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Crystal structure of 1,2,5-trimethyl-1,2,3,4-tetrahydropyrido[4,3-*b*]carbazole (guatambuine), $C_{18}H_{20}N_2$, *from Aspidosperma subincanum Mart.*

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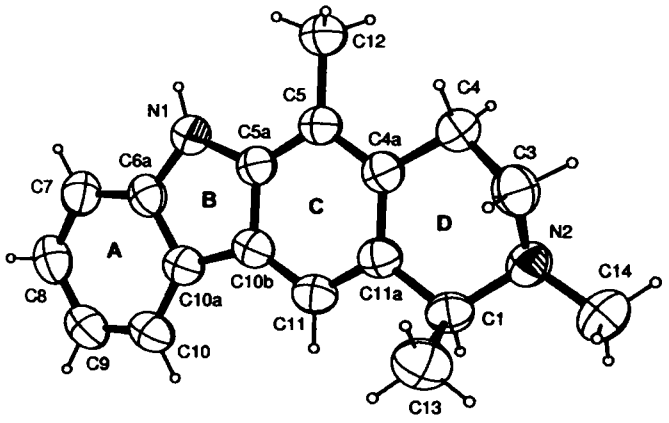

Abstract

$C_{18}H_{20}N_2$, orthorhombic, $P2_12_12_1$ (no. 19), a = 11.587(5) Å, b = 8.130(5) Å, c = 15.239(5) Å, V = 1435.5 Å^3, Z = 4, $R_{gt}(F)$ = 0.055, $wR_{ref}(F^2)$ = 0.137, T = 293 K.

Source of material

The title compound, guatambuine, a pyrido[4,3-*b*]carbazole alkaloid, was isolated from the chloroform fraction of the ethanol extract from the stem of *Aspidosperma subincanum*, a tree of the *Apocynaceae* family. The crystals of the guatambuine were obtained by recrystallization from MeOH at room temperature.

Experimental details

H atoms attached to C atoms were located on stereochemical grounds and refined with fixed bond lengths and angles, each riding on a carrier atom, with an isotropic displacement parameter amounting to 1.5 (for methyl) or 1.2 (for the other H atoms) times the value of the equivalent isotropic displacement parameter of the atoms to which they are bonded. The H atom attached to the N atom was refined freely.

Discussion

The title compound, 1,2,5-trimethyl-1,2,3,4-tetrahydropyrido[4,3-*b*]carbazole, belongs to the class of indole alkaloids, a large group of biologically active compounds of naturally occurrence. The indole alkaloids have numerous biological activities such as anti-tumor, anti-microbial, anti-hypertensive and stimulant for the central nervous system [1]. The occurrence of the indole alkaloids is described at the *Apocynaceae, Rubiaceae*, and *Loganiaceae* families [2]. Among the *Apocynaceae*, the genus *Aspidosperma* is especially rich in indole alkaloids [3]. *Aspidosperma subincanum* is a Brazilian medicinal plant used in the treatment of general debility, digestive tract disorders, dysentery and against fever [4]. Previous separate investigations of this plant revealed the presence of more than 15 indole alkaloids [5]. In our search for structurally unique and biologically interesting compounds from Brazilian medicinal plants we are working with the stem and stem bark of *A. subincanum*. From the ethanolic extract of stem of *A. subincanum* we isolated guatambuine or *N*-methyltetrahydroolivacine, which has received considerable interest as result of this anti-tumor activity [6]. Guatambuine was isolated previously from others *Aspidosperma* species [7].

All the bond lengths and angles are within experimental error in good agreement with literature values. The atoms of three rings A, B and C of the molecule are nearly coplanar; the largest deviation (0.040(3) Å) from the least-squares plane is exhibited by atom C5. The heterocyclic ring D is in a half-chair conformation, as indicated by the Cremer & Pople puckering parameters [8]: q_2 = 6.221 Å, q_3 = –5.103 Å, φ = 263.59°, Q = 8.046 Å and θ = 129.35°. The C1 and C4 atoms of the heterocyclic ring D are located on the same plane of the rings A, B and C, while atoms C3 and N2 are located above and below respectively of that plane. Neighbored molecules are held together through one strong hydrogen interaction: N–H···N, where d(N1···N2^i) = 3.015(4) Å, d(H1N···N2^i) = 2.148(4) Å, d(N1—H1N) = 0.98(2) Å, ∠N1–H1N···N2^i = 168(2)° (symmetry operation i: x–½,–y+½,–z).

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.09 × 0.12 × 0.18 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.81 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, ω
$2\theta_{max}$:	49.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4743, 2497
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1830
$N(param)_{refined}$:	185
Programs:	SHELXS-97 [9], SHELXL-97 [10], ORTEP-3 [11], WinGX [12]

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Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1N)	4*a*	0.758(4)	0.308(5)	−0.136(3)	0.10(1)
H(1)	4*a*	1.1958	−0.1424	0.0234	0.061
H(2)	4*a*	1.0625	−0.1496	−0.1027	0.062
H(3)	4*a*	0.9836	−0.1621	−0.2820	0.071
H(4)	4*a*	0.8872	−0.1235	−0.4120	0.077
H(5)	4*a*	0.7453	0.0755	−0.4241	0.076
H(6)	4*a*	0.6991	0.2426	−0.3066	0.070
H(8A)	4*a*	1.0477	0.2470	0.1500	0.063
H(8B)	4*a*	0.9307	0.1628	0.1754	0.063
H(9A)	4*a*	1.0954	0.0502	0.2496	0.070
H(9B)	4*a*	1.0189	−0.0828	0.2025	0.070
H(10A)	4*a*	1.3166	−0.1618	0.1537	0.105
H(10B)	4*a*	1.2182	−0.2302	0.2140	0.105
H(10C)	4*a*	1.2810	−0.0658	0.2387	0.105
H(11A)	4*a*	1.1349	−0.3653	0.1110	0.107
H(11B)	4*a*	1.0625	−0.3549	0.0243	0.107
H(11C)	4*a*	1.0097	−0.2926	0.1130	0.107
H(12A)	4*a*	0.7901	0.3938	−0.0010	0.091
H(12B)	4*a*	0.8964	0.4268	0.0602	0.091
H(12C)	4*a*	0.7964	0.3112	0.0918	0.091

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	4*a*	0.8045(2)	0.2237(3)	−0.1432(2)	0.050(2)	0.055(1)	0.052(2)	0.005(1)	−0.004(1)	−0.005(1)
N(2)	4*a*	1.1678(2)	−0.0391(3)	0.1405(2)	0.053(2)	0.050(1)	0.057(2)	0.003(1)	−0.003(1)	0.006(1)
C(1)	4*a*	1.1282(3)	−0.1283(3)	0.0614(2)	0.054(2)	0.043(2)	0.056(2)	0.002(1)	0.005(1)	0.004(1)
C(1A)	4*a*	1.0417(3)	−0.0246(3)	0.0106(2)	0.047(2)	0.041(2)	0.054(2)	−0.002(1)	0.004(2)	0.002(1)
C(2)	4*a*	1.0220(3)	−0.0638(3)	−0.0765(2)	0.057(2)	0.042(1)	0.057(2)	0.001(1)	0.004(2)	−0.002(1)
C(2A)	4*a*	0.9414(3)	0.0258(3)	−0.1252(2)	0.047(2)	0.044(2)	0.049(2)	−0.006(1)	0.008(1)	−0.002(1)
C(2B)	4*a*	0.8998(3)	0.0172(3)	−0.2143(2)	0.048(2)	0.047(2)	0.051(2)	−0.005(1)	0.006(2)	−0.001(1)
C(3)	4*a*	0.9269(3)	−0.0815(4)	−0.2865(2)	0.064(2)	0.056(2)	0.059(2)	−0.007(2)	0.011(2)	−0.010(2)
C(4)	4*a*	0.8693(3)	−0.0582(4)	−0.3638(2)	0.070(2)	0.066(2)	0.057(2)	−0.012(2)	0.005(2)	−0.013(2)
C(5)	4*a*	0.7839(3)	0.0624(4)	−0.3711(2)	0.062(2)	0.078(2)	0.050(2)	−0.014(2)	−0.004(2)	−0.004(2)
C(6)	4*a*	0.7559(3)	0.1624(4)	−0.3013(2)	0.050(2)	0.068(2)	0.058(2)	−0.003(2)	−0.004(2)	−0.001(2)
C(6A)	4*a*	0.8146(3)	0.1401(3)	−0.2229(2)	0.047(2)	0.054(2)	0.048(2)	−0.010(1)	0.002(2)	−0.001(1)
C(6B)	4*a*	0.8817(2)	0.1553(3)	−0.0838(2)	0.042(2)	0.043(1)	0.050(2)	−0.003(1)	0.002(1)	0.001(1)
C(7)	4*a*	0.9022(2)	0.2005(3)	0.0027(2)	0.046(2)	0.043(1)	0.052(2)	−0.000(1)	0.004(1)	−0.002(1)
C(7A)	4*a*	0.9818(2)	0.1066(3)	0.0502(2)	0.046(2)	0.043(2)	0.050(2)	−0.004(1)	0.001(1)	0.001(1)
C(8)	4*a*	1.0040(3)	0.1455(4)	0.1460(2)	0.048(2)	0.056(2)	0.055(2)	0.003(1)	−0.001(2)	−0.005(1)
C(9)	4*a*	1.0693(3)	0.0108(4)	0.1929(2)	0.062(2)	0.061(2)	0.051(2)	−0.005(2)	−0.002(2)	0.002(1)
C(10)	4*a*	1.2533(3)	−0.1323(4)	0.1911(2)	0.068(2)	0.069(2)	0.073(2)	0.010(2)	−0.007(2)	0.015(2)
C(11)	4*a*	1.0792(3)	−0.3017(4)	0.0791(2)	0.091(3)	0.049(2)	0.074(2)	−0.004(2)	0.006(2)	0.009(2)
C(12)	4*a*	0.8407(3)	0.3463(4)	0.0420(2)	0.064(2)	0.056(2)	0.062(2)	0.010(2)	−0.002(2)	−0.006(2)

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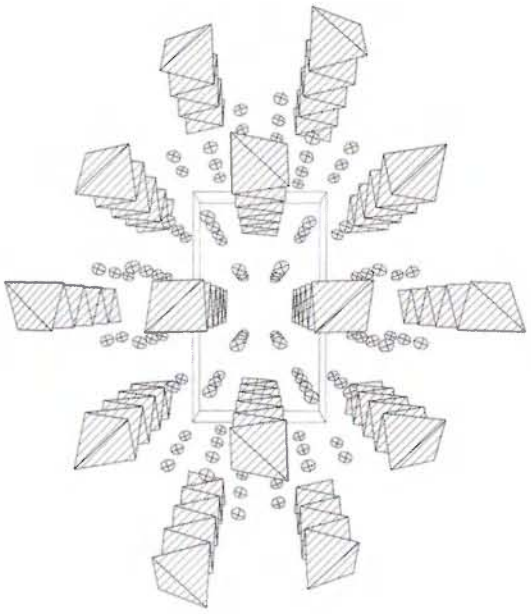

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