Crystal structure of 2-amino-4,6-dimethylpyrimidinium hydrogen oxalate, $C_8H_{11}N_3O_4$

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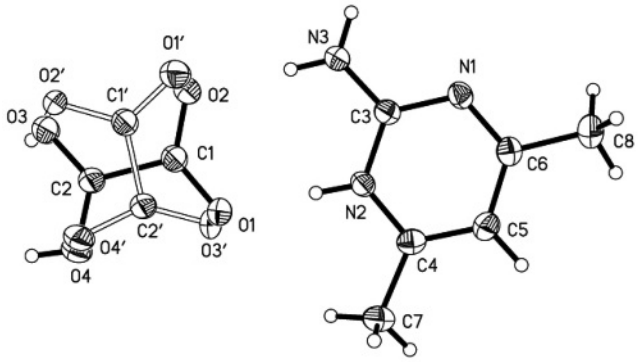

Abstract

$C_8H_{11}N_3O_4$, monoclinic, $P2_1/c$ (no. 14), a = 3.9213(7) Å, b = 14.556(1) Å, c = 17.268(2) Å, β = 102.410(2)°, V = 962.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.0659, $wR_{ref}(F^2)$ = 0.2386, T = 293 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.38×0.40×0.43 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.20 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6323, 1634
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1254
$N(param)_{refined}$:	193
Programs:	SHELX [4]

Source of material

All commercially available reagents were used as supplied. The title compound was synthesized by the following procedure. A mixture of the guanidine hydrochloride (0.1mol), acetyl acetone (0.2mol), sodium carbonate (0.03 mol), and oxalic acid (0.1mol) was stirred in water (30mL) for 2h to afford the title compound (yield 79%). Single crystals suitable for X-ray measurements were obtained by recrystallization from water at room temperature.

Discussion

Five- and six-membered heterocyclic compounds are important constituents that often exist in biologically active natural products and synthetic compounds of medicinal interest [1]. As useful precursors to potentially bioactive pyrimidine derivatives, methylpyrimidine have attracted considerable attention for many years [2]. In recent years, Ru complexes of the pyrimidine ring with $-NH_2$ have been synthesized [2]. In the crystal structure of the title compound, $C_6H_{10}N_3^+$ cations and $C_2HO_4^-$ anions are linked by intermolecular O–H···O and N–H···N hydrogen bonds to form a two-dimensional network. The N–C bond lengths in the range of (1.317(3) -1.363(3) Å) are in agreement with those observed previously [3]. There are some weak O–H···O and N–H···O intramolecular hydrogen bonds in the crystal structure. The $C_2HO_4^-$ anion is disordered.

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(2)	4*e*		0.1766	0.6337	0.2934	0.044
H(3A)	4*e*		0.5238	0.4778	0.4256	0.055
H(3B)	4*e*		0.4647	0.5033	0.3402	0.055
H(4)	4*e*	0.853	0.3786	0.5673	0.0096	0.104
H(2')	4*e*	0.147	0.5996	0.4521	0.0714	0.072
H(5)	4*e*		–0.1608	0.8018	0.4358	0.051
H(7A)	4*e*		0.0525	0.8306	0.2768	0.079
H(7B)	4*e*		–0.3361	0.8189	0.2846	0.079
H(7C)	4*e*		–0.1735	0.7495	0.2332	0.079
H(8A)	4*e*		0.0015	0.6494	0.5958	0.077
H(8B)	4*e*		–0.1492	0.7469	0.5693	0.077
H(8C)	4*e*		0.2540	0.7336	0.5997	0.077

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	4*e*		0.2417(6)	0.6093(1)	0.4760(1)	0.045(1)	0.037(1)	0.032(1)	–0.0051(9)	0.006(1)	–0.0015(8)
N(2)	4*e*		0.1526(6)	0.6480(1)	0.3402(1)	0.043(1)	0.036(1)	0.030(1)	–0.0009(9)	0.0063(9)	0.0009(8)
N(3)	4*e*		0.4413(7)	0.5151(2)	0.3876(1)	0.065(2)	0.040(1)	0.033(1)	0.012(1)	0.009(1)	0.0029(9)
O(1)	4*e*	0.853	0.284(4)	0.614(1)	0.2009(7)	0.087(7)	0.047(3)	0.036(5)	0.021(4)	0.018(4)	0.000(3)
O(2)	4*e*	0.853	0.589(5)	0.487(1)	0.241(1)	0.076(6)	0.051(6)	0.036(2)	0.016(3)	0.014(3)	0.005(3)
O(3)	4*e*	0.853	0.611(2)	0.4500(4)	0.0904(3)	0.108(4)	0.045(3)	0.041(3)	0.025(2)	0.025(3)	0.004(2)
O(4)	4*e*	0.853	0.349(5)	0.584(2)	0.053(1)	0.108(9)	0.063(3)	0.034(6)	0.036(5)	0.010(5)	0.011(4)
C(1)	4*e*	0.853	0.4444(9)	0.5428(2)	0.1908(2)	0.053(2)	0.034(2)	0.036(3)	–0.000(2)	0.011(2)	–0.001(2)
C(2)	4*e*	0.853	0.476(1)	0.5234(3)	0.1049(2)	0.054(2)	0.033(3)	0.038(3)	0.004(2)	0.011(2)	0.001(2)
O(1')	4*e*	0.147	0.66(3)	0.471(7)	0.241(8)	0.08(4)	0.05(3)	0.05(2)	0.01(2)	0.01(2)	0.00(2)
O(2')	4*e*	0.147	0.683(8)	0.433(2)	0.116(2)	0.07(1)	0.04(1)	0.04(1)	0.023(8)	0.02(1)	0.01(1)
O(3')	4*e*	0.147	0.21(2)	0.613(6)	0.182(4)	0.06(2)	0.05(1)	0.03(3)	0.01(2)	0.02(2)	–0.01(2)
O(4')	4*e*	0.147	0.44(3)	0.58(1)	0.069(6)	0.11(5)	0.06(2)	0.03(3)	0.04(3)	0.01(3)	0.01(2)
C(1')	4*e*	0.147	0.592(5)	0.489(1)	0.169(1)	0.05(1)	0.03(1)	0.04(1)	0.000(9)	0.01(1)	–0.001(9)
C(2')	4*e*	0.147	0.367(5)	0.572(1)	0.136(1)	0.05(1)	0.03(1)	0.04(2)	0.004(9)	0.01(1)	0.00(1)
C(3)	4*e*		0.2791(7)	0.5905(2)	0.4020(2)	0.038(1)	0.032(1)	0.034(1)	–0.0037(9)	0.004(1)	0.0009(9)
C(4)	4*e*		–0.0097(7)	0.7271(2)	0.3514(2)	0.036(1)	0.034(1)	0.045(2)	–0.003(1)	0.005(1)	0.002(1)
C(5)	4*e*		–0.0497(7)	0.7479(2)	0.4262(2)	0.044(2)	0.038(2)	0.046(2)	–0.001(1)	0.012(1)	–0.004(1)
C(6)	4*e*		0.0799(7)	0.6864(2)	0.4879(2)	0.037(1)	0.039(1)	0.038(2)	–0.008(1)	0.008(1)	–0.005(1)
C(7)	4*e*		–0.1272(9)	0.7869(2)	0.2801(2)	0.058(2)	0.048(2)	0.051(2)	0.010(1)	0.009(2)	0.012(1)
C(8)	4*e*		0.0433(9)	0.7058(2)	0.5705(2)	0.060(2)	0.053(2)	0.044(2)	–0.006(1)	0.017(2)	–0.009(1)

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