dewevrei (*C. dewevrei* De Wild. et Durand var. *excelsa* Chev. and *C. dewevrei* De Wild. et Durand var. *aruwimiensis* (De Wild.) Chev.).

The dried and finely ground leaf material (portions of 1 g) was boiled in 125 ml 0.01 n H_2SO_4 (20 min), mixed with 13 g MgO, cooled and filtered through glass filter G4. The filtrate was extracted with $CHCl_3$ (100 ml × 3). Following evaporation of $CHCl_3$, the concentrate was chromatographed by preparative TLC on Si gel ($CHCl_3$–MeOH 9:1). The zone with R_f 0.52 was eluted with MeOH and rechromatographed. Crystallization from MeOH (and few drops of H_2O) yielded needles (mp 202°) with mass, UV and IR spectra [1] identicl to those of the authentic sample of 3 with mp 205° prepared by methylation of 7,9-dimethyluric acid [3,4]. Thermal rearrangement of both the natural and synthesized sample gave 1,3,7,9-tetramethyluric acid (1).

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CHONDROFOLINE FROM *UVARIA OVATA**

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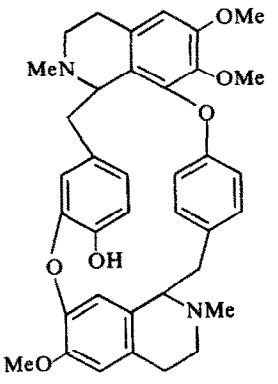
Key Word Index—*Uvaria ovata*; Annonaceae; chondrofoline; *bis*-1-benzyltetrahydroisoquinoline alkaloid.

Chondrofoline, a member of the rare group of 7,3‴–8′,4″-linked *bis*-benzylisoquinolines, has been found in *Uvaria ovata* (Annonaceae); it has previously been found only in *Chondrodendron platyphyllum* Miers (Menispermaceae) [1]. Alkaloids of this type have previously been isolated only from Menispermaceae and Lauraceae [2] and their discovery in another, closely allied, Ranalean family yet again illustrates the potential value of alkaloids in the systematics of the Ranales.

EXPERIMENTAL

Plant. Uvaria ovata A. DC; Voucher. Enti 1284, deposited at the herbarium of the Royal Botanic Garden, Edinburgh; Source. Achimota, Ghana.

Alkaloid isolation. Powdered leaf (650g) was extracted successively with petrol (40–60°), $CHCl_3$ and MeOH. Acid extraction of the $CHCl_3$ concn., basification of the acid extract with NH_3 and re-extraction into $CHCl_3$ gave a mixture of alkaloids. Col. chr. of the mixture over Al gave, on elution with $CHCl_3$–MeOH (99:1), a single alkaloid. Recrystallisation of the alkaloid from $CHCl_3$–Et_2O and finally Et_2O gave plates (97 mg) mp 136–140°. $[\alpha]_{546}^{20}$ − 257 (*c* 0.10 in 0.1 N HCl). Found, M^+ 608.2877; $C_{37}H_{40}N_2O_6$ requires 608.2886. UV λ_{max}^{EtOH} nm (log ε) 232 (4.57), 281 (3.97), undergoing a bathochromic shift on the addition of alkali. IR ν_{max} (KBr) cm^{-1} 3450 (OH). PMR ($CDCl_3$) δ 2.30 (3H, *s* N-Me), 2.58 (3H, *s* N-Me), 3.78 (3H, *s* OMe), 3.92 (6H, *s* 2 × OMe), 2.60–3.85 (14H, *m* CH_2 and CH), 6.00 (1H, *s* C-8′-H), 6.65–7.35 (9H, *m* Har). MS 608 (91), 607 (50), 487 (2), 312 (92), 311 (19), 299 (24), 298 (100), 266 (10), 204 (23), 192 (12), 190 (15), 176 (11.5), 174 (19), 161 (6.5), 159 (10), 146 (11), 145 (15). From UV, IR and PMR spectra and accurate mass measurement of the molecular ion, it appeared likely that the alkaloid was of the *bis*-1-benzyltetrahydroisoquinoline type, with one OH, 3 × OMe and 2 × NMe substituents. The significance of MS fragmentation patterns in the identification of *bis*-benzylisoquinoline alkaloids and the probable origin of the fragments observed have been thoroughly discussed [3]. The major ions, at *m/e* 312 ($C_{19}H_{22}NO_3$) and *m/e* 298 ($C_{18}H_{20}NO_3$), indicated that the two benzylisoquinoline moieties were linked head to tail. In addition, the relatively high abundance of an ion at *m/e* 204 ($C_{12}H_{14}NO_2$) suggested the presence of a 6,7-dimethoxyisoquinoline fragment, and the relatively low abundance of an ion corresponding to loss of Me from the other benzylisoquinoline subunit indicated MeO substitution in this isoquinoline unit rather than in the benzyl group attached to it. A detailed comparison of the complete MS with those of known head to tail linked *bis*-benzylisoquinolines [4] suggested that the alkaloid



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* Part 3 in the series 'Chemical Studies on the Annonaceae'. For Part 2 see Panichpol, K., Waigh, R. D. and Waterman, P. G. (1976) *J. Pharm. Pharmacol.* **28**, 71p.

was chondrofoline (1) or a stereoisomer thereof. The stereochemical coidentity of the alkaloid from *U. ovata* was ascertained by comparison of the ORD spectrum [1, 5] with that of authentic chondrofoline and the structure was finally confirmed by direct comparison (UV, IR, mmp, TLC).

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PHENETHYLAMINES FROM *ECHINOCEREUS CINERASCENS* AND *PILOSOCEREUS CHRYSACANTHUS**

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Key Word Index—*Echinocereus cinerascens*; *Pilosocereus chrysacanthus*; Cactaceae; alkaloids; *N,N*-dimethyl-3,4-dimethoxyphenethylamine; *N*-methyl-3,4-dimethoxyphenethylamine; *N*-methyl-3,4-dimethoxyphenethylamine.

In a field screening of Mexican cacti for the presence of alkaloids, *Echinocereus cinerascens* (DC.) Rümpler and *Pilosocereus chrysacanthus* (Web.) Byl. et Rowl. were found to give positive tests with the Dragendorff reagent [1]. Plants were collected and the alkaloids extracted and studied. The present report describes the isolation and identification of the major phenethylamine alkaloids of these two cactus species.

Although several alkaloid screening papers have listed various *Echinocereus* species [2–4], only one species, *E. merkeri*, has been investigated in more detail. *N,N*-dimethyl-3,4-dimethoxyphenethylamine was isolated for the first time from *E. merkeri*, which contains several additional phenethylamines and the tetrahydroisoquinoline salsoline [5, 6].

We have now identified the major alkaloid of *E. cinerascens* as *N,N*-dimethyl-3,4-dimethoxyphenethylamine. Alkaloid extraction followed by fractionation on an alumina column led to the isolation of this compound, as well as small amounts of *N*-methyl-3,4-dimethoxyphenethylamine. *E. cinerascens* has an edible fruit [7] and dry plants are used as fuel [8], but no medicinal uses seem to have been recorded for *E. cinerascens* or *Pilosocereus chrysacanthus*. The major alkaloid of the latter species was identified as *N*-methyl-3,4-dimethoxyphenethylamine.

The alkaloids now isolated were identified by comparison with synthetic reference materials using TLC, GC, IR, and MS. A part of the *N,N*-dimethyl-3,4-dimethoxyphenethylamine isolated from *E. cinerascens* was oxidized to the corresponding 3,4-dimethoxybenzoic acid, identified by IR and mp comparison with an authentic sample.

N-Methyl- and *N,N*-dimethyl-3,4-dimethoxyphenethylamine have been reported from *E. merkeri* [5], and are also found in other genera of the Cactaceae, e.g. *Coryphantha* [9] and *Ariocarpus* [10].

EXPERIMENTAL

Plant material. Echinocereus cinerascens (DC.) Rümpler (4.1 kg) was collected north of Pachuca, Hidalgo, and *Pilosocereus chrysacanthus* (Web.) Byl. et Rowl. (3.0 kg) near San Antonio Texcala, Puebla, by the authors

Alkaloid extraction. Fresh plant material was homogenized in EtOH. The filtered extracts were evaporated to dryness and dissolved in 3% HOAc. The aq. phases were extracted with $CHCl_3$ and the $CHCl_3$ discarded. Aq. phases were basified with NH_3 conc (pH 10) and alkaloids extracted with $CHCl_3$ and $CHCl_3$–EtOH (3:1). Crude alkaloids were purified on an acid diatomaceous earth column (Celite 545). Yield of alkaloids. *E. cinerascens* 585 mg; 0.014%; *P. chrysacanthus* 684 mg; 0.02%.

Isolation and identification. The alkaloid extract of *E. cinerascens* (525 mg) was fractionated on an aluminium oxide column (Merck, act. II–III acc. to Brockmann) as earlier described [5]. The eluates were analyzed by TLC and GC (5% SE-30 and 5% XE-60 columns, col. temp. 150°) [11]. MS were obtained with a combined GC–MS instrument (ion source 2.5 kV, electron energy 70 eV, and ionization current 60 μA). *N,N*-Dimethyl-3,4-dimethoxyphenethylamine was eluted with $CHCl_3$–C_6H_6 (1:2) and crystallized as the hydrochloride (292 mg) mp 193–197°; lit. mp 194–196° [4]. Alkaline permanganate oxidation of 50 mg of this compound gave 10 mg of 3,4-dimethoxybenzoic acid, mp 178–181°; lit. mp 181° [12]. *N*-methyl-3,4-dimethoxyphenethylamine was isolated from the $CHCl_3$–MeOH (4:1) fractions as the hydrochloride (yield 8 mg), mp 134–136°; lit. mp 136–137° [4]. Preparative TLC on Si gel GF plates in $CHCl_3$–EtOH–NH_3 conc (80:20:0.4) of 80 mg of the *P. chrysacanthus* alkaloids yielded *N*-methyl-3,4-dimethoxyphenethylamine, which was crystallized as the hydrochloride (yield 21 mg), mp 134–135°; lit. mp 136–137° [4].

*Cactaceae Alkaloids. 27.