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Original Article

A New Pseudoguaianolide from *Postia bombycina*

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ABSTRACT

The small genus *Postia* (tribe Inuleae) with four species, distributed over Syria and Iran, was previously placed in the subtribe Buphthalminae next to Anisopappus. Recently, however, both were transferred to the subtribe Inulinae sensu amplo in the Inula group. Further investigation of the aerial parts of *Postia bombycina* Boiss. & Hausskn. in addition to the known compounds, afforded a new sesquiterpene lactone type pseudoguaianolide. The aerial parts of *P. bombycina* were extracted with hexane/methanol/diethyl ether by immersion in the solvent. To determine the number of natural compounds in each fraction, thin layer chromatography was prepared. The obtained extract was evaporated to dryness, defatted with methanol and separated by column chromatography over silica gel. The structure of pseudoguaianolide was elucidated by high field ^{1}H-NMR, ^{13}C-NMR, 135-DEPT and FT-IR spectroscopy techniques. Previously, three pseudoguaianolides, four guaianolides, a Seco guaianolide, the known elemanolide and the xanthanolide tomentosin was isolated from *P. bombycina*. Therefore *P. bombycina* is a rich source of sesquiterpenes, especially guaianolide structure.

INTRODUCTION

Sesquiterpene lactones are the largest group of secondary metabolites comprising a large class of over 5500 known compounds in the plants with biologically significant, and many studies have been conducted on the Asteraceae family to find a natural compound with a new structure [1]. Sesquiterpenoids are commonly found in laticifers, which in most of the Asteraceae are specialized secretory cells, but can be found in the vacuoles of other cells in the plant, especially when produced in response to environmental stresses. Sesquiterpenoids are also one of the main constituents of latex in the latex producing plants and are very strong antimicrobials as well as antifeedants to chewing birds and insects. Some sesquiterpenoid lactones also have a range of other effects such as allelopathy, antimicrobial, stimulation of germination in the Orobanche, disrupting the cell wall of invasive bacteria and fungi [2].

The small genus *postia* of the Inuleae tribe has four species that are distributed in Iran and Syria. The Iranian flora includes two species: *P. puberula* and *P. bombycina*). The chemistry of this tribe has been studied previously, nothing is known about that genus *Postia* except our previous study that a seco Guainolide and other sesquiterpene lactones from *Postia bombycina* were identified (Fig. 1) [3].

We now present the result of a study of the new pseudoguaianolide from *P. bombycina* Boiss. & Hausskn. from Iran (Fig 2).

In other words, in 1989, from the investigation of the aerial parts of *P. bombycina*, four sesquiterpene lactones from guaianolide type 1-4, one seco-guaianolide 5, three sesquiterpene lactones pseudo-guaianolide type 6-8 [3,4] and also one eudesmanolide the names called granilin 9 [5] extracted with biological effects including antibacterial and natural substance xanthanolide called tomentosin 10 [6].

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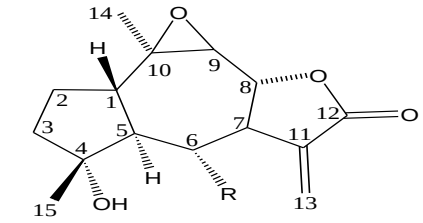
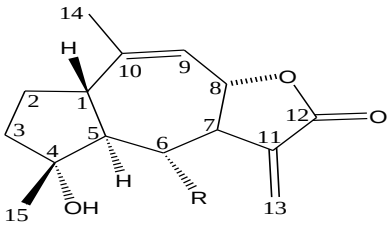
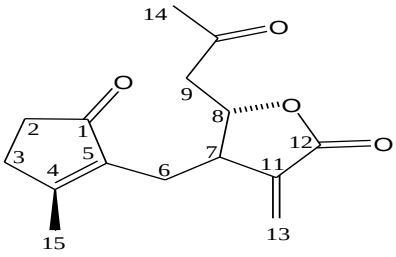


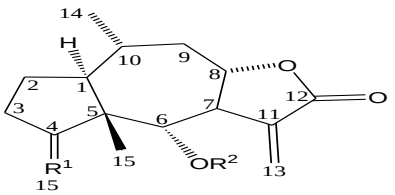
1 R=H
2 R=OAc
guaianolides



3 R=H
4 R=OAc
guaianolides

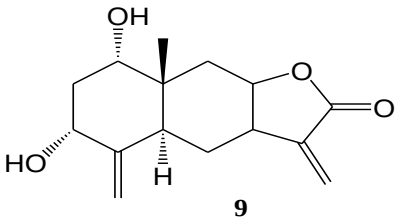


5
seco-guaianolide

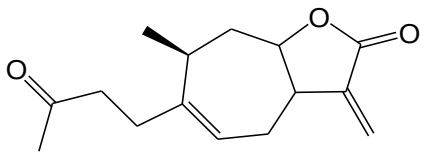


	R^1	R^2
6	**β-OH, H**	**Ac**
7	**β-OAc, H**	**H**
8	**=O**	**Ac**

pseudo-guaianolides



9
eudesmanolide



10
Xanthanolide tomentosin

Fig. 1 seco Guainolide and other sesquiterpene lactones from *P. bombycina* Boiss. & Hausskn. [3-6]

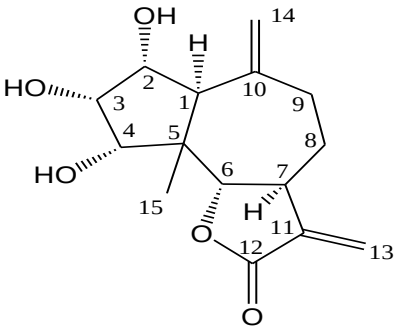


Fig. 2 A new pseudoguaianolide from *P. bombycina* Boiss. & Hausskn.

The structure of these natural compounds was identified and determined using the ^{1}H-NMR spectroscopy technique. The skeletons of these Guainolide 1-4, seco-Guainolide 5, and pseudo-Guainolides 6-8, granilin 9 and tomentosin 10 are given in Figure 1.

MATERIAL AND METHODS

Extraction and isolation

The air-dried aerial parts (1000g) were collected in August 2016 in the province of Khorasan, 60 km W of Mashhad, Iran. Voucher Number 324 deposited in the Herbarium of the Department of Botany, Shahid Beheshti University, Tehran, Iran.

The aerial parts of *P. bombycina* were extracted three times with 6 L each of hexane/methanol/ diethyl ether (in the ratio of 1:1:1) by immersion in the solvent at room temperature for 72 hours. The obtained extract was evaporated to dryness under low pressure, weighed (12.4 g) and suspended in boiling methanol (10 ml/g of extract) to remove long-chain saturated hydrocarbons. After standing at -15°C for 3 hours, the fatty and waxy material was removed by filtration, and the filtrate was evaporated in a vacuum. This yielded a dark-green dense oil (9.1 g) which was separated by column chromatography over silica gel. The dimensions of the chromatographic column were 1200 × 30 mm. Silica gel 230-400 mesh ASTM (Merck) was used for column chromatography. Silica gel was thoroughly mixed with hexane and was packed into a column. After the column was packed with Silica gel, a slurry of the silica gel and hexane was prepared, and the slurry was added to the column. The column was eluted with 360 mL each of hexane/ethyl acetate/methanol, respectively to give 41 fractions. The polarity of the mobile phase was

increased by changing the ratio of non-polar to polar solvents. To determine the number of compounds in each fraction, thin layer chromatography (TLC) was prepared with Kieselgel 60 prepared aluminum sheets. Then, the same fractions were added together. As a result, the number of fractions decreased to 24. In the next step, the residue from the fractions 14 + 15 + 16 + 17 was rechromatographed (Column dimensions: 800 × 20 mm) on silica gel (230-400 mesh ASTM) with 200 mL each of hexane/diethyl ether/methanol to give 21 fractions. From the fractions of 13 + 14 + 15, especially in fraction 14, colorless oil was purified by Thin Layer Chromatography (TLC) plates. ^{1}H-NMR spectrum was taken from these fractions. Fractions 13, 14 and 15 afforded pseudoguaianolide.

General experimental procedures

The structure of pseudoguaianolide was determined by FT-IR (Shimadzu, Japan), 500 MHz ^{1}H-NMR (Brucker, Germany), ^{13}C-NMR and 135-DEPT spectroscopy techniques.

RESULTS

The aerial parts of *P. bombycina* Boiss. & Hausskn. afforded in addition to the known compounds [4,5,6] a new pseudoguaianolide. The structure of the sesquiterpene lactone was followed from its high field spectroscopic data.

The molecular formula of the new pseudoguaianolide was suggested as $C_{15}H_{20}O_5$ by HREIMS.

The IR spectrum showed bonds demonstrated of carbonyl γ-lactone (1762 cm^{-1}) and OH (3493; 3394 cm^{-1}) and Carbon-Carbon double bonds (1667; 1660 cm^{-1}).

The ^{13}C-NMR spectrum of this compound contained resonances for 15 carbons, including four olefinic carbons and a Carbonyl carbon (approximately 170 ppm) (Table 1). The more detailed analysis was performed by 1H-NMR measurements. The typical low field signals at δ.

At δ 6.15 (d, H-13), δ 5.06 (d, H-13'), δ 5.61 (brs, H-14), δ 4.71 (brs, H-14') together with the signal H-15 (0.80, s, 3H).

The irradiation of the signal δ 2.73 showed that it was due to H-7 as the double doublet at 1.91 and 1.47 (H-8 and H-8') collapsed to a br doublet. Also, at δ 6.15 and δ 5.06 (H-13, H-13') to br singlets. Furthermore, as the signal of H-6 was only coupled with H-7 δ 2.73 (9.2 Hz) and this signal, in addition, is coupled with other signals H8, H8', H 13 and H 13'.

Table 1 ^{1}H-NMR and ^{13}C-NMR data of pseudoguaianolide (500 MHz, $CDCl_3$, δ- values)

No.	δ_C (ppm)	δ_H (ppm)	
1	33.62 d	2.48 d (10Hz)	
2	75.14 d	3.02 t (9 Hz)	
3	74.95 d	3.73 dd (9,10 Hz)	
4	78.53 d	4.22 d (10 Hz)	
5	40.35 s	-	
6	76.75 d	4.63 d (9 Hz)	
7	33.48 d	2.73 ddddd (8,9,10,0.5,0.5 Hz)	
8	26.80 t	H-8 α	1.91 ddd (12,10,10 Hz)
		H-8 β	1.47 ddd (12,8,10 Hz)
9	34.03 t	H-9 α	2.11 ddd (12,8,10 Hz)
		H-9 β	1.99 ddd (12,8,10 Hz)
10	142.18 s	-	
11	149.51 s	-	
12 (C=O)	170.51 s	-	
13	120.15 t	H-13	6.15 d (0.5 Hz)
		H-13'	5.06 d (0.5 Hz)
14	110.98 t	H-14	5.61 brs
		H-14'	4.71 brs
15	17.69 q	0.80 s 3H	

The presence of a pseudoguaianolide was very likely. The observed NOE indicated the presence of a trans-12,6-olide γ-lactone.

CONCLUSIONS

A new pseudoguaianolide was isolated from *P. bombycina*. Before that in 1989, three pseudoguaianolides, four guaianolides, a Seco guaianolide, the known elemanolide and the xanthanolide tomentosin was isolated from *P. bombycina* (3). Therefore *P. bombycina* is a rich source of sesquiterpenes, especially guaianolide-type.

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