Crystal structure of 17-(cyclopropylmethyl)-6,7-didehydro-4,5α-epoxy-3-hydroxy-14β-(3-phenylpropyloxy)indolo[2,3:6,7]morphinan hydroiodide, $(C_{35}H_{37}N_2O_3)I$

F. Schüllner[I], G. Laus[II], K. Wurst*[III] and H. Schmidhammer[I]

[I] University of Innsbruck, Department of Pharmaceutical Chemistry, Institute of Pharmacy, 6020 Innsbruck, Austria
[II] Immodal Pharmaka GmbH, 6111 Volders, Austria
[III] University of Innsbruck, Institute of Inorganic Chemistry, 6020 Innsbruck, Austria

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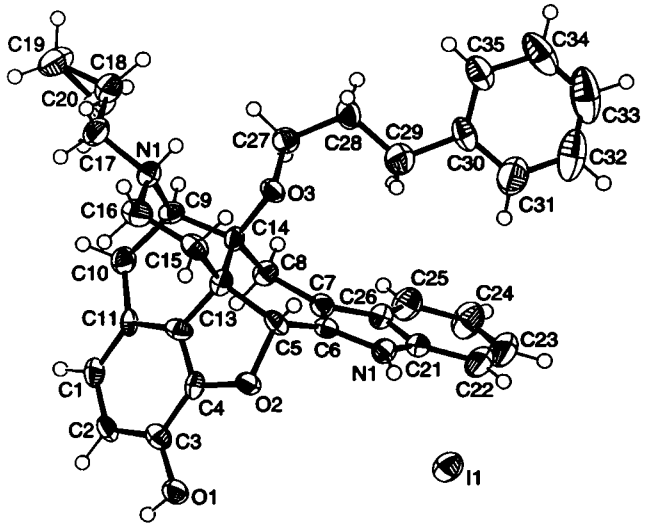

Abstract

$C_{35}H_{37}IN_2O_3$, monoclinic, $P12_11$ (no. 4), $a = 9.8202(3)$ Å, $b = 8.4511(4)$ Å, $c = 18.0106(7)$ Å, $\beta = 96.528(2)°$, $V = 1485.0$ Å^3, $Z = 2$, $R_{gt}(F) = 0.030$, $wR_{ref}(F^2) = 0.068$, $T = 233$ K.

Source of material

The indolo[2',3':6,7]morphinan was prepared from the corresponding morphinan-6-one [1] and phenylhydrazine in acetic acid [2] and was converted to the hydroiodide salt. Suitable crystals were obtained from acetone solution.

Experimental details

Hydrogen atoms at carbon atoms were calculated and included in the refinement using a riding model with $U_{iso} = 1.2$ or $1.5\,U_{eq}$ of the attached carbon atom. Hydrogen atoms at nitrogen and oxygen atoms were found and refined regularly with isotropic displacement parameters and bond restraints ($d = 0.85$ Å).

Discussion

The title compound crystallized with two molecules in the unit cell of a chiral space group. The Flack parameter x was –0.03(2), confirming the correct absolute structure. Intermolecular hydrogen bonding was observed between H(–O1) and O2' with an H···O distance of 2.14 Å. Short contacts were also found between the iodide I1 and both (N–)H donors, with distances of 2.95 Å to H(–N1') and 2.75 Å to the indole H(–N2).

* Correspondence author (e-mail: klaus.wurst@uibk.ac.at)

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.08 × 0.12 × 0.21 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	11.17 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, φ/ω
$2\theta_{max}$:	46°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6805, 3834
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3628
$N(param)_{refined}$:	382
Programs:	SHELXS-97 [3], SHELXL-97 [4]

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

Atom	Site	x	y	z	U_{iso}
H(1O)	2a	1.051(8)	0.647(4)	0.479(5)	0.12(4)
H(1N)	2a	0.326(6)	1.066(4)	0.339(3)	0.06(2)
H(2N)	2a	0.994(3)	1.290(4)	0.317(2)	0.01(1)
H(1)	2a	0.6560	0.4897	0.3767	0.040
H(2)	2a	0.8747	0.4953	0.4386	0.039
H(5)	2a	0.7720	1.2262	0.3971	0.034
H(8A)	2a	0.6811	0.8699	0.2328	0.035
H(8B)	2a	0.5968	0.9897	0.1772	0.035
H(9)	2a	0.3889	0.9009	0.2468	0.033
H(10A)	2a	0.4253	0.6815	0.3404	0.041
H(10B)	2a	0.5032	0.6782	0.2682	0.041
H(15A)	2a	0.6156	1.0818	0.4752	0.037
H(15B)	2a	0.5322	1.1949	0.4162	0.037
H(16A)	2a	0.4616	0.8770	0.4467	0.046
H(16B)	2a	0.3752	1.0291	0.4633	0.046
H(17A)	2a	0.2344	0.7819	0.3693	0.049
H(17B)	2a	0.1696	0.9451	0.3903	0.049
H(18)	2a	0.1087	1.0008	0.2605	0.056
H(19A)	2a	0.0178	0.6864	0.3016	0.066
H(19B)	2a	–0.0645	0.8030	0.2380	0.066
H(20A)	2a	0.1307	0.8023	0.1692	0.064
H(20B)	2a	0.2130	0.6856	0.2328	0.064
H(22)	2a	1.1002	1.4102	0.1945	0.057
H(23)	2a	1.0786	1.3826	0.0656	0.069
H(24)	2a	0.9106	1.2221	0.0045	0.066
H(25)	2a	0.7666	1.0736	0.0701	0.054
H(27A)	2a	0.3134	1.1772	0.2234	0.042
H(27B)	2a	0.4269	1.1292	0.1714	0.042
H(28A)	2a	0.3460	1.3841	0.1408	0.052
H(28B)	2a	0.3859	1.4400	0.2243	0.052
H(29A)	2a	0.5869	1.3304	0.1384	0.060
H(29B)	2a	0.6110	1.4283	0.2134	0.060
H(31)	2a	0.7630	1.5399	0.1122	0.077
H(32)	2a	0.7807	1.7484	0.0327	0.107
H(33)	2a	0.5974	1.9048	–0.0036	0.107
H(34)	2a	0.3902	1.8597	0.0403	0.085
H(35)	2a	0.3654	1.6482	0.1191	0.057

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}
I(1)	2a	1.24621(3)	1.38485(5)	0.38423(2)	0.0360(2)	0.0516(2)	0.0387(2)	−0.0058(3)	0.0028(1)	−0.0019(3)
O(1)	2a	1.0333(4)	0.7444(5)	0.4724(2)	0.032(2)	0.031(2)	0.045(2)	−0.002(2)	−0.008(2)	0.007(2)
O(2)	2a	0.8853(4)	1.0231(5)	0.4196(2)	0.028(2)	0.024(2)	0.031(2)	0.002(2)	−0.003(2)	0.002(2)
O(3)	2a	0.5030(3)	1.1786(4)	0.2760(2)	0.032(2)	0.026(2)	0.030(2)	0.003(2)	−0.002(1)	0.007(1)
N(1)	2a	0.3570(4)	0.9715(5)	0.3522(2)	0.029(2)	0.036(3)	0.035(2)	0.000(2)	0.012(2)	0.006(2)
N(2)	2a	0.9344(4)	1.2542(5)	0.2845(2)	0.027(2)	0.028(3)	0.039(3)	−0.009(2)	−0.006(2)	0.001(2)
C(1)	2a	0.6999(5)	0.5875(6)	0.3866(3)	0.038(3)	0.022(3)	0.040(3)	−0.009(3)	0.008(2)	0.002(2)
C(2)	2a	0.8314(5)	0.5909(6)	0.4234(3)	0.039(3)	0.019(3)	0.040(3)	0.004(2)	0.007(2)	0.009(2)
C(3)	2a	0.9014(7)	0.7339(8)	0.4383(4)	0.039(4)	0.026(4)	0.031(3)	0.006(3)	0.005(3)	0.001(3)
C(4)	2a	0.8334(4)	0.872(1)	0.4143(2)	0.031(2)	0.023(3)	0.029(2)	−0.010(4)	0.006(2)	−0.001(3)
C(5)	2a	0.7844(5)	1.1238(6)	0.3720(3)	0.037(3)	0.016(3)	0.030(3)	0.003(2)	−0.003(2)	0.002(2)
C(6)	2a	0.8313(4)	1.1516(6)	0.2969(3)	0.023(2)	0.026(3)	0.033(3)	0.003(2)	−0.001(2)	0.005(2)
C(7)	2a	0.7721(5)	1.0889(6)	0.2311(3)	0.028(3)	0.023(3)	0.030(3)	0.000(2)	0.001(2)	0.001(2)
C(8)	2a	0.6503(5)	0.9798(6)	0.2263(3)	0.029(3)	0.033(3)	0.026(3)	−0.001(2)	0.004(2)	−0.001(2)
C(9)	2a	0.4462(4)	0.905(1)	0.2957(2)	0.024(2)	0.032(4)	0.027(2)	0.004(3)	0.005(2)	−0.002(3)
C(10)	2a	0.4951(6)	0.7353(7)	0.3149(3)	0.033(3)	0.028(3)	0.042(3)	−0.004(3)	0.005(2)	−0.002(3)
C(11)	2a	0.6322(7)	0.7255(8)	0.3642(4)	0.030(3)	0.011(3)	0.040(4)	−0.011(3)	0.003(3)	0.000(3)
C(12)	2a	0.6995(4)	0.868(1)	0.3811(2)	0.031(2)	0.033(4)	0.025(2)	0.012(4)	0.004(2)	0.001(3)
C(13)	2a	0.6474(6)	1.0271(9)	0.3653(3)	0.029(3)	0.031(4)	0.022(3)	−0.007(3)	0.003(3)	−0.001(3)
C(14)	2a	0.5615(5)	1.0255(6)	0.2883(3)	0.025(3)	0.023(3)	0.028(3)	−0.007(2)	0.002(2)	0.003(2)
C(15)	2a	0.5602(5)	1.0850(6)	0.4264(3)	0.041(3)	0.025(3)	0.027(3)	0.008(2)	0.005(2)	−0.002(2)
C(16)	2a	0.4343(5)	0.9830(7)	0.4287(3)	0.042(3)	0.046(4)	0.029(3)	0.009(3)	0.006(2)	0.003(2)
C(17)	2a	0.2195(4)	0.892(1)	0.3533(2)	0.032(2)	0.037(3)	0.056(3)	−0.002(5)	0.017(2)	0.000(5)
C(18)	2a	0.1338(4)	0.894(1)	0.2802(3)	0.035(2)	0.041(3)	0.063(3)	−0.007(5)	0.005(2)	0.002(5)
C(19)	2a	0.0237(6)	0.7697(8)	0.2643(4)	0.032(3)	0.057(4)	0.077(4)	−0.003(3)	0.007(3)	−0.010(3)
C(20)	2a	0.1447(5)	0.7694(8)	0.2217(3)	0.033(3)	0.061(4)	0.065(4)	−0.003(3)	0.005(3)	−0.012(3)
C(21)	2a	0.9449(5)	1.2560(6)	0.2095(3)	0.027(3)	0.030(3)	0.042(3)	0.000(2)	0.004(2)	0.007(2)
C(22)	2a	1.0334(5)	1.3429(7)	0.1698(3)	0.036(3)	0.055(6)	0.054(3)	−0.008(3)	0.013(2)	0.004(3)
C(23)	2a	1.0195(6)	1.3267(8)	0.0936(4)	0.046(3)	0.069(5)	0.061(4)	−0.012(3)	0.019(3)	0.012(3)
C(24)	2a	0.9190(6)	1.2285(9)	0.0569(3)	0.056(4)	0.075(5)	0.039(3)	−0.008(4)	0.021(3)	0.002(3)
C(25)	2a	0.8322(5)	1.1411(8)	0.0955(3)	0.045(3)	0.051(4)	0.040(3)	−0.006(3)	0.011(3)	−0.004(3)
C(26)	2a	0.8435(5)	1.1546(6)	0.1734(3)	0.029(3)	0.034(3)	0.036(3)	−0.001(2)	0.007(2)	0.006(2)
C(27)	2a	0.4060(5)	1.2011(6)	0.2112(3)	0.031(3)	0.035(3)	0.036(3)	−0.005(2)	−0.008(2)	0.004(2)
C(28)	2a	0.4125(5)	1.370(1)	0.1851(2)	0.045(3)	0.047(4)	0.038(3)	−0.006(4)	0.001(2)	0.020(4)
C(29)	2a	0.5506(5)	1.4171(9)	0.1665(3)	0.046(3)	0.056(6)	0.048(3)	−0.003(3)	0.004(2)	0.012(3)
C(30)	2a	0.5594(6)	1.5667(6)	0.1223(3)	0.061(4)	0.024(3)	0.031(3)	−0.007(3)	−0.002(2)	0.002(2)
C(31)	2a	0.6856(7)	1.6023(8)	0.0969(4)	0.061(4)	0.051(5)	0.083(5)	−0.009(4)	0.025(4)	0.000(4)
C(32)	2a	0.696(1)	1.727(1)	0.0504(5)	0.105(7)	0.092(7)	0.078(6)	−0.035(6)	0.041(5)	0.016(5)
C(33)	2a	0.588(1)	1.820(1)	0.0292(4)	0.145(8)	0.076(6)	0.044(4)	−0.050(6)	−0.007(5)	0.016(3)
C(34)	2a	0.4650(9)	1.7926(8)	0.0546(4)	0.109(6)	0.042(4)	0.052(4)	−0.010(4)	−0.034(4)	0.013(3)
C(35)	2a	0.4503(6)	1.6666(7)	0.1013(3)	0.063(4)	0.036(4)	0.041(3)	−0.005(3)	−0.006(3)	0.010(3)

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