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Phenylethylamines from *Browningia candelaris* (Cactaceae)

[Jeniletilaminas de *Browningia candelaris* (Cactaceae)]

Javier ECHEVERRÍA & Hermann M. NIEMEYER

Abstract

The analysis by gas chromatography coupled with mass spectrometry (GC-MS) of the alkaloidal extract of *Browningia candelaris* (Cactaceae) showed the presence of N-acetyl-3,4-dimethoxyphenylethylamine; N,N-dimethyl-3,4-dimethoxyphenylethylamine; N,N-dimethyl-4-methoxyphenylethylamine; and 4-methoxyamphetamine. The presence of these psychoactive compounds is discussed in terms of their possible ritual use in Andean cultures of Northern Chile.

Keywords: *Browningia candelaris*, Cactaceae, phenylethylamines, GC-MS, psychoactive natural products.

Resumen

El análisis por medio de cromatografía de gases acoplada a espectrometría de masas (CG-EM) del extracto alcaloidal de *Browningia candelaris* (Cactaceae) mostró la presencia de N-acetil-3,4-dimetoxifeniletilamina; N,N-dimetil-3,4-dimetoxifeniletilamina; N,N-dimetil-4-metoxifeniletilamina y 4-metoxianfetamina. La presencia de estos compuestos psicoactivos se discute en términos de su posible utilización en ceremonias mágico-religiosas por culturas andinas del norte de Chile.

Palabras Clave: *Browningia candelaris*, Cactaceae, feniletilaminas, CG-EM, productos naturales psicoactivos.

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INTRODUCCIÓN
Archaeological, ethnographic and ethnohistoric studies have demonstrated a long tradition of consumption of hallucinogenic substances by pre-hispanic peoples of the Americas (Schultes and Hofmann, 1980). The main native sources that have been utilized are the trees Anadenanthera spp. (Fabaceae, tryptamines) and Virola spp. (Myristicaceae, tryptamines), the vine Banisteriopsis spp. (Malpighiaceae, β-carbolines), the shrubs Psychotria spp. (Rubiacées, tryptamines) and Brugmansia spp. (Solanaceae, tropane alkaloids), the herbs Ipomoea spp. (Convolvulaceae, ergot alkaloids) and Nicotiana spp. (Solanaceae, nicotine derivatives), and the cacti Lophophora sp. and Trichocereus spp. (Cactaceae, phenylethylamines) (Schultes et al., 1998). Thus, the importance of alkaloid usage in this context is overwhelming. Alkaloids are widespread in nature (Cordell et al., 2001); it has been estimated that at least 200 genera from 20 families in the flora of the Americas should contain them (Schultes and Hofmann, 1980). Flora presently unknown to us may have provided the necessary hallucinogenic ingredients that might have been used by aboriginal peoples (Torres and Repke, 2006). Hence, we have undertaken the analysis of native plants from Northern Chile in search for alkaloids.

Numerous genera of the Cactaceae are known sources of hallucinogenic alkaloids, albeit their use in cultural contexts is less widespread than that of Lophophora and Trichocereus (Schultes et al., 1998). We report herein a study on Browningia candelaris (Meyen) Britton & Rose, a cactus species found in areas of extreme aridity and poor rocky soils of the Western slopes of the Andes from Arequipa in southern Peru to Quebrada de Tarapacá in northern Chile (Anderson, 2005). To the best of our knowledge, no reports have been published on the chemical analysis of species of the genus Browningia.

MATERIAL AND METHODS
Plant material
Aerial parts of Browningia candelaris were collected at Cuesta Cardones, Región de Arica y Parinacota, Chile (18°25.9’S, 69°48.8’W, 2750 masl) in May 2010. Voucher specimens were deposited in the herbarium at Universidad de Concepción.

Extraction of alkaloids
Oven dried plant material (10 g) was ground in a knife mill and extracted with 50 mL of MeOH under reflux for 1 h. The suspension obtained was cooled down to room temperature and filtered under reduced pressure through a Buchner funnel using paper filter; the resulting methanolic extract was evaporated under reduced pressure on a rotary evaporator. The syrupy residue (0.40 g) was dissolved and stirred with 50 mL of 5% HCl for 1 h; the neutral compounds were extracted with CHCl3 (2x15 mL) in a separatory funnel. The acid aqueous phase was adjusted to pH 10 with NH3·OH and the alkaloids extracted with CHCl3 (3x10 mL) until the extracts gave negative Dragendorff reaction. Finally, the pooled organic extracts were dried with anhydrous Na2SO4; evaporation of the solvent yielded an extract potentially containing alkaloids (9.60 mg).

GC/MS analysis of alkaloids
GC/MS analysis was performed with a Shimadzu model GCMS-QP 2010 Ultra gas chromatograph (Shimadzu, Kyoto, Japon), equipped with a Rtx-5MS Crossbond 5% diphenyl 95% dimethyl polysiloxane (Restek, Bellefonte, PA, USA) capillary GC column (30 m length, 0.25 mm I.D., 0.25 μm film thickness). The GC was operated in the splitless injection mode using 2 μL injection volume. The column temperature was initially held at 30°C for 3 min, then raised at 25°C/min to 230°C, and maintained for 10 min at 230°C. The carrier gas was helium at a flow rate of 1.32 mL/min. The mass spectrometer used electron impact (EI) ionization mode (70 eV) with an emission current of 250 μA. The temperatures of the injection port, ion source and transfer line were set at 250°C, 250°C and 280°C, respectively. Qualitative analysis of compounds was carried out comparing the retention indices and MS spectra for the obtained peaks with the data in the NIST98 database.

RESULTS AND DISCUSSION
A capillary GC/MS analysis of the alkaloid extract from dry cladodes of B. candelaris yielded four alkaloids (Figure 1), identified as phenylethylamines on the basis of their retention times and mass spectra: N-acetyl-3,4-dimethoxyphenethylamine (compound 1); N,N-dimethyl-3,4-dimethoxyphenethylamine (compound 2); N,N-dimethyl-4-methoxyphenethylamine (compound 3) and p-methoxymphetamine (compound 4). The structures of these compounds are shown in Figure 2. The relative proportion of these alkaloids was determined as a percentage of total alkaloids identified using the integrated area of the GC/MS peaks (Table 1).

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Phenylethylamines found in the *B. candelaris* are related to alkaloids identified in other species of cacti such as mescaline (3,4,5-trimethoxyphenethylamine) (5), a psychoactive alkaloid present in peyote (*Lophophora williamsii*) and in San Pedro (*Trichocereus pachanoi*); these two cactus species have been used extensively since pre-hispanic times by peoples in Mesoamerica and in the central and southcentral Andes, respectively. The occasional use of *B. candelaris* as source of hallucinogens may be suggested, given its presence along the route connecting the settlements in the Azapa valley of Northern Chile with the Titicaca basin in the Bolivian altiplano, the site of the Tiwanaku state (Berenguer, 1998).

**FIGURE 1**

Gas chromatogram of alkaloidal fraction of *B. candelaris* showing the peaks identified (compounds 1 to 4).

![Gas chromatogram of alkaloidal fraction of B. candelaris showing the peaks identified (compounds 1 to 4).](image)

<table>
<thead>
<tr>
<th>Compound</th>
<th>RT (min)</th>
<th>RI_{exp}</th>
<th>R_{lit}</th>
<th>Relative area (%)</th>
<th>Identification</th>
</tr>
</thead>
<tbody>
<tr>
<td>N-Acetyl-3,4-dimethoxyphenethylamine</td>
<td>13.07</td>
<td>1996</td>
<td>1978</td>
<td>6.0</td>
<td>R_{lit}, MS</td>
</tr>
<tr>
<td>N,N-Dimethyl-3,4-dimethoxyphenylethylamine</td>
<td>11.00</td>
<td>1634</td>
<td>1615</td>
<td>25.5</td>
<td>R_{lit}, MS</td>
</tr>
<tr>
<td>N,N-Dimethyl-4-methoxyphenylethylamine</td>
<td>10.00</td>
<td>1445</td>
<td>1430</td>
<td>34.1</td>
<td>R_{lit}, MS</td>
</tr>
<tr>
<td>4-Methoxyamphetamine</td>
<td>9.94</td>
<td>1431</td>
<td>1403</td>
<td>34.4</td>
<td>R_{lit}, MS</td>
</tr>
</tbody>
</table>

MS: Mass spectrum.  
RT: Retention time.  
RI_{exp}: Experimental retention index.  
R_{lit}: Retention index on VF-5MS capillary column, from the literature (Todua, 2011).  
1: Relative area of compounds identified.
FIGURE 2
Phenylethylamines identified in the alkaloid fraction of cladodes of *B. candelaris* (Cactaceae) (1-4) and mescaline (5).

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