



A SECOIRIDOID GLUCOSIDE FROM *JASMINUM ODORATISSIMUM*

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(Received in revised form 27 November 1995)

Key Word Index—*Jasminum odoratissimum*; Oleaceae; secoiridoid glucoside; 10-acetoxyoleoside dimethyl ester; 10-hydroxyoleoside dimethyl ester.

Abstract—A new secoiridoid glucoside, 10-acetoxyoleoside dimethyl ester, together with the known 10-hydroxyoleoside dimethyl ester was isolated from leaves of *Jasminum odoratissimum*. The structures of these compounds have been characterized on the basis of spectroscopic evidence.

INTRODUCTION

A great number of secoiridoids have been isolated from species of the family Oleaceae [1]. In this communication we report on the isolation and structural elucidation of 10-acetoxyoleoside dimethyl ester (**1**), a secoiridoid glycoside, which is described for the first time along with 10-hydroxyoleoside dimethyl ester (**2**), previously isolated from *Osmanthus asiaticus* [2]. We also include the spectroscopic data for this latter compound, which has not hitherto been reported.

RESULTS AND DISCUSSION

From the ethanolic extract of leaves of *Jasminum odoratissimum* L., a species endemic to the Canary Islands [3], the secoiridoid glycoside **1** was isolated as an optically active syrupy oil, $[\alpha]_D^{25} -196.3$ (CHCl₃). The FAB mass spectrum of **1** shows $[M + H]^+$ at m/z 477. The HR mass spectrum presents the molecular ion

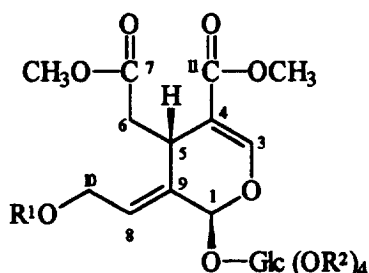
at m/z 296.0891 $[M - Glc]^+$, which is in agreement with the formula C₁₄H₁₆O₇ (calc. 296.0896). The ¹H NMR spectra of **1** and **2** are similar, the differences being an additional signal at δ 2.05 assignable to an acetate group and the downfield shift of the signal of the C-10 methylene group from δ 4.15 to 4.68 in the spectrum of the former compound. The ¹³C NMR spectrum of **1** shows two signals at δ 20.78 and 171.04, corresponding to an acetoxy group. Acetylation of both **1** and **2** yielded the same acetate (**3**) which was identified by direct comparison with authentic samples as well as by the mass spectrum, and ¹H and ¹³C NMR spectra [4].

EXPERIMENTAL

¹H and ¹³C NMR: 400 and 100 MHz, respectively. Chemical shifts are given relative to TMS as int. standard (δ scale). MS: 70 eV.

Extraction and isolation. Fresh leaves of *J. odoratissimum* (0.6 kg) were collected in September 1994, in San Andrés (Tenerife, Canary Islands). A voucher specimen is deposited at the Herbarium of the Department of Botany, Faculty of Biology, University of La Laguna (TFC31748). Air-dried leaves (380 g) were ground to a fine powder and extracted in a Soxhlet apparatus with EtOH. The extract was concd and chromatographed on silica gel, using *n*-hexane with gradually increasing proportions of EtOAc as eluent, to give **1** (250 mg) and **2** (40 mg).

10-Acetoxyoleoside dimethyl ester (1). ¹H NMR (CDCl₃): δ 2.05 (3H, s, OCOMe), 2.43 (1H, dd, $J = 14.6, 9.4$ Hz, H_A-6), 2.83 (1H, dd, $J = 14.6, 4.0$ Hz, H_B-6), 3.40–3.70 (2H, m, H-2', H-5'), 3.65 (3H, s, OMe), 3.72 (3H, s, OMe), 3.80 (2H, m, H-6'), 3.98 (1H, dd, $J = 9.4, 4.0$ Hz, H-5), 4.68 (1H, m, H_A-10), 4.78 (1H, dd, $J = 13.2, 6.5$ Hz, H_B-10), 4.85 (1H, d,



- 1** R¹ = Ac R² = H
2 R¹ = R² = H
3 R¹ = R² = Ac

$J = 7.5$ Hz, H-1'), 5.81 (1H, *s*, H-1), 6.11 (1H, *t*, $J = 6.5$ Hz, H-8), 7.49 (1H, *s*, H-3); ^{13}C NMR (CDCl_3): δ 20.78 (OCO-CH_3), 31.10 (C-5), 39.99 (C-6), 51.55 (OMe), 51.86 (OMe), 60.54 (C-10), 61.42 (C-6'), 69.53 (C-4'), 73.03 (C-2'), 76.07 (C-3', C-5'), 93.57 (C-1), 99.79 (C-1'), 107.94 (C-4), 123.45 (C-8), 132.13 (C-9), 153.41 (C-3), 166.36 (C-11), 171.04 (OC-OCH_3), 171.54 (C-7).

10-Hydroxyoleoside dimethyl ester (2). ^1H NMR (CD_3OD): δ 2.46 (1H, *dd*, $J = 14.8, 9.5$ Hz, H_A -6), 2.79 (1H, *dd*, $J = 14.8, 4.2$ Hz, H_B -6), 3.22–3.42 (2H, *m*, H-2', H-5'), 3.61 (3H, *s*, OMe), 3.64 (1H, *m*, H-5), 3.67 (3H, *s*, OMe), 3.85 (1H, *d*, $J = 10.0$ Hz, H_A -6'), 3.91 (1H, *dd*, $J = 10.0, 4.2$ Hz, H_B -6'), 4.15 (1H, *ddd*, $J = 13.5, 5.7, 1.4$ Hz, H_A -10), 4.28 (1H, *dd*, $J = 13.5, 7.0$ Hz, H_B -10), 4.78 (1H, *d*, $J = 7.5$ Hz, H-1'), 5.91 (1H, *s*, H-1), 6.12 (1H, *dd*, $J = 7.0, 5.7$ Hz, H-8), 7.49 (1H, *s*, H-3). ^{13}C NMR (CDCl_3): δ 32.31 (C-5), 40.96 (C-6), 52.00 (OMe), 52.31 (OMe), 59.12 (C-10), 62.65

(C-6'), 68.43 (C-11), 71.38 (C-4'), 74.67 (C-2'), 77.83 (C-5'), 78.34 (C-3'), 94.60 (C-1), 100.90 (C-1'), 109.17 (C-4), 129.43 (C-8), 130.92 (C-9), 155.04 (C-3), 173.47 (C-7).

Acknowledgement—This work was supported by Grant P.I. No. 92/136 from the Gobierno de Canarias.

REFERENCES

1. Boros, G. A. and Stermitz, F. R. (1991) *J. Nat. Prod.* **54**, 1173.
2. Sugiyama, M., Machida, K., Matsuda, N. and Kikuchi, M. (1993) *Phytochemistry* **34**, 1169.
3. Bramwell, D. and Bramwell, Z. (1990) *Flora Silvestre de las Islas Canarias*, p. 199. Editorial Rueda, Madrid.
4. Inouye, H., Inoue, K., Nishioka, T. and Kaniwa, M. (1975) *Phytochemistry* **14**, 2029.