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

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Abstract
Phytochemical analysis of the CH₂Cl₂ 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, ¹H and ¹³C – NMR, DEPT (90,135), ¹H-¹H 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].

2. Experimental section
2.1. Materials and Methods:
Melting points were measured on an Electrothermal Entineering melting point apparatus / LTD / and are uncorrected.
¹H-NMR, ¹³C-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 CH₂Cl₂. 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 Et₂O to give soluble part and another part is insoluble.
The part soluble in Et₂O concentrated under vacuum to give 9.02 g of its 3.02 g from the part soluble in Et₂O 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 docosahydropicene carboxylic 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 CHCl_3, and in hot N-Hexane and chloroform, m.p=134-135°C, 26 mg.

IR (KBr) cm⁻¹: 3442, 2920, 2850, 1737, 1636, 1466, 1384.

**1HNMR, 13CNMR (CDCl_3) δ (ppm) see Table (1).**

3. Result and discussion:

**Elucidation of structure of Benzyl hepta methyl docosahydropicene carboxylic acid [7-9]:**

Benzyl hepta methyl docosahydropicene carboxylic 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 docosahydropicene carboxylic acid extracted from Erica.

The determination of the structure of Benzyl hepta methyl docosahydropicene carboxylic acid, was based on the usual spectral methods. Thus, the IR spectrum of 3 shows a broad band at 3442 cm⁻¹ (O-H stretching), strong absorption band at 2850-2920 cm⁻¹ (C-H stretching), a weak band at 1634 cm⁻¹ (C=C stretching), and two medium bands at 1466 cm⁻¹ and 1384 cm⁻¹ (CH bending and CH_3 groups) and C=O stretching 1737 cm⁻¹.

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

Moreover, the 13C-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:** 13C-NMR of Benzyl hepta methyl docosahydropicene carboxylic acid in 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 5: DEPT- 135 of Benzyl hepta methyl docosahydropicene carboxylic acid in CDCl₃

Figure 6: DEPT- 90 of Benzyl hepta methyl docosahydropicene carboxylic acid in CDCl₃

$^1$H-$^1$H COSY and $^1$H-NMR spectrums also display the spin – spin coupling between different protons (Figure 7,8).

Table 1 and HETCOR spectrum show the correlations between hydrogen groups and carbon atoms adjacent to them in benzyl hepta methyl docosahydropicene carboxylic acid (Figure 9). These correlations are shown in (Figure 10)[10].

Figure 7: $^1$H-$^1$H COSY of Benzyl hepta methyl docosahydropicene carboxylic acid in CDCl₃
Figure 8: \( ^1\)H-NMR of Benzyl hepta methyl docosahydropicene carboxylic acid in CDCl\(_3\)

Figure 9: HETCOR of Benzyl hepta methyl docosahydropicene carboxylic acid in CDCl\(_3\)
**figure 10:** HETCOR spectroscopy for Benzyl hepta methyl docosahydropicene carboxylic acid.

**Tab(1):** $^1$H-NMR, $^{13}$C-NMR, DEPT, COSY and HETCOR data of Benzyl hepta methyl docosahydropicene carboxylic acid

<table>
<thead>
<tr>
<th>COSY</th>
<th>HETCOR</th>
<th>$^{13}$C NMR</th>
<th>Dept 90-135</th>
<th>N o</th>
</tr>
</thead>
<tbody>
<tr>
<td>H$_{13a}$ = 0.9867</td>
<td>H$_{10}$ = 1.3602</td>
<td>39.75</td>
<td>CH$_2$</td>
<td>1</td>
</tr>
<tr>
<td>-----------</td>
<td>------------</td>
<td>-----------</td>
<td>-----------</td>
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</tr>
<tr>
<td>H$_{14}$ = 1.3602</td>
<td>H$_{11b}$ = 1.8040</td>
<td>H$_{2a}$ = 0.9867</td>
<td>25.43</td>
<td>CH$_2$</td>
</tr>
<tr>
<td>H$_{3}$ = 0.9062</td>
<td>H$_{3b}$ = 1.6506</td>
<td>H$_{3}$ = 1.4540</td>
<td>33.55</td>
<td>C</td>
</tr>
<tr>
<td>H$_{11a}$ = 1.3056</td>
<td>H$_{11b}$ = 1.3056</td>
<td>H$_{3}$ = 0.9062</td>
<td>40.39</td>
<td>CH</td>
</tr>
<tr>
<td>H$_{12a}$ = 0.9453</td>
<td>H$_{12b}$ = 1.8040</td>
<td>H$_{9}$ = 0.9453</td>
<td>20.17</td>
<td>CH$_2$</td>
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<tr>
<td>H$_{13}$ = 1.1600</td>
<td>H$_{14}$ = 0.8705</td>
<td>H$_{1}$ = 0.8705</td>
<td>27.97</td>
<td>CH$_2$</td>
</tr>
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<td>H$_{14}$ = 1.5940</td>
<td>H$_{15}$ = 1.8940</td>
<td>H$_{8}$ = 0.9453</td>
<td>H$_{7}$ = 1.1600</td>
<td>20.17</td>
</tr>
<tr>
<td>H$_{15}$ = 0.8705</td>
<td>H$_{16}$ = 0.8811</td>
<td>H$_{7}$ = 1.1600</td>
<td>H$_{3}$ = 1.4540</td>
<td>33.55</td>
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<tr>
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<td>H$_{17}$ = 1.5940</td>
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<td>20.17</td>
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</tr>
<tr>
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<td>H$_{18}$ = 0.9453</td>
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<td>48.65</td>
<td>CH</td>
</tr>
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<td>H$_{19}$ = 1.0451</td>
<td>H$_{11}$ = 1.1227</td>
<td>H$_{9}$ = 0.8952</td>
<td>H$_{2}$ = 0.8811</td>
<td>37.40</td>
</tr>
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</table>
Conclusions
In summary, We demonstrated in this article compound identity vertic Benzyl hepta methyl docosahydropicene 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 CHCl₃: N- hexane ( 50 : 50 , Rₖ = 0.83) mixture.

Acknowledgment
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4. Reference

[4]. Jaber Al-Qahtani, forums center social forums.