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Butylammonium tetrahydroxypentaborate

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Key indicators

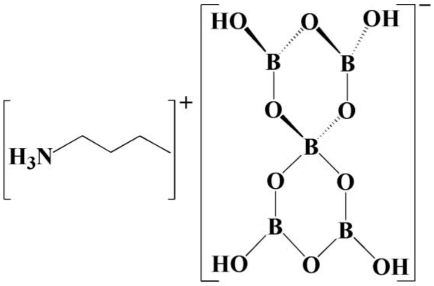
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.010 Å
R factor = 0.054
wR factor = 0.145
Data-to-parameter ratio = 14.0

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

The title compound, butylammonium 1,3,5,7,9,11-hexaoxa-2,4,6,8,10-pentaboraspiro[5.5]undecane-2,4,8,10-tetraol(1−), $C_4H_{12}N^+\cdot B_5O_6(OH)_4^-$, was synthesized under mild solvothermal conditions in the presence of 1-butylamine acting as a template. The structure consists of pentaborate $[B_5O_6(OH)_4]^-$ anions connected through O—H···O hydrogen bonds into a three-dimensional framework, with channels running along one direction. The $[C_4H_{12}N]^+$ cations lie in the channels, interacting with the framework through N—H···O hydrogen bonds.

Comment

Research into borate materials has expanded rapidly in the past decades, mainly because of their rich structural chemistry and potential applications in mineralogy and industry (Burns *et al.*, 1995; Chen *et al.*, 1995; Grice *et al.*, 1999; Touboul *et al.*, 2003). In borates, B atoms can exhibit threefold coordination (BO_3, triangular) or fourfold coordination (BO_4, tetrahedral), and these BO_3 and BO_4 groups can polymerize to form more complex polynuclear anions, including isolated rings or cages, infinite chains, sheets and frameworks. Most borates synthesized and studied to date have been prepared under the templating effect of inorganic cations, such as alkali-metal, alkaline-earth, rare-earth or transition-metal cations. In contrast, very little structural information is available regarding possible analogues containing organic templating agents. To our knowledge, only a few examples with polyanions $[B_4O_5(OH)_4]$ (Batsanov *et al.*, 1982), $[B_5O_6(OH)_4]$ (Wang *et al.*, 2004), $[B_7O_9(OH)_5]$ (Liu & Li, 2006; Liu, Li & Zhang, 2006), $[B_9O_{12}(OH)_6]$ (Schubert *et al.*, 2000) and $[B_{14}O_{20}(OH)_6]$ (Liu, Li & Zhang, 2006) have been reported to date. Continuing our work on organically templated borates, we have obtained a new pentaborate, (I), using 1-butylamine as the structure-directing agent.



(I)

The structure of (I) (Fig. 1) consists of $[B_5O_6(OH)_4]^-$ pentaborate anions and $[C_4H_{12}N]^+$ cations. The $[B_5O_6(OH)_4]^-$

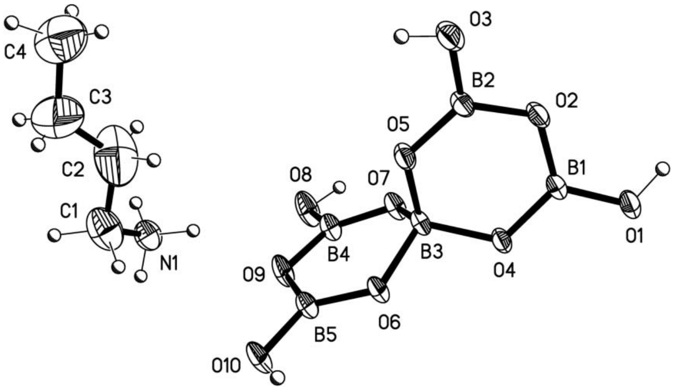


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level for non-H atoms.

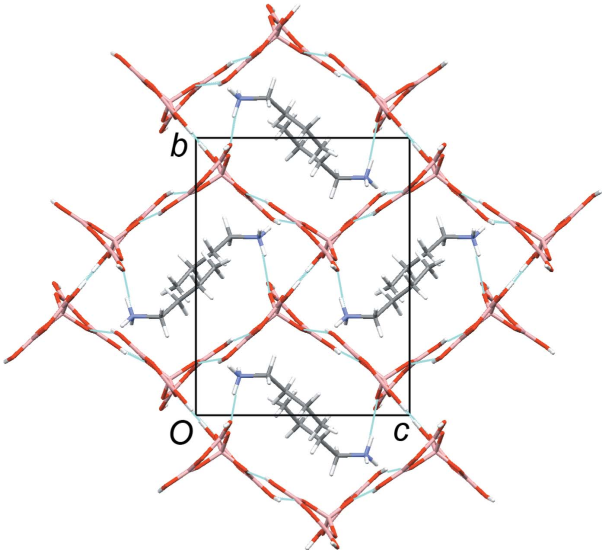


Figure 2
Projection along *a*, showing $[B_5O_6(OH)_4]^-$ anions linked into a three-dimensional framework, with $[C_4H_{12}N]^+$ cations occupying channels. Pale-blue lines denote hydrogen bonds.

polyanion is made up of two B_3O_3 rings, each containing two BO_3 triangles and one BO_4 tetrahedron. The B—O bond distances are in the range 1.347 (3)–1.387 (3) Å for the BO_3 triangles and 1.456 (3)–1.473 (2) Å for the BO_4 tetrahedron, in good agreement with those reported previously for other borate compounds. The O—B—O bond angles of the BO_3 triangles lie in the range 114.97 (19)–123.6 (2)°, while those of the BO_4 tetrahedron range from 107.2 (2) to 110.8 (2)°. The anionic $[B_5O_6(OH)_4]^-$ clusters are connected through intermolecular O—H···O hydrogen bonds (Table 1), forming a three-dimensional framework with channels running along the *a* axis. The organic cations reside in these channels, interacting with the framework through N—H···O hydrogen bonds (Fig. 2 and Table 1).

Experimental

A mixture of H_3BO_3 (1.00 g), 1-butylamine (0.40 ml) and H_2O (0.80 ml) (molar ratio 1:0.25:2.7) was sealed in a Teflon-lined steel autoclave, heated at 453 K for 2 d, then cooled to room temperature. Colourless crystals were recovered by filtration, washed with distilled water and dried in air.

Crystal data

$C_4H_{12}N^+\cdot B_5H_4O_{10}^-$
$M_r = 292.23$
Monoclinic, $P2_1/c$
$a = 9.4025\ (3)$ Å
$b = 13.6395\ (5)$ Å
$c = 10.5395\ (4)$ Å
$\beta = 91.956\ (2)^\circ$
$V = 1350.85\ (8)$ Å^3

$Z = 4$
$D_x = 1.437$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.13$ mm^{-1}
$T = 293\ (2)$ K
Prism, colourless
0.25 × 0.22 × 0.15 mm

Data collection

Siemens SMART CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.969$, $T_{\max} = 0.981$

9341 measured reflections
2750 independent reflections
2274 reflections with $I > \breve{2}I)$
$R_{\text{int}} = 0.019$
$\theta_{\max} = 26.5^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.145$
$S = 1.07$
2750 reflections
196 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0651P)^2 + 0.8653P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.56$ e Å^{-3}
$\Delta\rho_{\min} = -0.50$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*D*···O8	0.89	2.26	3.127 (3)	165
N1—H1*D*···O9	0.89	2.55	3.262 (3)	137
N1—H1*C*···O2$^{\text{i}}$	0.89	2.39	3.123 (3)	140
N1—H1*C*···O3$^{\text{i}}$	0.89	2.33	3.117 (3)	148
N1—H1*E*···O4$^{\text{ii}}$	0.89	2.02	2.886 (3)	163
O1—H1···O10$^{\text{iii}}$	0.84 (2)	1.90 (2)	2.741 (2)	175 (3)
O3—H3···O7$^{\text{iv}}$	0.84 (2)	1.99 (2)	2.814 (2)	170 (4)
O8—H8···O5$^{\text{v}}$	0.84 (2)	1.97 (3)	2.778 (2)	162 (4)
O10—H10···O6$^{\text{vi}}$	0.84 (2)	1.83 (2)	2.662 (2)	176 (3)

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

H atoms of the hydroxy groups were located in difference Fourier maps and their positions were refined with all O—H distances restrained to a single refined value with a standard uncertainty of 0.01, and with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{iso}}(\text{O})$. H atoms of the butylammonium cation were positioned geometrically, with C—H = 0.97 (CH_2) or 0.96 Å (CH_3), and with N—H = 0.89 Å, and allowed to ride during refinement with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{iso}}(\text{C})$ for the CH_2 groups or $1.5U_{\text{iso}}(\text{C,N})$ for the CH_3 and NH_3 groups. The NH_3 group was allowed to rotate about its local threefold axis. The 1,2- and 1,3-distances in the butylammonium cation were restrained, and the relatively large displacement ellipsoids suggest some disorder, which was not explicitly modelled.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics:

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SHELXTL (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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