Crystal structure of (3a*S*-*cis*)-1,2,3,3a,8,8a-hexahydro-1,3a,8-trimethyl-pyrrolo[2,3-*b*]indol-5-yl 3,4-dihydro-2(1*H*)-isoquinolinecarboxylate, $C_{23}H_{27}N_3O_2$

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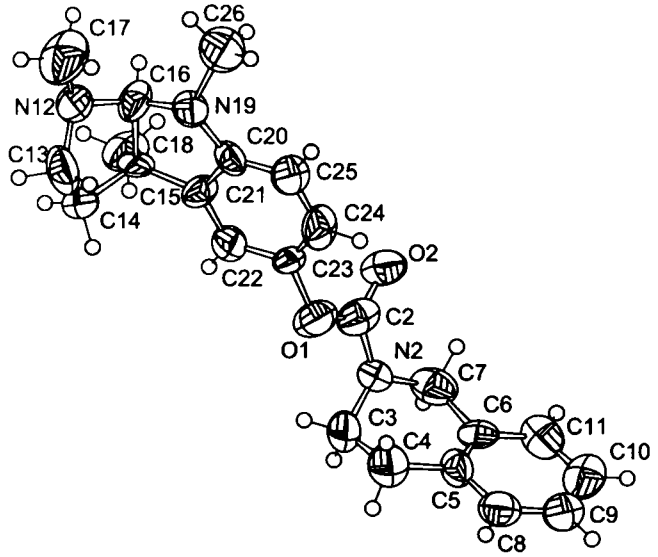

Abstract

$C_{23}H_{27}N_3O_2$, orthorhombic, $P2_12_12_1$ (No. 19), a = 8.88(2) Å, b = 11.574(5) Å, c = 19.72(2) Å, V = 2026.1 Å^3, Z = 4, $R_{gt}(F)$ = 0.059, $wR_{ref}(F^2)$ = 0.099, T = 293 K.

Source of material

The compound is a commercially available.

Experimental details

The crystals of the title compound did not diffract strongly even with the use of copper radiation. The high internal R-value (0.123) is connected with the large subset of weak reflections. The internal R-value calculated from the significant reflections ($I \geq 2\sigma(I)$) is 0.0412. All non-hydrogen atoms were refined with anisotropic displacement parameters using a 'rigid-bond' restraint to U_{ij} of two bonded atoms [1], implemented as the DELU-instruction in SHELXL-97 [2]. The H atoms were positioned geometrically and allowed to ride during the least-square refinements.

Discussion

The compound is an important pharmacologically active substance. The system of three fused rings like those of the pyrrolo-indol part of the molecule are known. Up to date in the CSD database [3], there are 43 different structures containing the same pyrrolo-indol ring system as the title compound. The indol moiety (C20-C25, C15, C16 and N19) are planar within 0.03 Å, except C16 that deviates approximately 0.20(2) Å from the plane. The pyrrolo ring has an envelope conformation with the atoms C14, C15, C16 and N12 within 0.06 Å from a common plane. The fifth atom, C13, is deviating 0.59 Å from this plane. The angle between these planes are 58(1)°. The nitrogen N19 is 0.34(1) Å out of the plane of the nearest three carbons and the N12 is 0.38(1) Å out of the plane of the corresponding three nearest carbons. Both nitrogens can thus be considered as sp^3-hybridized. The third nitrogen, N2 is only 0.10(1) Å out of plane of the three nearest atoms.

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.32 × 0.39 × 0.71 mm
Wavelength:	Cu K_α radiation (1.54184 Å)
μ:	6.34 cm^{-1}
Diffractometer, scan mode:	STOE AED4, ω/2θ
$2\theta_{max}$:	90.68°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4525, 1669
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 805
$N(param)_{refined}$:	254
Programs:	SHELX-97 [2], SHELXS-97 [4], DIAMOND [5]

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

Atom	Site	x	y	z	U_{iso}
H(3A)	4*a*	0.0901	0.9545	0.3930	0.100
H(3B)	4*a*	0.1619	0.8430	0.4250	0.100
H(4A)	4*a*	-0.0608	0.7800	0.4651	0.106
H(4B)	4*a*	-0.0642	0.9126	0.4807	0.106
H(7A)	4*a*	-0.0844	0.7996	0.2597	0.096
H(7B)	4*a*	-0.0370	0.9277	0.2752	0.096
H(8)	4*a*	-0.3442	0.8795	0.4887	0.087
H(9)	4*a*	-0.5664	0.8972	0.4292	0.081
H(10)	4*a*	-0.5683	0.8960	0.3151	0.096
H(11)	4*a*	-0.3439	0.8837	0.2555	0.091
H(13A)	4*a*	0.9106	0.4175	0.4896	0.109
H(13B)	4*a*	0.7382	0.3829	0.4887	0.109
H(14A)	4*a*	0.7223	0.5708	0.4519	0.106

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Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(14B)	4*a*	0.8760	0.5626	0.4120	0.106
H(16)	4*a*	0.7606	0.3235	0.3114	0.078
H(17A)	4*a*	0.9017	0.1664	0.3827	0.183
H(17B)	4*a*	0.8037	0.1781	0.4485	0.183
H(17C)	4*a*	0.9756	0.2089	0.4504	0.183
H(18A)	4*a*	0.8679	0.5389	0.2983	0.129
H(18B)	4*a*	0.7333	0.6263	0.3025	0.129

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(18C)	4*a*	0.7178	0.5134	0.2592	0.129
H(22)	4*a*	0.4825	0.6580	0.3603	0.078
H(24)	4*a*	0.1241	0.4541	0.3886	0.087
H(25)	4*a*	0.2767	0.2915	0.3889	0.078
H(26A)	4*a*	0.6368	0.1338	0.3528	0.150
H(26B)	4*a*	0.5233	0.1917	0.3023	0.150
H(26C)	4*a*	0.4675	0.1493	0.3735	0.150

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(2)	4*a*	0.056(1)	0.8134(9)	0.3383(5)	0.059(6)	0.063(6)	0.078(9)	0.004(6)	–0.009(6)	–0.026(7)
C(3)	4*a*	0.074(1)	0.8728(9)	0.4013(7)	0.057(8)	0.079(8)	0.12(1)	0.007(7)	–0.026(9)	–0.020(9)
C(4)	4*a*	–0.065(1)	0.8560(9)	0.4445(6)	0.080(8)	0.085(9)	0.10(1)	0.003(8)	–0.02(1)	–0.009(9)
C(5)	4*a*	–0.208(2)	0.8667(9)	0.4066(8)	0.06(1)	0.056(8)	0.07(1)	0.002(7)	–0.02(1)	0.001(9)
C(6)	4*a*	–0.210(2)	0.8715(8)	0.3382(8)	0.07(1)	0.022(6)	0.07(1)	0.010(7)	0.00(1)	–0.001(8)
C(7)	4*a*	–0.066(1)	0.8548(9)	0.2958(5)	0.11(1)	0.055(8)	0.08(1)	0.004(8)	–0.01(1)	–0.003(8)
C(8)	4*a*	–0.343(2)	0.8788(9)	0.4415(6)	0.076(9)	0.067(8)	0.07(1)	–0.007(7)	0.01(1)	–0.016(8)
C(9)	4*a*	–0.476(2)	0.890(1)	0.4057(8)	0.06(1)	0.053(8)	0.09(1)	–0.015(7)	0.02(1)	0.00(1)
C(10)	4*a*	–0.477(2)	0.890(1)	0.3383(9)	0.08(1)	0.052(8)	0.11(1)	0.007(8)	–0.00(1)	0.02(1)
C(11)	4*a*	–0.344(2)	0.8821(9)	0.3026(6)	0.10(1)	0.059(8)	0.07(1)	0.000(8)	–0.03(1)	0.015(8)
O(1)	4*a*	0.1993(8)	0.6687(7)	0.3815(4)	0.083(5)	0.102(6)	0.073(8)	0.039(5)	–0.005(5)	–0.016(6)
O(2)	4*a*	0.1127(8)	0.6558(6)	0.2743(4)	0.086(5)	0.077(6)	0.071(7)	0.017(5)	0.011(5)	–0.011(5)
C(2)	4*a*	0.126(1)	0.710(1)	0.3270(8)	0.063(9)	0.05(1)	0.09(1)	–0.001(8)	0.03(1)	0.01(1)
N(12)	4*a*	0.8503(9)	0.3306(7)	0.4052(6)	0.070(7)	0.060(6)	0.094(9)	0.016(5)	–0.021(6)	–0.008(6)
C(13)	4*a*	0.822(1)	0.409(1)	0.4611(6)	0.074(9)	0.13(1)	0.07(1)	–0.017(9)	–0.030(8)	–0.01(1)
C(14)	4*a*	0.785(1)	0.521(1)	0.4240(7)	0.056(7)	0.081(8)	0.13(1)	0.006(8)	0.002(8)	–0.05(1)
C(15)	4*a*	0.700(1)	0.4833(6)	0.3605(7)	0.060(8)	0.019(6)	0.08(1)	0.000(5)	0.007(9)	–0.014(7)
C(16)	4*a*	0.731(1)	0.3482(7)	0.3571(6)	0.044(6)	0.050(8)	0.10(1)	0.007(6)	–0.004(7)	0.015(8)
C(17)	4*a*	0.886(1)	0.2106(9)	0.4232(6)	0.11(1)	0.087(9)	0.17(1)	0.03(1)	–0.015(9)	0.03(1)
C(18)	4*a*	0.760(1)	0.5461(8)	0.2997(6)	0.082(8)	0.043(6)	0.13(1)	0.006(6)	0.016(8)	0.007(8)
N(19)	4*a*	0.5853(9)	0.2968(6)	0.3781(4)	0.065(6)	0.030(5)	0.101(8)	0.002(5)	–0.020(7)	–0.002(6)
C(20)	4*a*	0.470(1)	0.3790(8)	0.3772(6)	0.058(8)	0.052(7)	0.045(9)	–0.003(6)	–0.020(8)	–0.005(8)
C(21)	4*a*	0.534(1)	0.4892(8)	0.3664(6)	0.043(7)	0.050(7)	0.07(1)	0.000(6)	0.020(8)	–0.001(8)
C(22)	4*a*	0.442(1)	0.5844(8)	0.3657(6)	0.058(8)	0.051(7)	0.087(9)	–0.001(5)	–0.01(1)	–0.010(8)
C(23)	4*a*	0.290(1)	0.569(1)	0.3732(7)	0.054(8)	0.066(8)	0.071(9)	0.030(8)	–0.02(1)	–0.032(8)
C(24)	4*a*	0.228(1)	0.462(1)	0.3828(6)	0.046(8)	0.096(8)	0.08(1)	–0.018(8)	0.014(8)	0.00(1)
C(25)	4*a*	0.318(1)	0.3647(9)	0.3838(6)	0.059(8)	0.057(7)	0.08(1)	–0.015(7)	0.014(8)	0.004(8)
C(26)	4*a*	0.5502(9)	0.1831(8)	0.3492(6)	0.092(8)	0.066(7)	0.14(1)	–0.010(7)	–0.003(8)	–0.01(1)

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