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Poly[sodium(I)-μ_3-bis(ethylenedioxy)borato]

Graeme J. Gainsford* and Tim Kemmitt

Industrial Research Limited, PO Box 31-310, Lower Hutt, New Zealand

Correspondence e-mail: g.gainsford@irl.cri.nz

Key indicators

Single-crystal X-ray study
T = 113 K
Mean σ(C–C) = 0.004 Å
R factor = 0.049
wR factor = 0.140
Data-to-parameter ratio = 13.6

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

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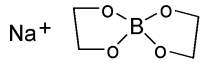
In the title compound, $[Na(C_4H_8BO_4)]_n$, the sodium cations are bound to six (ethylenediolato)borate O atoms, forming polymeric sheets normal to the b axis. The structure was solved using data from a multiply twinned crystal.

Comment

This study is part of a programme aimed at investigating boron diolates and alkoxides. Anionic complexes are known to form between the tetrahedral monoborate ion and many polyols in aqueous solution (Weser, 1967) but structural data are sparse. The basic polymeric fragment of the title compound, $[Na^+(C_4H_8BO_4)^-]_n$, (I), is illustrated in Fig. 1.



(I)

There are several compounds with five-membered dioxygen-boron-containing rings in the Cambridge Structural Database (CSD, Version 5.25; Allen, 2002), but most involve acid moieties [*e.g.* caesium bis(citrato)borate, refcode HAGTIS01; Marsh, 1997]. There are six reported sodium salts with anions based on BO_4^- tetrahedra (Allen, 2002), and they follow the normal (non-polymeric) pattern for monomeric

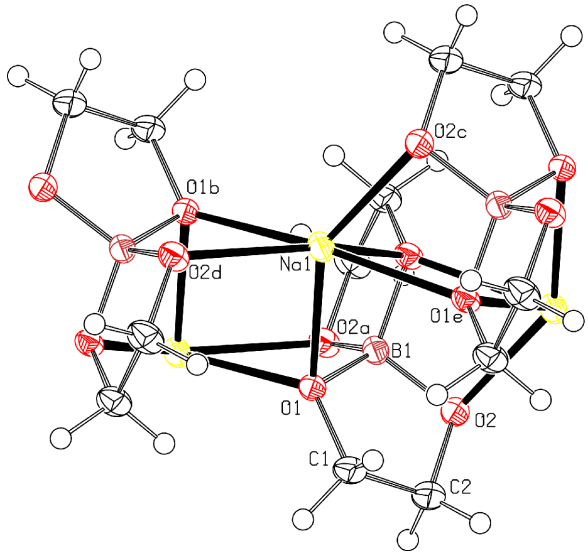


Figure 1
Part of the polymeric structure of (I), with ellipsoids at the 50% probability level (Spek, 2003). Asymmetric unit atoms plus O atoms bound to Na1 are labelled. [Symmetry codes (*a*) $2-x, y, \frac{1}{2}-z$; (*b*) $2-x, 2-y, -z$; (*c*) $2-x, 2-y, 1-z$; (*d*) $x, 2-y, z-\frac{1}{2}$; (*e*) $x, 2-y, \frac{1}{2}+z$.]

salts. One exception is sodium bis(methyl β-D-ribofuranosid-2,3-ato)borate dihydrate (refcode GUNSOX; Benner & Klufers, 2000), where the presence of two additional O-atom donors (water molecules) leads to the formation of dimeric units. In the title compound, the Na—O distances range from 2.371 (2) to 2.451 (3) Å. The molecules pack in two-dimensional sheets normal to the *b* axis (Fig. 2), with a closest intermolecular contact C2—H2*B*···H2*B*^{i} of 2.33 Å [symmetry code: (i) $\frac{3}{2}-x, \frac{3}{2}-y, -z$]; this is similar to only one other related compound (HAGTIS01), which has polymeric sheets normal to the *a* axis.

Experimental

The title compound was prepared by the addition of excess ethylenediol to a solution of sodium metaborate (13.8 g) in water (10 ml). Water and some ethylenediol were removed on a rotary evaporator equipped with a high-temperature oil bath, leaving a concentrated solution of the product in ethylene glycol. Acetonitrile was floated on top of the ethylenediol layer, allowing slow diffusion to promote the growth of crystals on the walls of the flask within the acetonitrile layer.

Crystal data

$[Na(C_4H_8BO_4)]$	D_x = 1.576 Mg m^{-3}
M_r = 153.90	Mo $K\alpha$ radiation
Monoclinic, *C*2/*c*	Cell parameters from 677 reflections
a = 7.990 (7) Å	θ = 2.8–26.4°
b = 14.675 (13) Å	μ = 0.19 mm^{-1}
c = 5.707 (5) Å	*T* = 113 (2) K
β = 104.271 (13)°	Needle, white
V = 648.5 (9) Å^3	0.52 × 0.11 × 0.08 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	641 independent reflections
φ and ω scans	478 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (Blessing, 1995)	R_{int} = 0.044
T_{min} = 0.386, T_{max} = 0.990	θ_{max} = 26.4°
1418 measured reflections	$h = -7 \rightarrow 9$
	$k = -18 \rightarrow 14$
	$l = -7 \rightarrow 7$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.049	$w = 1/[\sigma^2(F_o^2) + (0.0853P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.141	$(\Delta/\sigma)_{max} < 0.001$
S = 1.04	$\Delta\rho_{max}$ = 0.31 e Å^{-3}
641 reflections	$\Delta\rho_{min}$ = −0.28 e Å^{-3}
47 parameters	

Table 1
Selected geometric parameters (Å, °).

Na1—O2^{ii}	2.370 (2)	Na1—O1	2.450 (3)
Na1—O1^{iii}	2.415 (2)	O1—B1	1.483 (3)
O2^{ii}—Na1—O1^{iii}	59.67 (7)	O2^{ii}—Na1—O1	145.60 (6)
O1^{iii}—Na1—O1^{iv}	168.34 (10)	O1^{iii}—Na1—O1	88.94 (7)
B1—O2—C2—C1	−27.4 (3)	O1—C1—C2—O2	24.2 (3)

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

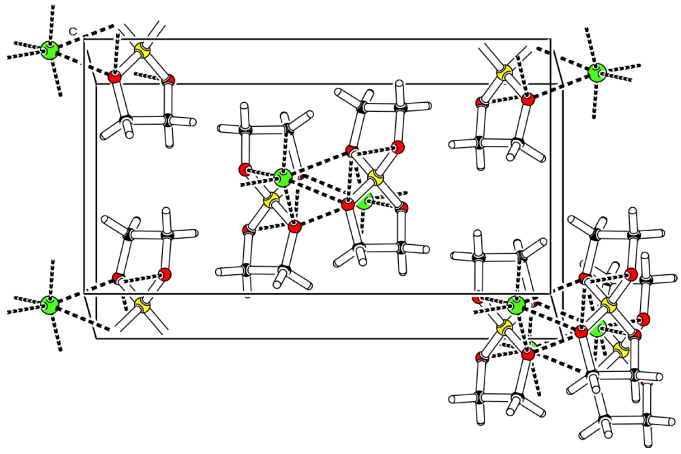


Figure 2
The cell packing, viewed down the *a* axis (*b* to the right) (Spek, 2003). Cation–anion interactions are shown as dashed lines. For clarity, only atoms within the cell and the lower right corner atom set are shown.

All crystals appeared to be multiples or twinned. *XPREP* (Siemens, 1996) identified a monoclinic *C*-centred cell and data were extracted from the largest of three identified domains using *GEMINI* (Bruker, 2000). Data from the second component were also integrated, but with worse internal agreement. Reflections 400, 110, 310, 510, 710, 220, 330 and 620, which had $F_o \gg F_c$, were excluded from the final refinement. The large ratio of maximum/minimum transmission calculated by the procedure of *SADABS* (Sheldrick, 1996) is thought to relate to the alignment of the major and minor twin domains in the crystal, coupled with sinusoidal variations in beam intensity. All H atoms were constrained to their expected geometries (C—H = 0.99 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT* and *SADABS* (Sheldrick, 1996); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97.

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