Crystal structure of adamantylammonium bicarbonate, $[C_{10}H_{18}N]HCO_3$

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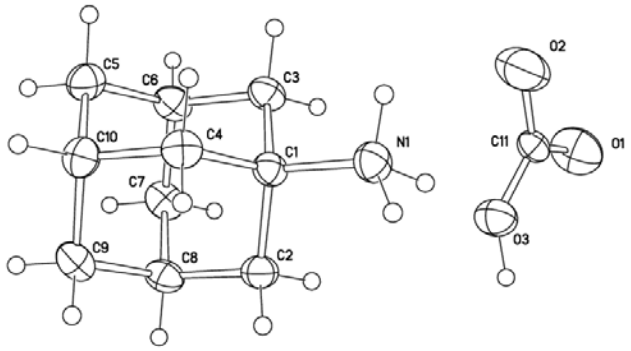

Abstract

$C_{11}H_{19}NO_3$, orthorhombic, $P2_12_12_1$ (no. 19), a = 6.3919(1) Å, b = 7.3659(2) Å, c = 22.8347(5) Å, V = 1075.1 Å^3, Z = 4, $R_{gt}(F)$ = 0.044, $wR_{ref}(F^2)$ = 0.129, T = 296 K.

Source of material

Reagents and solvents used were of commercially available quality. Adamantaneamine was not a primary reactant in this investigation. It was produced from decomposition of the *N*-2-hydroxy-3-methoxy-benzaldehyde-1-aminoadamanantane. The latter was synthesized according to [1]. $NaHCO_3$ (2 mmol) and *N*-2-hydroxy-3-methoxybenzaldehyde-1-aminoadamanantane (2 mmol) were dissolved in methanol (30 ml) solution and the mixture was stirred for 8 h at room temperature. The yellow solution was kept aside, and the yellow crystal was obtained after several days.

Experimental details

The crystal structure was solved by direct methods and successive Fourier difference syntheses. The H atoms bonded to C and N atoms were positioned geometrically and refined using a riding model [d($C_{aliphatic}$—H) = 0.97(2) Å and d(N—H) = 0.86 Å, U_{iso}(H) = 1.2 U_{eq}(C)]. The H atoms bonded to O atoms were located in a difference Fourier maps and refined with O—H distance restraints of 0.85(2) Å and U_{iso}(H) = 1.5 U_{eq}(O).

Discussion

Adamantane has a highly-symmetric and steady structure. Adamantaneamine has an obvious effect on controlling the exuviating the influenza A virus. Especially, it can be used to prevent virus going into host cells and influenza A_2 in asia. In addition it can alleviate the Parkinson symptom [2].

The title crystal structure is formed by adamantaneamine cations and bicarbonate anions. All the C—C bonds (C1-C10) are in the range of 1.516(3) Å - 1.535(3) Å in the adamantyl group, with C–C bond angles of 108.7(2)°- 110.3(2)°, which is consistent with diamond-like unit. The C—N bond length is 1.503(3) Å. The bicarbonate anion and the adamantaneamine cation are connected with each other with hydrogen bonds. These hydrogen bonds contribute to the formation of a two-dimensional network.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.089 × 0.108 × 0.297 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.95 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, ω
$2\theta_{max}$:	49.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4726, 1905
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1593
$N(param)_{refined}$:	145
Programs:	SHELXS-97 [3], SHELDXL-97 [4], SHELXTL [5]

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

Atom	Site	x	y	z	U_{iso}
H(3D)	4*a*	0.0607	0.0226	0.3060	0.086
H(2A)	4*a*	–0.1910	0.1037	0.1604	0.050
H(2B)	4*a*	–0.0512	–0.0423	0.1919	0.050
H(3A)	4*a*	0.3342	–0.0594	0.1702	0.051
H(3B)	4*a*	0.4354	0.0766	0.1253	0.051
H(4A)	4*a*	0.2103	0.3116	0.0755	0.049
H(4B)	4*a*	–0.0306	0.3207	0.0891	0.049
H(5A)	4*a*	0.2046	–0.1062	–0.0045	0.057
H(5B)	4*a*	0.3558	0.0473	0.0177	0.057
H(6A)	4*a*	0.3860	–0.2097	0.0794	0.054
H(7A)	4*a*	0.0421	–0.3222	0.0671	0.057
H(7B)	4*a*	0.0921	–0.3052	0.1343	0.057
H(8A)	4*a*	–0.2406	–0.1817	0.1150	0.053
H(9A)	4*a*	–0.1792	–0.0873	0.0173	0.057
H(9B)	4*a*	–0.2682	0.0768	0.0535	0.057
H(10A)	4*a*	0.0222	0.1737	–0.0020	0.052
H(1A)	4*a*	0.057(5)	0.348(4)	0.201(1)	0.053
H(1B)	4*a*	0.282(4)	0.338(4)	0.181(1)	0.053
H(1C)	4*a*	0.170(4)	0.197(4)	0.228(1)	0.053

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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*	0.1635(4)	0.2651(3)	0.18947(9)	0.046(1)	0.043(1)	0.047(1)	0.001(1)	–0.0020(9)	–0.007(1)
O(1)	4*a*	0.4171(4)	–0.0886(3)	0.3463(1)	0.074(1)	0.080(2)	0.099(2)	0.010(1)	–0.017(1)	0.022(1)
O(2)	4*a*	0.5026(3)	0.0954(4)	0.2802(1)	0.064(1)	0.114(2)	0.100(2)	–0.022(1)	–0.009(1)	0.015(2)
O(3)	4*a*	0.1822(3)	0.0268(3)	0.29098(7)	0.044(1)	0.065(1)	0.064(1)	0.003(1)	–0.0012(8)	0.0039(9)
C(1)	4*a*	0.1226(3)	0.1329(3)	0.14066(9)	0.034(1)	0.033(1)	0.040(1)	–0.001(1)	–0.0010(9)	–0.0043(9)
C(2)	4*a*	–0.0723(3)	0.0231(3)	0.15550(9)	0.035(1)	0.047(1)	0.044(1)	–0.001(1)	0.0022(9)	0.004(1)
C(3)	4*a*	0.3108(3)	0.0064(3)	0.1340(1)	0.033(1)	0.043(1)	0.051(1)	0.004(1)	–0.0039(9)	–0.002(1)
C(4)	4*a*	0.0875(4)	0.2393(3)	0.0844(1)	0.043(1)	0.034(1)	0.047(1)	–0.000(1)	0.001(1)	0.002(1)
C(5)	4*a*	0.2314(4)	–0.0221(4)	0.0273(1)	0.049(1)	0.045(1)	0.049(1)	–0.003(1)	0.007(1)	–0.010(1)
C(6)	4*a*	0.2668(4)	–0.1273(3)	0.0841(1)	0.038(1)	0.038(1)	0.058(1)	0.009(1)	–0.003(1)	–0.007(1)
C(7)	4*a*	0.0702(4)	–0.2368(3)	0.0985(1)	0.051(2)	0.030(1)	0.061(1)	–0.004(1)	–0.008(1)	0.004(1)
C(8)	4*a*	–0.1151(4)	–0.1106(3)	0.1060(1)	0.036(1)	0.041(1)	0.055(1)	–0.009(1)	–0.002(1)	0.004(1)
C(9)	4*a*	–0.1492(4)	–0.0038(4)	0.0490(1)	0.041(1)	0.046(1)	0.055(1)	0.001(1)	–0.011(1)	–0.006(1)
C(10)	4*a*	0.0448(4)	0.1063(3)	0.0345(1)	0.050(1)	0.038(1)	0.041(1)	0.002(1)	–0.001(1)	0.0050(9)
C(11)	4*a*	0.3676(3)	0.0120(3)	0.30633(9)	0.033(1)	0.031(1)	0.042(1)	0.000(1)	–0.0052(9)	–0.000(1)

References

1. Zhao, G. L.; Zhang, P. H.; Feng, Y. L.: Studies on the synthesis, characterization and antibacterial activity of rare earth complexes with Schiff base derived from *o*-vanillin and adamantaneamine. Chinese J. Inorg. Chem. **21** (2005) 421-424.
2. Hayden, F. G.; Belshe, R. B.; Clover, R. D.; Hay, A. J.; Oates, M. G.; Soo, W.: Emergence and apparent transmission of rimantadine-resistant influenza A virus in families. N. Engl. J. Med. **321** (1989) 1696-702.
3. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
4. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
5. Sheldrick, G. M.: SHELXTL. Structure Determination Software Suite. Version 6.14. Bruker AXS, Madison, Wisconsin, USA 2000.