Chemistry and Materials Research
ISSN 2224- 3224 (Print) ISSN 2225- 0956 (Online)
Vol.8 No.3, 2016
www.iiste.org

Isolation a Novel One Derivatives Taraxastene Ethyl benzoate from Erica verticillata

Dr. Thanaa shriteh , Suhair Ali
Dr. for organic chemistry : Department of chemistry-Faculty of science-albaath university, SYRIA

Abstract
The present paper discusses the elucidation of the structure of the novel taraxastene Ethyl benzoate, as well as, the identification of Taraxastenes.The compound have been isolated for the first time from Erica verticillata a plant collected from Hama area in Syria. The structure elucidation of the Taraxastene Ethyl benzoate was based on spectroscopy data IR, 1H-NMR, 13C-NMR, DEPT (90,135) , HETCOR , and 1H-1HCOSY.
Keywords: Erica verticillata , Ethyl benzoate , Taraxastenes

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 verticillata is one of the perennial species. The branches and flowers headers contain active substances medically, they tannins , Alorboaten , Ericolin acid, Aarsen, Kobrkstin and Ersenaul, these are the material diuretic, analgesic for pain, to treat inflammation of the bladder and prostate problems, neurological disorders, and to address insomnia and some pharmacological interventions chemical cancer treatment, where it is used effectively drink one cup twice a day[3,4].

In 1920 it has been noted that there Aloorcolik acid in Erica tetralix.L [3], in 1930 confirmed Fisher and Lauer presence Aloorcolik acid in many types of the genus Erica [5], and in 1975 conducted a study in Alterpnat triple to plant heather ((Erica Verticillata Forssk As a result of this study was to isolate and prove the identities of four Terpnat trio of Conclusion Alkruformah the husks and leaves and flowers of this plant are: Alfredelan and Alleopol and acid Aloorcelik and Albotlin[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.
1H-NMR,13 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:

In addition to the round-bottom flask provider condenser for boiling the apostate (12mmole) of azido benzyl or its derivatives, quomarine (8mmole) and solvent P- xylene or THF (depending on the solubility of the reactants). And leave the mix for a period of time (the time required to form enaminolactones) at the boiling point of the solvent

(the track the progress of the reaction using thin layer chromatography), separated the products and separated by filtration.

Green parts of Erica verticillata were collected from Hama in Syria, in 2014, and air-dried (500 g) were extracted three times with CH_2Cl_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 Et_2O to give soluble part and another part is insoluble.

The part soluble in Et_2O concentrated under vacuum to give 9.02 g of its

3.02 g from the part soluble in Et_2O 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.).

Hydroxy methyl taraxastene Ethyl benzoate: 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, 164-166°C, 39 mg.

IR (KBr) cm^{-1} : 3444 , 2919 , 2850 , 1634, 1470 , 1384 , 1350.

^{1}HNMR , ^{13}CNMR ($CDCl_3$) δ (ppm) see Table (1).

3. Result and discussion:

Elucidation of structure of Hydroxy methyl taraxastene Ethyl benzoate [7-9]*:*

Hydroxy methyl taraxastene Ethyl benzoate, was isolated from the concentrated dichloromethane extract of the air – dried leaves and flowers of the plant using silica gel column chromatography.

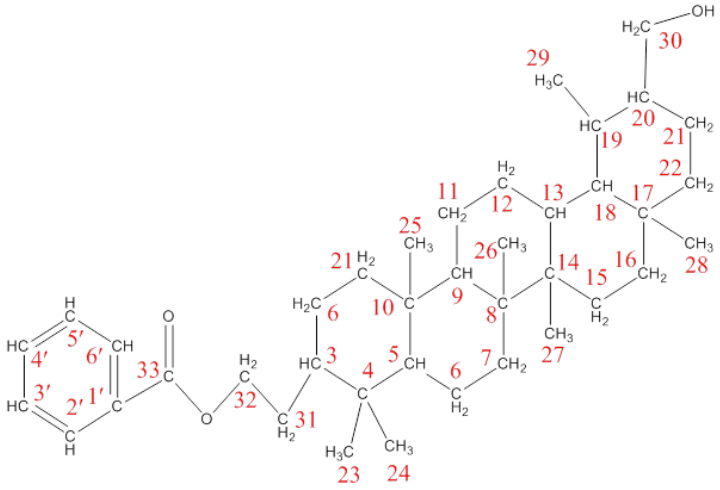


Figure 2: isolate compound from Erica Verticillata.

The determination of the structure of Hydroxy methyl taraxastene Ethyl benzoate, was based on the usual spectral methods. Thus, the IR spectrum of **3** shows a broad band at 3444 cm^{-1} (O-H stretching), strong absorption band at 2919-2850 cm^{-1} (C-H stretching), a weak band at 1634 cm^{-1} (C=C stretching), and two medium bands at 1470 cm^{-1}and1350 cm^{-1} (CH bending and CH_3 groups),C-O stretching 1384 $cm^{-1}$and C=O stretching 1737cm^{-1}.

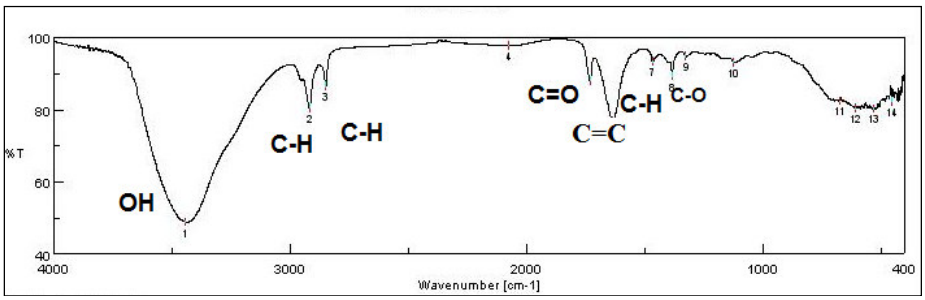


Figure 3: IR of Hydroxy methyl taraxastene Ethyl benzoate in KBr

Moreover, the ^{13}C-NMR of the isolated compound, exhibits 37 signals indicating the presence of at least 37 carbon atoms in the molecule . (Table 1, Figure 4).

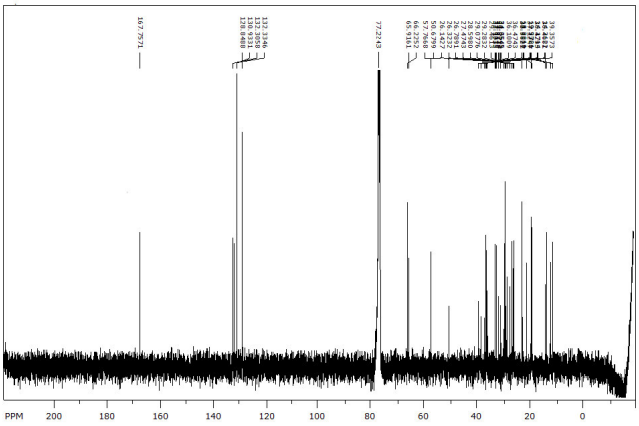


Figure 4: ^{13}C-NMR of Hydroxy methyl taraxastene Ethyl benzoate in $CDCl_3$

DEPT - 135, and DEPT- 90, however, show that these include 13 secondary, 10 tertiary, 7 quaternary and seven primary carbons (Figure 5,6).

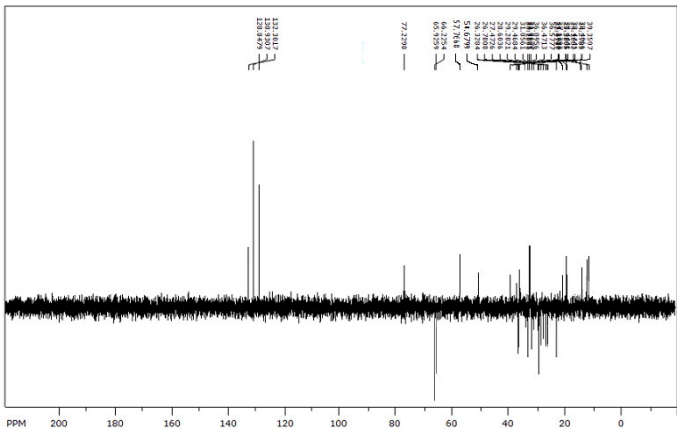


Figure 5: DEPT- 135 of Hydroxy methyl taraxastene Ethyl benzoate in $CDCl_3$

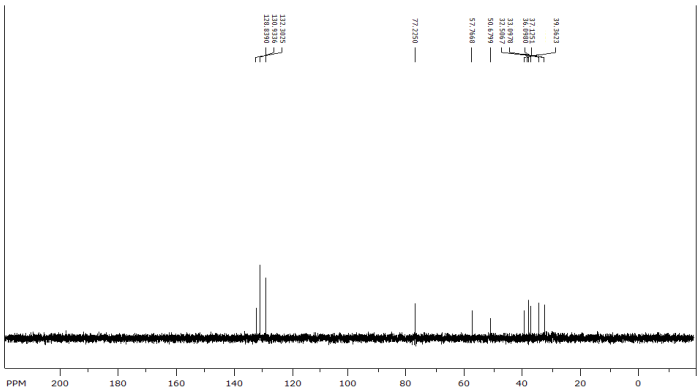


Figure 6: DEPT- 90 of Hydroxy methyl taraxastene Ethyl benzoate in $CDCl_3$

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

Table 1and HETCOR spectrum show the correlations between hydrogen groups and carbon atoms adjacent to them in Hydroxy methyl taraxastene Ethyl benzoate (Figure 9). these correlations are shown in (Figure 10)[10].

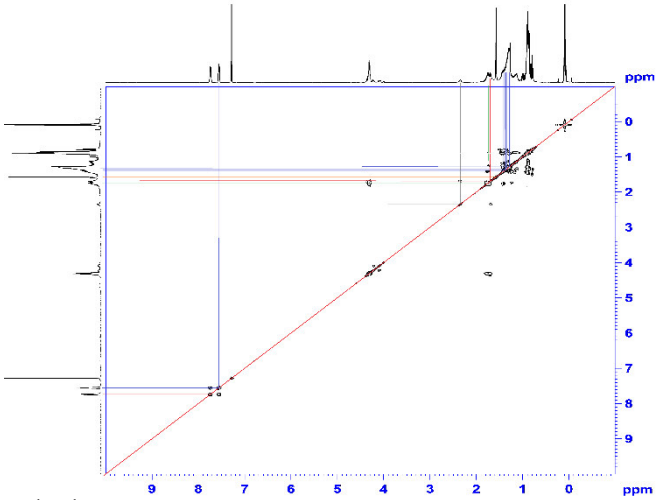


Figure 7: ^{1}H-^{1}H COSY of Hydroxy methyl taraxastene Ethyl benzoate in $CDCl_3$

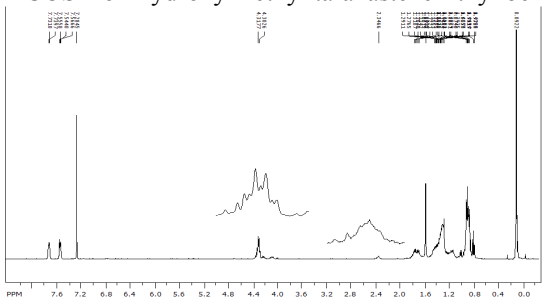


Figure 8: ^{1}H-NMR of Hydroxy methyl taraxastene Ethyl benzoate in $CDCl_3$

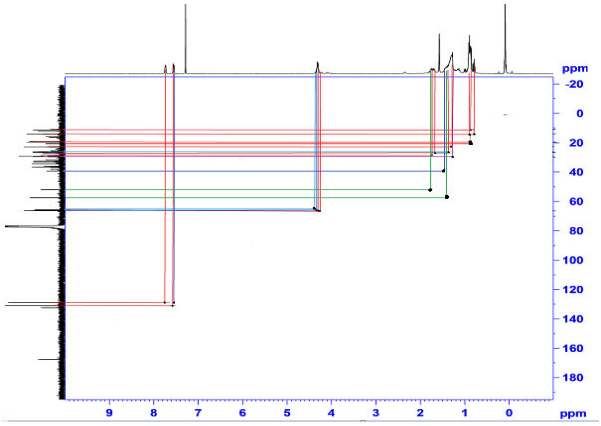
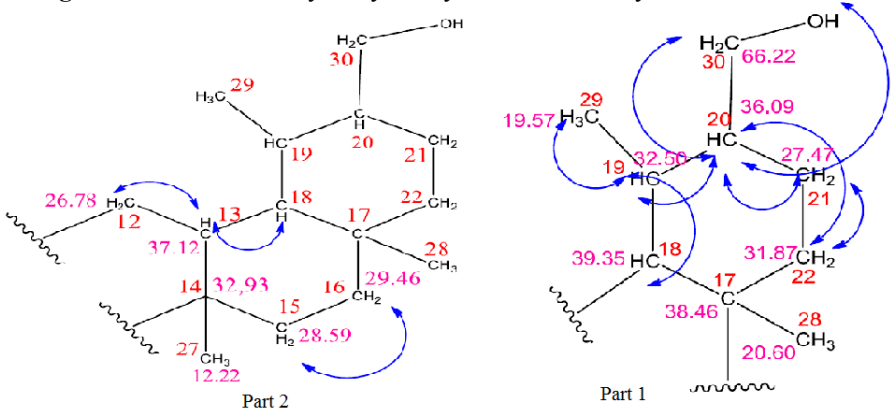


Figure 9: HETCOR of Hydroxy methyl taraxastene Ethyl benzoate in $CDCl_3$



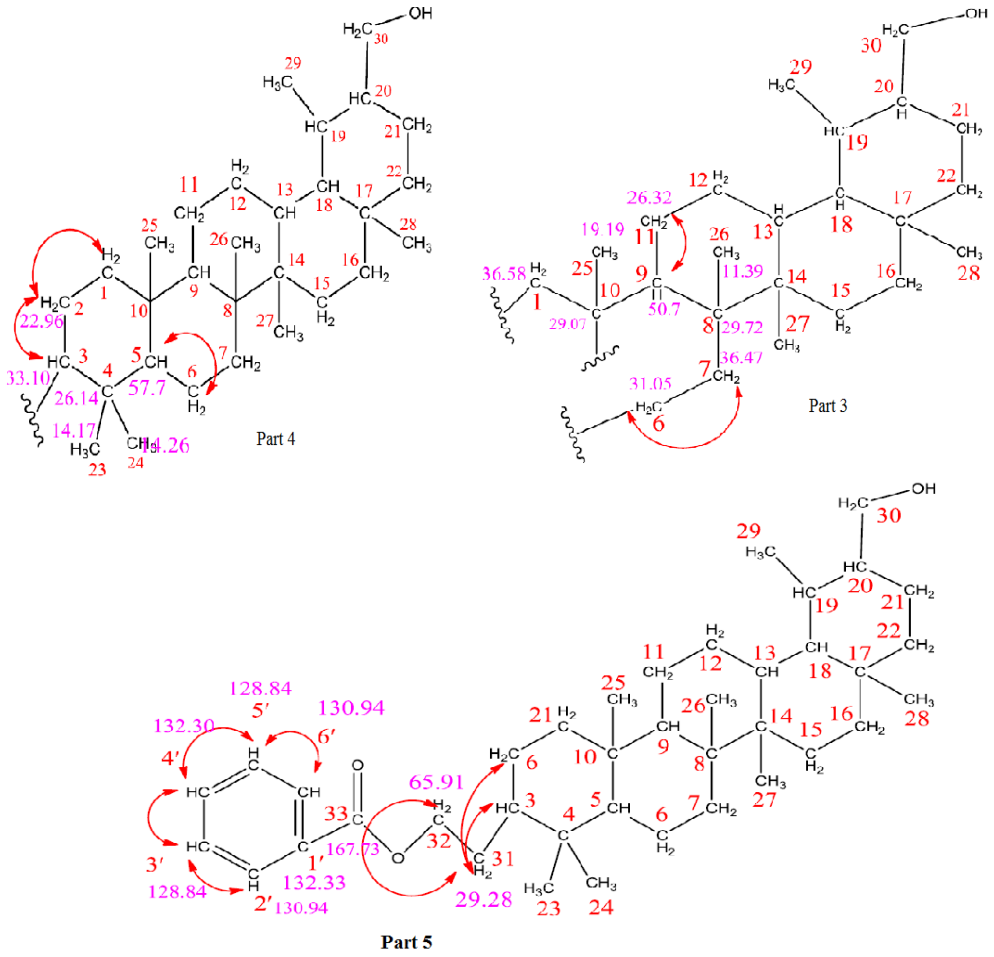


figure 10: HETCOR spectroscopy for the compound.

Tab(1): ^{1}H-NMR, ^{13}C-NMR, DEPT, , COSY and HETCOR data of Hydroxy methyl taraxastene Ethyl benzoate

^{1}H-^{1}H COSY	HETCOR	^{13}C NMR	Dept 90-135	No
1.3187	1.3829 m_2	36.58	CH_2	1
1.3829 , 1.4308 , 1.7048	1.3187 m_2	22.96	CH_2	2
1.3187 , 1.7048	1.4308 m_2	33.10	CH	3
----------	----------	26.14	C	4
1.4194	1.3935 m_2	57.7	CH	5
1.3691 , 1.3935	1.4194 m_2	31.05	CH_2	6
1.4194	1.3691 m_2	36.47	CH_2	7
----------	----------	29.72	C	8
1.4073	1.7555 q(18.48HZ)	50.7	CH	9
----------	----------	29.07	C	10
1.7555	1.4073 m_2	26.32	CH_2	11
1.7221	1.2911 m_2	26.78	CH_2	12
1.3626 , 1.2911	1.7221 t_2 (14.04 HZ)	37.12	CH	13
----------	----------	32,93	C	14
1.3439	1.3342 m_2	28.59	CH_2	15
1.3342	1.3439 m_2	29.46	CH_2	16
----------	----------	38.46	C	17
1.7395 , 1.7221	1.3626 m_2	39.35	CH	18
1.3626 , 1.6879	1.7395 q(18.48HZ)	32.50	CH	19
, 4.3177 1.2765 , 1.7395	1.6879 t_2 (14.04 HZ)	36.09	CH	20
1.3553 , 1.6879	1.2765 m_2	27.47	CH_2	21
1.2765	1.3553 m_2	31.87	CH_2	22
----------	0.7917 t_1 (13.67HZ)	14.17	CH_3	23
----------	0.7739 t_1 (13.67 HZ)	14.26	CH_3	24
----------	0.8746 m_1	19.19	CH_3	25
----------	0.8949 m_1	11.39	CH_3	26
----------	0.8559 m_1	12.22	CH_3	27
----------	0.9103 m_1	20.6	CH_3	28
----------	0.8851 m_1	19.57	CH_3	29
1.6879	4.3177 m_3	66.22	CH_2	30
1.3187 ,1.4308 , 4.3026	1.7048 t_2 (14.04 HZ)	29.28	CH_2	31
1.7048	4.3026 m_3	65.91	CH_2	32
----------	----------	167.73	OCO	33
----------	----------	132.33	C	1'
7.5440	7.7310 d(3.53HZ)	130.94	CH	2'
7.5558 , 7.7310	7.5440 dd(2.07HZ)(9.15HZ)	128.84	CH	3'
7.5666 , 7.5440	7.5558 dd(2.07HZ)(9.15HZ)	132.30	CH	4'
7.5558 , 7.7397	7.5666 dd(2.07HZ)(9.15HZ)	128.84	CH	5'
7.5666	7.7397 d(3.53HZ)	130.94	CH	6'

Conclusions

In summary, We demonstrated in this article compound identity verticillata which is a new compound. the compound seem a babble precipitate a white, fully dissolved in chloroform, and was R_f plants account for this compound in a sentence (N- hexane: chloroform) (50:50) was R_f=0.25.

Acknowledgment
The authors would like to thank Prof . Dr. Anwar Al- Khatib for his help in classifying the studied plant.

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]. Akkola E., Yeşiladab E., Güvençc A., Journal of Ethnopharmacology, 2008, Vol. 116, issue (2) ,p. 251–257.
[6]. Fieser,L., Fieser,M., Steroids (Book Rustion Tran) isd . Mir Mockow, 1964 .
[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]. 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.