Crystal structure of isobutyl 3-(4-amino-2-oxo-1,3,5-triazin-1(2*H*)-yl) propanoate, $C_{10}H_{16}N_4O_3$

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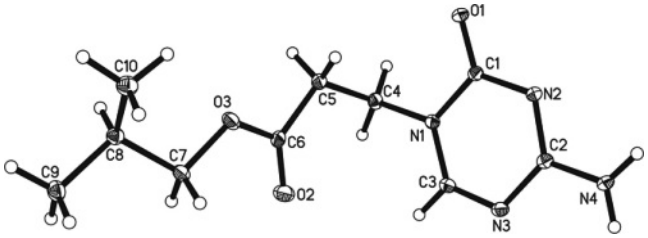

Abstract

$C_{10}H_{16}N_4O_3$, triclinic, $P\bar{1}$ (No. 2), a = 5.7929(8) Å, b = 7.348(1) Å, c = 14.222(2) Å, α = 98.649(3)°, β = 91.512(3)°, γ = 102.139(3)°, V = 584.1 Å^3, Z = 2, $R_{gt}(F)$ = 0.0390, $wR_{ref}(F^2)$ = 0.1469, T = 298 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.22×0.25×0.33 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.03 cm^{-1}
Diffractometer, scan mode:	Bruker APEX II area-detector, φ and ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4829, 2135
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1892
$N(param)_{refined}$:	156
Programs:	SHELX [13], SADABS [14]

Source of material

All reactant are purchased and used without further purification. In a typical reaction 4-amino-1,3,5-triazin-2(1*H*)-one (1.12 g, 10 mmol), isobutyl acrylate (2.2 ml,15 mmol), and 0.06g boric acid (1 mmol) were placed into a flask. Water (40 ml) was added and the reaction mixture was stirred at 333 K for several hours. The progress of the reaction was monitored by thin-layer chromatography (*TLC*). The solvent was removed under reduced pressure. Then the solid material was washed with ethyl acetate (40 ml) and the solution was filtered to afford the pure product. The purified product was dissolved in the mixture of ethyl alcohol and *N,N*-dimethylformamide (v/v = 2/1). The resulting solution was left in air for a few days, yielding colourless crystals.

Experimental details

All H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of Csp^2–H = 0.93 Å with U_{iso} = 1.2U_{eq} (parent atom), Csp^3–H = 0.97/0.98 Å with U_{iso} = 1.5/1.2 U_{eq} (parent atom) and N–H = 0.86 Å with U_{iso} = 1.2U_{eq} (parent atom) [13, 14].

Discussion

Among various analogs of cytosine, the 5-azacytosine(5-*azaC*) analogs have attracted more attention than the normal cytosine or other analogs [1, 2]. For using as anti-cancer drugs, 5-azacytidine (*AC*) and 2'-deoxy-5-azacytidine (*DAC*), demonstrate their active in inhibition aflatoxin biosynthesis [3], the treatment of leukemia [4] and myelodysplastic syndrome (MDS) [5], acute myeloid leukemia (AML) [6], respectively. In anti-viral range, HPMP-5-*azaC*, its cyclic form and ester prodrugs, all show remarkable activity against various DNA viruses [7]. As analogs of HPMP-cytosine [8, 9], they both have extinct functions and anti-virus potential. As *AC* and *DAC* are unstable in water, further solutions are put forward, that is, saturation of the 5,6-double bond, which leads to 5,6-dihydro-5-azacytidine (*DHAC*) and 2'-deoxy-5,6-dihydro-5-azacytidine (*DHDAC*), former one could inhibit RNA synthesis [10], while the later exhibits unique anti-HIV potential [11]. Beside their mainly applications in medical chemistry, they have shown distinctive lanthanides/actinides separation efficacy. Recently, report on the separation of uranium from multi-ion system has been discovered [12], broadening their scope to hazardous pollutants detection. Consequently, more effort and passion should be spend on the research of cytosine analogues. Herein we report the structure of isobutyl 3-(4-amino-2-oxo-1,3,5-triazin-1(2*H*)-yl)propanoate, also an cytosine analog. The bond lengths and angles in the title molecule (Fig.), are within normal ranges. The N1–C1 and N1–C3 bond distances [1.4158(19) Å and 1.3494(19) Å], N2–C1 bond distance [1.3482(18) Å], N3–C2 bond distance [1.3829(19) Å], N4–C2 bond distance [1.3269(19) Å] are shorter than N–C single bond of [1.443(4) Å], but longer than a typical N=C double bond of [1.269(2) Å]. The C6–O3 bond length [1.3373(8) Å] is shorter than a typical C–O bond length [1.439(2) Å], but longer than a typical C=O double bond [1.173(5) Å]. The partial double bond character of the structure is presumed as a result of the electron delocalization. The dihedral angle formed by the heterocyclic ring and the plane defined by N1/C4/C5 is 80.85°. The atoms C1, C2, C3, N1, N2 and N3 in the heterocyclic ring are slightly puckered, with an r.m.s. deviation of 0.093 Å. It should be noted that the hydrogen bonding interactions plays an important role in the crystal structure of the title compound.

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(4A)	2*i*	1.4393	1.6543	0.4611	0.023
H(4B)	2*i*	1.2444	1.7281	0.4206	0.023
H(3)	2*i*	0.6807	1.2468	0.3197	0.021
H(4C)	2*i*	0.5997	0.9298	0.3377	0.023
H(4D)	2*i*	0.7859	0.8547	0.3945	0.023
H(5A)	2*i*	0.9954	0.8434	0.2455	0.025
H(5B)	2*i*	0.7775	0.6796	0.2522	0.025
H(7A)	2*i*	0.7591	0.9222	–0.0243	0.026
H(7B)	2*i*	0.5213	0.7881	–0.0047	0.026
H(8)	2*i*	0.6719	0.5233	–0.0632	0.023
H(9A)	2*i*	0.4593	0.6439	–0.1724	0.036
H(9B)	2*i*	0.6513	0.5510	–0.2243	0.036
H(9C)	2*i*	0.6943	0.7710	–0.1983	0.036
H(10A)	2*i*	1.0767	0.7700	–0.1134	0.036
H(10B)	2*i*	1.0326	0.5494	–0.1310	0.036
H(10C)	2*i*	1.0785	0.6609	–0.0269	0.036

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(1)	2*i*	1.2198(2)	1.0009(1)	0.41875(7)	0.0246(6)	0.0136(5)	0.0251(6)	0.0065(4)	–0.0033(4)	0.0041(4)
O(2)	2*i*	0.5559(2)	0.9441(2)	0.16235(8)	0.0436(7)	0.0416(7)	0.0220(6)	0.0266(6)	–0.0007(5)	0.0028(5)
O(3)	2*i*	0.7946(2)	0.7799(2)	0.08348(7)	0.0267(6)	0.0334(6)	0.0173(6)	0.0142(5)	–0.0033(4)	0.0005(4)
N(1)	2*i*	0.9127(2)	1.1145(2)	0.36406(8)	0.0178(6)	0.0151(6)	0.0154(6)	0.0032(5)	–0.0007(5)	0.0030(4)
N(2)	2*i*	1.2696(2)	1.3188(2)	0.43051(8)	0.0187(6)	0.0136(6)	0.0184(6)	0.0043(5)	–0.0005(5)	0.0029(5)
N(3)	2*i*	0.9539(2)	1.4394(2)	0.36428(8)	0.0212(6)	0.0175(6)	0.0191(6)	0.0073(5)	0.0012(5)	0.0049(5)
N(4)	2*i*	1.3008(2)	1.6346(2)	0.43317(9)	0.0222(6)	0.0125(6)	0.0241(7)	0.0044(5)	–0.0014(5)	0.0047(5)
C(1)	2*i*	1.1439(2)	1.1404(2)	0.40611(9)	0.0196(7)	0.0149(7)	0.0134(7)	0.0048(5)	0.0015(5)	0.0031(5)
C(2)	2*i*	1.1743(2)	1.4599(2)	0.40945(9)	0.0200(7)	0.0164(7)	0.0125(7)	0.0059(5)	0.0037(5)	0.0041(5)
C(3)	2*i*	0.8338(2)	1.2671(2)	0.3466(1)	0.0177(7)	0.0206(7)	0.0167(7)	0.0067(6)	0.0004(5)	0.0045(5)
C(4)	2*i*	0.7647(3)	0.9224(2)	0.3425(1)	0.0207(7)	0.0157(7)	0.0190(7)	–0.0006(6)	–0.0004(6)	0.0043(6)
C(5)	2*i*	0.8253(3)	0.8135(2)	0.2502(1)	0.0269(8)	0.0148(7)	0.0206(8)	0.0037(6)	–0.0039(6)	0.0031(6)
C(6)	2*i*	0.7085(3)	0.8562(2)	0.1626(1)	0.0225(8)	0.0137(7)	0.0192(7)	0.0019(6)	–0.0014(6)	0.0010(5)
C(7)	2*i*	0.6907(3)	0.7996(2)	–0.0079(1)	0.0233(8)	0.0253(8)	0.0171(8)	0.0093(6)	–0.0029(6)	0.0026(6)
C(8)	2*i*	0.7421(2)	0.6449(2)	–0.0820(1)	0.0185(8)	0.0180(7)	0.0201(8)	0.0037(5)	0.0013(6)	0.0042(6)
C(9)	2*i*	0.6260(3)	0.6535(2)	–0.1782(1)	0.0232(8)	0.0288(8)	0.0184(8)	0.0067(6)	0.0010(6)	0.0007(6)
C(10)	2*i*	1.0070(3)	0.6575(2)	–0.0890(1)	0.0210(8)	0.0258(8)	0.0267(8)	0.0061(6)	0.0012(6)	0.0046(6)

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