Crystal structure of 6-amino-3-methyl-5-nitrosopyrimidine-2,4(1*H*,3*H*)-dione monosodium dihydrate, $C_5H_5N_4O_3Na \cdot 2H_2O$

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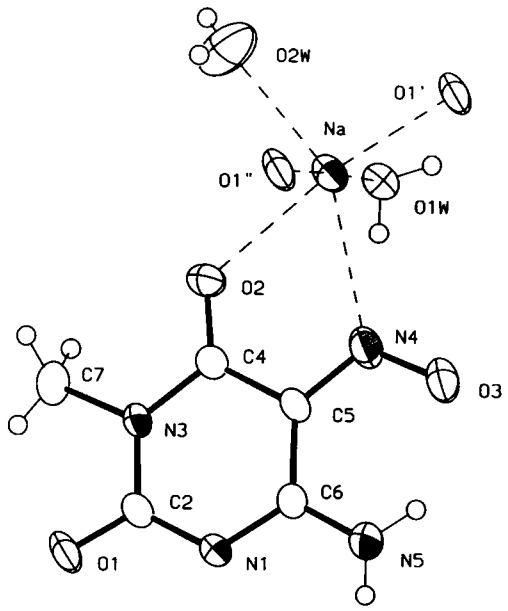

Source of material: 6-amino-3-methylpyrimidine-2,4(1*H*,3*H*)-dione was obtained by regioselective methylation of 6-amino-uracil (see ref. 1). The compound was dissolved in dimethyl-formamide, and nitrosation was performed by the addition of excess sodium nitrite dissolved in water, and a few drops of aqueous concentrated hydrochloric acid solution as described (see ref. 2). The solvent was removed by rotary evaporation, and the residue was suspended in water and collected by filtration. Slow recrystallization from a mixture of dimethylsulfoxide and water over a period of several weeks at room temperature yielded crystals suitable for X-ray analysis.

$C_5H_9N_4NaO_5$, triclinic, $P\bar{1}$ (No. 2), a =7.119(2) Å, b =8.220(2) Å, c =8.761(2) Å, α =64.01(3)°, β =89.90(3)°, γ =88.32(3)°, V =460.6 Å^3, Z =2, $R(F)$ =0.038, $R_w(F^2)$ =0.100.

Table 1. Parameters used for the X-ray data collection

Crystal:	red prism, size 0.07 x 0.26 x 0.44 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	1.82 cm^{-1}
Diffractometer:	Siemens P3
Scan mode:	ω
$T_{measurement}$:	293 K
$2\theta_{max}$:	50°
N(hkl)$_{unique}$:	1620
Criterion for I_o:	$I_o > 2\,\sigma(I_o)$
N($param$)$_{refined}$:	173
Programs:	SHELXS-86, SHELXL-93

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

Atom	Site	x	y	z	U_{iso}
H(1N5)	2*i*	–0.423(4)	0.948(4)	–0.261(4)	0.043(7)
H(2N5)	2*i*	–0.436(4)	0.892(4)	–0.076(4)	0.063(9)
H(1C7)	2*i*	–0.193(6)	0.102(6)	0.053(5)	0.11(1)
H(2C7)	2*i*	0.015(6)	0.169(5)	–0.002(5)	0.11(1)
H(3C7)	2*i*	–0.133(5)	0.183(5)	–0.129(5)	0.09(1)
H(1OW)	2*i*	–0.512(5)	0.252(5)	0.612(5)	0.07(1)
H(2OW)	2*i*	–0.540(5)	0.240(4)	0.473(4)	0.062(9)
H(3OW)	2*i*	0.052(8)	0.015(8)	0.649(7)	0.14(2)
H(4OW)	2*i*	0.12(1)	0.12(1)	0.56(1)	0.26(5)

Table 3. Final 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}
Na	2*i*	–0.1691(1)	0.3512(1)	0.4613(1)	0.0497(6)	0.0365(5)	0.0244(5)	0.0039(4)	0.0004(3)	–0.0163(4)
O(1)	2*i*	–0.1556(2)	0.4818(2)	–0.3523(2)	0.059(1)	0.0417(9)	0.0263(8)	0.0045(7)	0.0053(7)	–0.0229(7)
O(3)	2*i*	–0.3963(3)	0.7523(2)	0.1698(2)	0.068(1)	0.0406(9)	0.0355(9)	0.0123(8)	0.0039(8)	–0.0224(7)
O(2)	2*i*	–0.1701(2)	0.2840(2)	0.2180(2)	0.0513(9)	0.0305(8)	0.0226(8)	0.0058(7)	–0.0046(6)	–0.0101(7)
N(3)	2*i*	–0.1843(2)	0.3754(2)	–0.0672(2)	0.0348(9)	0.0241(8)	0.0237(8)	0.0029(7)	–0.0001(7)	–0.0144(7)
N(1)	2*i*	–0.2875(2)	0.6757(2)	–0.2616(2)	0.037(1)	0.0297(9)	0.0202(8)	0.0045(7)	–0.0009(7)	–0.0129(7)
N(4)	2*i*	–0.3214(3)	0.6023(2)	0.1851(2)	0.042(1)	0.035(1)	0.032(1)	–0.0004(8)	0.0047(8)	–0.0204(8)
N(5)	2*i*	–0.4038(3)	0.8709(3)	–0.1612(3)	0.052(1)	0.030(1)	0.029(1)	0.0115(8)	–0.0013(8)	–0.0137(9)

Table 3. (Continued)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(2)	2*i*	–0.2085(3)	0.5131(3)	–0.2331(2)	0.031(1)	0.031(1)	0.023(1)	0.0003(8)	0.0008(8)	–0.0148(8)
C(4)	2*i*	–0.2119(3)	0.4035(3)	0.0754(2)	0.0250(9)	0.027(1)	0.023(1)	–0.0008(8)	0.0008(7)	–0.0123(8)
C(5)	2*i*	–0.2926(3)	0.5809(3)	0.0442(2)	0.029(1)	0.028(1)	0.0204(9)	–0.0024(8)	0.0023(7)	–0.0137(8)
C(6)	2*i*	–0.3293(3)	0.7113(3)	–0.1303(2)	0.026(1)	0.026(1)	0.025(1)	–0.0003(8)	0.0008(7)	–0.0138(8)
C(7)	2*i*	–0.1158(4)	0.1954(3)	–0.0435(3)	0.053(2)	0.029(1)	0.042(1)	0.008(1)	–0.001(1)	–0.022(1)
O(1W)	2*i*	–0.4622(2)	0.2186(2)	0.5514(2)	0.046(1)	0.0452(9)	0.0254(8)	0.0098(7)	–0.0010(7)	–0.0166(7)
O(2W)	2*i*	0.0515(4)	0.1122(3)	0.5984(4)	0.068(2)	0.042(1)	0.076(2)	0.011(1)	–0.004(1)	–0.005(1)

References

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