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FLAVONOL-3-GLUCOSIDES IN *BACCHARIS ANGUSTIFOLIA* AND *BERLANDIERA PUMILA*

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Plants. (1) *Baccharis angustifolia* Mich. and (2) *Berlandiera pumila* Nutt *Source* (1) Collected by Gary Anderson, in November 1966 at St Mark's Wild Life Refuge, Florida, (2) Junction Truck Route and Spring Hill Road, Leaon County, Tallahassee *Previous work* (1) Sister species[1,2] (terpenoids), (2) sister species[3] (amines)

Compounds isolated Quercetin 3-β-glucoside (isoquercitrin) and kaempferol 3-β-glucoside (astragalin) were isolated from the methanolic extracts of the plant by methods described previously[4] and identified by direct comparison with authentic material by mixed m p , co-chromatography (TLC, 3 solvents) and UV and IR analysis

Acknowledgement—This investigation was supported in part by U S Public Health Service (RG-GM-05814)

[1] F BOHLMANN and C ZDERO, *Tetrahedron Letters* **28,** 2419 (1969)
[2] Y NAVES, *Bull Soc Chim Fr* 1871 (1959)
[3] T A WHEATON and I STEWART, *Lloydia* **33,** 244 (1970)
[4] H WAGNER, M A IYENGAR, E MICHAHELLES and W HERZ, *Phytochem* **10,** 2824 (1971)

Key Word Index—Baccharis angustifolia, *Berlandiera pumila*, Compositae, quercetin and kaempferol-3-glucosides

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VIRGININ. A SESQUITERPENE LACTONE FROM *ENCELIA VIRGINENSIS*

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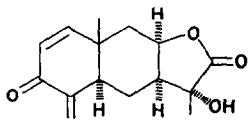
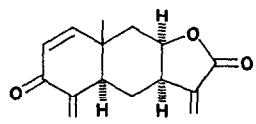
Encelia virginensis is a composite shrub which grows in the southwestern desert We have found it growing at the base of hills upon the slopes of which grow *E farinosa.* Since these two related plants may be found in close proximity and *E farinosa* has been shown[1] to contain the sesquiterpene lactones farinosin (I) and encelin (II), we investigated samples

[1] T A GEISSMAN and R MUKHERJEE, *J Org Chem* **33,** 656 (1968)

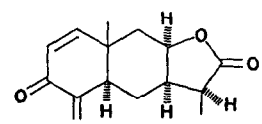
of plants taken from an area where the two species were growing together but with no obvious overlap. Only farinosin was isolated from *E. farinosa*; no encelin was detected, although thin layer chromatograms of the crude sesquiterpene extract suggested the presence of small amounts of other compounds



Farinosin (I)



Encelin (II)



Virginin (III)

Farinosin and a new sesquiterpene lactone, virginin (III), were isolated from leaves of *E. virginensis*. Virginin $[\alpha]_D^{24}$ −128° (C., 0·14, $CHCl_3$), m.p. 241–242°, has strong IR absorption at 5·66 (δ-lactone), 6·02 (cyclohexenone), 6 21 (conjugated double bond) and 11·82 nm exocyclic methylene) It gives UV absorption at 240 nm (ε 9560) indicative of an α,β-unsaturated ketone The molecular ion appears at *m/e* 246 in its mass spectrum, along with intense peaks at *m/e* 91 and 105 The latter two have been pointed out as characteristic of sesquiterpene lactones.[1] The NMR spectrum shows a three proton singlet at δ1·00 (methyl at C-10); a three proton doublet at δ1·27 (methyl at C-11), a broad, one proton multiplet at δ4 60 (H at c-8); a pair of doublets, one proton each at δ5·30 and 6·14, $J = 3$ cps (exocyclic methylene); and a pair of one proton doublets at δ6·02 and 6·88, $J = 10$ cps (vinyl protons at C-1 and C-2).

All these spectral data are very similar to those shown by farinosin.[1] The significant differences are the lack of OH absorption in the IR and NMR spectra of III, the absence of a three proton singlet at δ1·52 and its replacement by a three proton doublet at δ1·27 In addition the molecular ion of III is 16 mass units less than that of farinosin. This evidence indicates that virginin differs from farinosin by the replacement of the C-11 hydroxyl group by a hydrogen.

EXPERIMENTAL

Isolation of virginin and farinosin *Encelia virginensis* was collected in June of 1969 The leaves and young shoots were dried at 56° and ground in a Wiley mill The extraction procedure was that of Geissman [3] From 794 g of plant material 2 93 g of crude yellow oil was obtained Chromatography over 90 g of alumina (Woelm, activity II) using the solvent series benzene, Et_2O, $CHCl_3$, gave several contiguous crystalline fractions The crystalline fractions were combined and recrystallized from $CHCl_3$ to yield 158 mg (0 02%) of farinosin m p 199–200 (lit 200–201 [1]). Farinosin gave IR, UV, NMR, and mass spectra identical to those reported [1]

The mother liquors from the recrystallization of farinosin were placed on a preparative TLC plate of silical gel H (1 50 mm and developed with $CHCl_3$) The silica gel between R_f 0 5 and 0 6 was scraped off and the dihydroencelin extracted with EtOAc and recrystallized from Et_2O to yield 2 mg of product m p 241–242°

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[2] W HERZ, P S. SUBRAMANIAM and T A GEISSMAN, *J Org Chem* **33**, 3743 (1968).
[3] T A GEISSMAN, *J Org Chem* **31**, 656 (1966)

Key Word Index—Encelin virginensis, Compositae, sesquiterpenoid lactones, virginin, farinosin