =1.6506	H <sub>2a</sub> = 0.9867	25.43	CH <sub>2</sub>	2
H <sub>31a</sub> = 1.3056	H <sub>2b</sub> =1.4540			
H <sub>2a</sub> = 0.9867 H <sub>31a</sub> = 1.3056	H <sub>3</sub> =0.9062	40.39	CH	3
-----	-----	33.55	C	4
H <sub>6b</sub> =0.9453	H <sub>5</sub> =1.8940	53.05	CH	5
H <sub>7a</sub> = 1.1600	H <sub>6a</sub> = 0.8705	20.17	CH <sub>2</sub>	6
H <sub>7b</sub> =1.5940 H <sub>5</sub> = 1.8940	H <sub>6b</sub> =0.9453			
H <sub>6a</sub> = 0.8705 H <sub>8</sub> = 0.8811	H <sub>7a</sub> = 1.1600	27.97	CH <sub>2</sub>	7
H <sub>6b</sub> =0.9453 H <sub>8</sub> = 0.8811	H <sub>7b</sub> =1.5940			
H <sub>7a</sub> = 1.1600 H <sub>7b</sub> =1.5940 H <sub>9</sub> = 0.8952	H <sub>8</sub> =0.8811	46.80	CH	8
H <sub>11a</sub> =1.0451 H <sub>11b</sub> =1.1227 H <sub>8</sub> = 0.8811	H <sub>9</sub> = 0.8952	48.65	CH	9
-----	-----	37.40	C	10

H <sub>9</sub> = 0.8952	H <sub>11a</sub> =1.0451	20.63	CH <sub>2</sub>	11
H <sub>9</sub> = 0.8952	H <sub>11b</sub> =1.1227			
H <sub>11a</sub> =1.0451 H <sub>11b</sub> =1.1227	H <sub>12a</sub> =1.3318	31.91	CH <sub>2</sub>	12
-----	H <sub>12b</sub> = 1.7984			
-----	-----	38.78	C	13
-----	-----	38.98	C	14
H <sub>16a</sub> =1.0719 H <sub>16b</sub> =1.0969	H <sub>15a</sub> =1.4501	29.70	CH <sub>2</sub>	15
-----	H <sub>15b</sub> =1.5311			
H <sub>15a</sub> =1.4501	H <sub>16 a</sub> =1.0719	29.49	CH <sub>2</sub>	16
H <sub>15a</sub> =1.4501	H <sub>16 b</sub> =1.0969			
-----	-----	37.88	C	17
H <sub>19a</sub> =1.2829	H <sub>18</sub> = 1.2590	37.61	CH	18
H <sub>18</sub> = 1.2590	H <sub>19a</sub> =1.2829	35.42	CH <sub>2</sub>	19
-----	H <sub>19b</sub> = 1.4644			
-----	-----	29.07	C	20
H <sub>22 b</sub> =1.2701	H <sub>21a</sub> =1.2400	34.15	CH <sub>2</sub>	21
	H <sub>21b</sub> = 1.3515			
-----	H <sub>22a</sub> =0.9607	29.39	CH <sub>2</sub>	22
H <sub>21 a</sub> = 1.2400	H <sub>22 b</sub> =1.2701			
-----	H <sub>23</sub> = 1.3411 s	22.71	CH <sub>3</sub>	23
-----	H <sub>24</sub> = 1.2949 s	22.71	CH <sub>3</sub>	24
-----	H <sub>25</sub> = 1.1126 s	14.14	CH <sub>3</sub>	25
-----	H <sub>26</sub> = 1.1430 s	19.11	CH <sub>3</sub>	26
-----	H <sub>27</sub> = 0.7582 s	14.14	CH <sub>3</sub>	27
-----	H <sub>28</sub> = 1.4042 s	31.65	CH <sub>3</sub>	28
-----	H <sub>29</sub> = 1.3194 s	31.65	CH <sub>3</sub>	29
-----	10.07 s	192.29	C	30
H <sub>2 b</sub> =1.4540 H <sub>3</sub> = 0.9062	H <sub>31a</sub> = 1.3056	36.09	CH <sub>2</sub>	31
H <sub>2 a</sub> = 0.9867	H <sub>31b</sub> =1.6506			
-----	-----	136.43	C	1'
7.50	7.9 d	128.97	CH	2'
7.25 , 7.9	7.50 d	129.73	CH	3'
7.50	7.25 d	134.42	CH	4'
7.25 ,7 .9	7.50 d	129.73	CH	5'
7.50	7.9 d	128.97	CH	6'

### Conclusions

In summary, We demonstrated in this article compound identity vertic Benzyl hepta methyl docosahydronicene carboxylic acid which is a new compound. the compound seem a white crystal, fully dissolved in chloroform, and purified on preparative TLC by using of CHCl<sub>3</sub>: N- hexane ( 50 : 50 , R<sub>f</sub> = 0.83) mixture.

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