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A NEW NICOTINOYL GLUCOSIDE FROM *CRYPTOLEPIS BUCHANANI*

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Key Word Index—*Cryptolepis buchanani;* Asclepiadaceae; nicotinoyl glucoside; alkaloid.

Abstract—A new alkaloid was isolated from the stems of *Cryptolepis buchanani*. Its structure was determined as 1,3,6-*O*-trinicotinoyl-α-D-glucopyranose by chemical and spectroscopic investigations.

INTRODUCTION

Previously we reported [1] the isolation of a new nicotinoyl glucoside from the stems of *Cryptolepis buchanani* Roem. & Schult. (Asclepiadaceae). Further examination of the stems has now led to the isolation of another new nicotinoyl glucoside, designated as CB-2.

RESULTS AND DISCUSSION

Dried and milled stems were defatted with petrol and then extracted with EtOH. The EtOH extract was concentrated under reduced pressure and then partitioned between $CHCl_3$ and H_2O. The $CHCl_3$ soluble fraction on chromatography over Si gel yielded CB-2, mp 131–133°.

CB-2, $C_{24}H_{21}N_3O_9$, showed UV $\lambda_{\max}^{\mathrm{EtOH}}$ 222, 260 (*sh*), 265 and 272 (*sh*) nm (log ε 4.41, 3.90, 3.92 and 3.84, respectively). There was no shift in acid. The UV absorption was very similar to that of buchananine [1] except for the ε values which were *ca* 3 times greater than those of buchananine. The IR spectrum exhibited peaks for OH (3350 cm^{-1}), ester carbonyl (1735 cm^{-1}) and an aromatic double bond (1600 cm^{-1}). Hydrolysis of CB-2 yielded glucose and nicotinic acid similar to buchananine.

The ^1H NMR spectrum (90 MHz, $CDCl_3$) of CB-2 showed 12 aromatic protons for 3 nicotinoyl moieties. A 3H *m* centered around δ 7.4; a 3H *dt* at 8.3 ($J_{4',5'} = 7$; $J_{4',6'} = J_{2',4'} = 2$ Hz); a 3H *dd* at 8.8 ($J_{5',6'} = 5$ Hz; $J_{4',6'} = 2$ Hz); a 3H *d* at 9.27 ($J_{2',4'} = 2$ Hz) were assignable respectively to C-5′, C-4′, C-6′ and C-2′ protons of the three nicotinoyl moieties. The non-aromatic part of the spectrum showed signals at δ 4.7–6.1 (7H, *m*, sugar protons) and at 2.98 (2H, *br*, exchangeable, C-2 OH and C-4 OH). The *J* value (3.5 Hz) of the anomeric proton indicated that the glucose moiety was in the α-form.

The above information indicated that 3 molecules of nicotinic acid were esterified with 1 molecule of glucose in CB-2. Since the alkaloid did not reduce Fehling's and Benedict's solution, the C-1 OH of glucose was in glycosidic linkage with nicotinic acid. When the alkaloid was treated with periodic acid in bicarbonate buffer [1], no formaldehyde–dimedone was obtained. This suggested that —CH_2OH group was involved in the ester linkage with nicotinic acid. When the periodic acid reaction was monitored by TLC, no spots corresponding to any compounds produced by vicinal OH cleavage were obtained. No isopropylidene derivative could be obtained when CB-2 was treated with dry Me_2CO and dry $FeCl_3$ [2]. This also showed that no vicinal OH groups were present. Therefore, the C-3 OH of the glucose moiety was preferred for the location of the third nicotinoyl moiety. CB-2 was thus characterized as 1,3,6-*O*-trinicotinoyl-α-D-glucopyranose.

CB-2 did not exhibit a M^+ peak in its MS (70 eV). Peaks at *m/e* 446 ($M^+ - H_2O - H - HCHO$; 0.2%), 372 (M^+ − nicotinic acid; 0.4), 328 (0.2), 223 (0.4), 186 (1.2), 149 (4.8), 124 (7.3) and 123 (100) were obtained. These can be rationalized following a mechanism similar to that given for buchananine [1].

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