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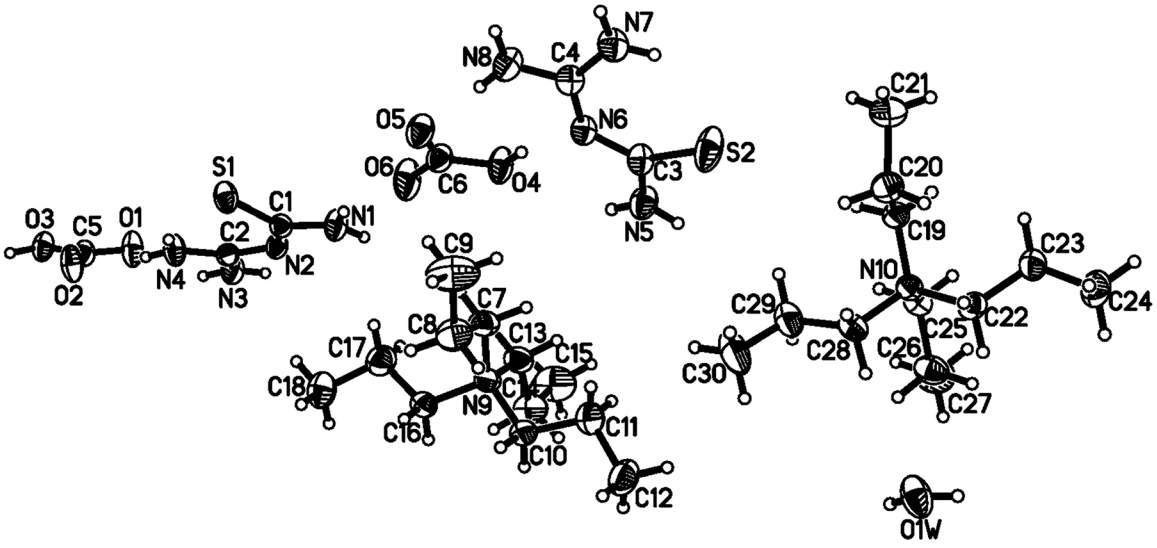
Crystal structure of tetrapropylammonium bicarbonate–1-(diaminomethylene)thiourea – water (2/2/1), $C_{30}H_{72}N_{10}O_7S_2$



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Abstract

$C_{30}H_{72}N_{10}O_7S_2$, triclinic, $P\bar{1}$ (no. 2), a = 8.75510(10) Å, b = 15.1170(2) Å, c = 16.8843(2) Å, α = 76.272 (1)°, β = 78.397 (1)°, γ = 88.277 (1)°, V = 2126.09(5) Å^3, Z = 2, $R_{gt}(F)$ = 0.0407, $wR_{ref}(F^2)$ = 0.1282, 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:	0.26 × 0.24 × 0.12 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.18 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	19,173, 7464, 0.018
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 5867
$N(param)_{refined}$:	454
Programs:	Bruker [1], SHELX [2,3]

1 Source of materials

Amidinothiourea and tetrapropylammonium hydroxide (25% aqueous solution), which are commercially available, were mixed in a molar ratio of 1:2. The mixture was dissolved in a minimum amount of ethanol and water, then the mixture was vigorously stirred for about 60 min. Subsequently the clean solution was set aside to allow it slow evaporation at room temperature. Colorless block crystals were obtained. It can be concluded that the bicarbonate anions are present in the crystal structure due to the absorption of CO_2 of the air under basic conditions.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
S1	0.54199 (6)	−0.19846 (3)	0.19276 (3)	0.06188 (16)
C1	0.54831 (19)	−0.11339 (11)	0.10362 (10)	0.0458 (4)
N1	0.47763 (19)	−0.03618 (10)	0.11265 (9)	0.0610 (4)
H1A	0.4746	0.0073	0.0697	0.073*
H1B	0.4350	−0.0298	0.1614	0.073*
O1	0.8247 (2)	−0.34784 (10)	−0.11645 (9)	0.0828 (5)
O1W	0.3863 (2)	0.88541 (13)	0.35088 (10)	0.0912 (5)
H1WA	0.341 (3)	0.9353 (15)	0.3311 (18)	0.137*
H1WB	0.432 (4)	0.8641 (19)	0.3107 (16)	0.137*
C2	0.69574 (19)	−0.18183 (11)	0.00015 (11)	0.0479 (4)
S2	−0.11863 (11)	0.41500 (4)	0.40623 (4)	0.1043 (3)
N2	0.60970 (17)	−0.11424 (9)	0.02440 (8)	0.0490 (3)
O2	0.91976 (19)	−0.40157 (10)	−0.00259 (10)	0.0818 (5)
C3	−0.0259 (2)	0.33659 (12)	0.35660 (11)	0.0569 (4)
N3	0.73681 (19)	−0.17486 (10)	−0.08130 (9)	0.0628 (4)
H3A	0.7935	−0.2159	−0.0998	0.075*
H3B	0.7067	−0.1293	−0.1156	0.075*
O3	0.93551 (17)	−0.48140 (8)	−0.09911 (8)	0.0660 (4)
H3	0.989 (3)	−0.5175 (16)	−0.0642 (14)	0.099*
C4	−0.1134 (2)	0.19808 (12)	0.45466 (10)	0.0509 (4)
N4	0.7456 (2)	−0.25345 (10)	0.04963 (10)	0.0654 (4)
H4A	0.8021	−0.2930	0.0287	0.078*
H4B	0.7216	−0.2604	0.1027	0.078*
O4	0.14137 (19)	0.15667 (9)	0.26780 (8)	0.0720 (4)
H4	0.089 (3)	0.1770 (17)	0.3088 (14)	0.108*
C5	0.8903 (2)	−0.40666 (12)	−0.07089 (12)	0.0585 (5)
N5	0.0617 (2)	0.36858 (11)	0.28120 (10)	0.0708 (5)
H5A	0.1121	0.3316	0.2543	0.085*
H5B	0.0678	0.4262	0.2595	0.085*
O5	0.15811 (18)	0.03631 (9)	0.37020 (8)	0.0728 (4)
C6	0.1959 (2)	0.07336 (12)	0.29528 (12)	0.0581 (5)
N6	−0.02533 (17)	0.24580 (9)	0.38305 (8)	0.0518 (3)
O6	0.2823 (2)	0.04111 (11)	0.24221 (10)	0.0946 (6)
C7	0.5107 (2)	0.24778 (14)	0.25670 (12)	0.0639 (5)
H7A	0.4265	0.2907	0.2604	0.077*
H7B	0.4696	0.1937	0.2463	0.077*
N7	−0.2301 (2)	0.23033 (12)	0.50136 (9)	0.0672 (4)
H7C	−0.2819	0.1951	0.5458	0.081*
H7D	−0.2545	0.2866	0.4875	0.081*
C8	0.5611 (3)	0.22199 (17)	0.33973 (13)	0.0805 (6)
H8A	0.6437	0.1779	0.3374	0.097*
H8B	0.6020	0.2756	0.3511	0.097*
N8	−0.0785 (2)	0.11143 (10)	0.47743 (10)	0.0677 (4)
H8C	−0.1316	0.0772	0.5220	0.081*
H8D	−0.0026	0.0892	0.4477	0.081*
C9	0.4276 (4)	0.1824 (3)	0.40841 (16)	0.1242 (12)
H9A	0.4625	0.1667	0.4603	0.186*
H9B	0.3464	0.2264	0.4114	0.186*
H9C	0.3880	0.1288	0.3977	0.186*
N9	0.63831 (16)	0.28956 (10)	0.18284 (9)	0.0525 (4)
C10	0.7093 (2)	0.37362 (12)	0.19616 (12)	0.0576 (4)
H10A	0.7598	0.3553	0.2435	0.069*
H10B	0.7896	0.3981	0.1478	0.069*
N10	−0.04455 (15)	0.75384 (9)	0.31284 (8)	0.0474 (3)
C11	0.5979 (3)	0.44880 (15)	0.21093 (16)	0.0788 (6)

Table 2: (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
H11A	0.5399	0.4647	0.1664	0.095*
H11B	0.5240	0.4278	0.2629	0.095*
C12	0.6856 (3)	0.53132 (15)	0.21442 (18)	0.0917 (7)
H12A	0.6134	0.5786	0.2238	0.138*
H12B	0.7418	0.5157	0.2590	0.138*
H12C	0.7577	0.5525	0.1627	0.138*
C13	0.5642 (2)	0.31320 (14)	0.10672 (12)	0.0652 (5)
H13A	0.4740	0.3504	0.1183	0.078*
H13B	0.5271	0.2573	0.0977	0.078*
C14	0.6702 (3)	0.36336 (18)	0.02699 (13)	0.0820 (6)
H14A	0.7005	0.4221	0.0334	0.098*
H14B	0.7640	0.3287	0.0164	0.098*
C15	0.5905 (4)	0.3772 (2)	−0.04558 (17)	0.1165 (10)
H15A	0.6600	0.4090	−0.0951	0.175*
H15B	0.5619	0.3191	−0.0526	0.175*
H15C	0.4985	0.4124	−0.0356	0.175*
C16	0.7699 (2)	0.22289 (12)	0.17130 (12)	0.0573 (4)
H16A	0.8494	0.2522	0.1242	0.069*
H16B	0.8165	0.2107	0.2202	0.069*
C17	0.7257 (3)	0.13335 (14)	0.15734 (16)	0.0774 (6)
H17A	0.6777	0.1443	0.1090	0.093*
H17B	0.6498	0.1017	0.2051	0.093*
C18	0.8659 (3)	0.07447 (16)	0.14427 (16)	0.0870 (7)
H18A	0.8343	0.0179	0.1355	0.131*
H18B	0.9404	0.1053	0.0965	0.131*
H18C	0.9125	0.0627	0.1925	0.131*
C19	−0.1834 (2)	0.68854 (12)	0.34653 (11)	0.0531 (4)
H19A	−0.2740	0.7189	0.3285	0.064*
H19B	−0.1635	0.6367	0.3216	0.064*
C20	−0.2228 (3)	0.65383 (14)	0.43936 (13)	0.0705 (5)
H20A	−0.2242	0.7043	0.4658	0.085*
H20B	−0.1436	0.6119	0.4573	0.085*
C21	−0.3801 (3)	0.60597 (18)	0.46553 (18)	0.0950 (8)
H21A	−0.4039	0.5841	0.5249	0.142*
H21B	−0.4585	0.6478	0.4484	0.142*
H21C	−0.3781	0.5555	0.4399	0.142*
C22	−0.0699 (2)	0.83633 (12)	0.35043 (11)	0.0541 (4)
H22A	−0.0632	0.8172	0.4087	0.065*
H22B	0.0143	0.8800	0.3230	0.065*
C23	−0.2225 (2)	0.88361 (13)	0.34452 (14)	0.0651 (5)
H23A	−0.3081	0.8416	0.3738	0.078*
H23B	−0.2315	0.9027	0.2866	0.078*
C24	−0.2327 (3)	0.96604 (15)	0.38187 (16)	0.0823 (6)
H24A	−0.3305	0.9953	0.3776	0.123*
H24B	−0.2252	0.9470	0.4394	0.123*
H24C	−0.1488	1.0080	0.3523	0.123*
C25	−0.0293 (2)	0.78225 (13)	0.21897 (10)	0.0557 (4)
H25A	−0.0163	0.7279	0.1974	0.067*
H25B	−0.1262	0.8098	0.2070	0.067*
C26	0.1033 (2)	0.84809 (16)	0.17260 (12)	0.0727 (6)
H26A	0.2012	0.8224	0.1848	0.087*
H26B	0.0887	0.9046	0.1906	0.087*
C27	0.1087 (3)	0.8668 (2)	0.07964 (15)	0.1033 (9)
H27A	0.1931	0.9086	0.0505	0.155*
H27B	0.1243	0.8108	0.0619	0.155*
H27C	0.0121	0.8928	0.0677	0.155*

Table 2: (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C28	0.1022 (2)	0.70833 (13)	0.33586 (12)	0.0581 (4)
H28A	0.1860	0.7536	0.3184	0.070*
H28B	0.0852	0.6883	0.3960	0.070*
C29	0.1557 (3)	0.62808 (16)	0.29953 (15)	0.0781 (6)
H29A	0.1926	0.6487	0.2399	0.094*
H29B	0.0690	0.5858	0.3093	0.094*
C30	0.2850 (3)	0.5810 (2)	0.3393 (2)	0.1150 (10)
H30A	0.3187	0.5299	0.3160	0.172*
H30B	0.3709	0.6228	0.3290	0.172*
H30C	0.2476	0.5601	0.3982	0.172*

2 Experimental details

Crystal data, data collection and structure refinement details are summarized in Table 1.

3 Comment

Amidinothiourea is a derivative of thiourea, which can be utilized in the crystal engineering to explore supramolecular architecture [4,5]. It can be seen that amidinothiourea is a good hydrogen bond acceptor and donor due to it functional groups. Till now, the similar organic crystal structure of tetrabutylammonium sulfanilate – 1-(diaminomethylene)thiourea [6] has been reported.

In the asymmetric unit of the title structure, there are two independent 1-(diaminomethylene)thiourea, two bicarbonate anions, two tetrapropylammonium cations and one water molecule. Two amidinothiourea molecules firstly interact with each other to form a dimer by a pair of N–H···N hydrogen bonds. At the same time, two bicarbonate anions also link with each other to generate the related dimer by O–H···O contacts. Then these two types of dimers utilize N–H···O hydrogen bonding to extend to form the hydrogen-bonded chains, and these neighboring chains are connected with each other by the linkage of water molecules by different N–H···O and O–H···S hydrogen bonds to finally generate a 2-dimensional hydrogen-bonded layer. Between the adjacent layers, tetrapropylammonium cations are contained to construct the final stable crystal structure.

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