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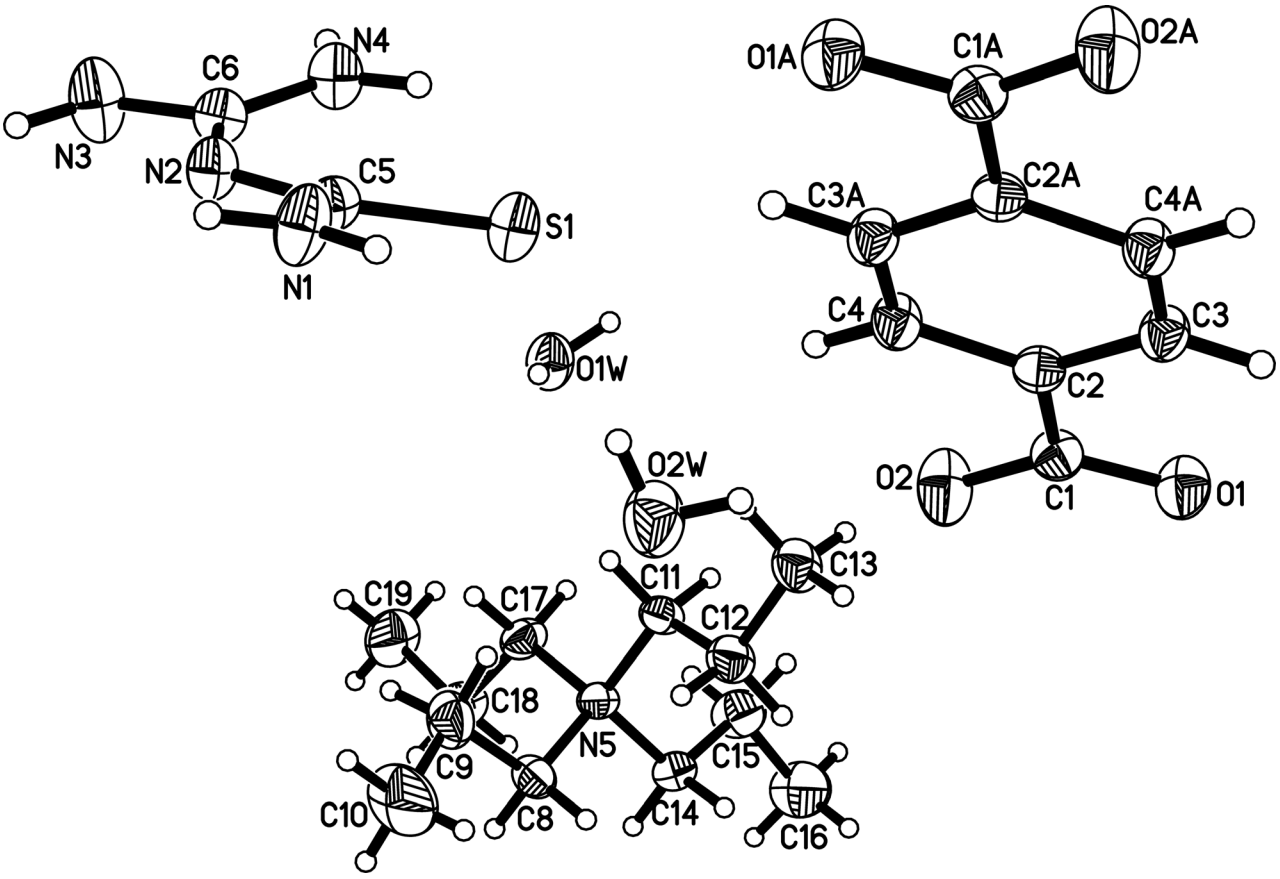
Crystal structure of bis(tetrapropylammonium) terephthalate – 1-(diaminomethylene)thiourea – water (1/2/4) $C_{18}H_{40}N_5O_4S$



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Abstract

$C_{18}H_{40}N_5O_4S$, monoclinic, $P2_1/n$ (no. 14), a = 8.4254(9) Å, b = 8.4254(9) Å, c = 16.1470(17) Å, β = 98.302(2)°, V = 2411.9(4) Å^3, Z = 4, $R_{gt}(F)$ = 0.0409, w$R_{ref}(F^2)$ = 0.1162, 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:	Colorless block
Size:	0.40 × 0.20 × 0.20 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.17 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)u_{nique}$, R_{int}:	12,101, 4245, 0.021
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3591
$N(param)_{refined}$:	253
Programs:	Bruker [1], SHELX [2, 3]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.34682 (19)	0.85885 (9)	0.53201 (10)	0.0377 (4)
N1	0.4709 (2)	1.01283 (8)	0.11194 (10)	0.0543 (4)
H1A	0.4659	1.0325	0.0630	0.065*
H1B	0.4503	1.0394	0.1534	0.065*
O1	0.32977 (15)	0.84318 (6)	0.60614 (7)	0.0491 (3)
S1	0.51596 (6)	0.90591 (3)	0.22298 (3)	0.05203 (16)
O1W	0.47721 (18)	0.15818 (9)	0.22663 (9)	0.0674 (4)
H1WA	0.4518	0.2044	0.2190	0.101*
H1WB	0.5438	0.1569	0.2720	0.101*
C2	0.42669 (17)	0.93211 (8)	0.51590 (9)	0.0328 (3)
N2	0.53983 (18)	0.90651 (7)	0.05306 (8)	0.0421 (3)
O2	0.30074 (18)	0.81831 (7)	0.46993 (8)	0.0618 (4)
O2W	0.2278 (2)	0.79113 (9)	0.29901 (9)	0.0752 (4)
H2WA	0.3080	0.8084	0.2790	0.113*
H2WB	0.2592	0.8023	0.3499	0.113*
C3	0.47187 (19)	0.98258 (9)	0.57998 (10)	0.0382 (4)
H3A	0.4538	0.9713	0.6341	0.046*
N3	0.6209 (2)	0.81132 (9)	−0.02399 (10)	0.0659 (5)
H3B	0.6705	0.7699	−0.0287	0.079*
H3C	0.5766	0.8345	−0.0680	0.079*
C4	0.45618 (19)	0.95030 (9)	0.43571 (10)	0.0388 (4)
H4A	0.4272	0.9169	0.3920	0.047*
N4	0.68277 (18)	0.80151 (8)	0.11661 (9)	0.0485 (4)
H4B	0.7312	0.7603	0.1093	0.058*
H4C	0.6800	0.8180	0.1664	0.058*
C5	0.51118 (19)	0.94056 (9)	0.12356 (10)	0.0392 (4)
N5	0.00279 (15)	0.05238 (8)	0.25538 (9)	0.0419 (3)
C6	0.6126 (2)	0.83952 (9)	0.05139 (10)	0.0404 (4)
C8	−0.1172 (2)	0.10458 (11)	0.20593 (12)	0.0510 (4)
H8A	−0.1671	0.1345	0.2451	0.061*
H8B	−0.2007	0.0748	0.1741	0.061*
C9	−0.0480 (3)	0.15670 (13)	0.14604 (14)	0.0720 (6)
H9A	0.0477	0.1804	0.1750	0.086*
H9B	−0.0174	0.1279	0.1000	0.086*
C10	−0.1652 (4)	0.2150 (2)	0.1125 (2)	0.1264 (13)
H10A	−0.1178	0.2469	0.0750	0.190*
H10B	−0.2592	0.1917	0.0829	0.190*
H10C	−0.1942	0.2441	0.1579	0.190*
C11	0.1380 (2)	0.09682 (10)	0.30580 (11)	0.0461 (4)
H11A	0.2018	0.0629	0.3439	0.055*
H11B	0.2068	0.1161	0.2675	0.055*
C12	0.0861 (2)	0.16127 (12)	0.35598 (13)	0.0590 (5)
H12A	0.0270	0.1971	0.3184	0.071*
H12B	0.0150	0.1430	0.3937	0.071*
C13	0.2278 (3)	0.19921 (12)	0.40571 (13)	0.0638 (5)
H13A	0.1914	0.2398	0.4369	0.096*
H13B	0.2854	0.1640	0.4436	0.096*
H13C	0.2973	0.2180	0.3684	0.096*
C14	−0.0891 (2)	0.00804 (10)	0.31340 (11)	0.0470 (4)
H14A	−0.1317	0.0427	0.3507	0.056*
H14B	−0.1795	−0.0159	0.2798	0.056*
C15	0.0067 (3)	−0.05105 (13)	0.36585 (16)	0.0713 (6)
H15A	0.0561	−0.0843	0.3296	0.086*
H15B	0.0915	−0.0274	0.4039	0.086*
C16	−0.0984 (3)	−0.09537 (15)	0.41519 (19)	0.0928 (9)
H16A	−0.0350	−0.1324	0.4479	0.139*
H16B	−0.1460	−0.0626	0.4517	0.139*
H16C	−0.1813	−0.1195	0.3776	0.139*
C17	0.0802 (2)	0.00133 (11)	0.19762 (12)	0.0537 (5)
H17A	0.1340	0.0321	0.1607	0.064*
H17B	0.1618	−0.0282	0.2314	0.064*
C18	−0.0324 (3)	−0.05121 (14)	0.14445 (16)	0.0775 (7)
H18A	−0.0841	−0.0838	0.1804	0.093*
H18B	−0.1151	−0.0226	0.1103	0.093*
C19	0.0577 (3)	−0.09743 (16)	0.08885 (17)	0.0900 (8)
H19A	−0.0155	−0.1304	0.0556	0.135*
H19B	0.1076	−0.0651	0.0527	0.135*
H19C	0.1385	−0.1262	0.1227	0.135*

Source of materials

Amidinothiourea, terephthalic acid and tetrapropylammonium hydroxide (25% aqueous solution), which are commercially available, were mixed in a molar ratio of 1:1:2. The mixture was dissolved in a minimum amount of ethanol and water, then stirred for about 35 min. Subsequently the clean solution was set aside to allow slow evaporation at room temperature. Colorless block crystals were obtained about seven days.

Comment

Amidinothiourea, a derivative of thiourea, can be utilized to synthesize the related organic crystals to explore supramolecular architecture [4, 5]. The amidinothiourea as an approximately planar molecule is composed of two triangular motifs, and it would form various hydrogen bonds by the NH_2 groups if the appropriate hydrogen bond acceptor were provided. Searching in the related literatures, it can be seen the crystal structures of tetrabutylammonium sulfanilate – 1-(diaminomethylene)thiourea [6] and terephthalate-1-(diaminomethylene)thiourea [7] are reported.

In the asymmetric unit of the title compound, there exist one independent 1-(diaminomethylene)thiourea, one half terephthalate positioned on the symmetry center, one tetrapropylammonium cation and two water molecules. Analyzing the crystal structure, the neutral amidinothiourea molecules firstly form a dimer by a pair of N–H···N hydrogen bonds, then the dimer links with the terephthalate by N–H···O hydrogen bonds to generate a zigzag chain extended along the *b* axis. Consecutively, the hydrogen-bonded chains interact with two independent water molecules by varied O–H···O contacts to regularly arrange to yield a 3-dimensional hydrogen-bonded framework with channels.

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