

using hexane-Me<sub>2</sub>CO (4:1). One crystalline compound was isolated mp 165°, M<sup>+</sup> 236 (Found: C, 61.5; H, 5.3. C<sub>12</sub>H<sub>12</sub>O<sub>4</sub> requires C, 61.0; H, 5.1). IR  $\nu_{\max}$  (cm<sup>-1</sup>) 1715 (C=O). NMR (CDCl<sub>3</sub>) 3.81 and 2.03 (two d; J = 10 Hz) 3- and 4-H of coumarin, 3.62 (s, 6-H); 6 and 6.1 (s; 3 OMe). The C<sub>6</sub>H<sub>6</sub>-induced solvent shifts of OMe groups are seen at 6.2, 6.5 and 6.6, indicating that two OMe groups have suffered a significant upfield shift, suggesting that at least one adjacent position to the two OMe groups is unsubstituted. MS (m/e) 236 (100%); 221 (80), M-15; 195 (11), M-41; 194 (93) M-42; 178 (2), M-58;

165 (5) M-71; 150 (11), M-86; 135 (4) M-101; 107 (3) M-129. The compound is therefore 5,7,8-trimethoxycoumarin.

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CHRYSOERIOL 7-O-RHAMNOSIDE FROM *SEDUM FORMOSANUM*

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**Key Word Index**—*Sedum formosanum*; Crassulaceae; sterols; flavonoid; chrysoeriol 7-o-rhamnoside.

*Sedum formosanum* Hay. has been used in folk-medicine for the treatment of diabetes [1]. We have isolated from it campesterol, stigmasterol and sitosterol and a new flavone glycoside, identified as chrysoeriol 7-o-rhamnoside. The sugar was identified as rhamnose by co-chromatography with an authentic sample and confirmed by oxidation of the glycoside with periodate [2]. The NMR spectrum of the glycoside showed the characteristic rhamnoside H-1" proton and rhamnosyl C-Me group [3]. The aglycone was identified as chrysoeriol from UV, NMR [3] and MS data [4] and this was confirmed by its demethylation to give luteolin. The glycoside lost its sugar on acid hydrolysis, indicating that it was an o-glycoside. Comparing the NMR spectrum of glycoside and aglycone showed a downfield shift for H-8 and H-6 indicating that the sugar unit was attracted to oxygen at C-7, a fact confirmed by the absence of a UV shift with NaOAc. Thus the compound is chrysoeriol 7-o-rhamnoside. Although a number of chrysoeriol glycoside are known, this is the first report of the 7-rhamnoside.

## EXPERIMENTAL

Air-dried whole plants of *Sedum formosanum* were obtained from the beach of Yee-Leou (Taiwan) in May, 1973. NMR spectra were recorded in DMSO-d<sub>6</sub>. GLC was used with 3% SE-30 column. Mp's are uncorrected.

**Extraction and isolation.** Plants (5.3 kg) were extracted with n-hexane and EtOH successively. Evaporation of the n-hexane extract left 11. of viscous residue, which was then deposited a precipitate at 4°. The supernatant was further concentrated and then subjected to column chromatography on Si gel and eluted with n-hexane-Me<sub>2</sub>CO (40:1), giving 1.6 g of the sterol mixture. The EtOH extract was concentrated and the syrupy mass was dissolved in 3% HOAc. The filtered acidic soln was extracted with Et<sub>2</sub>O, and a brown ppt. formed. This was dis-

solved in EtOH, filtered, and the filtrate was conc. and dried. The yellow mass was subjected to column chromatography over Si gel and eluted with 25% MeOH in CHCl<sub>3</sub>, and MeOH, giving the flavone glycoside, eventually as fine yellow needles from MeOH (15 mg). The sterol mixture crystallized from n-hexane as needles, mp 139–40°, which gave a positive Liebermann-Burchard test. It was identified as a mixture of campesterol, stigmasterol and sitosterol by GLC comparison with soybean sterols [5]. Chrysoeriol 7-rhamnoside had mp 287–9° (Found: C, 54.82; H, 5.13, C<sub>22</sub>H<sub>32</sub>O<sub>10</sub>·2H<sub>2</sub>O requires: C, 54.77; H, 5.43%). It showed a single spot of polyamide plate (EtOH, FeCl<sub>3</sub>, brown-gray), and gave a violet colour with Mg-HCl,  $\lambda_{\max}$  350, 268 sh, 253; AlCl<sub>3</sub> 388, 355, 296 sh, 276, 261; NaOAc 418, 356, 268 sh, 254; NaOAc + H<sub>3</sub>BO<sub>3</sub> 351, 268 sh, 254, EtONa 418, 261. NMR  $\delta$ : 7.5 (2H, m.) 6.93 (1H, d, J, 9 Hz,) 6.75 (1H, d, J 2 Hz,) 6.38 (1H, d, J 2 Hz,) 5.18 (1H, br,) 1.25 (3H, m.). Its identification was carried out by standard procedures [3].

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