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CACTUS ALKALOIDS, LXI. IDENTIFICATION OF MESCALINE AND RELATED COMPOUNDS IN EIGHT ADDITIONAL SPECIES USING TLC AND MS/MS

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The well-known cactus alkaloid mescaline (3,4,5-trimethoxy-β-phenethyamine) is present in *Lophophora* and some species of *Trichocereus* in sufficient concentrations to impart hallucinogenic activity upon ingestion by humans (1). It is less well known that additional cactus species, representing all three cactus subfamilies, can also contain this hallucinogen although in lesser concentrations (2). In this paper we report the detection of small traces of mescaline in eight additional species (Table 1), using a combination of tlc (3) and mass spectrometry (ms/ms) (4). Quantitative tlc estimated the maximum concentration in the richest of these species (*Stenocereus stellatus*) to be at the level of 0.01% of the dry weight, an insufficient amount to cause hallucinations upon ingestion. This is the first report of the detection of mescaline in *Polaskia*, *Pterocereus*, and *Stenocereus*. *Neoraimondia macrostibas* is an ingredient, with *Trichocereus pachanoi* and several noncactaceous plant species, of the Peruvian hallucinogenic drink, "cimora" (1); the absence of mescaline in *Neoraimondia arequipensis* var. *roseiflora*, formerly treated as a variety of *N. macrostibas* (5), leaves unexplained this folkloric use.

The presence of the biosynthetically-related compounds (6), 3,4-dimethoxy-β-phenethylamine and 3,5-dimethoxy-4-hydroxy-β-phenethylamine, was simultaneously determined. The tlc separation of mescaline and the former compound was difficult (7), but their separation by ms/ms and detection in the alkaloid extract was facile because the protonated molecules of each yielded characteristic daughter ion spectra. Ms/ms can thus be a rapid means of direct screening of crude extracts for series of trace compounds, including the detection of minute amounts of biosynthetic precursors.

TABLE 1. Tlc and Ms/ms Identifications of Mescaline, 3,4-Dimethoxy-β-phenethylamine, and 3,5-Dimethoxy-4-hydroxy-β-phenethylamine[a]

Plant Species	Source[b]	Mescaline		3,4-dimethoxy-β-phenethylamine		3,5-dimethoxy-4-hydroxy-β-phenethylamine	
		tlc	ms/ms	tlc	ms/ms	tlc	ms/ms
Escontria chiotilla (Web.) Rose	1	−	−	−	−	−	±
Melocactus maxonii (Rose) Gurke	2	−	−	±	−	+	−
Neoraimondia arequipensis var. *roseiflora* (Werd. and Backeb.) Rauh.	3 (No. 85055)	−	−	−	±	−	±
Opuntia acanthocarpa Engelm. and Bigel.	4 (No. 8320)	+	+	+	+	−	±
Opuntia basilaria Engelm. and Bigel.	4 (No. 8504)	−	+	−	−	−	±
Opuntia bigelovii Engelm	4 (No. 8508)	−	−	−	−	−	−
Opuntia echinocarpa Engelm. and Bigel.	4 (No. 8327 and 8328)	±	+	±	+	−	+
Opuntia exaltata (Berg.) Backeb.	3 (No. 84284)	−	−	−	±	−	±
Opuntia ramosissima Engelm.	4 (No. 8501)	−	−	−	±	−	−
Polaskia chende (Gossel.) Gibs. and Horak	5 (HBG 6301)	±	+	±	+	+	+
Pterocereus foetidus MacDoug. and Mir.		−	−	−	+	−	±
Pterocereus gaumeri (Britt. and Rose) MacDoug. and Mir.	5	±	±	±	+	+	±
Stenocereus beneckei (Ehrenb.) Buxb.	5 (HBG 32973)	±	±	±	+	+	+
Stenocereus eruca (Brandeg.) Gibs. and Horak	6 (Gib. 3625)	±	−	±	−	+	−
Stenocereus stellatus (Pfeiff.) Rice.	5 (HBG 34963)	+	+	+	+	+	+
Stenocereus treleasei[c] (Britt. and Rose) Backeb.	5	+	+	+	+	+	+

[a]+ = Alkaloid readily apparent at concentration ca. 0.01% of dry weight; ± = alkaloid appears to be present at concentrations less than 0.01% of dry weight; and − = alkaloid not detected with lower limit of detection less than 0.001% of dry weight.

[b]Source 1: Cactus Ranchito, Tarzana, California.
Source 2: purchased from Abbey Garden, 4620 Carpinteria Avenue, Carpinteria, California 93013.
Source 3: cuttings collected by C.N.O.
Source 4: cuttings collected by F.Z.
Source 5: cuttings from plants growing at the Huntington Botanical Garden, 1151 Oxford Road, Marino, California 81108.
Source 6: cuttings collected in Baja California, Mexico by A.C.G.

[c]This plant is an unvouchered specimen growing at the Huntington Botanical Garden and may be a form of *S. stellatus*.

EXPERIMENTAL

PLANT MATERIALS AND EXTRACTION.—Cuttings were obtained from authenticated plants as indicated in Table 1. After freeze-drying, the materials were reduced to a 2 mm powder in a Wiley mill. Samples of 5 g were basified, extracted with $CHCl_3$, and partitioned to yield extract A (alkaloids) (8).

THIN LAYER CHROMATOGRAPHY.—Analytical plates of Si gel (Merck F-254) were developed in one of three solvent systems: Me_2CO-Et_2O-MeOH-NH_4OH (6:6:5:1), Et_2O-MeOH-NH_4OH (8:4:1), and $CHCl_3$-EtOH-NH_4OH (7:7:1). Traces of the primary amines were detected by their characteristic yellow-green uv fluorescence after spraying with fluorescamine (3). Overspraying with tetrazotized benzidine or iodoplatinic acid reagents then produced visual chromophores (9). Cochromatography of reference alkaloids with the alkaloids in the extracts was taken as tlc evidence for alkaloid presence. Quantities of mescaline, ranging from 1-24 μg, were chromatographically compared with known dilutions of extract A from *S. stellatus* to estimate mescaline concentration.

TANDEM MASS SPECTROMETRY.—A Finnigan TSQ (Model 4500) triple quadrupole mass spectrometer was used (10,11). This system consists of three coaxially arranged quadrupole rod assemblies. The first and third quadrupoles are conventional mass analyzers, and the second quadrupole is used as a focusing

collision cell to generate daughter spectra from selected parent ions. Samples were introduced into the source by the direct chemical ionization method (12). Isobutane and, independently, isobutane together with NH_3, were employed as the chemical ionization reagent gases (0.4-0.7 torr), and argon was used as the collision gas at a gauge pressure of 2.0 mtorr. The collision energy was 20 eV. Characteristic parent and daughter ions were observed and recorded as follows; mescaline: *m/z* 212 $(M+H)^+$, 195 $(M+H\text{-}NH_3)^+$, 180 $(M+H\text{-}NH_3\text{-}CH_3)^+$, 168 (M+H-side chain)$^+$, 165 $(M+H\text{-}NH_3\text{-}CH_2O)^+$; 3,4-dimethoxy-β-phenethylamine: *m/z* 182 $(M+H)^+$, 165 $(M+H\text{-}NH_3)^+$, 150 $(M+H\text{-}NH_3\text{-}CH_3)^+$, 138 (M+H-side chain)$^+$; 3,5-dimethoxy-4-hydroxy-β-phenethylamine: *m/z* 198 $(M+H)^+$, 181 $(M+H\text{-}NH_3)^+$, 166 $(M+H\text{-}NH_3\text{-}CH_3)$, 153 (M+H-side chain-H)$^+$, 121 $(M+H\text{-}NH_3\text{-}2CH_2O)^+$. The daughter spectra recorded with NH_3 reagent gas did not display the peak due to loss of the side chain but were otherwise very similar to those recorded using isobutane.

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FLAVONOIDS FROM *STEPHANODORIA TOMENTELLA*

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In a continuation of our chemotaxonomic studies of the '*Gutierrezia-Xanthocephalum* complex' (tribe Astereae, Compositae) (1-6), we have now investigated *Stephanadoria tomentella* Greene. This taxon is a monotypic genus at one time thought to be related to *Gutierrezia* and *Xanthocephalum*. In this study we report eight flavonoids, namely: kaempferol 3-*O*-β-glucoside, kaempferol 3-*O*-β-galactoside, kaempferol 3-*O*-β-glucuronide, quercetin 3-*O*-β-glucuronide, quercetin 3-*O*-β-glucoside, vitexin, vicenin-2, and quercetin 3-methyl ether. We also include previously unreported uv data for the natural product kaempferol 3-*O*-β-glucuronide and ^{1}H-nmr data for its trimethylsilyl ether.

EXPERIMENTAL

PLANT MATERIAL.—Leaves and heads of *S. tomentella* (500 g) were collected from the state of San Luis Potosi, Mexico, near the railroad station at Gerritos between Hwy. 57 and Hwy. 80 by Mark Leidig and Meredith Lane in June 1981. A voucher specimen (Mark, S.N.) is on deposit in the Plant Resources Center at the University of Texas at Austin, Austin, Texas.

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