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The crystal structure of acetoximium 1'-hydroxy-1*H*,1*H*'-5,5'-bitetrazole-1-olate monohydrate, C₅H₁₁N₉O₄

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.15 × 0.11 × 0.08 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.14 mm ⁻¹
Diffractometer, scan mode:	D8 VENTURE, φ and ω
$\theta_{\max}$, completeness:	26.3°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	10747, 2181, 0.083
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1375
$N(\text{param})_{\text{refined}}$:	186
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3], Diamond [4]

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Abstract

C₅H₁₁N₉O₄, monoclinic, $P2_1/c$ (no. 14), $a = 7.4259(5)$ Å, $b = 6.6115(5)$ Å, $c = 21.9579(14)$ Å, $\beta = 93.073(2)^\circ$, $V = 1076.50(13)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0545$, $wR_{\text{ref}}(F^2) = 0.1234$, $T = 170$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

An amount of 4.0 g of dihydroxylammonium 5,5'-bistetrazole-1,1'-diolate (TKX-50) was added into the mixed solution

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.4678(4)	0.6962(4)	0.56104(13)	0.0223(7)
C2	0.3537(4)	0.7525(4)	0.50891(13)	0.0212(7)
C3	0.0270(4)	0.8860(4)	0.69939(15)	0.0311(8)
H3A	0.1253	0.9148	0.6726	0.047*
H3B	0.0422	0.9684	0.7364	0.047*
H3C	−0.0887	0.9185	0.6781	0.047*
C4	0.0303(4)	0.6696(5)	0.71606(13)	0.0240(7)
C5	−0.0812(4)	0.5872(5)	0.76410(15)	0.0349(8)
H5A	−0.2047	0.5658	0.7473	0.052*
H5B	−0.0824	0.6831	0.7981	0.052*
H5C	−0.0304	0.4581	0.7787	0.052*
H1A	0.169(5)	0.171(6)	0.5809(18)	0.058(13)*
H1B	0.353(5)	0.185(5)	0.5877(17)	0.050(12)*
N1	0.6478(3)	0.6824(4)	0.56229(11)	0.0226(6)
N2	0.7105(3)	0.6261(4)	0.61801(11)	0.0275(6)
N3	0.5677(3)	0.6063(4)	0.65030(12)	0.0297(7)
N4	0.4165(3)	0.6487(4)	0.61626(11)	0.0254(6)
N5	0.1736(3)	0.7569(4)	0.50839(11)	0.0239(6)
N6	0.1059(3)	0.8152(4)	0.45375(11)	0.0290(6)
N7	0.2454(3)	0.8460(4)	0.42073(12)	0.0297(6)
N8	0.4007(3)	0.8085(4)	0.45372(11)	0.0265(6)
N9	0.1312(3)	0.5542(4)	0.68651(12)	0.0254(6)
H9	0.205(4)	0.601(5)	0.6563(13)	0.050(11)*
O1	0.2621(3)	0.1638(3)	0.60785(11)	0.0307(6)
O2	0.1442(3)	0.3512(3)	0.69916(10)	0.0331(6)
H2	0.188(5)	0.291(6)	0.6664(14)	0.084(16)*
O3	0.7553(3)	0.7118(3)	0.51674(9)	0.0324(6)
O4	0.0703(3)	0.7072(4)	0.55419(10)	0.0349(6)
H4	−0.051(3)	0.711(7)	0.542(2)	0.102(17)*

containing 50 mL water, 75 mL acetic acid and 4 mL acetone below 42 °C. The reaction mixture was kept at 42 °C for 2 h. After the reaction, the reaction liquid was moved to the wild-mouth bottle while it was hot, and the cap was closed tightly. After standing for 36 h, crystals of the title compound were obtained.

Experimental details

All hydrogen atoms were identified in difference Fourier syntheses. The O–H bond lengths were constrained to 0.87 Å for the hydrogen atoms of the heterocyclic ring. The C–H bond, N–H bond and O–H bond lengths were constrained to 0.98 Å, 0.90 Å, 0.84 Å for the hydrogen atoms of acetoximium.

Comment

TKX-50 was synthesized as a new energetic material with high energy and low sensitivity by Fischer *et al.* in 2012 [5, 6]. The spheroidization of energetic materials is an important way to increase energy and reduce sensitivity [7]. Solvents such as acetic acid, formic acid, acetone and water are used for TKX-50 crystallization [8, 9]. The title compound was obtained from the crystallization of TKX-50 in acetone, acetic acid and water. The title compound was crystallized to determine its structure by means of X-ray diffraction. The crystal structure of the title compound was analyzed and refined using the SHELXL program [1–4].

As shown in the figure, the title compound has similar heterocyclic rings structure to TKX-50. The asymmetric unit of the title compound contains one acetoximium cation, one 1'-hydroxy-1H,1H'-5,5'-bistetrazole-1-olate anion and one water molecule. The heterocyclic rings in the organic anion are planar and the dihedral angle of the two tetrazole rings is 1.4°. The oxygen atom of N-1 and hydroxyl group of N-5 are almost

coplanar with the heterocyclic ring. All bond lengths and angles are in the expected ranges.

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