Crystal structure of isotetrandrine, $C_{38}H_{42}N_2O_6$

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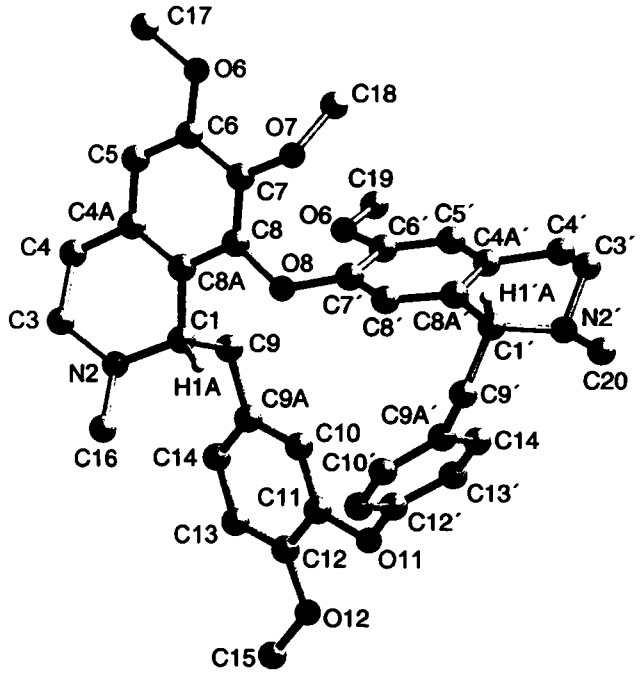

Abstract

$C_{38}H_{42}N_2O_6$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 12.024(2)$ Å, $b = 15.637(2)$ Å, $c = 17.553(3)$ Å, $V = 3300.2$ Å^3, $Z = 4$, $R_{gt}(F) = 0.041$, $wR_{ref}(F^2) = 0.109$, $T = 293$ K.

Source of material

Isotetrandrine was isolated from *Limaciopsis loangensis* (*Menispermaceae*) and was a kind gift of Prof. M. Leboeuf [1]. The impure free base was crystallized in 2-propanol to afford the material used in this study.

Discussion

This compound is a diastereomer of the bis(benzylisoquinoline) alkaloid tetrandrine [2], from which it differs only in having the (*R*) configuration at C1, leading to a *cis* relationship of H1A and H1'A. Its structure consists of two 1,2,3,4-tetrahydroisoquinoline units joined through the O8 and O11 atoms bonded to carbons C7', C8 and C11, C12', respectively. As in the case of tetrandrine, one benzylisoquinoline moiety adopts an extended conformation, while the other one is more folded. The angles formed by the mean planes of each isoquinoline and its corresponding benzyl group are 15.8° and 129.3°. The torsion angles C8A–C1–C9–C9A and C8A'–C1'–C9'–C9A' are 163.2° and 40.0°, respectively. Consequently, the overall shape of the molecule approximates that of a triangle. The bond distances and angles are in normal ranges.

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Table 1. Data collection and handling.

Crystal:	white block, size 0.20 × 0.80 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.85 cm^{-1}
Diffractometer, scan mode:	Bruker AXS SMART CCD, φ/ω
$2\theta_{max}$:	55.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	24138, 7344
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 6052
$N(param)_{refined}$:	415
Programs:	SHELXS-97 [3], SHELXL-97 [4], DIAMOND [5]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	4*a*	0.5159	0.4386	1.0029	0.08
H(3A)	4*a*	0.7037	0.3721	1.1078	0.08
H(3B)	4*a*	0.7926	0.4454	1.1126	0.08
H(4A)	4*a*	0.8482	0.4149	0.9868	0.08
H(4B)	4*a*	0.8647	0.3347	1.0396	0.08
H(5A)	4*a*	0.8333	0.2214	0.9459	0.08
H(9A)	4*a*	0.6719	0.5183	0.9045	0.08
H(9B)	4*a*	0.5632	0.4769	0.8721	0.08
H(10A)	4*a*	0.3824	0.5397	0.9041	0.08
H(13A)	4*a*	0.5599	0.7850	0.9844	0.08
H(14A)	4*a*	0.6611	0.6619	0.9614	0.08
H(15A)	4*a*	0.3360	0.9240	1.0076	0.08
H(15B)	4*a*	0.4488	0.9005	0.9680	0.08
H(15C)	4*a*	0.4236	0.8676	1.0506	0.08
H(16A)	4*a*	0.6479	0.5352	1.1451	0.08
H(16B)	4*a*	0.5485	0.4734	1.1273	0.08
H(16C)	4*a*	0.5573	0.5617	1.0853	0.08
H(17A)	4*a*	0.8193	0.0259	0.8545	0.08
H(17B)	4*a*	0.8265	0.0749	0.9323	0.08
H(17C)	4*a*	0.8709	0.1180	0.8576	0.08
H(18A)	4*a*	0.4610	0.1211	0.7505	0.08
H(18B)	4*a*	0.5894	0.1224	0.7662	0.08
H(18C)	4*a*	0.5287	0.2058	0.7393	0.08
H(19A)	4*a*	0.6529	0.4127	0.7058	0.08
H(19B)	4*a*	0.5368	0.4496	0.6819	0.08
H(19C)	4*a*	0.5674	0.3546	0.6626	0.08
H(20A)	4*a*	−0.1501	0.3144	0.7012	0.08
H(20B)	4*a*	−0.1413	0.3284	0.7894	0.08
H(20C)	4*a*	−0.1164	0.2382	0.7544	0.08
H(1'A)	4*a*	0.0599	0.2382	0.8159	0.08
H(3'A)	4*a*	0.0025	0.2846	0.6294	0.08
H(3'B)	4*a*	0.0643	0.2196	0.6829	0.08
H(4'A)	4*a*	0.1500	0.3766	0.6306	0.08
H(4'B)	4*a*	0.1991	0.2858	0.6126	0.08
H(5')	4*a*	0.3775	0.3567	0.6621	0.08
H(8')	4*a*	0.2416	0.3013	0.9021	0.08
H(9'A)	4*a*	−0.0489	0.3529	0.8748	0.08
H(9'B)	4*a*	0.0489	0.3138	0.9219	0.08
H(10')	4*a*	0.0974	0.4255	1.0027	0.08
H(11')	4*a*	0.1854	0.5546	1.0226	0.08
H(13')	4*a*	0.1659	0.6065	0.8008	0.08
H(14')	4*a*	0.0725	0.4785	0.7813	0.08

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}
O(6)	4a	0.7093(1)	0.11653(9)	0.86535(8)	0.0707(9)	0.0429(7)	0.0657(9)	0.0166(7)	0.0031(7)	−0.0100(6)
O(7)	4a	0.5011(1)	0.17768(9)	0.84739(7)	0.0536(8)	0.0523(8)	0.0558(8)	−0.0041(7)	−0.0005(6)	−0.0145(6)
O(8)	4a	0.44363(9)	0.33692(8)	0.90965(6)	0.0347(6)	0.0547(7)	0.0386(6)	0.0051(5)	−0.0025(5)	−0.0078(5)
O(11)	4a	0.2437(1)	0.66788(8)	0.9242(1)	0.0407(7)	0.0340(7)	0.118(1)	0.0041(6)	−0.0124(8)	−0.0154(7)
O(12)	4a	0.3421(1)	0.80541(7)	0.97101(8)	0.0448(7)	0.0309(6)	0.0764(9)	0.0013(5)	0.0027(6)	−0.0094(6)
N(2)	4a	0.6630(1)	0.47515(9)	1.04645(9)	0.0433(8)	0.0413(8)	0.0489(8)	0.0013(6)	−0.0028(7)	−0.0065(7)
C(1)	4a	0.5925(1)	0.4420(1)	0.98423(9)	0.0336(8)	0.0357(8)	0.0452(9)	0.0013(7)	0.0011(7)	−0.0018(7)
C(3)	4a	0.7429(2)	0.4152(1)	1.0784(1)	0.051(1)	0.050(1)	0.053(1)	0.0018(9)	−0.0107(9)	−0.0034(9)
C(4)	4a	0.8097(2)	0.3723(1)	1.0171(1)	0.0374(9)	0.048(1)	0.066(1)	0.0031(8)	−0.0090(8)	−0.0024(9)
C(5)	4a	0.7621(2)	0.2426(1)	0.9375(1)	0.0383(9)	0.0428(9)	0.052(1)	0.0087(8)	0.0023(8)	0.0047(8)
C(6)	4a	0.6876(2)	0.1956(1)	0.8954(1)	0.052(1)	0.0345(9)	0.0441(9)	0.0053(8)	0.0079(8)	0.0017(7)
C(7)	4a	0.5799(2)	0.2261(1)	0.88331(9)	0.044(1)	0.0391(9)	0.0399(9)	−0.0035(8)	0.0035(7)	−0.0025(7)
C(8)	4a	0.5513(1)	0.3052(1)	0.91393(9)	0.0357(9)	0.0395(9)	0.0363(8)	0.0017(7)	0.0026(7)	0.0018(7)
C(9)	4a	0.5951(2)	0.5050(1)	0.9163(1)	0.044(1)	0.0387(9)	0.0498(9)	0.0069(8)	0.0033(8)	0.0025(8)
C(10)	4a	0.4187(2)	0.5889(1)	0.9204(1)	0.049(1)	0.0318(8)	0.055(1)	0.0001(8)	−0.0057(8)	−0.0019(7)
C(11)	4a	0.3576(2)	0.6617(1)	0.9344(1)	0.0411(9)	0.0330(9)	0.055(1)	0.0011(7)	−0.0017(8)	−0.0017(8)
C(12)	4a	0.4103(2)	0.7364(1)	0.9591(1)	0.045(1)	0.0307(8)	0.0471(9)	0.0005(7)	0.0033(8)	0.0005(7)
C(13)	4a	0.5235(2)	0.7357(1)	0.9685(1)	0.045(1)	0.0348(9)	0.055(1)	−0.0041(8)	−0.0004(8)	−0.0020(8)
C(14)	4a	0.5845(2)	0.6614(1)	0.9543(1)	0.040(1)	0.043(1)	0.063(1)	−0.0005(8)	−0.0003(9)	0.0019(9)
C(15)	4a	0.3916(2)	0.8805(1)	1.0018(1)	0.063(1)	0.0344(9)	0.069(1)	−0.0009(9)	0.001(1)	−0.0094(9)
C(16)	4a	0.5988(2)	0.5147(2)	1.1060(1)	0.071(2)	0.072(1)	0.061(1)	0.010(1)	−0.007(1)	−0.024(1)
C(17)	4a	0.8149(2)	0.0810(1)	0.8785(1)	0.070(2)	0.046(1)	0.086(2)	0.021(1)	0.015(1)	0.001(1)
C(18)	4a	0.5218(2)	0.1549(2)	0.7694(1)	0.099(2)	0.071(2)	0.064(1)	0.009(2)	−0.011(1)	−0.026(1)
C(19)	4a	0.5755(2)	0.3996(2)	0.6994(1)	0.067(2)	0.126(2)	0.062(1)	−0.019(2)	0.018(1)	0.012(1)
C(20)	4a	−0.1103(2)	0.2988(2)	0.7465(1)	0.057(1)	0.080(2)	0.069(1)	−0.012(1)	−0.020(1)	0.005(1)
C(4A)	4a	0.7320(1)	0.3219(1)	0.9676(1)	0.0359(8)	0.0372(8)	0.0472(9)	0.0023(7)	0.0014(7)	0.0043(7)
C(8A)	4a	0.6261(1)	0.3536(1)	0.95598(9)	0.0368(9)	0.0342(8)	0.0380(8)	0.0019(7)	0.0026(7)	0.0020(7)
C(9A)	4a	0.5332(2)	0.5875(1)	0.9301(1)	0.043(1)	0.0350(9)	0.0464(9)	0.0043(7)	0.0028(7)	0.0029(7)
O(6′)	4a	0.5306(1)	0.3731(1)	0.76929(7)	0.0468(8)	0.087(1)	0.0481(7)	−0.0104(7)	0.0096(6)	0.0038(7)
N(2′)	4a	0.0061(1)	0.3219(1)	0.73821(9)	0.0488(9)	0.0455(9)	0.0498(9)	−0.0042(7)	−0.0141(7)	0.0012(7)
C(1′)	4a	0.0715(2)	0.2993(1)	0.8063(1)	0.046(1)	0.0331(8)	0.0463(9)	−0.0051(8)	−0.0079(8)	0.0014(7)
C(3′)	4a	0.0535(2)	0.2798(1)	0.6720(1)	0.070(1)	0.052(1)	0.049(1)	−0.003(1)	−0.021(1)	−0.0065(9)
C(4′)	4a	0.1628(2)	0.3200(2)	0.6514(1)	0.070(1)	0.062(1)	0.041(1)	0.003(1)	−0.0103(9)	−0.0024(9)
C(5′)	4a	0.3499(2)	0.3466(1)	0.7108(1)	0.059(1)	0.058(1)	0.0354(9)	0.001(1)	0.0037(8)	0.0014(8)
C(6′)	4a	0.4205(2)	0.3515(1)	0.7721(1)	0.042(1)	0.050(1)	0.0445(9)	0.0002(8)	0.0044(8)	−0.0031(8)
C(7′)	4a	0.3794(1)	0.3310(1)	0.84421(9)	0.0407(9)	0.0393(9)	0.0365(8)	0.0025(7)	−0.0036(7)	−0.0055(7)
C(8′)	4a	0.2686(1)	0.3132(1)	0.85353(9)	0.0427(9)	0.0349(8)	0.0371(8)	−0.0005(7)	−0.0005(7)	0.0004(7)
C(9′)	4a	0.0314(2)	0.3480(1)	0.8774(1)	0.0411(9)	0.046(1)	0.050(1)	−0.0091(8)	−0.0003(8)	−0.0019(8)
C(10′)	4a	0.1116(2)	0.4613(1)	0.9616(1)	0.045(1)	0.046(1)	0.049(1)	0.0045(8)	−0.0025(8)	−0.0039(8)
C(11′)	4a	0.1648(2)	0.5383(1)	0.9736(1)	0.051(1)	0.048(1)	0.060(1)	0.0069(9)	−0.0142(9)	−0.0165(9)
C(12′)	4a	0.1872(2)	0.5907(1)	0.9129(1)	0.0356(9)	0.0333(9)	0.079(1)	0.0050(7)	−0.0062(9)	−0.0095(9)
C(13′)	4a	0.1527(2)	0.5697(1)	0.8415(1)	0.060(1)	0.043(1)	0.065(1)	−0.0041(9)	0.000(1)	0.0030(9)
C(14′)	4a	0.0974(2)	0.4925(1)	0.8299(1)	0.062(1)	0.045(1)	0.052(1)	−0.005(1)	−0.0071(9)	−0.0021(9)
C(4A′)	4a	0.2374(2)	0.3267(1)	0.7199(1)	0.053(1)	0.0415(9)	0.0395(9)	0.0003(8)	−0.0045(8)	−0.0007(7)
C(8A′)	4a	0.1954(2)	0.3123(1)	0.79202(9)	0.0437(9)	0.0346(8)	0.0412(9)	−0.0022(7)	−0.0046(7)	−0.0033(7)
C(9A′)	4a	0.0790(1)	0.4364(1)	0.8894(1)	0.0338(9)	0.0387(9)	0.051(1)	0.0015(7)	−0.0010(7)	−0.0048(7)

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