Crystal structure of bis(hydroxylammonium) 5,5′-azotetrazolate dihydrate, $(NH_3OH)_2(C_2N_{10})\cdot 2H_2O$

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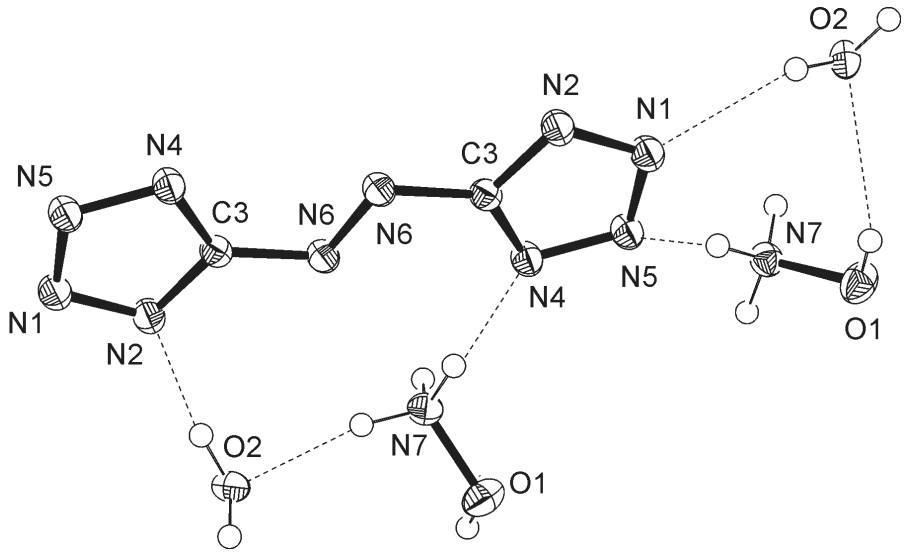

Abstract

$C_2H_{12}N_{12}O_4$, monoclinic, *C*12/*c*1 (no. 15), $a = 20.620(4)$ Å, $b = 4.3100(8)$ Å, $c = 15.051(3)$ Å, $\beta = 124.30(1)°$, $V = 1105.0$ Å^3, $Z = 4$, $R_{gt}(F) = 0.028$, $wR_{ref}(F^2) = 0.071$, $T = 173$ K.

Source of material

The yellow title compound was crystallized from aqueous solutions of sodium 5,5′-azotetrazolate and hydroxylamine hydrochloride, as described in [1]. Nitrogen-rich materials of this type are of considerable interest as energetic salts [2,3] and for pyrotechnic applications [4]. The crystals decompose at 125 °C without any residue.

Experimental details

Hydrogen atoms on water molecules were located and refined isotropically with O—H bond restraints of 0.85(2) Å and an H···H distance restraint of 1.35(2) Å.

Discussion

The azotetrazolate anion is planar and contains a center of inversion. Each tetrazolate ring receives four H bonds: N1 and N2 from two water molecules, N4 and N5 from two hydroxylammonium ions. In turn, each water molecule donates H bonds to N1 and N2 of two azotetrazolate anions and accepts H bonds from two hydroxylammonium cations. Hydrogen bond characteristics (donor···acceptor distance, H···acceptor distance, donor–H···acceptor angle) are as follows: for N7–H···N5 — 2.875 Å, 1.98 Å, 168°; for N7–H···N4^{i} — 2.847 Å, 1.95 Å, 170°; for N7–H···O2^{ii} — 2.843 Å, 1.95 Å, 168°; for O1–H···O2^{iii} — 2.668 Å, 1.86 Å, 162°; for O2–H···N1^{iii} — 2.813 Å, 1.99 Å, 161°; and for O2–H···N2^{iv} — 2.822 Å, 1.96 Å, 172°. These interactions form a three-dimensional network in the crystal. Symmetry code (i): 1/2–*x*,–1/2+*y*,1/2–*z*; (ii): *x*,1–*y*,–1/2+*z*; (iii): –*x*,*y*,1/2–*z*; (iv): *x*,–1+*y*,*z*, respectively.

Table 1. Data collection and handling.

Crystal:	yellow fragment, size 0.16 × 0.16 × 0.32 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.43 cm^{-1}
Diffractometer, scan mode:	STOE IPDS II, rotation method
$2\theta_{max}$:	50.08°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3305, 970
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 895
$N(param)_{refined}$:	92
Programs:	SIR2002 [4], SHELXL-97 [5]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(7A)	8*f*	0.1392	0.5280	0.1456	0.032
H(7B)	8*f*	0.0992	0.5217	0.0270	0.032
H(7C)	8*f*	0.1798	0.3935	0.0985	0.032
H(1)	8*f*	0.0647	0.1433	0.0950	0.044
H(22)	8*f*	–0.010(1)	0.439(4)	0.338(1)	0.046(5)
H(21)	8*f*	0.037(1)	0.179(4)	0.379(2)	0.060(6)

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(4)	8*f*	0.21143(6)	0.8568(3)	0.35579(8)	0.0183(5)	0.0222(6)	0.0185(5)	–0.0022(4)	0.0095(5)	–0.0029(4)
N(5)	8*f*	0.15360(6)	0.6781(3)	0.27996(8)	0.0185(5)	0.0232(6)	0.0187(5)	–0.0018(4)	0.0092(4)	–0.0021(5)
N(1)	8*f*	0.08965(6)	0.7171(3)	0.27942(9)	0.0191(5)	0.0238(6)	0.0206(5)	–0.0012(5)	0.0102(4)	–0.0030(5)
N(6)	8*f*	0.21477(6)	1.2233(3)	0.48336(8)	0.0171(5)	0.0203(6)	0.0186(5)	–0.0010(4)	0.0088(4)	–0.0007(4)
N(2)	8*f*	0.10450(6)	0.9223(3)	0.35533(9)	0.0184(5)	0.0232(6)	0.0216(6)	–0.0018(5)	0.0104(5)	–0.0042(5)
C(3)	8*f*	0.17925(7)	1.0030(3)	0.4001(1)	0.0165(6)	0.0185(7)	0.0171(6)	0.0000(5)	0.0086(5)	0.0008(5)
N(7)	8*f*	0.13260(6)	0.4179(3)	0.08944(9)	0.0194(5)	0.0229(6)	0.0222(6)	–0.0016(5)	0.0124(5)	–0.0013(5)
O(1)	8*f*	0.10074(6)	0.1231(2)	0.08518(8)	0.0332(6)	0.0217(5)	0.0411(6)	–0.0035(4)	0.0263(5)	–0.0024(4)
O(2)	8*f*	0.01163(5)	0.3142(2)	0.39091(7)	0.0202(5)	0.0256(6)	0.0207(5)	0.0051(4)	0.0109(4)	0.0029(4)

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