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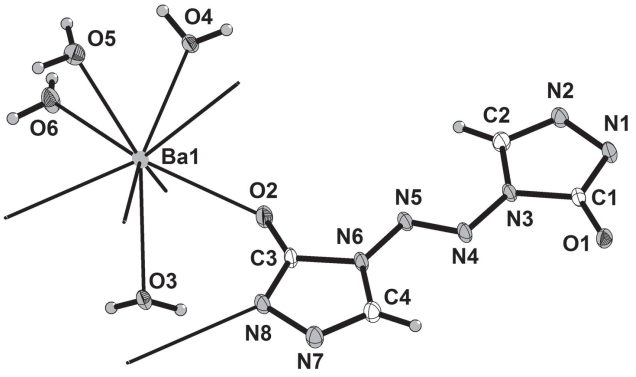
Crystal structure of tetraqua((*E*)-4,4′-(diazene-1,2-diyl)bis(5-oxo-4,5-dihydro-1,2,4-triazol-1-ide)-κ²*N*:*O*)barium(II), $C_4H_{10}N_8O_6Ba$



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Abstract

$C_4H_{10}BaN_8O_6$, triclinic, $P\bar{1}$ (No. 2), $a = 7.6249(18)$ Å, $b = 7.8460(16)$ Å, $c = 11.723(3)$ Å, $\alpha = 70.520(8)°$, $\beta = 85.079(10)°$, $y = 64.007(7)°$, $V = 592.792$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0332$, $wR_{ref}(F^2) = 0.0734$, $T = 153$ K.

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless, block, size 0.08×0.12×0.22 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	33.92 cm^{-1}
Diffractometer, scan mode:	CCD, φ and ω scans
$2\theta_{max}$:	63°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8849, 3870
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3645
$N(param)_{refined}$:	204
Programs:	SHELX [4], Diamond [5], SADABS [6]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(4)	2*i*	0.5339	0.7117	0.4688	0.022
H(2)	2*i*	−0.0971	1.3700	0.4936	0.021
H(03A)	2*i*	0.270(5)	0.396(5)	1.055(2)	0.018(9)
H(03B)	2*i*	0.237(5)	0.489(6)	0.931(2)	0.04(1)
H(04A)	2*i*	0.056(5)	1.229(2)	1.045(2)	0.06(2)
H(04B)	2*i*	0.073(5)	1.266(4)	0.922(2)	0.02(1)
H(05A)	2*i*	0.599(5)	0.825(4)	1.206(3)	0.06(2)
H(05B)	2*i*	0.429(2)	1.002(4)	1.180(3)	0.04(1)
H(06A)	2*i*	−0.012(3)	0.927(5)	1.241(3)	0.05(1)
H(06B)	2*i*	0.153(5)	0.748(4)	1.286(3)	0.05(2)

Source of material

The title compound was prepared as follows: ZTO (4,4′-azobistriazolone) is easily available by a literature known synthesis [1, 2]. ZTO (1.00 g) was suspended in 5 mL of water and to it a solution of $Ba(OH)_2 \cdot 8H_2O$ (1.61 g, in 10 mL of water) was added dropwise. After a reaction time of 30 min at room temperature, 50 mL of methanol was also added dropwise, and the resulting mixture was slowly cooled to room temperature. The bright yellow precipitate formed, which was filtered, washed with methanol and dried under vacuum, yielding 0.82 g (70.5%). ^{1}H—NMR (d_6—DMSO): = 8.14 ppm (CH); IR (KBr pellet): 3184, 3115, 1612, 1507, 1345, 1203, 1032, 936, 854, 820 cm^{-1}, Elemental Analysis (%) calcd. for $C_4H_{10}N_8O_6Ba$: C, 11.91; H, 2.50; N, 27.77; found: C, 11.89; H, 2.52; N, 27.80.

Experimental details

Single-crystal X-ray diffraction data were collected using a Bruker-AXS SMART APEX2 CCD diffractometer. Absorption correction was performed by multiscan method implemented in SADABS.

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Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ba(1)	2*i*	0.31217(2)	0.79106(2)	1.02878(1)	0.01155(9)	0.00931(8)	0.01247(9)	−0.00120(6)	0.00051(6)	−0.00305(6)
C(1)	2*i*	0.0677(4)	1.3280(4)	0.2443(2)	0.014(1)	0.013(1)	0.010(1)	−0.007(1)	−0.001(1)	0.000(1)
O(1)	2*i*	0.1841(3)	1.2412(3)	0.1767(2)	0.018(1)	0.0149(9)	0.0108(9)	−0.0042(8)	0.0020(8)	−0.0037(8)
C(4)	2*i*	0.5127(5)	0.6921(4)	0.5523(3)	0.020(2)	0.016(1)	0.013(1)	−0.003(1)	0.002(1)	−0.004(1)
O(2)	2*i*	0.2719(3)	0.8233(3)	0.7940(2)	0.025(1)	0.016(1)	0.012(1)	−0.0021(9)	0.0020(9)	−0.0031(8)
N(5)	2*i*	0.2156(4)	1.0107(4)	0.5409(2)	0.017(1)	0.011(1)	0.012(1)	−0.0025(9)	−0.0016(9)	−0.0002(9)
N(4)	2*i*	0.2284(4)	1.0604(4)	0.4288(2)	0.015(1)	0.011(1)	0.012(1)	−0.0006(9)	−0.0032(9)	0.0007(9)
N(3)	2*i*	0.0794(4)	1.2454(3)	0.3701(2)	0.015(1)	0.010(1)	0.010(1)	−0.0016(9)	−0.0003(9)	0.0000(9)
N(6)	2*i*	0.3646(4)	0.8262(4)	0.5979(2)	0.016(1)	0.011(1)	0.010(1)	−0.0006(9)	−0.0014(9)	0.0000(9)
N(1)	2*i*	−0.0860(4)	1.5074(4)	0.2127(2)	0.017(1)	0.014(1)	0.011(1)	−0.005(1)	0.0007(9)	0.0004(9)
N(2)	2*i*	−0.1712(4)	1.5416(4)	0.3193(2)	0.017(1)	0.013(1)	0.014(1)	−0.002(1)	0.001(1)	−0.0030(9)
N(8)	2*i*	0.5415(4)	0.5593(4)	0.7519(2)	0.018(1)	0.011(1)	0.012(1)	−0.0009(9)	−0.001(1)	−0.0011(9)
N(7)	2*i*	0.6187(4)	0.5345(4)	0.6417(2)	0.019(1)	0.016(1)	0.015(1)	−0.001(1)	0.000(1)	−0.004(1)
C(3)	2*i*	0.3854(4)	0.7382(4)	0.7254(3)	0.015(1)	0.013(1)	0.009(1)	−0.004(1)	−0.003(1)	0.001(1)
C(2)	2*i*	−0.0710(5)	1.3855(4)	0.4110(3)	0.018(2)	0.016(1)	0.014(1)	−0.004(1)	0.003(1)	−0.005(1)
O(3)	2*i*	0.3119(3)	0.4527(3)	0.9925(2)	0.014(1)	0.0125(9)	0.0114(9)	−0.0042(8)	0.0015(8)	−0.0005(8)
O(4)	2*i*	0.0620(3)	1.1850(3)	0.9873(2)	0.017(1)	0.0118(9)	0.013(1)	−0.0054(8)	0.0018(8)	−0.0021(8)
O(5)	2*i*	0.5191(3)	0.9217(3)	1.1511(2)	0.015(1)	0.017(1)	0.021(1)	0.0014(9)	−0.0012(9)	−0.0066(9)
O(6)	2*i*	0.0996(4)	0.8537(4)	1.2263(2)	0.027(1)	0.018(1)	0.015(1)	0.001(1)	0.006(1)	−0.0017(9)

Discussion

This contribution is part of our continuing interest in nitrogen-rich coordination compounds [3]. The crystallographic studies reveal that the asymmetric unit is made up of one Ba, one (*E*)-4,4′-(diazene-1,2-diyl)bis(5-oxo-4,5-dihydro-1,2,4-triazol–1–ide) (ZTO^{2-}) anion and four coordinated water molecules. In crystal structure, Ba1 is coordinated by two adjacent ZTO2− anion through one Ba—O [Ba1—O2 = 2.709(2) Å], one Ba—N [Ba1—N8# = 2.915(3) Å] and seven water ligands (see the figure). Consequently each Ba1 is nine-coordinated. Ba—O distances of the water ligands are: Ba1—O3 = 2.827(2) Å, Ba1—O4 = 2.724(2) Å, Ba1—O5 = 2.895(3) Å and Ba1—O6 = 2.766(2) Å. These connections lead to a two dimensional coodination polymer.

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