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Crystal structure of bis(amino(carbamothioylamino)methaniminium) 3-nitrophthalate monohydrate, $C_{12}H_{19}N_9O_7S_2$

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 :	0.34 mm ⁻¹
Diffraction, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	9554, 3339, 0.041
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3095
$N(\text{param})_{\text{refined}}$:	277
Programs:	SHELX [1, 2], Bruker [3, 4]

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.

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.18117 (11)	0.21275 (2)	0.79951 (9)	0.0411 (2)
C1	0.8048 (3)	0.07343 (6)	0.5654 (3)	0.0311 (5)
H1A	0.8191	0.0504	0.5289	0.037*
O1	0.7322 (3)	0.18333 (4)	0.9003 (2)	0.0337 (4)
N1	1.0721 (3)	0.05997 (5)	0.8883 (3)	0.0289 (4)
O1W	0.5484 (3)	0.86633 (5)	0.0718 (3)	0.0469 (5)
H1WA	0.4617	0.8493	0.0624	0.070*
H1WB	0.6108	0.8598	0.0236	0.070*
S2	0.77203 (10)	0.96808 (2)	0.54773 (9)	0.0422 (2)
C2	0.9200 (3)	0.08492 (5)	0.7548 (3)	0.0232 (4)
O2	0.6683 (3)	0.20212 (4)	0.6048 (2)	0.0373 (4)
N2	0.2030 (3)	0.28108 (5)	0.7422 (3)	0.0345 (5)
H2A	0.2117	0.2989	0.6764	0.041*
H2B	0.1994	0.2846	0.8508	0.041*
O3	0.9552 (2)	0.11576 (4)	1.1467 (2)	0.0320 (4)
C3	0.9012 (3)	0.11869 (5)	0.8215 (3)	0.0207 (4)
N3	0.2024 (3)	0.24733 (5)	0.5019 (3)	0.0271 (4)
H3	0.208 (4)	0.2698 (4)	0.455 (4)	0.041*
C4	0.7607 (3)	0.14186 (5)	0.6821 (3)	0.0230 (4)
O4	1.2240 (2)	0.07121 (5)	1.0265 (3)	0.0414 (4)
N4	0.1968 (3)	0.22647 (5)	0.2244 (3)	0.0385 (5)
H4A	0.1894	0.2096	0.1458	0.046*
H4B	0.2082	0.2482	0.1958	0.046*
C5	0.6492 (3)	0.13076 (6)	0.4897 (3)	0.0305 (5)

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Abstract

$C_{12}H_{19}N_9O_7S_2$, monoclinic, $P2_1/n$ (no. 14), $a = 7.3376(8)$ Å, $b = 37.536(4)$ Å, $c = 7.7149(8)$ Å, $\beta = 117.1190(10)^\circ$, $V = 1891.3(3)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0453$, $wR_{\text{ref}}(F^2) = 0.1321$, $T = 298$ K.

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Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H5A	0.5596	0.1466	0.3979	0.037*
O5	1.0423 (3)	0.02799 (5)	0.8544 (3)	0.0539 (5)
N5	0.1737 (3)	0.18599 (5)	0.4325 (3)	0.0391 (5)
H5B	0.1661	0.1689	0.3551	0.047*
H5C	0.1700	0.1814	0.5401	0.047*
O6	1.1684 (2)	0.14948 (4)	1.0835 (2)	0.0320 (4)
C6	0.6684 (4)	0.09681 (6)	0.4320 (3)	0.0335 (5)
H6A	0.5896	0.0897	0.3035	0.040*
N6	0.7657 (3)	0.90039 (5)	0.4622 (3)	0.0326 (4)
H6B	0.7194	0.8825	0.3844	0.039*
H6C	0.8637	0.8974	0.5777	0.039*
C7	0.7185 (3)	0.17892 (5)	0.7331 (3)	0.0244 (4)
N7	0.5304 (3)	0.93216 (5)	0.2146 (3)	0.0269 (4)
H7	0.509 (4)	0.9107 (4)	0.149 (3)	0.040*
C8	1.0192 (3)	0.12912 (5)	1.0354 (3)	0.0240 (4)
N8	0.2533 (3)	0.94923 (6)	−0.0616 (3)	0.0355 (5)
H8A	0.1683	0.9650	−0.1358	0.043*
H8B	0.2416	0.9274	−0.0990	0.043*
C9	0.1951 (3)	0.24830 (6)	0.6765 (3)	0.0256 (4)
N9	0.4250 (3)	0.99152 (5)	0.1720 (3)	0.0387 (5)
H9A	0.3422	1.0078	0.1008	0.046*
H9B	0.5231	0.9969	0.2842	0.046*
C10	0.1910 (3)	0.21902 (6)	0.3865 (3)	0.0280 (5)
C11	0.6868 (3)	0.93218 (6)	0.4046 (3)	0.0265 (4)
C12	0.4018 (3)	0.95860 (6)	0.1096 (3)	0.0268 (5)

Source of material

Amidinothiourea (0.25 mmol, A. R.), 3-nitrophthalic acid (0.25 mmol, A. R.) and tetrapropylammonium hydroxide (30% aqueous solution) were dissolved (ratio: 1/2/2) in a small amount of water/ethanol (v/v, 1:2) to yield a clean solution. After stirring for an hour, the solution was set aside to generate colorless block crystals of the title compound after about two weeks. Tetrapropylammonium hydroxide was not present in the final structure.

Experimental details

Hydrogen atoms were positioned using riding model options of the SHELX system [2].

Comment

Amidinothiourea, which is a medical intermediate of famotidine [5], can be utilized to coordinate varied metal ions to yield novel compounds [6]. Amidinothiourea can yield plenty of hydrogen bonds because it has several −NH₂

groups and one S atom. Therefore, the title compound was prepared to explore the hydrogen bonding patterns.

In the asymmetric unit of the title compound, there are two monoprotonated amidinothiourea cations, one nitrophthalate and one water molecule. It is obvious that the 3-nitrophthalic acid throws its two carboxylic protons to generate the corresponding anion [7, 8], and two N atoms derived from two amidinothiourea molecules spontaneously accept the two protons to yield the two cations of the asymmetric unit. From the packing diagram, it can be seen that these two cations connect with each other to form a hydrogen-bonded ribbon by N–H...S contacts, then the ribbons are further linked with the 3-nitrophthalate to give a 3-dimensional framework with cavities by different N–H...O hydrogen bonds. The water molecule further consolidates the hydrogen-bonded framework by different N–H...O and O–H...O interactions. It is clear that amidinothiourea can be used as a qualified hydrogen-bonding synthon to generate novel crystal structures [7].

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