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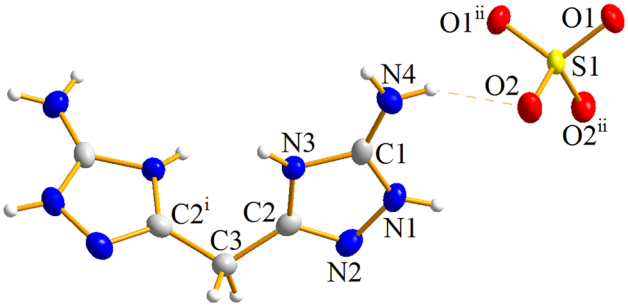
Crystal structure of bis(5-amino-1,2,4-triazol-4-ium-3-yl)methane sulfate, $C_5H_{10}N_8O_4S$



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Abstract

$C_5H_{10}N_8O_4S$, Orthorhombic, *Pba2* (no. 32), $a = 7.7320(11)$ Å, $b = 12.8686(19)$ Å, $c = 5.0657(8)$ Å, $\beta = 90°$, $V = 504.04(13)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0298$, $wR_{ref}(F^2) = 0.0748$, $T = 296(2)$ 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.

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]. In a typical experiment 98% H_2SO_4 (0.2 ml, 3.68 mmol) was added dropwise at room temperature to BATZM (0.631 g, 3.5 mmol) in deionized water (20 ml). The resulting solution was stirred for 2 h. Colorless rod crystals of the title compound were obtained by slow evaporation from water.

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Table 1: Data collection and handling.

Crystal:	Rodlike, colorless
Size:	0.36 × 0.27 × 0.13 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.35 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
θ_{max}, completeness:	30°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	2909, 1218, 0.027
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1144
$N(param)_{refined}$:	104
Programs:	Bruker programs [1], SHELX [2–4], DIAMOND [5]

Experimental details

All hydrogen atoms were originally found in difference maps, but were treated differently in the refinement, with N–H distances restrained to 0.85 (1) Å, and with $U_{iso}(H) = 1.2U_{eq}$ (N), while C–H distances were restrained to 0.96 Å, with $U_{iso}(H) = 1.2U_{eq}(C)$ for methylene H atoms.

Comment

Triazoles are five-membered nitrogen-containing cyclic compounds: two isomers are 1,2,3-trizole and 1,2,4-trizole. Triazole derivatives have a wide range of applications in medical treatment, agriculture and industry, especially in medicine [7, 8], pesticides [9, 10], and energetic materials [11, 12]. Bis-triazoles are an important class of energetic compounds [13, 14]. We have also already reported some bis-triazole compounds [6, 15, 16, 17].

The asymmetric unit of the title compound structure contains one half of the title compound (see the figure; ^{i} = 2–*x*, –*y*, *z*; ^{ii} = 2–*x*, 1–*y*, *z*). The cation has an internal twofold axis which passes through atom C3 and is parallel to the *c* axis. Also the anion is located on a twofold axis. The combination of H atoms with N3, also indicates that N3 has the highest charge density among all N atoms in the base BATZM. Neighbouring ion are linked by electrostatic attractions and hydrogen bonds: N4—H4A···O2 ($d_{N4\cdots O2}$ = 2.830(3) Å, 153(4)°); N1—H1···O1^{iii} ($d_{N1\cdots O1}$ = 2.823(3) Å, 139(4)°, symmetry code: (iii) –*x* + 2, –*y* + 1, *z* + 1); N3—H3···O2^{iv} ($d_{N3\cdots O2}$ = 2.713(3) Å,

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	1.000000	0.500000	0.0012 (2)	0.0207 (2)
N4	0.7524 (3)	0.2499 (2)	0.1393 (6)	0.0301 (6)
N3	0.8528 (3)	0.10502 (17)	0.3898 (5)	0.0233 (5)
N1	0.9411 (3)	0.25576 (18)	0.5071 (5)	0.0275 (5)
N2	1.0196 (3)	0.18705 (19)	0.6768 (5)	0.0290 (6)
O1	0.9325 (3)	0.58532 (14)	−0.1606 (5)	0.0312 (5)
O2	0.8585 (3)	0.45990 (16)	0.1732 (5)	0.0318 (5)
C1	0.8421 (3)	0.2077 (2)	0.3321 (6)	0.0228 (6)
C2	0.9617 (4)	0.0969 (2)	0.6034 (6)	0.0236 (6)
C3	1.000000	0.000000	0.7544 (8)	0.0271 (9)
H4A	0.768 (5)	0.3118 (14)	0.099 (9)	0.050 (12)*
H1	0.960 (5)	0.3193 (13)	0.538 (8)	0.044 (10)*
H4B	0.689 (4)	0.217 (2)	0.034 (5)	0.031 (9)*
H3A	1.093 (4)	0.017 (3)	0.871 (8)	0.050 (11)*
H3	0.782 (4)	0.061 (3)	0.329 (9)	0.061 (13)*

175(4)°); N4—H4B···O1^{iv}($d_{N4\cdots O1}$ = 2.973(4) Å, 159(3)°, symmetry code: (iv) $-x + 3/2, -y + 1/2, z$); N4—H4B···N2^{v} ($d_{N4\cdots N2}$ = 3.064(4) Å, 120(3)°, symmetry code: (v) $x - 1/2, -y + 1/2, z - 1$) into a three-dimensional network. The geometric parameters of the title compound structure are all in the expected ranges [18].

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

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