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Rubidium metaborate, $Rb_3B_3O_6$

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Rubidium metaborate, $Rb_3B_3O_6$, was obtained by the reaction of Rb_2CO_3 and BN using a radiofrequency furnace at a maximum reaction temperature of 1173 K. The crystal structure has been determined by single-crystal X-ray diffraction. The space group is $R\overline{3}c$, with all atoms positioned on a twofold axis (Wyckoff site 18*e*). The ionic compound is isotypic with $Na_3B_3O_6$, $K_3B_3O_6$ and $Cs_3B_3O_6$.

Comment

A wide variety of alkali borates have been reported. The title compound, $Rb_3B_3O_6$, belongs to the series of alkali metaborates $M_3B_3O_6$ (M = Li, Na, K, Rb and Cs). Surprisingly, a single-crystal structure determination of $Rb_3B_3O_6$ has not been published previously; Schneider & Carpenter (1970) and Schlaeger & Hoppe (1994) assumed $Rb_3B_3O_6$ to be isotypic with the other alkali metaborates, but the structure has never been examined by single-crystal X-ray diffraction. We present here the crystal structure of $Rb_3B_3O_6$, solved and refined from X-ray diffraction data.

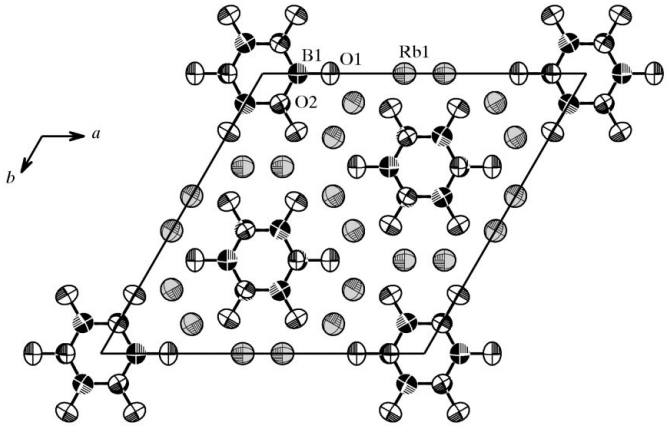

Figure 1
The crystal structure of $Rb_3B_3O_6$, viewed along the crystallographic *c* axis, with 99% probability displacement ellipsoids.

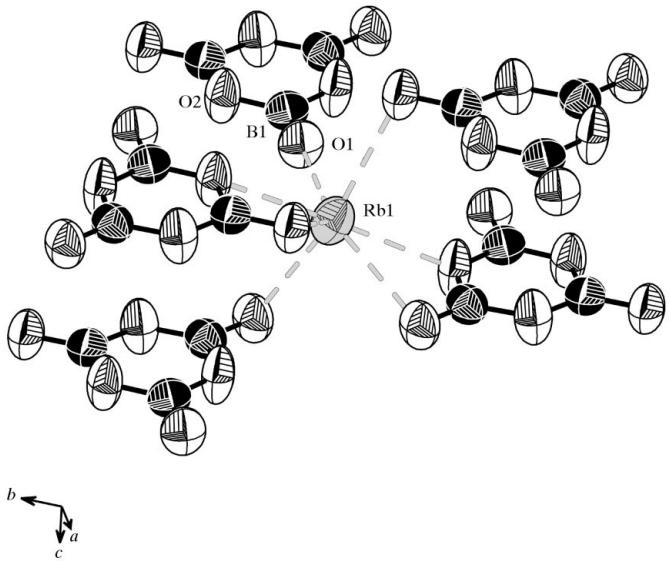

Figure 2
The Rb atom surrounded by $B_3O_6^{3-}$ rings, with 99% probability displacement ellipsoids.

We confirm that $Rb_3B_3O_6$ is isotypic with $Na_3B_3O_6$ (Marezio *et al.*, 1963), $K_3B_3O_6$ (Schneider & Carpenter, 1970) and $Cs_3B_3O_6$ (Schlaeger & Hoppe, 1994).

The lattice parameters increase from Na to Cs, along with the size of the cations. The previously published lattice parameters of $Rb_3B_3O_6$ (Schneider & Carpenter, 1970) are nearly equivalent to those found in the present investigation. The characteristic building units are cyclic planar $B_3O_6^{3-}$ anions, which can be described as comprising three corner-sharing BO_3^{3-} groups. The B—O bond length for terminal atom O1 is 1.315 (6) Å, while the B—O bond to bridging atom O2 [1.407 (3) Å] is significantly longer. The B—O distances of the $M_3B_3O_6$ (M = Na, K, Rb and Cs) series are compared in Table 1. The $B_3O_6^{3-}$ rings in $Rb_3B_3O_6$ are stacked in a staggered manner along [001], with consecutive rings rotated with respect to one another by 60° (Figs. 1 and 2). Each Rb atom is surrounded by seven O atoms, with Rb—O distances of ∼3 Å.

Experimental

$Rb_3B_3O_6$ was obtained by the high-temperature reaction of Rb_2CO_3 (2.0 mmol) and BN (2.0 mmol) using a radiofrequency (rf) furnace. Details of the experimental set-up are given in Schnick *et al.* (1999). Under an atmosphere of pure argon, the starting compounds were placed in a tungsten crucible, which was positioned at the center of the induction coil of an rf furnace. The reaction was performed under an atmosphere of pure nitrogen (purified by silica gel, potassium hydroxide, molecular sieve, P_4O_{10} and a BTS catalyst). The reaction batch was heated to 1173 K at a rate of 7.3 K min^{-1}. The temperature was maintained for 2 h and then the product was cooled at a rate of 0.2 K min^{-1} to 473 K. Subsequently, the mixture was quenched to room temperature. $Rb_3B_3O_6$ was obtained as a coarse crystalline white solid mixed with RbCN.

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Crystal data

$Rb_3B_3O_6$
M_r = 384.85
Trigonal, $R\bar{3}c$
a = 13.1572 (19) Å
c = 7.7434 (15) Å
V = 1160.9 (3) Å^3
Z = 6
D_x = 3.303 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8606 reflections
θ = 3.1–40.3°
μ = 18.87 mm^{-1}
T = 293 (2) K
Block, colourless
0.38 × 0.19 × 0.17 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: numerical (*X-SHAPE*; Stoe & Cie, 1999), T_{min} = 0.056, T_{max} = 0.154
5001 measured reflections
231 independent reflections
226 reflections with $I > 2\sigma(I)$
R_{int} = 0.082
θ_{max} = 25°
$h = -15 \rightarrow 15$
$k = -15 \rightarrow 15$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
$R(F)$ = 0.019
$wR(F^2)$ = 0.046
S = 1.22
231 reflections
22 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0139P)^2 + 8.1199P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.00105 (19)

Table 1
A comparison of B—O distances (Å) and O—B—O angles (°) in $Na_3B_3O_6$, $K_3B_3O_6$, $Rb_3B_3O_6$ and $Cs_3B_3O_6$.

Compound	B—O1	B—O2	O2—B—O2	O2—B—O1
$Na_3B_3O_6$	1.28 (2)	1.43 (1)	114.5 (6)	122.8 (7)
$K_3B_3O_6$	1.33 (1)	1.398 (5)	117.3 (8)	121.3 (4)
$Rb_3B_3O_6$	1.315 (6)	1.407 (3)	115.7 (5)	122.1 (2)
$Cs_3B_3O_6$	1.298 (8)	1.416 (4)	114.8 (3)	122.6 (3)

Data collection: *COLLECT* (Nonius, 1997–2000); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *PLATON* (Spek, 2003) and *WinGX* (Farrugia, 2003).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: IZ1042). Services for accessing these data are described at the back of the journal.

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