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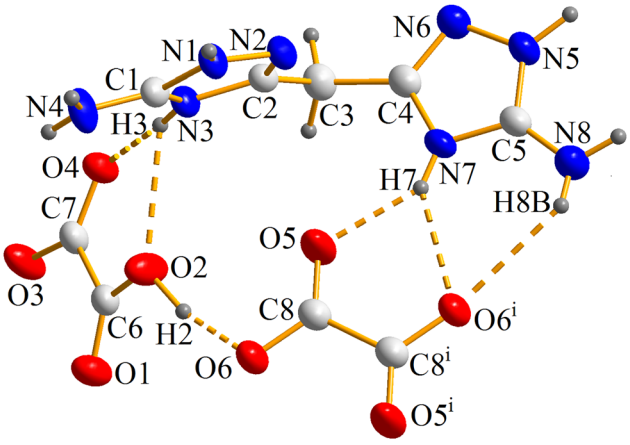
Crystal structure of the double salt bis(5-amino-1,2,4-triazol-4-ium-3-yl)methane hydrogen oxalate hemioxalate, $C_8H_{11}N_8O_6$



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Abstract

$C_8H_{11}N_8O_6$, monoclinic, $P2_1/n$ (no. 14), a = 5.6348(8) Å, b = 19.485(3) Å, c = 11.5316(17) Å, β = 101.013(2)°, V = 1242.8(3) Å^3, Z = 4, $R_{gt}(F)$ = 0.0396, w$R_{ref}(F^2)$ = 0.1039, T = 296(2) K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size	0.37 × 0.28 × 0.14 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.15 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.6°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7096, 2808, 0.026
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 2258
$N(param)_{refined}$:	243
Programs:	Bruker [1], SHELX [2–4], Diamond [5]

Source of material

Bis(5-amino-1*H*-1,2,4-triazol-3-yl)methane (BATZM) was synthesized and purified according to a reported method [6], oxalic acid dihydrate (0.630 g, 7.0 mmol) was added at room temperature to BATZM (0.631 g, 3.5 mmol) in deionized water (30 mL). The resulting solution was stirred for 2 h. Colourless block crystals of the title compound were obtained by slow evaporation from water.

Experimental details

All hydrogen atoms were originally found in electron density difference maps, but were treated differently in the refinement, only O–H distances restrained to 0.86(1) Å, and with U_{iso}(H) = 1.2U_{eq}(O).

Comment

Triazoles are widely present in energetic materials due to higher nitrogen content [7–9], bis-triazoles are an important class of energetic compounds [10, 11], we have also reported some bis-triazole compounds [6, 12–14].

The cation of the title compound has no symmetry, unlike that was found in the structure of the pure component and other salts [6, 12–14]. The combination of H atoms with N3, indicates that N3 has the highest charge density among all N atoms in the base BATZM, this is the same as other salts. The midpoint of the O8–O8^i bond is the center of symmetry of the two asymmetric units Symmetry codes: (i) –x, –y, –z + 1. Neighbouring ion are further linked by electrostatic attractions and hydrogen bonds: O2–H2···O6 ($d_{O2\cdots O6}$ = 2.4862(16)

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1276 (3)	0.25988 (7)	0.40800 (12)	0.0280 (3)
C2	0.4367 (3)	0.19870 (7)	0.49056 (12)	0.0272 (3)
C3	0.6448 (3)	0.15019 (9)	0.50425 (14)	0.0348 (4)
H3A	0.608 (4)	0.1142 (10)	0.4450 (16)	0.049 (5)*
H3B	0.786 (4)	0.1748 (9)	0.4923 (16)	0.044 (5)*
C4	0.6985 (3)	0.11877 (8)	0.62456 (13)	0.0310 (3)
C5	0.6522 (3)	0.05632 (7)	0.77502 (13)	0.0320 (3)
C6	0.0030 (3)	0.10290 (7)	0.13383 (12)	0.0306 (3)
C7	0.2004 (3)	0.14191 (8)	0.08432 (12)	0.0314 (3)
C8	0.0189 (3)	0.02596 (8)	0.45215 (13)	0.0329 (3)
O1	−0.1230 (2)	0.05986 (6)	0.07569 (10)	0.0440 (3)
O2	−0.0087 (3)	0.12053 (7)	0.23992 (10)	0.0506 (4)
H2	−0.070 (5)	0.0867 (10)	0.275 (2)	0.110 (10)*
O3	0.1993 (2)	0.13452 (7)	−0.02278 (9)	0.0494 (3)
O4	0.3493 (2)	0.17700 (6)	0.15438 (9)	0.0365 (3)
O5	0.2007 (3)	0.06157 (8)	0.47122 (12)	0.0641 (4)
O6	−0.1458 (2)	0.02807 (6)	0.36035 (9)	0.0384 (3)
N1	0.1635 (2)	0.26599 (7)	0.52508 (10)	0.0307 (3)
H1	0.063 (4)	0.2880 (10)	0.5663 (17)	0.050 (5)*
N2	0.3582 (2)	0.22682 (6)	0.57786 (10)	0.0307 (3)
N3	0.3035 (2)	0.21850 (6)	0.38434 (10)	0.0283 (3)
H3	0.310 (4)	0.2034 (10)	0.3128 (18)	0.050 (5)*
N4	−0.0496 (3)	0.28839 (8)	0.33243 (12)	0.0417 (4)
H4A	−0.152 (4)	0.3144 (11)	0.3634 (18)	0.061 (6)*
H4B	−0.065 (4)	0.2813 (10)	0.254 (2)	0.055 (6)*
N5	0.8560 (2)	0.09183 (7)	0.79969 (12)	0.0361 (3)
H5	0.963 (4)	0.0964 (10)	0.8718 (18)	0.053 (5)*
N6	0.8865 (2)	0.13246 (7)	0.70495 (11)	0.0369 (3)
N7	0.5498 (2)	0.07197 (6)	0.66370 (11)	0.0331 (3)
H7	0.415 (4)	0.0549 (11)	0.622 (2)	0.066 (7)*
N8	0.5715 (3)	0.01313 (8)	0.84789 (14)	0.0444 (4)
H8A	0.649 (4)	0.0123 (10)	0.921 (2)	0.057 (6)*
H8B	0.425 (4)	−0.0044 (11)	0.8271 (18)	0.061 (6)*

Å, 170(3)°); N3–H3···O2 ($d_{N3\cdots O2}$ = 2.8971(17) Å, 112.5(16)°); N3–H3···O4 ($d_{N3\cdots O4}$ = 2.8308(16) Å, 174.2(19)°); N7–H7···O5 ($d_{N7\cdots O5}$ = 2.6773(18) Å, 142(2)°); N7–H7···O6^{i} ($d_{N7\cdots O6}$ = 2.9692(18) Å, 138.3(19)°); N8–H8B···O1^{i} ($d_{N8\cdots O1}$ = 3.168(2) Å, 137.5(18)°); N8–H8B···O6^{i} ($d_{N8\cdots O6}$ = 3.157(2) Å, 135.9(18)°); N5–H5···O1^{ii} ($d_{N5\cdots O1}$ = 3.2230(18) Å, 125.7(15)°, symmetry code: (ii) *x* + 1, *y*, *z* + 1; N5–H5···O3^{ii} ($d_{N5\cdots O3}$ = 2.6666(18) Å, 156.4(18)°); N8–H8A···O1^{ii} ($d_{N8\cdots O1}$ = 2.994(2) Å, 152.4(19)°); N4–H4B···N2^{iii} ($d_{N4\cdots N2}$ = 2.8970(19) Å, 171.9(19)°, symmetry code: (iii) *x* − 1/2, −*y* + 1/2, *z* − 1/2); N1–H1···O3^{iv} ($d_{N1\cdots O3}$ = 3.2176(18) Å, 126.1(15)°, symmetry code: (iv) *x* − 1/2, −*y* + 1/2, *z* + 1/2); N1–H1···O4^{iv} ($d_{N1\cdots O4}$ = 2.7568(17) Å, 173.7(18)°); N4–H4A···O3^{iv} ($d_{N4\cdots O3}$ = 2.820(2) Å, 161.6(19)°) connecting the title compounds into a three-dimensional network. The geometric parameters of the title structure are all in the expected ranges [15].

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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