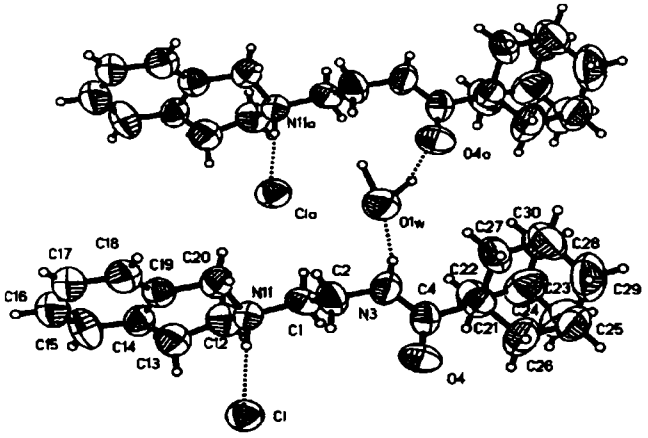
Crystal structure of 2-[2-(1-adamantanecarboxamido)ethyl]-1,2,3,4-tetrahydroisoquinolinium chloride monohydrate, $(C_{22}H_{31}N_2O)Cl \cdot H_2O$

J. Karolak-Wojciechowska*,[I], A. Fruziński[I], M. Paluchowska[II] and M. Mokrosz[II]

[I] Technical University of Łódź, Institute of General and Ecological Chemistry, ul. Żwirki 36, 90-924 Łódź, Poland
[II] Institute of Pharmacology, Department of Medicinal Chemistry, PAS, ul. Smętna 12, 31-343 Kraków, Poland

Received February 7, 2002, accepted and available on-line April 24, 2002; CCDC-No. 1267/803



Abstract

$C_{22}H_{33}ClN_2O_2$, monoclinic, $P12_1/c1$ (No. 14), $a = 17.157(3)$ Å, $b = 17.426(3)$ Å, $c = 7.211(1)$ Å, $\beta = 94.12(3)°$, $V = 2150.4$ Å^3, $Z = 4$, $R_{gt}(F) = 0.077$, $wR_{ref}(F^2) = 0.154$, $T = 293$ K.

Source of material

The synthesis of the title compound has been described previously [1] as a part of our research program focused on serotonin $5HT_{1A}$ receptor ligands. The crystals for X-ray studies were obtained from ethanol/acetone (1:1) mixture by slow evaporation. However, most of the obtained samples have been classified as amorphous and were not appropriate for single-crystal structures determination.

Discussion

The independent unit of the crystal structure contains the protonated main molecule, Cl^- anion and one water molecule. As in previously studied structures of potential $5HT_{1A}$ and $5HT_{2A}$ receptors ligands [2,3], the main molecule under discussion incorporates two cyclic moieties 1,2,3,4-tetrahydroisoquinoline and adamantane-linked via four-membered aliphatic spacer, which is composed of ethylene and amide groups. The heterocyclic ring in tetrahydroisoquinoline system adopts half chair conformation with spacer located in equatorial position and proton occupied axial position at N11, respectively. Extended spacer attends *trans-trans-gauche-trans-trans* conformation. It is of wider interest that two parent-nitrogen hydrogens are *trans* with respect to extended spacer direction accompanied by partial charges equal (0.25 e$^-$) on both hydrogens (semiempirical PM3 approximation [6]). The ion Cl^- interacts with nitrogen from tetrahydroisoquinoline, d(N11–H11A···Cl) = 3.051(3) Å. The molecules of the title compound (including the amide group) are bridged via two H-bond by the water molecule to an infinite chain along the c-axis, d(O1w–H1w···O4^i) = 2.705(4) Å, d(N3–H3A···O1w) = 2.860(5) Å; $i = x, y, -1+z$.

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.3 × 0.2 × 0.2 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	17.10 cm^{-1}
Diffractometer, scan mode:	KUMA Diffraction KM-4, $\omega/2\theta$
$2\theta_{max}$:	156.3°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8772, 4386
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1137
$N(param)_{refined}$:	246
Programs:	SHELXS-97 [4], SHELXL-97[5], SHELXTL-PC [6]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	4e	0.5411	0.5581	–0.2259	0.117
H(1B)	4e	0.5146	0.6046	–0.4035	0.117
H(2A)	4e	0.5756	0.6736	–0.0725	0.127
H(2B)	4e	0.5540	0.7178	–0.2577	0.127
H(3A)	4e	0.6554	0.6521	–0.3856	0.125
H(11A)	4e	0.4432	0.6170	–0.0697	0.090
H(12A)	4e	0.4148	0.5016	–0.2343	0.133
H(12B)	4e	0.3774	0.5519	–0.3953	0.133
H(13A)	4e	0.3217	0.5357	–0.0503	0.136
H(13B)	4e	0.2766	0.5148	–0.2376	0.136
H(15A)	4e	0.1645	0.5894	–0.1232	0.152
H(16A)	4e	0.1064	0.7097	–0.1380	0.169
H(17A)	4e	0.1758	0.8200	–0.1840	0.157
H(18A)	4e	0.3069	0.8084	–0.2435	0.137
H(20A)	4e	0.4014	0.6896	–0.4040	0.126
H(20B)	4e	0.4281	0.7327	–0.2216	0.126
H(22A)	4e	0.7328	0.5392	–0.4304	0.178
H(22B)	4e	0.7614	0.4973	–0.2473	0.178
H(23A)	4e	0.8454	0.4669	–0.4821	0.197
H(24A)	4e	0.9579	0.4837	–0.3170	0.194
H(24B)	4e	0.8920	0.4663	–0.1857	0.194
H(25A)	4e	0.9679	0.5723	–0.0628	0.203
H(26A)	4e	0.8357	0.5685	0.0068	0.201

* Correspondence author (e-mail: jkarolak@p.lodz.pl)

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(26B)	4e	0.8582	0.6537	–0.0214	0.201
H(27A)	4e	0.8189	0.7223	–0.3007	0.159
H(27B)	4e	0.7674	0.6831	–0.4604	0.159
H(28A)	4e	0.9014	0.6849	–0.5440	0.182
H(29A)	4e	0.9998	0.6354	–0.3328	0.246

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(29B)	4e	0.9485	0.6906	–0.2232	0.246
H(30A)	4e	0.9175	0.5596	–0.6014	0.176
H(30B)	4e	0.8288	0.5800	–0.6334	0.176
H(1W)	4e	0.6730	0.6362	–0.7775	0.146
H(2W)	4e	0.5985	0.6699	–0.8161	0.146

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}
Cl	4e	0.45278(6)	0.60965(6)	0.2209(2)	0.1006(9)	0.0876(8)	0.0756(8)	0.0116(6)	0.0090(6)	–0.0008(6)
C(1)	4e	0.5189(2)	0.6058(2)	–0.2700(5)	0.086(3)	0.092(3)	0.056(3)	0.001(3)	0.018(2)	0.000(2)
C(2)	4e	0.5729(2)	0.6700(3)	–0.2056(5)	0.066(3)	0.108(4)	0.082(3)	0.002(3)	0.021(2)	–0.015(3)
N(3)	4e	0.6489(2)	0.6527(2)	–0.2629(5)	0.063(3)	0.103(3)	0.084(2)	0.015(2)	0.001(2)	0.000(2)
C(4)	4e	0.7088(3)	0.6353(2)	–0.1440(7)	0.057(3)	0.079(3)	0.085(4)	0.007(2)	0.008(3)	–0.012(3)
O(4)	4e	0.7056(2)	0.6344(2)	0.0249(4)	0.121(3)	0.172(3)	0.052(2)	0.010(2)	0.024(2)	0.007(2)
N(11)	4e	0.4391(2)	0.6156(2)	–0.2031(4)	0.061(2)	0.047(2)	0.071(2)	–0.001(2)	0.003(2)	–0.004(2)
C(12)	4e	0.3878(3)	0.5488(2)	–0.2629(6)	0.089(3)	0.072(3)	0.105(3)	–0.021(3)	0.007(3)	–0.002(3)
C(13)	4e	0.3120(3)	0.5513(2)	–0.1775(6)	0.086(4)	0.092(4)	0.094(3)	–0.012(3)	0.005(3)	–0.005(2)
C(14)	4e	0.2741(3)	0.6276(3)	–0.1847(5)	0.060(3)	0.097(4)	0.068(3)	–0.003(3)	–0.007(2)	–0.009(2)
C(15)	4e	0.1944(3)	0.6341(3)	–0.1498(7)	0.078(4)	0.102(4)	0.127(4)	–0.022(3)	0.035(3)	–0.025(3)
C(16)	4e	0.1612(3)	0.7060(5)	–0.1563(7)	0.075(4)	0.156(6)	0.107(4)	–0.003(4)	0.009(3)	0.013(4)
C(17)	4e	0.2002(3)	0.7705(4)	–0.1883(6)	0.075(4)	0.142(5)	0.097(4)	0.014(4)	0.012(3)	0.018(3)
C(18)	4e	0.2778(3)	0.7625(3)	–0.2231(5)	0.092(4)	0.094(4)	0.088(3)	0.010(3)	0.014(3)	0.005(3)
C(19)	4e	0.3157(3)	0.6948(3)	–0.2283(5)	0.070(3)	0.077(3)	0.063(3)	–0.001(3)	0.006(2)	–0.008(2)
C(20)	4e	0.3997(2)	0.6889(2)	–0.2712(6)	0.074(3)	0.081(3)	0.097(3)	0.006(2)	0.007(3)	0.014(3)
C(21)	4e	0.7860(2)	0.6142(2)	–0.2294(5)	0.073(3)	0.081(3)	0.062(3)	0.011(2)	0.003(2)	–0.009(2)
C(22)	4e	0.7745(3)	0.5359(3)	–0.3347(7)	0.101(4)	0.114(4)	0.146(5)	0.015(3)	0.045(3)	–0.003(4)
C(23)	4e	0.8524(4)	0.5133(4)	–0.4113(9)	0.151(6)	0.142(5)	0.099(4)	0.033(4)	–0.011(4)	–0.014(4)
C(24)	4e	0.9108(3)	0.5033(3)	–0.2704(8)	0.138(5)	0.138(5)	0.110(5)	0.054(4)	0.002(4)	–0.019(4)
C(25)	4e	0.9282(3)	0.5791(5)	–0.1621(8)	0.081(4)	0.242(8)	0.081(4)	0.029(4)	–0.004(3)	0.010(5)
C(26)	4e	0.8505(3)	0.6053(4)	–0.0834(7)	0.092(4)	0.220(6)	0.089(4)	0.050(4)	–0.005(3)	–0.009(4)
C(27)	4e	0.8091(3)	0.6750(3)	–0.3664(7)	0.072(3)	0.120(4)	0.131(4)	0.008(3)	0.033(3)	0.022(3)
C(28)	4e	0.8864(4)	0.6487(4)	–0.4527(9)	0.103(5)	0.129(5)	0.139(5)	–0.006(4)	0.049(4)	0.019(4)
C(29)	4e	0.9486(3)	0.6430(4)	–0.291(1)	0.082(5)	0.243(8)	0.165(7)	–0.036(5)	–0.005(4)	–0.023(6)
C(30)	4e	0.8717(3)	0.5748(4)	–0.5389(7)	0.078(4)	0.189(7)	0.089(4)	0.026(4)	0.030(3)	0.024(5)
O(1W)	4e	0.6298(2)	0.6438(2)	–0.6596(4)	0.103(2)	0.105(2)	0.085(2)	0.003(2)	0.012(2)	0.002(2)

Acknowledgment. The partial support (synthesis) of Polish State Committee for Scientific Research, project No 4 P05F 005 18, is greatly acknowledged.

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