

FLAVONOIDS AND A HELIANGOLIDE, NIVEUSIN-C,
FROM *MELAMPODIUM CAMPHORATUM*

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Melampodium campboratum (L.F.) Baker (Compositae) is the only member of this well-investigated genus (1-3) occurring in Guyana and is used medicinally by the local Amerindians (4). Fractionation of the hexane and CH_2Cl_2 extracts of *M. campboratum* yielded the flavonoids, apigenin dimethyl ether and acacetin, as well as the unusual heliangolide, niveusin-C, previously isolated from *Helianthus maximiliani* (5) and *Helianthus niveus* (6). This is the first reported isolation of a sesquiterpene lactone of this type from *Melampodium*.

EXPERIMENTAL

PLANT MATERIAL.—The entire plant was collected in October 1982, on the white sands at Soesdyke on the Soesdyke-Linden Highway, Demerara, Guyana. Voucher specimens were deposited in the Herbarium of the University of Guyana and at the Institute of Systematic Botany, University of Utrecht, Netherlands.

ISOLATION OF APIGENIN DIMETHYL ETHER.—Air-dried and ground plant material (1.2 kg) was exhaustively extracted with hexane in a Soxhlet apparatus (in batches of 230 g) to afford a tar (44 g) after evaporation of the solvent. The crude extract was subjected to Büchner funnel and flash column chromatography to give apigenin dimethyl ether (0.01% of plant material), recrystallized from MeOH, and identified by comparison of its mp, uv, ^1H nmr, ms, and mp of its acetate with published values (7-9).

ISOLATION OF ACACETIN AND NIVEUSIN-C.—Air-dried and ground plant material (2 kg) was exhaustively extracted with CH_2Cl_2 in a Soxhlet apparatus (in batches of 230 g) for 3 days. Removal of the solvent yielded a gum (160 g), which was worked up in batches by the following procedure: the gum (60 g) was dissolved in hot EtOH (400 ml) and stirred with an equal volume of hot H_2O for 15 min, and the suspension was refrigerated for 24 h; the supernatant liquor was decanted, filtered through Celite, most of the EtOH was removed by evaporation, and the aqueous solution was extracted with CHCl_3 (5×50 ml); the combined organic extracts were dried, filtered and evaporated to give a syrup (17 g) which, on column chromatography ($\text{CH}_2\text{Cl}_2/\text{EtOAc}$ mixtures of increasing polarity), afforded acacetin (0.0003% of plant material), identified by comparison of its mp and ir, uv, and ^1H -nmr spectra with published values (10), and niveusin-C (0.002% of plant material), identified by mp, mmp, and comparison of ir, uv, and ^1H -nmr spectra with those of an authentic sample (5), supplied by Professor W. Herz. Details of isolation and identification procedures are available on request.

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TRICIN FROM *VERNONIA REMOTIFLORA*

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The genus *Vernonia* Schreb. (Compositae), with more than 1,000 species, has attracted a wide range of interests. The diversity of its metabolites has been of chemotaxonomic interest (1,2), and the structural complexity and biological activity of some of the metabolites have been of considerable chemical and pharmacological interest; the tumor inhibitors vernolepin and vernomenin provide notable examples (3,4). *Vernonia remotiflora* Rich. is a representative of the genus found in Guyana and does not appear to have been investigated previously. We have examined this species, but the only secondary metabolite that we have been able to isolate and identify is the flavone triclin (5,6).

EXPERIMENTAL

PLANT MATERIAL.—The aerial parts of *V. remotiflora* were collected in June 1984, in a locality at Long Creek on the Soesdyke-Linden Highway, Demerara, Guyana. Voucher specimens were deposited in the Herbarium of the University of Guyana and at the Institute of Systematic Botany, University of Utrecht, Netherlands.

EXTRACTION AND ISOLATION.—Air-dried and ground plant material (1 kg) was exhaustively extracted by cold percolation with CHCl_3 . Removal of the solvent under reduced pressure afforded a gum (45.5 g) that was dissolved in hot EtOH (40 ml). The solution was stirred for 15 min with an equal volume of hot H_2O , and the resulting suspension was refrigerated overnight. The supernatant liquor was decanted, filtered, concentrated, and extracted with CHCl_3 (5×100 ml). The residue (7.2 g) after evaporation of the solvent was fractionated on a column of Si gel (200 g). Crystallization of the fractions eluted with CHCl_3 -EtOAc (9:1) yielded triclin, identified by comparison of its mp, uv, and ^1H -nmr data with published values (5,6).

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