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GENKWANIN 4'-GALACTOSIDE AND OTHER CONSTITUENTS FROM *DUABANGA SONNERATIOIDES**

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Key Word Index—*Duabanga sonneratioides*; Lythraceae.

Our initial investigations[1,2] on the stem bark of *D. sonneratioides*. Ham. (Lythraceae) showed the presence of several known constituents. Further work on this plant, because of its activity against Walker's carcinosarcoma[3] has led to the isolation of ellagic acid, tetramethylellagic acid, epioleonolic acid and a new flavone galactoside.

The flavone galactoside (**1**) $C_{22}H_{22}O_{10}$, m.p. 260° (dec.), ν_{max} 3400, 1650, 1600, 1555, 1248, 1060 and 830 cm^{-1}. λ_{max} (EtOH) 226, 270 and 325 nm ($AlCl_3$) 280, 300, 335 and 385 nm (NaOAc) 270 and 325 nm (NaOAc–boric acid) 226, 270 and 325 nm is sparingly soluble in $CHCl_3$, Me_2CO, EtOAc and MeOH and gave positive Fiegels' test for glycoside. On acidic hydrolysis, **1** furnished an aglycone (**2**), $C_{16}H_{12}O_5$, m.p. 267°, M^+ 284, λ_{max} 225, 270 and 329 nm (NaOAc) 225, 270 and 329 nm identical with genkwanin[4] and a sugar, galactose (PC). **2** formed a diacetate, $C_{20}H_{16}O_7$, m.p. 204° (IR, NMR and MS).

The shift in the UV maxima of **1** in the presence of $AlCl_3$ indicated the presence of a free 5-hydroxyl. This shows that the sugar moiety must be at position 7. It is known that glycosidation of the 4'-hydroxyl group produces a 3–10 nm hypsochromic shift in band **I**.[5] Analogous with this, λ_{max} 329 nm of **2** shifts to 325 nm in **1**. The chemical and spectroscopic evidence thus indicated **1** to be 4'-*O*-galactoside of 5,4'-dihydroxy-7-methoxyflavone.

Ellagic acid $C_{14}H_6O_8$, m.p. >360°, M^+302, λ_{max} 258 and 368 nm gave a positive Griessmayer test (IR and m.m.p.). It formed a tetraacetate $C_{22}H_{14}O_{12}$, m.p. 336° and a tetramethyl ether $C_{18}H_{14}O_8$, m.p. 340°. Tetramethyl ellagic acid $C_{18}H_{14}O_8$, m.p. 340° was characterized on the basis of IR, UV, MS and m.m.p.

Epioleonolic acid $C_{30}H_{48}O_3$, m.p. 302° has been identified through its IR, MS and NMR and acetate $C_{32}H_{50}O_4$, m.p. 268°.

EXPERIMENTAL

UV spectra were determined in EtOH, IR spectra in KBr and 60 MHz NMR spectra in $CDCl_3$ with TMS as internal standard. Uncorrected capillary m.ps are reported.

Extraction and separation of constituents. Air dried powdered stem bark (6 kg) were extracted with 50% EtOH

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(40 l.) and the extract concentrated *in vacuo* below 50°. The syrupy material obtained was extracted successively with C_6H_6 (6 l.), EtOAc (6 l.) and *n*-BuOH (4 l.). C_6H_6 and EtOAc fraction gave compounds reported earlier[1,2] and also tetramethylellagic acid (10% EtOAc in C_6H_6) and epioleonolic acid (20% EtOAc in C_6H_6). *n*-BuOH extract was freed of the solvent and the residue treated with Me_2CO. Me_2CO soluble fraction gave **1** while Me_2CO insoluble part afforded ellagic acid.

Genkwanin 4′-galactoside (**1**). Me_2CO soluble part of the *n*-BuOH extract, deposited a solid which was recrystallized from EtOH (Found: C, 58·70; H, 5·20. $C_{22}H_{22}O_{10}$ requires: C, 59·10; H, 4·91%). Acid hydrolysis and extraction with Et_2O afforded genkwanin, m.p. 267° (3 crystallization gave m.p. 280°) (Found: C, 67·10; H, 4·30. $C_{16}H_{12}O_5$ requires: C, 67·60; H, 4·20%).

Diacetate. M.p. 204° (Found: C, 64·10; H, 4·10. $C_{20}H_{16}O_7$ requires: C, 64·60; H, 4·30%). The aq. hydrolysis soln was neutralized ($BaCO_3$) and PC indicated that the only sugar present was galactose.

Ellagic acid. The Me_2CO insoluble fraction from the *n*-BuOH extract was extracted (Soxhlet) with hot Me_2CO. Concentration and standing in the cold furnished a yellow solid. This on recrystallization gave yellow needles, m.p. >360° (Found: C, 56·10; H, 2·10. $C_{14}H_6O_8$ requires: C, 56·60; H, 1·98%).

Tetramethyl ellagic acid. Obtained as needles when crystallized from MeOH and dioxane m.p. 340° (Found: C, 59·80; H 4·20. $C_{18}H_{14}O_8$ required: C, 60·33; H, 3·91%).

Epioleonolic acid. Crystallized from MeOH, m.p. 302° (Found: C, 78·50; H, 9·86. $C_{30}H_{48}O_3$ requires: C, 78·94; H, 9·86%).

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ACORENONE-B IN *ANGELICA LUCIDA* OIL

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Plant. Angelica lucida L. (≡ *Coelopleurum actaeifolium* (Michx.) Coult. & Rose, *Archangelica gmelini* (specimen retained in Herbarium at University of Waterloo, NA 5422). *Source.* Blue Rocks, Lunenburg, Nova Scotia, Canada. *Uses.* Tender parts of plant used as food and tonic[1] by Eskimos.

Present work. The dried seeds were water distilled to yield 0·15% essential oil. The oil, which was analysed by a combination of techniques described previously[2] was found to contain acorenone-B (43·1%), β-phellandrene (16·1%), myrcene (8·0%), caryophyllene (7·5%), α-pinene (2·0%), phenyl ethyl isovalerate (2·0%), bornyl acetate (1·6%), benzyl isovalerate (1·1%), camphene (0·3%), β-pinene (0·2%) and sabinene (0·2%).

Comment. Acorenone B, which was recently isolated from *Bothriochloa intermedia* (R.Br.) A. Camus (Gramineae) and synthesized,[3] was found to be identical to acorenone previously isolated from *Acorus calamus* L. (Araceae).[4]

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