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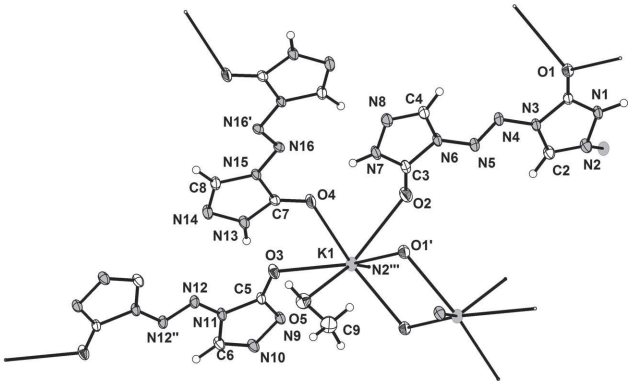
Crystal structure of potassium (*E*)-5-oxo-4-((5-oxo-1*H*-1,2,4-triazol-4(5*H*)-yl)diazenyl)-4,5-dihydro-1,2,4-triazol-1-ide – (*E*)-4,4-diazene-1,2-diylbis(2,4-dihydro-3*H*-1,2,4-triazol-3-one) – methanol (1/1/1), $C_9H_{11}N_{16}KO_5$



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Abstract

$C_9H_{11}N_{16}K_1O_5$, triclinic, $P\bar{1}$ (no. 2), $a = 7.983(2)$ Å, $b = 9.731(2)$ Å, $c = 12.300(4)$ Å, $\alpha = 84.689(9)°$, $\beta = 86.779(9)°$, $\gamma = 69.450(7)°$, $V = 890.5(4)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0396$, $wR_{ref}(F^2) = 0.0878$, $T = 153$ K.

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Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

ZTO (4,4′-azobis(1,2,4-triazolone), 1.00 g) [1, 2] was suspended in 5 mL of water and to it a solution of KOH (1.20 g in 2 mL of water) was added dropwise. After 1 h at room temperature, 50 mL of methanol was added dropwise, and the resulting mixture was slowly cooled to room temperature. The bright yellow precipitate was filtered, washed with methanol and dried under vacuum, yielding 1.0 g (83.7%). IR (KBr pellet): 3222, 3121, 1619, 1500, 1334, 1253, 1203, 1017, 916, 823, 799, 748 cm^{-1}.

Table 1: Data collection and handling.

Crystal:	Yellow, prism, size 0.09×0.18×0.28 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.68 cm^{-1}
Diffractometer, scan mode:	AFC10/Saturn724+, φ and ω scans
$2\theta_{max}$:	63°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	13542, 5812
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4527
$N(param)_{refined}$:	297
Programs:	SHELX [3], Diamond [4], SADABS [5]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2)	2*i*	0.7103	−0.1898	0.5846	0.024
H(4)	2*i*	1.1680	−0.0655	0.2966	0.022
H(6)	2*i*	−0.3193	1.0054	0.0820	0.023
H(8)	2*i*	0.7779	0.7278	−0.1226	0.026
H(9A)	2*i*	0.5053	0.9165	0.3945	0.045
H(9B)	2*i*	0.5930	0.7441	0.4291	0.045
H(9C)	2*i*	0.3848	0.8290	0.4536	0.045
H(13N)	2*i*	0.383(3)	0.702(2)	0.063(2)	0.046(6)
H(7N)	2*i*	0.799(3)	0.289(2)	0.197(2)	0.046(6)
H(1N)	2*i*	1.074(3)	−0.554(2)	0.693(2)	0.048(6)
H(5O)	2*i*	0.537(3)	0.805(2)	0.252(2)	0.055(7)

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Table 3: Fractional coordinates and atomic displacement parameters (Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
K(1)	2*i*	0.41498(4)	0.51624(3)	0.32988(3)	0.0163(1)	0.0241(2)	0.0210(2)	−0.0080(1)	0.0019(1)	0.0053(1)
O(1)	2*i*	1.2723(1)	−0.4475(1)	0.54115(8)	0.0158(5)	0.0232(5)	0.0237(5)	−0.0022(4)	0.0032(4)	0.0053(4)
O(2)	2*i*	0.6038(1)	0.1882(1)	0.34141(9)	0.0208(5)	0.0254(6)	0.0330(6)	−0.0005(4)	0.0022(4)	0.0081(5)
O(3)	2*i*	0.1966(1)	0.6709(1)	0.15157(8)	0.0124(4)	0.0220(5)	0.0278(5)	−0.0029(4)	−0.0001(4)	0.0096(4)
O(4)	2*i*	0.6400(1)	0.4820(1)	0.15753(9)	0.0181(5)	0.0206(5)	0.0267(5)	−0.0002(4)	0.0055(4)	0.0113(4)
O(5)	2*i*	0.4444(2)	0.7907(1)	0.30044(9)	0.0203(5)	0.0315(6)	0.0313(6)	−0.0112(5)	0.0032(4)	0.0010(5)
N(1)	2*i*	1.0293(2)	−0.4660(1)	0.6500(1)	0.0162(5)	0.0163(5)	0.0197(6)	−0.0044(4)	−0.0001(4)	0.0069(4)
N(2)	2*i*	0.8498(2)	−0.3811(1)	0.6626(1)	0.0154(5)	0.0198(6)	0.0258(6)	−0.0060(4)	0.0005(4)	0.0055(5)
N(3)	2*i*	0.9782(2)	−0.2748(1)	0.53584(9)	0.0149(5)	0.0143(5)	0.0169(5)	−0.0039(4)	−0.0008(4)	0.0050(4)
N(4)	2*i*	1.0105(2)	−0.1837(1)	0.45120(9)	0.0186(5)	0.0153(5)	0.0173(5)	−0.0066(4)	−0.0020(4)	0.0051(4)
N(5)	2*i*	0.8696(2)	−0.0776(1)	0.42903(9)	0.0207(6)	0.0147(5)	0.0186(5)	−0.0067(4)	−0.0022(4)	0.0046(4)
N(6)	2*i*	0.8994(2)	0.0151(1)	0.34523(9)	0.0186(5)	0.0136(5)	0.0188(6)	−0.0047(4)	−0.0023(4)	0.0059(4)
N(7)	2*i*	0.8455(2)	0.2071(1)	0.2330(1)	0.0228(6)	0.0143(5)	0.0233(6)	−0.0024(5)	−0.0020(5)	0.0074(5)
N(8)	2*i*	1.0254(2)	0.1265(1)	0.2200(1)	0.0230(6)	0.0182(6)	0.0216(6)	−0.0065(5)	−0.0003(5)	0.0030(5)
N(9)	2*i*	−0.0991(2)	0.7124(1)	0.21162(9)	0.0130(5)	0.0147(5)	0.0211(6)	−0.0031(4)	0.0012(4)	0.0051(4)
N(10)	2*i*	−0.2622(2)	0.8241(1)	0.1849(1)	0.0135(5)	0.0170(5)	0.0271(6)	−0.0028(4)	0.0025(4)	0.0054(5)
N(11)	2*i*	−0.0502(2)	0.8741(1)	0.08843(9)	0.0133(5)	0.0151(5)	0.0184(5)	−0.0048(4)	−0.0002(4)	0.0064(4)
N(12)	2*i*	0.0490(2)	0.9389(1)	0.02110(9)	0.0166(5)	0.0161(5)	0.0166(5)	−0.0066(4)	−0.0001(4)	0.0050(4)
N(13)	2*i*	0.4914(2)	0.6820(1)	0.0371(1)	0.0119(5)	0.0199(6)	0.0227(6)	−0.0008(4)	0.0025(4)	0.0067(5)
N(14)	2*i*	0.5367(2)	0.7599(1)	−0.0526(1)	0.0169(6)	0.0244(6)	0.0237(6)	−0.0016(5)	0.0014(5)	0.0108(5)
N(15)	2*i*	0.7743(1)	0.5815(1)	0.01133(9)	0.0113(5)	0.0151(5)	0.0173(5)	0.0001(4)	0.0023(4)	0.0042(4)
N(16)	2*i*	0.9439(1)	0.4837(1)	0.03118(9)	0.0120(5)	0.0152(5)	0.0185(5)	0.0000(4)	0.0018(4)	0.0011(4)
C(1)	2*i*	1.1155(2)	−0.4052(1)	0.5730(1)	0.0173(6)	0.0147(6)	0.0153(6)	−0.0048(5)	−0.0012(5)	0.0025(5)
C(2)	2*i*	0.8221(2)	−0.2675(2)	0.5939(1)	0.0158(6)	0.0196(6)	0.0227(7)	−0.0059(5)	0.0000(5)	0.0042(5)
C(3)	2*i*	0.7589(2)	0.1453(2)	0.3099(1)	0.0215(6)	0.0150(6)	0.0197(6)	−0.0035(5)	−0.0030(5)	0.0043(5)
C(4)	2*i*	1.0553(2)	0.0111(2)	0.2876(1)	0.0186(6)	0.0166(6)	0.0194(6)	−0.0055(5)	−0.0015(5)	0.0028(5)
C(5)	2*i*	0.0325(2)	0.7416(1)	0.1528(1)	0.0149(6)	0.0132(6)	0.0155(6)	−0.0043(5)	−0.0012(4)	0.0042(4)
C(6)	2*i*	−0.2313(2)	0.9188(2)	0.1129(1)	0.0131(6)	0.0163(6)	0.0252(7)	−0.0029(5)	−0.0002(5)	0.0047(5)
C(7)	2*i*	0.6326(2)	0.5713(1)	0.0788(1)	0.0119(5)	0.0153(6)	0.0191(6)	−0.0019(5)	0.0024(5)	0.0019(5)
C(8)	2*i*	0.7079(2)	0.6979(2)	−0.0668(1)	0.0175(6)	0.0213(7)	0.0193(7)	−0.0016(5)	0.0021(5)	0.0075(5)
C(9)	2*i*	0.4849(3)	0.8224(2)	0.4019(2)	0.0355(9)	0.038(1)	0.036(1)	−0.0099(8)	0.0005(7)	−0.0007(8)

Experimental details

Absorption correction was performed by multiscan method implemented in SADABS. The nitrogen and oxygen bonded hydrogen atoms were refined freely. The carbon-bound hydrogen atoms were placed on calculated positions using a riding model (AFIX 43 or AFIX 137 option of the SHELX program [3]).

Discussion

The crystallographic studies reveal that the asymmetric unit is made up of K^+ cation, an (*E*)-5-oxo-4-((5-oxo-1*H*-1,2,4-triazol-4(5*H*)-yl)diazenyl)-4,5-dihydro-1,2,4-triazol-1-ide anion, two half of (*E*)-4,4-diazene-1,2-diylbis(2,4-dihydro-3*H*-1,2,4-triazol-3-one) molecule and one methanol molecule (cf. the figure; ′ = 2−*x*, 1−*y*, −*z*; ″ −*x*, 2−*y*, −*z*). In the crystal structure, K(1) is coordinated by one ZTO^{2-} anion through two K—O [K(1)—O(4) = 2.6674(12) Å] and one K—N [K(1)—N(2)‴ = 2.8483(13) Å] (‴ = 1−*x*, −*y*, 1−*z*), as well as two ZTO molecules through three K—O [K(1)—O(1) = 2.7802(13) Å, K(1)—O(2) = 3.0073(13) Å, K(1)—O(4) = 2.6674(12) Å,] and one methanol through two K—O [K(1)—O(5) = 2.7512(13) Å]. These connections lead to a three-dimensional coodination polymer.

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