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Crystal structure of bis(amino(carbamothioylamino)methaniminium) 5-hydroxyisophthalate monohydrate, $C_{12}H_{20}N_8O_6S_2$

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.40 × 0.30 × 0.20 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.33 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	9182, 3307, 0.024
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3000
$N(\text{param})_{\text{refined}}$:	263
Programs:	Bruker [1], SHELX [2, 3]

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Abstract

$C_{12}H_{20}N_8O_6S_2$, monoclinic, $P2_1/c$ (no. 14), $a = 15.8571(11)$ Å, $b = 7.6581(5)$ Å, $c = 16.9166(12)$ Å, $\beta = 113.334(1)^\circ$, $V = 1886.3(2)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0317$, $wR_{\text{ref}}(F^2) = 0.1053$, $T = 298$ 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.

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.33357 (3)	−0.00541 (6)	0.21849 (3)	0.04291 (17)
O1	0.10189 (9)	0.05052 (16)	0.26704 (8)	0.0454 (3)
O1W	0.29233 (10)	0.66884 (17)	0.04495 (8)	0.0426 (3)
H1WA	0.2775 (17)	0.622 (3)	−0.0049 (9)	0.064*
H1WB	0.3342 (13)	0.745 (3)	0.0525 (16)	0.064*
N1	0.46447 (11)	0.04277 (19)	0.37236 (10)	0.0401 (4)
H1A	0.5027	0.1073	0.4120	0.048*
H1B	0.4620	−0.0675	0.3807	0.048*
S2	0.89945 (3)	0.71566 (7)	−0.03159 (3)	0.0540 (2)
O2	0.21117 (9)	−0.07118 (16)	0.37867 (8)	0.0454 (3)
N2	0.42281 (9)	0.29022 (17)	0.29635 (8)	0.0291 (3)
H2A	0.4642	0.3321	0.3426	0.035*
C3	0.22425 (10)	0.2323 (2)	0.35587 (10)	0.0268 (3)
O3	0.46600 (8)	0.32013 (16)	0.58223 (8)	0.0402 (3)
N3	0.41147 (10)	0.57218 (17)	0.25229 (9)	0.0343 (3)
H3A	0.3882	0.6542	0.2153	0.041*
H3B	0.4543	0.5956	0.3016	0.041*
C4	0.29954 (11)	0.2540 (2)	0.43380 (10)	0.0267 (3)
H4A	0.3208	0.1607	0.4717	0.032*
O4	0.43760 (8)	0.60496 (15)	0.56368 (7)	0.0370 (3)
N4	0.31658 (10)	0.3718 (2)	0.15904 (9)	0.0400 (4)
H4B	0.2926	0.4524	0.1214	0.048*
H4C	0.2978	0.2657	0.1477	0.048*
C5	0.34267 (10)	0.4157 (2)	0.45464 (9)	0.0256 (3)
O5	0.20727 (9)	0.66419 (15)	0.26058 (8)	0.0428 (3)

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H5	0.2233 (16)	0.759 (2)	0.2896 (14)	0.064*
N5	1.04724 (11)	0.6640 (2)	0.10754 (10)	0.0498 (4)
H5B	1.0876	0.6937	0.1571	0.060*
H5C	1.0514	0.5646	0.0857	0.060*
C6	0.31186 (11)	0.5537 (2)	0.39685 (10)	0.0275 (3)
H6A	0.3411	0.6615	0.4104	0.033*
N6	0.98156 (9)	0.92456 (19)	0.10816 (9)	0.0354 (3)
H6B	1.0272	0.9348	0.1571	0.043*
C7	0.23794 (11)	0.5309 (2)	0.31940 (10)	0.0293 (4)
N7	0.94029 (11)	1.1896 (2)	0.14213 (10)	0.0439 (4)
H7A	0.9058	1.2806	0.1309	0.053*
H7B	0.9864	1.1809	0.1907	0.053*
C8	0.19361 (11)	0.3709 (2)	0.29857 (10)	0.0290 (3)
H8A	0.1436	0.3565	0.2465	0.035*
N8	0.85303 (11)	1.0716 (2)	0.01024 (10)	0.0466 (4)
H8B	0.8173	1.1610	−0.0028	0.056*
H8C	0.8431	0.9872	−0.0259	0.056*
C9	0.17556 (11)	0.0591 (2)	0.33242 (10)	0.0314 (4)
C10	0.42190 (11)	0.4464 (2)	0.54001 (10)	0.0289 (3)
C11	0.40936 (11)	0.1134 (2)	0.29826 (10)	0.0293 (4)
C12	0.38154 (10)	0.4105 (2)	0.23346 (9)	0.0269 (3)
C13	0.97930 (11)	0.7703 (2)	0.06524 (11)	0.0350 (4)
C14	0.92257 (11)	1.0640 (2)	0.08512 (11)	0.0351 (4)

Source of material

Amidinothiourea (0.25 mmol, A. R.), 5-hydroxy isophthalic acid (0.25 mmol, A. R.) and tetrapropylammonium hydroxide (30% aqueous solution) were dissolved in small amount of water/ethanol (v/v, 1:2) to yield a clean solution with the molar ratio of 1:1:2. After stirring for an hour, the solution was set aside to generate colorless block crystals of the title compound after about 2 weeks. Tetrapropylammonium hydroxide was not present in the final structure.

Experimental details

Hydrogen atoms were positioned geometrically.

Comment

Amidinothiourea is the medical intermediate of famotidine that is a common acid inhibitor [4]. Also, amidinothiourea was used very early to coordinate with different metal ions to synthesize interesting compounds [5]. Noticeably, the organic adducts involving amidinothiourea were rarely

reported. As a almost coplanar molecule, amidinothiourea can yield various hydrogen bonding interactions due to its N and S atoms.

In the asymmetric unit of the title compound, there are two amidinothiourea cations, one isophthalate dianion and one water molecule. Obviously, isophthalic acid loses its two carboxylic protons to become the corresponding dianion, and two N atoms derived from two amidinothiourea molecules then accept the two protons to yield the two cations mentioned above. These two cations firstly connect with each other to form a dimer by N–H⋯S hydrogen bonding, then the dimer interacts with isophthalate to generate a hydrogen-bonded ribbon by N–H⋯O contacts. The water molecule is an important linking molecule that connects the neighboring hydrogen-bonded ribbons to form the final 3-dimensional network by different N–H⋯O and O–H⋯O interactions. It is shown that amidinothiourea [6] is a good hydrogen-bonding synthon that can interact with many molecules to generate novel crystal structures.

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