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Crystal structure of bis(amino(thioureido) methaniminium) terephthalate, $C_{12}H_{18}N_8O_4S_2$

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Abstract

$C_{12}H_{18}N_8O_4S_2$, monoclinic, $P2_1/c$ (no. 14), $a = 13.378(4)$ Å, $b = 5.0129(14)$ Å, $c = 13.396(4)$ Å, $\beta = 96.118(4)^\circ$, $V = 893.3(4)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0561$, $wR_{ref}(F^2) = 0.1201$, $T = 298$ 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

Amidinothiourea (0.25 mmol, A. R.), terephthalic acid (0.25 mmol, A. R.) and tetrabutylammonium hydroxide (25% aqueous solution) were dissolved in small amount of water/ethanol (v/v, 1:2) to yield a clear solution with the molar ratio of 1:1:2. After stirring half an hour, the solution

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Table 1: Data collection and handling.

Crystal:	Colorless prism
Size:	$0.22 \times 0.08 \times 0.04$ mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.34 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max} , completeness:	27.5° , 59%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	1859, 1311, 0.027
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 714
$N(\text{param})_{\text{refined}}$:	139
Programs:	Bruker [1], SHELX [2, 3]

was set aside to generate colorless prism crystals of the title compound after about 10 days.

Experimental details

H atoms were attached using geometric considerations.

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.96414 (8)	1.18783 (16)	0.64455 (8)	0.0533 (3)
C1	0.5782 (3)	0.1799 (5)	0.5265 (3)	0.0372 (7)
O1	0.7201 (2)	0.4298 (4)	0.4905 (2)	0.0531 (7)
N1	0.8757 (3)	0.8046 (6)	0.5335 (3)	0.0553 (9)
H1C	0.832 (2)	0.666 (5)	0.513 (3)	0.083*
H1D	0.929 (2)	0.828 (7)	0.496 (3)	0.083*
C2	0.5169 (3)	0.0908 (6)	0.5967 (3)	0.0468 (9)
H2A	0.5280	0.1515	0.6626	0.056*
O2	0.6718 (2)	0.4639 (5)	0.6427 (2)	0.0577 (8)
N2	0.8056 (2)	0.8714 (5)	0.6756 (2)	0.0443 (7)
H2	0.768 (2)	0.723 (5)	0.654 (3)	0.067*
C3	0.4398 (3)	−0.0860 (6)	0.5705 (3)	0.0479 (9)
H3A	0.3994	−0.1427	0.6188	0.057*
N3	0.7072 (3)	0.8459 (6)	0.8021 (3)	0.0557 (9)
H3C	0.673 (3)	0.712 (6)	0.766 (3)	0.084*
H3D	0.691 (4)	0.908 (6)	0.8636 (18)	0.084*
C4	0.6623 (3)	0.3711 (5)	0.5562 (3)	0.0424 (8)
N4	0.8337 (3)	1.1562 (5)	0.8160 (3)	0.0499 (8)
H4A	0.809 (3)	1.219 (8)	0.8738 (19)	0.075*
H4B	0.889 (2)	1.228 (7)	0.789 (3)	0.075*
C5	0.8793 (3)	0.9461 (6)	0.6161 (3)	0.0403 (8)
C6	0.7838 (3)	0.9663 (6)	0.7659 (3)	0.0411 (8)

*Hydrogen atoms were refined using a riding model.

Comment

Amidinothiourea is the medical intermediate of famotidine [4]. Amidinothiourea derivatives are also utilized to generate new metal-organic frameworks with the existence of different metal ions [5]. Noticeably, the organic adducts involving amidinothiourea were rarely reported. As we can see, amidinothiourea can form varied hydrogen bonds due to its geometry and functional groups. The title compound was prepared to explore the hydrogen bonding modes, which may be helpful for us to further study the packing patterns of similar systems.

In the asymmetric unit of the title compound, there are one amidinothiourea cation and half a terephthalate that is positioned at the symmetry center. Clearly, terephthalic acid becomes the related anion by losing its protons and one N atom derived from the independent amidinothiourea molecule forms the corresponding cation by accepting the proton. Observing the packing mode of the title structure, it can be seen that the neighboring amidinothiourea cations firstly interact with each other to generate ribbons by a pair of $N-H\cdots S$ hydrogen bonds, and then the hydrogen-bonded ribbons can link with terephthalate anions by $N-H\cdots O$ hydrogen bonds to obtain the final network. Obviously, in this structure, the amidinothiourea derivative cation is a qualified hydrogen bond donor because of its multiple $N-H$ groups and acceptor by its $C=S$ group. That is to say, the title amidinothiourea derivative tends to form sophisticated hydrogen bonds, typical for such a cation in its salts [6].

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