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Dimethylammonium tetrahydropentaborate

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Key indicators

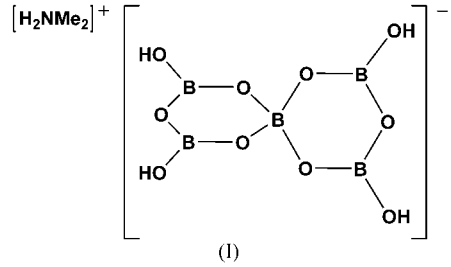
Single-crystal X-ray study
T = 100 K
Mean σ(N–C) = 0.002 Å
R factor = 0.034
wR factor = 0.088
Data-to-parameter ratio = 12.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound [systematic name: dimethylammonium 1,1′-spiro-bis(3,5,-dihydroxy-2,4,6-trioxa-1,3,5-triboracyclohexane)borate], $C_2H_8N^+\cdot B_5H_4O_{10}^-$, contains the $[B_5O_6(OH)_4]^-$ tetrahydropentaborate anion, which possesses typical geometrical parameters, accompanied by dimethylammonium cations. The packing of these species is influenced by cation-to-anion N—H···O and anion-to-anion O—H···O hydrogen bonds.

Comment

The tetrahydropentaborate anion, $[B_5O_6(OH)_4]^-$, has been crystallized with a variety of ammonium cations: $[NH_4]^+$ (Loboda *et al.*, 1993); $[H_2NC_5H_{10}]^+$, $[NMe_4]^+$ and $[NEt_4]^+$ (Wiebcke *et al.*, 1993); $[HNEt_3]^+$ (Loboda *et al.*, 1994); $[HNBu^n_3]^+$ (Turdybekov *et al.*, 1992) and $[NPr^n_4]^+$ (Freyhardt *et al.*, 1994). In this paper, we report the crystal structure of a dimethylammonium salt of this anion, $[H_2NMe_2]^+$-$[B_5O_6(OH)_4]^-$, (I) (Fig. 1).



The anion consists of a central BO_4 tetrahedron fused to four trigonal planar $BO_2(OH)$ units and shows normal geometrical parameters (Table 1). Hydrogen bonding (Table 2) between adjacent $[B_5O_6(OH)_4]^-$ units results in $R_2^2(8)$ (Etter, 1990) dimers (Fig. 2). This anion-to-anion hydrogen-bonding framework is supplemented by the formation of two hydrogen bonds from each dimethylammonium cation to two adjacent $[B_5O_6(OH)_4]^-$ anions.

Experimental

A large excess of $B(OH)_3$ (55.6 mmol, 3.44 g, dried by the Dean–Stark method) was added to a stirred solution of $B_2(NMe_2)_4$ (1 ml, 5.56 mmol) in tetrahydrofuran (25 ml), and the solution left to stir overnight. After removal of the solvent *in vacuo*, a white solid remained, which was shown to contain some $B_2(OH)_4$ and a majority of $B(OH)_3$ by $^{11}B\{^1H\}$ NMR spectroscopy. Dissolution of this solid in degassed water followed by slow evaporation over several days afforded a small crop of thin needle-like crystals approximately 5 mm long, a fragment of one of which was shown to be $[H_2NMe_2][B_5O_6(OH)_4]$.

Crystal data

$C_2H_8N^+\cdot B_5H_4O_{10}^-$
$M_r = 264.18$
Monoclinic, $C2/c$
$a = 13.3664$ (3) Å
$b = 11.4709$ (3) Å
$c = 17.1147$ (4) Å
$\beta = 112.160$ (1)°
$V = 2430.27$ (10) Å^3
$Z = 8$
$D_x = 1.444$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 4393 reflections
$\theta = 5.3$–70.2°
$\mu = 1.19$ mm^{-1}
$T = 100$ (2) K
Block, colourless
0.18 × 0.10 × 0.10 mm

Data collection

Bruker Proteum CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.792$, $T_{max} = 0.886$
9127 measured reflections
2225 independent reflections
1847 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 70.2°$
$h = -15 \rightarrow 16$
$k = -13 \rightarrow 13$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.088$
$S = 0.99$
2225 reflections
177 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0624P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

B1—O1	1.4623 (16)	B3—O4	1.3612 (18)
B1—O6	1.4679 (16)	B3—O3	1.3849 (17)
B1—O10	1.4680 (16)	B4—O6	1.3530 (18)
B1—O5	1.4726 (18)	B4—O7	1.3550 (17)
B2—O2	1.3506 (18)	B4—O8	1.3843 (17)
B2—O1	1.3628 (18)	B5—O9	1.3522 (17)
B2—O3	1.3860 (18)	B5—O10	1.3643 (18)
B3—O5	1.3571 (18)	B5—O8	1.3813 (17)
B3—O3—B2	118.78 (11)	B5—O8—B4	119.22 (11)
B3—O5—B1	123.11 (10)	B5—O10—B1	123.93 (10)
B4—O6—B1	123.62 (10)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.92	1.86	2.7707 (15)	170
N1—H1B···O4^{ii}	0.92	1.96	2.8765 (15)	173
O2—H2A···O7^{iii}	0.848 (17)	1.852 (17)	2.6972 (15)	175.1 (16)
O4—H4A···O5^{iv}	0.814 (16)	1.926 (16)	2.7340 (12)	171.6 (17)
O7—H7A···O10^{v}	0.841 (19)	1.862 (18)	2.7015 (13)	175.8 (18)
O9—H9A···O6^{vi}	0.822 (18)	1.942 (18)	2.7526 (13)	168.8 (19)

Symmetry codes: (i) $1-x, y-1, \frac{1}{2}-z$; (ii) $x-1, 1-y, z-\frac{1}{2}$; (iii) $\frac{1}{2}+x, \frac{3}{2}-y, \frac{1}{2}+z$; (iv) $2-x, y, \frac{1}{2}-z$; (v) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (vi) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.

The methyl H atoms of the cation were located using a rotating group refinement, with C—H bond lengths constrained to 0.96 Å and displacement parameters equal to 1.5 times U_{eq} of their parent C atom. The remaining H atoms of the cation were constrained to ideal geometries (Table 2) and refined with displacement parameters equal to 1.2 times U_{eq}(N). All hydroxyl H atoms were located in Fourier difference maps, assigned displacement parameters equal to 1.5U_{eq}(O) and refined with a distance restraint of 0.84 (3) Å on the O—H bonds.

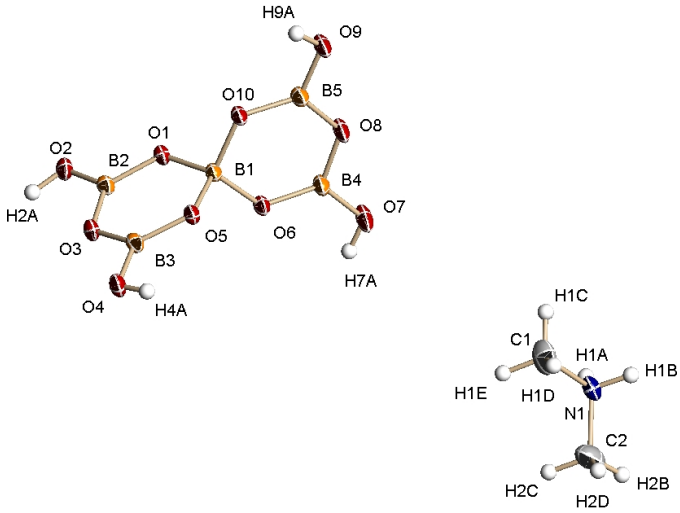


Figure 1
The molecular structure of (I), showing the atom labelling scheme (50% displacement ellipsoids).

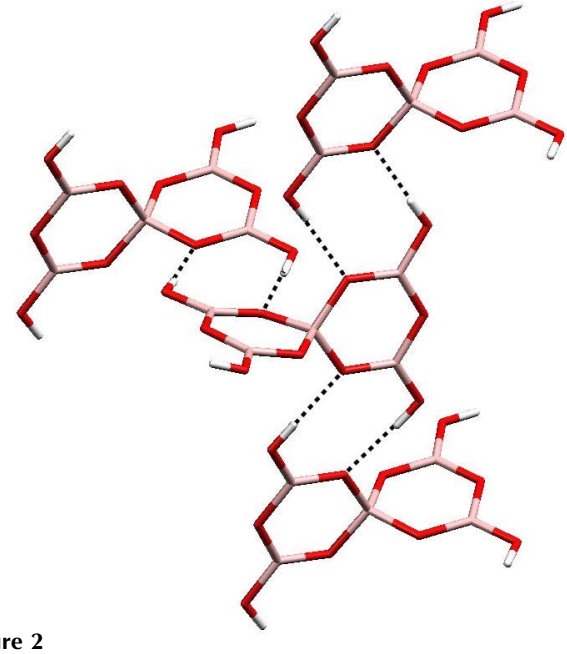


Figure 2
Detail of (I) in stick representation (key: B pink, O red and H white) illustrating the dimeric $R_2^2(8)$ hydrogen-bonding motif linking adjacent $[B_5O_6(OH)_4]^-$ anions.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT* and *SHELXTL* (Bruker, 2002); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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