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MINOR ALKALOIDS FROM NICOTIANA TABACUM

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Systematic alkaloid investigations of various cultivated strains (Coker-319, Virginia Delcrest, Kallo, Palmonostor) of Nicotiana tabacum L. (Solanaceae) were carried out with hplc. The presence of 2,4'-dipyridyl and 4,4'-dipyridyl could be presumed in the Palmonostor strain due to the retention data and uv spectra obtained by diode array detection. The other three dipyridyl isomers have been known for a long time (1,2). In 1980, Saint-Jalm and Moree-Testa (3) identified 2,4'-dipyridyl in tobacco smoke with a gc-ms method. Now 2,4'-dipyridyl and 4,4'-dipyridyl are reported for the first time being present in N. tabacum.

EXPERIMENTAL

PLANT MATERIAL.—Leaves of the *N. tabacum* strain Palmonostor were cultivated and collected in Albertirsa, 60 km from Budapest, Hungary. A voucher specimen is deposited in the Institute of Tobacco-Research, Debrecen, Hungary.

EXTRACTION AND ISOLATION.—Air dried, finely powdered leaves (40 kg) were exhaustively extracted with $CHCl_3$. The $CHCl_3$ extract was concentrated in vacuo and mixed with 5% HCl. The acidic extract was alkalinized with NaOH to pH 9 and extracted with $CHCl_3$, this being repeated twice.

The CHCl₃ phase was then washed with H_2O and dried, and the solvent was removed to give an alkaloid mixture which was then chromatographed on Si gel. The solvent strength (4) of the mobile phase (containing EtOH, dioxane, hexane, and 0.5% NH₃) was gradually increased from 0.4 to 1.8. The dipyridyl fraction (containing 2,3'-dipyridyl, 2,4'-dipyridyl, 3,3'-dipyridyl, and 4,4'-dipyridyl) was concentrated in vacuo. The mobile phase was optimized with the help of "PRISMA"-model (4, 5) on tlcplates. The optimized mobile phase was transferred to the sequential centrifugal layer chromatography (sclc) (6) using a diluting factor (7) which was calculated from the tlc pre-assay. The isolation of the 2,4'dipyridyl and 4,4'-dipyridyl was carried out by sclc with the recycling technique (8), using 1.9% THF, 1.59% dioxane, 1.39% methoxyethanol, and 95.12% hexane as mobile phase.

IDENTIFICATION.—Both alkaloids were identified by standard methods: hptlc, hplc, mp, spectral data (uv, ¹H nmr, eims) and compared with authentic samples (9). Full details on isolation and identification of the compounds are available on request.

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8-C-PRENYLATED FLAVONES FROM THE ROOTS OF TEPHROSIA HILDEBRANDTII

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Tepbrosia hildebrandtii Vatke (Leguminosae) is a herbaceous plant occurring in East Africa (1). From the roots of *T. hildebrandtii*, we have previously described the isolation and identification of hildecarpin, a new 6a-hydroxypterocarpan with insect antifeedant and antifungal properties (2,3), and the isolation and identification of four new β -substituted flavans (4). Here we report the isolation of two 8-*C*-prenylated flavones during a further study of the roots of *T. hildebrandtii*. They have been identified as 5,7-dimethoxy-8-(3"-hydroxy-3"-methyl-*trans*-but-1-enyl) flavone and 5,7-dimethoxy-8-(3"-methyl-*trans*-but-1,3dienyl)-flavone, named *trans*-tephrostachin and *trans*-anhydrotephrostachin, respectively (5).

Both *trans*-tephrostachin and *trans*-anhydrotephrostachin have been previously isolated from *Tephrosia bracteolata* Guill. et Pert. (5). Although *trans*-anhydrotephrostachin might reasonably be suspected as an artifact generated in the isolation procedure by dehydration of *trans*-tephrostachin, this does not, in fact, appear to be the case. *Trans*-anhydrotephrostachin appeared in the tlc of the original cold MeOH extract. Under the same conditions, no *trans*-anhydrotephrostachin was evident as a dehydration product occurring during the tlc of pure *trans*-tephrostachin.

EXPERIMENTAL

PLANT MATERIAL.—The roots of T. *bildebrandtii* were collected from Kilimambogo in Kenya; a voucher specimen is deposited at the Herbarium of the Botany Department, University of Nairobi, under the number 2418.

EXTRACTION, ISOLATION, AND IDENTIFICATION.—The air-dried roots (1.22 kg) were ground and extracted with MeOH in the cold, and the extract was evaporated in vacuo to give a gummy residue (69 g). The residue was partitioned between H₂O and CHCl₃. The CHCl₃ fraction (20.7 g) was then partitioned