Crystal structure of 4,5-dihydro-7-amino-5-oxo-[1,2,4]-triazolo-[1,5-*a*]pyrimidine, $C_5H_5N_5O$

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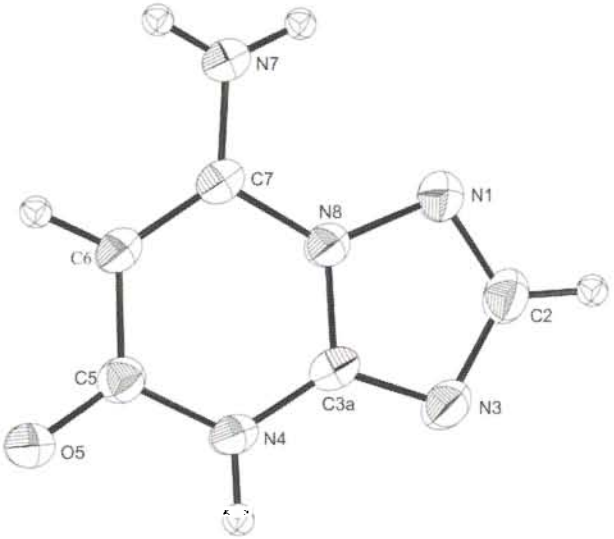
Received May 20, 1999, CCDC-No. 1267/193



Abstract

$C_5H_5N_5O$, monoclinic, $P12_1/n1$ (No. 14), a = 5.0756(6) Å, b = 8.9631(8) Å, c = 13.4484(7) Å, β = 92.509(8)°, V = 611.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.036, $wR(F)$ = 0.055, T = 293 K.

Source of material

The product was synthesised using the literature procedure [1].

Discussion

The molecule is planar with the mean deviation through the least-squares plane through all atoms being 0.02 A. Within the five-membered ring, the double bonds are localised at N(1)–C(2) (1.315(2)Å) and N(3)–C(3a) (1.316(2)Å). By constrast, significant delocalisation of π-electron density is found in the six-membered ring which extends to include the amino group. Thus, the range of bond distances within the ring is 1.349(2) Å to 1.418(2) Å and d(C(7)–N(7)) is 1.333(2) Å. The lattice is stabilised by a number of hydrogen bonding interactions, however, no evidence is found for extensive base-stacking. The structure may be described as being comprised of alternatively orientated layers stacked along the c-axis. Within each layer centrosymmetrically related molecules associate via N(4)–H(4)···O(5)i interactions such that d(H(4)···O(5)i) is 1.83(2) Å, d(N(4)···O(5)i) is 2.756(2) Å and the N(4)–H(4)···O(5)i angle is 175(2)°; symmetry operation i: $-x$, 1$-y$, 1$-z$. Further intra-layer contacts involve N(7)–H(7a)···N(3)ii with d(H(7a)···N(3)ii) of 2.26(2) Å, d(N(7)···N(3)ii) of 3.092(2) Å and N(7)–H(7a)···N(3)ii of 159(2)°; symmetry operation ii: 0.5+x, 0.5$-y$, 0.5+z. Interlayer contacts are afforded by N(7)–H(7b)···N(1)iii interactions such that d(H(7b)···N(1)iii) is 2.24(2) Å, d(N(7)···N(1)iii) is 2.821(2) Å and N(7)–H(7b)···N(1)iii is 153(2)°; symmetry operation iii: 2$-x$, $-y$, 1$-z$.

Table 1. Data collection and handling.

Crystal:	tan wedge, size 0.17 × 0.18 × 0.32 mm
Wavelength:	Cu K_α radiation (1.5418 Å)
μ:	10.63 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC7R, ω/2θ
$2\theta_{max}$:	130.14°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1241, 1109
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 3\ \sigma(I_{obs})$, 939
$N(param)_{refined}$:	120
Programs:	SIR97 [2], TEXSAN [3], DIFABS [4]

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

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	0.640(2)	0.054(1)	0.2860(9)	0.037(3)
H(4)	4*e*	0.123(3)	0.411(2)	0.444(1)	0.053(4)
H(6)	4*e*	0.546(2)	0.354(1)	0.7031(8)	0.036(3)
H(7a)	4*e*	0.944(3)	0.071(2)	0.596(1)	0.055(4)
H(7b)	4*e*	0.900(3)	0.161(1)	0.694(1)	0.045(3)

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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}
O(5)	4*e*	0.1614(1)	0.49680(8)	0.61686(5)	0.0375(4)	0.0423(4)	0.0294(4)	0.0127(3)	–0.0030(3)	–0.0013(3)
N(1)	4*e*	0.7329(2)	0.0933(1)	0.42800(6)	0.0341(5)	0.0391(5)	0.0277(5)	0.0050(4)	0.0008(4)	–0.0048(4)
N(3)	4*e*	0.4039(2)	0.2176(1)	0.34161(6)	0.0358(5)	0.0437(5)	0.0245(4)	0.0012(4)	–0.0035(4)	–0.0010(4)
N(4)	4*e*	0.2592(2)	0.3630(1)	0.48012(6)	0.0286(5)	0.0367(5)	0.0251(4)	0.0057(4)	–0.0050(3)	0.0013(4)
N(7)	4*e*	0.8600(2)	0.1410(1)	0.63184(7)	0.0377(5)	0.0406(5)	0.0276(5)	0.0112(4)	–0.0081(4)	–0.0030(4)
N(8)	4*e*	0.6054(2)	0.19151(9)	0.48878(6)	0.0270(4)	0.0333(4)	0.0235(4)	0.0033(3)	–0.0004(3)	–0.0004(3)
C(2)	4*e*	0.6020(2)	0.1151(1)	0.34267(8)	0.0380(6)	0.0437(6)	0.0246(5)	0.0014(5)	0.0020(4)	–0.0057(4)
C(3)	4*e*	0.4110(2)	0.2623(1)	0.43491(7)	0.0267(5)	0.0329(5)	0.0238(5)	–0.0014(4)	–0.0033(4)	0.0043(4)
C(5)	4*e*	0.3026(2)	0.3994(1)	0.57978(7)	0.0292(5)	0.0314(5)	0.0257(5)	0.0007(4)	0.0006(4)	0.0018(4)
C(6)	4*e*	0.5092(2)	0.3225(1)	0.63254(7)	0.0324(6)	0.0369(6)	0.0225(5)	0.0030(4)	–0.0039(4)	–0.0004(4)
C(7)	4*e*	0.6621(2)	0.2183(1)	0.58847(7)	0.0279(5)	0.0305(5)	0.0240(5)	–0.0017(4)	–0.0034(4)	0.0022(4)

Acknowledgment. The Australian Research Council is thanked for the support of the X-ray facility.

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