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O-METHYLPELLOTINE, A NEW PEYOTE ALKALOID FROM LOPHOPHORA DIFFUSA*

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Key Word Index--Lophophora diffusa; Cactaceae; alkaloids; O-methylpellotine.

The Mexican cactus Lophophora diffusa (Croizat) H. Bravo has only recently been recognized as a Lophophora species in its own right [1,2]. Scores of chemical papers [3,4] have been published on its sister species L. williamsii (Lem.) Coulter, the mescaline-containing and vision-producing "peyote". L. diffusa, which is also called "peyote", has however not been extensively investigated.

In a TLC investigation of *Lophophora* populations, Todd [5] found pellotine to be the major alkaloid of *L. diffusa* and reported low amounts of lophophorine and anhalamine, and traces only of anhalonidine and mescaline. During a recent reinvestigation of *L. williamsii* and *L. diffusa* (Bruhn and Holmstedt. to be published) we were able to largely confirm Todd's findings, but we also found some minor unknown compounds. In the present communication we wish to report the identification of one of these unknown compounds as the tetrahydroisoquinoline alkaloid *O*-methylpellotine, not previously known to occur in nature.

The extremely low yields of non-phenolic alkaloids from *L. diffusa* did not permit the isolation of any compound. However, comparison with reference materials using TLC and GC indicated the presence of *O*-methylpellotine. After purification

by preparative TLC the band corresponding to reference O-methylpellotine was used for GC-MS. The identification of O-methylpellotine is based on identical MS and the same chromatographic behaviour (TLC, GC) as an authentic sample. O-Methylpellotine (1,2-dimethyl-6,7.8-trimethoxytetrahydroisoquinoline) has not earlier been identified in Lophophora species, although its presence could be expected, several closely related alkaloids already being found [3].

EXPERIMENTAL

Plant material. Lophophora diffusa (Croizat) H. Bravo was collected north of Vizarrón, Querétaro, on 29 June 1971 by Jan G. Bruhn and Sr. Hernando Sánchez-Mejorada, Departamento de Botánica, Instituto de Biología, Universidad Nacional Autónoma de México. Mexico City, who also identified the plants. A herbarium specimen has been placed in the Department of Pharmacognosy, Biomedicum, Uppsala.

Isolation of alkaloids. Experimental details have been described in earlier publications [6,7]. A part of the purified alkaloid extract (500 mg) was fractionated on an ion-exchange column (IRA 400) into phenolic and non-phenolic alkaloids. These fractions were then studied by TLC and GC. Only 2% (10 mg) of the total alkaloids were recovered as non-phenolic alkaloids. The non-phenolic alkaloids were subjected to preparative TLC on Si gel G plates. System: CHCl₃--EtOH-NH₃ conc (80:20:0,4). The band corresponding to O-methylpelotine was scraped off and eluted with hot EtOH. After filtration and evaporation, the residue was dissolved in a small amount of abs. EtOH and studied by GC.

Identification of O-methylpellotine. GC on 2 columns (5% SE-30 and 5% XE-60 on Gas Chrom Q, 100/120 mesh. col. temp. 150°) showed a peak with the same retention times as O-methyl-

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pellotine. A MS of this peak was then obtained using a combined GC-MS instrument (ion source 3.5 kV, electron energy 70 eV and ionization current 60 μ A). Column: 3% XE-60 on Gas Chrom Q. 100/120 mesh, 150°. MS data: Compound in L. diffusa, major peaks, m/e 251 (M⁺, 0.5%), 236 (100%), 220 (23%), 206 (30%). Reference O-methylpellotine, major peaks, m/e 251 (M⁺, 0.5%), 236 (100%), 220 (8%), 206 (20%). The MS are in accord with published data [8].

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FLAVONES FROM PEACOCK AND REGAL ANNE CHRYSANTHEMUM FLOWERS

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Key Word Index—Chrysanthemum morifolium; Compositae; flavones; apigenin 7-O-glucoside; acacetin 7-O-glucoside; luteolin 7-O-glucoside; diosmetin 7-O-glucoside.

Plants. Chrysanthemum morifolium Ramat. cv. Regal Anne and Peacock. Source. Yoder Bros., Inc., Barberton, OH 44203, U.S.A. Uses. Ornamental. Previous work. Acacetin 7-rhamnoglucoside from the flowers of Chrysanthemum sinense Sab [1].

Present work. Fresh flowers of each cv. were extracted with hot MeOH and the concentrated extract taken up in citrate-phosphate buffer pH 3·0. Flavones were separated into several fractions by column chromatography on polyvinylpyrrolidone (PVP) [2] with 30% aqueous MeOH. Isolation of the individual flavones was by preparative TLC on microcrystalline cellulose (C₆H₆-HOAc-H₂O, 6:7:0·9; iso-PrOH-HCOOH-H₂O, 2:5:5; and 20% HOAc) and identification was by cochromatography with authentic samples, UV spectra and MS fragmentation patterns.

The same four flavones were isolated from each cv. Under UV radiation compound (1) was deep purple and the color did not change in ammonia vapor. Spectral data were those of a 4',7-disubsti-

tuted apigenin. The λ_{max} in EtOH were 324 and 268 nm; with NaOEt 365 (decrease in extinction), 286, and 245 (sh) nm; with AlCl₃ 382, 338, 300 and 278 nm; and with NaOAc or NaOAc-H₃BO₃ no appreciable change. Controlled acid hydrolysis yielded no intermediate glycoside, and the final products were glucose and an aglycone spectrally and chromatographically indistinguishable from apigenin 4'-methyl ether (acacetin) [3]. The MS fragmentation pattern of the aglycone and an authentic sample of acacetin were identical and showed that the molecular ion was at m/e 284. Principal fragments were observed at m/e 269, 256, and 241. Compound (1) was acacetin 7-O-glucoside and was indistinguishable from an authentic sample.

Compound (2) was deep purple under UV radiation and the color changed to yellow-green in ammonia vapor. Spectral data were those of a 7-substituted apigenin. The λ_{max} in EtOH were 335 and 268 nm; with NaOEt 386 (increase in extinction) 355 (sh), 295 (sh), and 268 nm; with AlCl₃ 383,