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# A NEW ACYLATED SECOIRIDOID FROM GENTIANA FORMOSANA<sup>1</sup>

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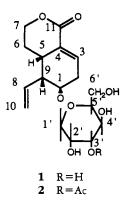
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ABSTRACT.—A mixture of  $\alpha$ - and  $\beta$ -amyrin palmitates, lupeol palmitate, uvaol palmitate,  $\beta$ -sitosterol,  $\beta$ -sitosterol- $\beta$ -D-glucoside, ursolic acid, uvaol,  $2\alpha$ -hydroxyursolic acid, sweroside, and a new acylated secoiridoid, 3'-acetylsweroside [2], were isolated and identified from the whole herb of *Gentiana formosana*.

As part of a search for active constituents in various genera of Formosan Gentianaceae, a mixture of  $\alpha$ - and  $\beta$ amyrin palmitates, along with lupeol palmitate, uvaol palmitate,  $\beta$ -sitosterol,  $\beta$ -sitosterol- $\beta$ -D-glucoside, ursolic acid, uvaol,  $2\alpha$ -hydroxyursolic acid, sweroside [1], and a new acylated secoiridoid, 3'acetylsweroside [2], were isolated from the fresh whole herbs of *Gentiana formosana* Hayata. In this paper, we report the characterization of 2.

# **RESULTS AND DISCUSSION**

Compound 2, a yellowish oil, showed a uv absorption curve similar to that of 1 (1,2). Its ir spectrum exhibited absorption bands at 1750 (ester C=O), 1720, and  $1640 \text{ cm}^{-1}$ . The eims showed a molecular ion peak at m/z 400. The <sup>1</sup>H-nmr spectrum of 2 indicated a singlet (3H) at  $\delta$  2.00 for an acetyl group and other proton signals of the aglycone corresponding well to those of the aglycone of 1(1,2). The characterization of **2** was also confirmed by its <sup>13</sup>C nmr (Experimental), in which all <sup>13</sup>C signals were assigned by means of <sup>1</sup>H decoupling data, DEPT pulse, and comparison with the data of  $\mathbf{1}$  (1,3). The shift values for  $^{13}C$  signals of 2 (except for C-3') and the acetyl signals corresponded very well to those of 1 (Experimental). A high field shift of glucosyl C-



3' (Experimental), compared with that of 1, indicated that the acetyl group is located at glucosyl C-3' of 2. Based on the above evidence, 2 was characterized as 3'-acetylsweroside [2].

## **EXPERIMENTAL**

GENERAL EXPERIMENTAL PROCEDURES.—All mp's are uncorrected. Ft nmr were performed on a Varian VXR-300151 Superconducting High Resolution Ft nmr System; ir spectra on a Hitachi model 260-30; uv spectra on Shimadzu UV-Visible Recording Spectrophotometer UV-240; MS on a JMS-HX 110 Mass spectrometer; and optical rotation on a Jasco model DIP-181 digital polarimeter.

EXTRACTION AND SEPARATION.—The fresh plants (1.3 kg) of *G. formosana* were collected at Ali Shian, Chiayi Hsien, Taiwan, during July 1978, chipped, and extracted with hot MeOH. The H<sub>2</sub>O-soluble portion of the MeOH extract was successively extracted with EtOAc and *n*-BuOH. The EtOAc extract was chromatographed on a Si gel column. Elution with Cyclohexane-CH<sub>2</sub>Cl<sub>2</sub> (9:1) yielded a mixture of  $\alpha$ - and  $\beta$ -amyrin palmitates. Elution with cyclohexane-CH<sub>2</sub>Cl<sub>2</sub> (1:2) yielded uvaol palmitate. Elution with C<sub>6</sub>H<sub>6</sub>-EtOAc (9:1) yielded  $\beta$ -sitosterol. Elution with CH<sub>2</sub>Cl<sub>2</sub>-EtOAc (6:1) yielded uvaol. Elution with CH<sub>2</sub>Cl<sub>2</sub>

<sup>&</sup>lt;sup>1</sup>Part XV in the series "Studies on the Constituents of Formosan Gentianaceous Plants." For part XIV see W.P. Tome, I.J. Chen, S.J. Liou, M.K. Cheng, and C.N. Lin, *J. Chin. Med.*, **2**, 51 (1992).

EtOAc (4:1) yielded ursolic acid. Elution with cyclohexane-CH<sub>2</sub>Cl<sub>2</sub>-EtOAc-MeOH (5:4:1) yielded  $\beta$ -sitosterol- $\beta$ -D-glucoside. Elution with cyclohexane-CH<sub>2</sub>Cl<sub>2</sub>-EtOAc-MeOH (4:3:2:1) yielded 2 $\alpha$ -hydroxyursolic acid. The *n*-BuOH extract also was chromatographed on a Si gel column. Elution with CH<sub>2</sub>Cl<sub>2</sub>-MeOH(4:1) yielded **1** and **2**. The above known compounds were identified by uv, ir, nmr, ms and comparison of the mmp's and spectral data with those of the authentic samples.

3'-Acetylsweroside [2].—Yellowish oil:  $[α]^{23}$ D -126.1° (MeOH, c=1.05); ir ν max (KBr) cm<sup>-1</sup> 3450, 1750, 1720, 1640; uv λ max (MeOH) nm (log ε) 240 (3.93); <sup>1</sup>H nmr (CD<sub>3</sub>OD) δ 1.60–1.85 (2H, m, H-6), 2.00 (3H, s, OAc), 2.65–2.97 (2H, m, H-5 and H-9), 4.35–4.48 (2H, m, H-7), 5.24– 5.35 (3H, m, H-8 and H-10), 5.46 (1H, d, J=1.5Hz, H-1), 7.57 (1H, d, J=2.4 Hz, H-3); <sup>13</sup>C nmr (CD<sub>3</sub>OD) δ 98.6 (C-1), 155.0 (C-3), 107.4 (C-4), 28.9 (C-5), 26.0 (C-6), 70.6 (C-7), 133.9 (C-8), 43.7 (C-9), 122.5 (C-10), 169.6 (C-11), 99.1 (C-1'), 75.5 (C-2'), 76.1 (C-3'), 72.1 (C-4'), 79.1 (C-5'), 63.1 (C-6'), 21.5 (Ac), 173.5 (Ac); eims (75) eV) (rel. int.) [M]<sup>+</sup> 400 (12), 288 (10), 246 (37), 229 (22), 205 (30), 187 (36), 169 (16), 145 (23), 127 (100), 109 (28), 97 (22), 85 (26), 73 (23). *Anal.* calcd for C<sub>18</sub>H<sub>24</sub>O<sub>10</sub>, 400.1368, found 400.1345.

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