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Guaianolide–type sesquiterpene lactones from the aqueous extract of *Salcedoa mirabaliarum*

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ABSTRACT

The phytochemical study of the aqueous extract of *Salcedoa mirabaliarum* (aerial parts), afforded five guaianolide–type sesquiterpene lactones: dihydroestafiatone, zaluzanin C, dihydroestafiatol, isoamberboin, and 4–*epi*–dihydroestafiatol, detected for first time in this species. The chemical structures were identified using ^{13}C Nuclear magnetic resonance by comparison with previously reported data.

INTRODUCTION

Salcedoa mirabaliarum F. Jiménez Rodríguez *et al.* [1] (Asteraceae), is an endemic species to the island of Hispaniola. Continuing with the interest of our research group in the Asteraceae botanical family present on Hispaniola [2–4] a phytochemical study of the aqueous extract of the aerial parts of *S. mirabaliarum* was performed affording five guaianolide–type sesquiterpene lactones detected for the first time in this species: dihydroestafiatone, zaluzanin C, dihydroestafiatol, isoamberboin, and 4–*epi*–dihydroestafiatol.

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MATERIALS AND METHODS

General experimental procedures

Nuclear magnetic resonance (NMR) spectra were obtained on a Bruker AVIII HD spectrometer with cryoprobe operating at 800 MHz in ^{1}H and 200 MHz in ^{13}C NMR, respectively. The chemical shift (δ) values are given in ppm. $CDCl_3$ was used as a solvent. Column chromatography (CC) was performed on a Biotage Isolera One flash purification system (Biotage, Charlotte, North Carolina, USA) using a SNAP ULTRA SiO_2 (100 g) cartridge. Analytical and preparative thin layer chromatography (TLC) was developed on silica gel 60 F_{254} plates (Merck KGaA, Darmstadt, Germany).

Plant material

The aerial parts of *S. mirabaliarum* were collected at Provincia Hermanas Mirabal, Municipio Tenares, Distrito Municipal Blanco Arriba, sección La Jíbara, paraje Mundo Nuevo (767 meters high) Dominican Republic, growing naturally with the following associated species: *Tabebuia ricardii*, *Gesneria viridiflora*, *Pouteria domingensis*, and *Bombacopsis emarginata*. The plant material was identified

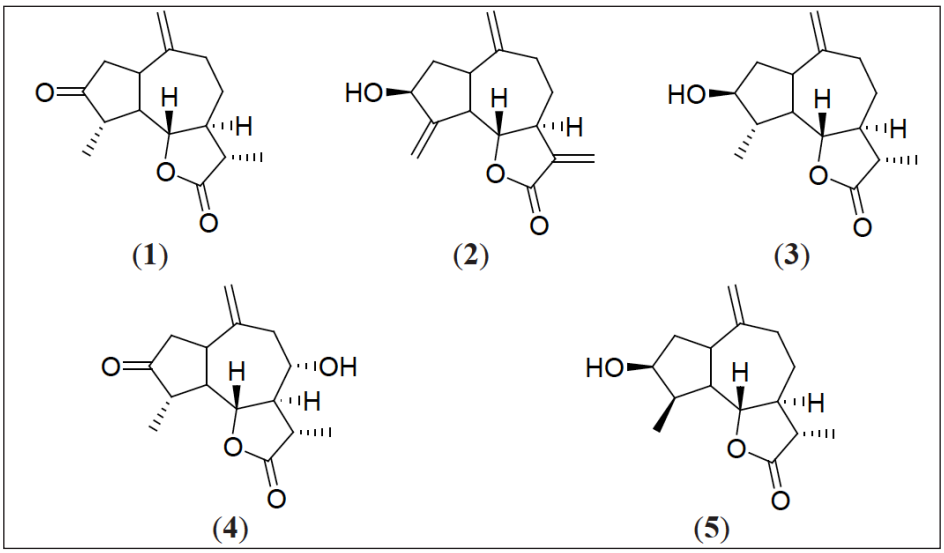

Figure 1. Chemical structures of compounds **1-5**.

by Mr. Teodoro Clase, a botanist at Jardín Botánico Nacional "Dr. Rafael Ma. Moscoso", Santo Domingo, Dominican Republic, where a voucher specimen (JBSD 121461) has been deposited.

Extraction and isolation

Dried and powdered aerial parts of S. mirabaliarum (300 g) were boiled with distilled water (4 L) for 2 hours, after which the resulting decoction was cooled, filtered, and extracted (3 × 3 L) with ethyl acetate (EA). After filtration, volume reduction and treatment successively with NaCl 1% and anhydrous Na_2SO_4, from the EA residue, it was possible to obtain 2.53 g of aqueous extract, which was subjected to CC (SiO_2) eluting with increasing mixtures of acetone-hexane, affording 97 fractions. Preparative TLC over selected eluted fractions, allowed to isolate compounds **1–5** (Fig. 1).

RESULTS AND DISCUSSION

The EA residue (2.53 g) of the aqueous extract from *S. mirabaliarum*, afforded, after different chromatographic techniques, 10.9 mg of dihydroestafiatone (**1**), [5], 2.6 mg of zaluzanin C (**2**), 3.8 mg of dihydroestafiatol (**3**), 2.6 mg of isoamberboin (**4**), [6], and 1.8 mg of 4–*epi*–dihydroestafiatol (**5**), [7]. Their chemical structures were identified using ^{13}C NMR by comparison with reported data.

***Dihydroestafiatone* (1)**

^{13}C NMR (200 MHz, $CDCl_3$) δ_C = 219.2 (C–3), 178.2 (C–12), 149.1 (C–10), 112.6 (C–14), 88.5 (C–6), 50.9 (C–5), 48.6 (C–7), 47.3 (C–4), 44.0 (C–2), 41.8 (C–11), 39.8 (C–1), 39.1 (C–9), 32.9 (C–8), 14.0 (C–15), 13.4 (C–13).

***Zaluzanin C* (2)**

^{13}C NMR (200 MHz, $CDCl_3$) δ_C = 170.0 (C–12), 153.1 (C–4), 147.9 (C–10), 139.7 (C–11), 120.2 (C–13), 114.4 (C–14), 111.3 (C–15), 83.9 (C–6), 73.6 (C–3), 50.0 (C–7), 45.6 (C–5), 44.2 (C–1), 39.1 (C–2), 34.3 (C–9), 30.6 (C–8).

***Dihydroestafiatol* (3)**

^{13}C NMR (200 MHz, $CDCl_3$) δ_C = 178.6 (C–12), 149.3 (C–10), 112.5 (C–14), 86.0 (C–6), 78.4 (C–3), 52.9 (C–7), 50.6 (C–5), 47.0 (C–4), 42.2 (C–11), 42.1 (C–1), 38.4 (C–2), 37.0 (C–9), 32.8 (C–8), 18.1 (C–15), 13.1 (C–13).

***Isoamberboin* (4)**

^{13}C NMR (200 MHz, $CDCl_3$) δ_C = 218.9 (C–3), 178.4 (C–12), 143.7 (C–10), 115.0 (C–14), 83.1 (C–6), 75.8 (C–8), 54.0 (C–7), 51.4 (C–5), 49.2 (C–9), 47.3 (C–4), 43.6 (C–2), 41.1 (C–11), 39.6 (C–1), 16.4 (C–13), 14.4 (C–15).

***4–epi–dihydroestafiatol* (5)**

^{13}C NMR (200 MHz, $CDCl_3$) δ_C = 178.8 (C–12), 148.4 (C–10), 111.8 (C–14), 83.0 (C–6), 73.8 (C–3), 51.5 (C–7), 47.2 (C–5), 42.1 (C–11), 41.3 (C–1), 40.4 (C–4), 39.3 (C–2), 34.7 (C–9), 32.8 (C–8), 13.3 (C–13), 8.1 (C–15).

CONCLUSION

In summary, we have reported the isolation of five guaianolide–type sesquiterpene lactones (**1–5**) from the aqueous extract of *S. mirabaliarum*. All compounds are reported for the first time in this species.

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AUTHOR CONTRIBUTIONS

All authors made substantial contributions to conception and design, acquisition of data, or analysis and interpretation of data; took part in drafting the article or revising it critically for important intellectual content; agreed to submit to the current journal; gave final approval of the version to be

published; and agree to be accountable for all aspects of the work. All the authors are eligible to be an author as per the International Committee of Medical Journal Editors (ICMJE) requirements/guidelines.

CONFLICTS OF INTEREST

The authors report no financial or any other conflicts of interest in this work.

ETHICAL APPROVALS

This study does not involve experiments on animals or human subjects.

DATA AVAILABILITY

All data generated and analyzed are included in this research article.

USE OF ARTIFICIAL INTELLIGENCE (AI)-ASSISTED TECHNOLOGY

The authors declares that they have not used artificial intelligence (AI)-tools for writing and editing of the manuscript, and no images were manipulated using AI.

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