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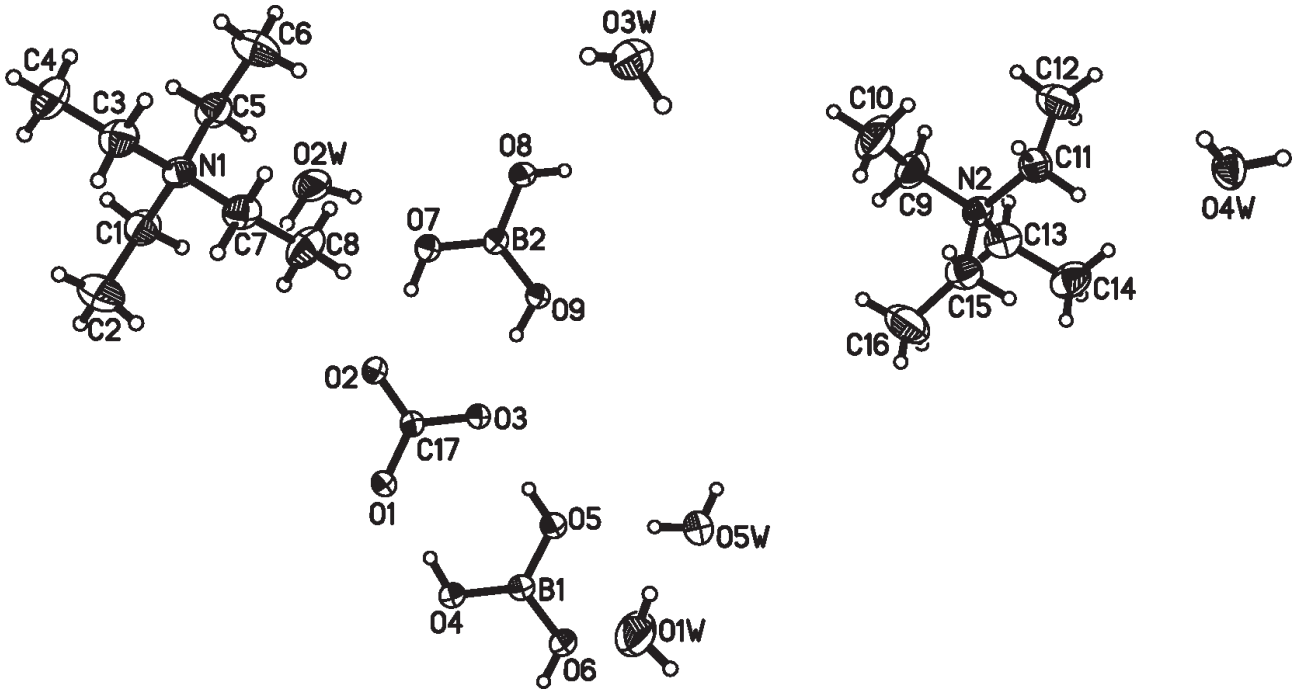
Bis(tetraethylammonium) carbonate – boric acid – water (1/2/5), $C_{17}H_{56}B_2N_2O_{14}$



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Abstract

$C_{17}H_{56}B_2N_2O_{14}$, monoclinic, $P2_1/c$ (no. 14), $a = 14.3049(6)$ Å, $b = 14.5382(7)$ Å, $c = 15.0065(7)$ Å, $\beta = 103.185(3)°$, $V = 3038.6(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0556$, $wR_{ref}(F^2) = 0.1836$, $T = 296(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.51 × 0.42 × 0.38 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.0 cm^{-1}
Diffractometer, scan mode:	Bruker CCD, φ and ω
$2\theta_{max}$, completeness:	55°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	16461, 6922, 0.032
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4322
$N(param)_{refined}$:	367
Programs:	SADABS [1], SHELX [2], Bruker programs [3]

Source of materials

The title compound was prepared by accident. Firstly, an aromatic acid (A. R.), boric acid (A. R.) and tetramethylammonium (25% aqueous solution, C. R.) were dissolved in small amount of H_2O/EtOH (v/v, 2:1) with the molar ratio of 1:2:2 to evaporate under room temperature to prepare the cocrystal of the related acid. About after 14 days, colorless block crystals appeared in the clean solution. Determined by single crystal X-ray diffraction, the crystal was proved to be the

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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}
C1	0.79334(16)	0.20899(16)	0.02193(16)	0.0685(6)
H1A	0.8403	0.1642	0.0122	0.082*
H1B	0.7453	0.1763	0.0460	0.082*
N1	0.84290(10)	0.27691(10)	0.09287(11)	0.0479(4)
O1	0.31306(9)	0.00102(10)	0.07003(8)	0.0559(4)
O1W	0.10356(16)	0.4933(2)	0.1582(2)	0.1412(12)
H1WA	0.148(2)	0.518(3)	0.198(3)	0.212*
H1WB	0.0492(16)	0.514(3)	0.163(4)	0.212*
B1	0.09669(15)	0.00990(15)	0.12558(14)	0.0449(5)
N2	0.32973(11)	0.22341(10)	0.87185(11)	0.0503(4)
O2	0.46194(9)	0.01154(11)	0.15217(8)	0.0574(4)
O2W	0.73286(11)	0.06453(16)	0.23149(12)	0.0856(6)
H2WA	0.7013(19)	0.067(3)	0.1760(8)	0.128*
H2WB	0.6936(18)	0.055(3)	0.2656(17)	0.128*
C2	0.7457(2)	0.2496(3)	−0.0685(2)	0.1154(12)
H2A	0.7161	0.2015	−0.1092	0.173*
H2B	0.6975	0.2928	−0.0603	0.173*
H2C	0.7927	0.2806	−0.0942	0.173*
B2	0.54853(14)	0.00945(15)	0.38186(13)	0.0425(4)
C3	0.91710(16)	0.33208(17)	0.05817(17)	0.0695(6)
H3A	0.8842	0.3689	0.0066	0.083*
H3B	0.9481	0.3741	0.1061	0.083*
O3	0.34043(8)	0.00924(10)	0.22129(8)	0.0507(3)
O3W	0.80495(12)	0.93980(14)	0.47664(12)	0.0779(5)
H3WA	0.8311(19)	0.966(2)	0.4377(18)	0.117*
H3WB	0.7484(11)	0.962(2)	0.469(2)	0.117*
C4	0.99350(18)	0.2764(2)	0.0289(2)	0.0937(9)
H4A	1.0370	0.3169	0.0081	0.141*
H4B	1.0281	0.2410	0.0798	0.141*
H4C	0.9641	0.2357	−0.0199	0.141*
O4	0.12762(9)	−0.00504(10)	0.04862(9)	0.0550(4)
H4	0.1888(7)	−0.0065(17)	0.0570(18)	0.083*
O4W	0.39566(11)	0.92532(12)	0.93726(10)	0.0645(4)
H4WA	0.3843(18)	0.9501(18)	0.9852(13)	0.097*
H4WB	0.4445(13)	0.9498(18)	0.9252(18)	0.097*
C5	0.89022(16)	0.22149(15)	0.17669(16)	0.0640(6)
H5A	0.8413	0.1851	0.1956	0.077*
H5B	0.9356	0.1791	0.1596	0.077*
O5	0.15703(9)	0.01917(13)	0.20915(9)	0.0692(5)
H5	0.2182(8)	0.015(2)	0.214(2)	0.104*
O5W	0.07435(12)	0.07431(14)	0.35216(11)	0.0785(5)
H5WA	0.099(2)	0.054(2)	0.3097(14)	0.118*
H5WB	0.1088(19)	0.063(2)	0.4046(9)	0.118*
C6	0.9422(2)	0.2774(2)	0.25723(19)	0.1044(10)
H6A	0.9698	0.2370	0.3069	0.157*
H6B	0.9922	0.3124	0.2401	0.157*
H6C	0.8978	0.3184	0.2761	0.157*
O6	0.00081(9)	0.01707(13)	0.12269(10)	0.0670(4)
H6	−0.0375(18)	0.012(2)	0.0706(11)	0.100*
C7	0.77162(15)	0.34436(15)	0.11630(16)	0.0617(5)
H7A	0.8063	0.3875	0.1612	0.074*
H7B	0.7432	0.3791	0.0616	0.074*
O7	0.58139(10)	0.01172(13)	0.30520(9)	0.0702(5)
H7	0.5399(17)	0.011(2)	0.2546(12)	0.105*
O8	0.61543(9)	0.01024(11)	0.46357(9)	0.0573(4)
H8	0.5942(18)	0.0028(17)	0.5121(12)	0.086*
C8	0.69235(18)	0.30268(19)	0.1532(2)	0.0843(8)
H8A	0.6510	0.3505	0.1660	0.126*
H8B	0.6560	0.2612	0.1087	0.126*
H8C	0.7193	0.2697	0.2085	0.126*
C9	0.41160(18)	0.17226(18)	0.8477(2)	0.0797(7)
H9A	0.3859	0.1292	0.7989	0.096*
H9B	0.4440	0.1368	0.9006	0.096*
O9	0.45376(8)	0.00604(10)	0.38185(8)	0.0482(3)
H9	0.4178(15)	0.0035(16)	0.3281(9)	0.072*
C10	0.4848(2)	0.2331(3)	0.8175(3)	0.1158(12)
H10A	0.5347	0.1955	0.8033	0.174*
H10B	0.4540	0.2673	0.7642	0.174*
H10C	0.5122	0.2750	0.8659	0.174*
C11	0.36487(17)	0.28704(15)	0.95156(15)	0.0650(6)
H11A	0.3099	0.3186	0.9650	0.078*
H11B	0.4058	0.3332	0.9334	0.078*
C12	0.4199(2)	0.2414(2)	1.03840(19)	0.0994(9)
H12A	0.4393	0.2870	1.0852	0.149*
H12B	0.3796	0.1968	1.0583	0.149*
H12C	0.4757	0.2114	1.0267	0.149*
C13	0.26188(17)	0.15157(16)	0.89619(18)	0.0702(6)
H13A	0.2380	0.1135	0.8428	0.084*
H13B	0.2983	0.1122	0.9438	0.084*
C14	0.17776(18)	0.1893(2)	0.9281(2)	0.0873(8)
H14A	0.1393	0.1395	0.9418	0.131*
H14B	0.2002	0.2257	0.9822	0.131*
H14C	0.1398	0.2270	0.8809	0.131*
C15	0.27718(18)	0.28342(17)	0.79418(16)	0.0693(6)
H15A	0.2250	0.3143	0.8134	0.083*
H15B	0.3210	0.3304	0.7822	0.083*
C16	0.2367(3)	0.2323(3)	0.7063(2)	0.1162(12)
H16A	0.2046	0.2749	0.6605	0.174*
H16B	0.2880	0.2028	0.6856	0.174*
H16C	0.1919	0.1867	0.7168	0.174*
C17	0.37179(12)	0.00750(12)	0.14750(11)	0.0398(4)

title compound. At the same time, it can be concluded that carbonate should be derived from the vigorous stirring during the experiment.

Experimental details

All hydrogen atoms were identified in difference Fourier syntheses. The H atoms bonded to C were localized in the ideal positions and all H atoms bonded to O atoms were refined with the O—H distance restrained to 0.86 Å. The U_{iso} values of the hydrogen atoms bonded to O were set to $1.5U_{eq}$(O).

Comment

Boric acid is a triangular planar molecule with hydrogen bond donors and acceptors due to its three hydroxyl groups.

Meanwhile, carbonate is also a perfect triangular planar ion that can act as hydrogen bond donor. Searching in the related literature, it can be found that boric acid can interact with tetraalkylammonium, such as tetraethylammonium, to form the stable crystal structure [4], and tetraethylammonium bicarbonate is also known [5]. Interestingly, in the actual experiment, the crystal structure composed of tetraethylammonium carbonate and boric acid has been prepared, which has two neutral boric acid molecules, one carbonate anion, five water molecules and two tetraethylammonium cations. Herein we reported the new structure to further explore the packing rules of triangular planar molecules and tetraalkylammonium.

In the asymmetric unit of the title compound, there are two boric acid molecules, one carbonate dianion, five water molecules and two counterions of tetraethylammonium cations. It can be clearly found that the neutral acid molecules can connect with the carbonate ion to form the acid-anion unit by several O—H···O hydrogen bonds, then the unit can further link five crystallographically independent water molecules to produce 2D layers in tha *ac* plane. Each carbonate anion accepts six hydrogen bonds. One crystallographic boric acid molecule (B1) is involved in five, the other one (B2) in six hydrogen bonds. Each of the five water molecules is involved in three hydrogen bonds. The interlayer distance between the hydrogen bonded layers is about $b/2 = 7.27$ Å. To form the final stable structure, two independent tetraethylammonium ions are contained between the layers to yield the neutral structure. Bond lengths and angles of the ions and molecules are in the expected ranges [6, 7].

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