

Strontium tetrafluoroborate. Erratum.

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In the paper by Bunič, Tavčar, Goreshnik & Žemva [*Acta Cryst.* (2007), **C63**, i75–i76], the structure reported as Sr(BF₄)₂ is actually that of Cd(BF₄)₂. The correct structure of Sr(BF₄)₂ is now reported.

Comment

This erratum is to correct the report of the crystal structure of strontium tetrafluoroborate (Bunič *et al.*, 2007). The investigated compound was Cd(BF₄)₂ and not the reported Sr(BF₄)₂ because of experimental error. We report here the correct structure of strontium tetrafluoroborate, which appears to be isomorphous with the previously published structures of Ca(BF₄)₂ (Jordan *et al.*, 1975) and Cd(BF₄)₂ (Tavčar & Žemva, 2005). In the Sr(BF₄)₂ structure, the metal atom possesses a coordination number of eight with a square-antiprismatic coordination polyhedron. The Sr–F distances lie in the narrow range 2.490 (4)–2.538 (4) Å, compared with Ca–F distances in the range 2.330 (2)–2.401 (2) Å in Ca(BF₄)₂ and Cd–F distances in the range 2.296 (2)–2.381 (3) Å in Cd(BF₄)₂. The Sr metal center is bonded to eight BF₄[−] units. In turn, each anion is connected to four Sr atoms. All four F atoms in each anion act as μ₂-bridges between B and Sr atoms, resulting in similar B–F bond lengths of 1.376 (7)–1.402 (7) Å.

Experimental

Routine crystallization of strontium tetrafluoroborate from different solvents usually gives crystals of various solvates. However, crystals of the anhydrous salt were grown by dissolving Sr(BF₄)₂·2H₂O, prepared by the reaction between SrCO₃ (Aldrich, 99.99%) and excess aqueous HF (Aldrich, 40%), in acetone and further very slow crystallization.

Crystal data

Sr(BF ₄) ₂	$V = 1235.0 (10) \text{ \AA}^3$
$M_r = 261.24$	$Z = 8$
Orthorhombic, <i>Pbca</i>	Mo $K\alpha$ radiation
$a = 9.602 (5) \text{ \AA}$	$\mu = 8.83 \text{ mm}^{-1}$
$b = 9.259 (5) \text{ \AA}$	$T = 296 \text{ K}$
$c = 13.890 (6) \text{ \AA}$	$0.1 \times 0.1 \times 0.08 \text{ mm}$

Data collection

Rigaku Mercury CCD (2×2 bin mode) diffractometer	9319 measured reflections
Absorption correction: multi-scan (Blessing, 1995)	1534 independent reflections
$T_{\min} = 0.427$, $T_{\max} = 0.504$	1348 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.055$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.059$	101 parameters
$wR(F^2) = 0.111$	$\Delta\rho_{\max} = 1.49 \text{ e \AA}^{-3}$
$S = 1.34$	$\Delta\rho_{\min} = -0.76 \text{ e \AA}^{-3}$
1534 reflections	

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993) and *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); software used to prepare material for publication: *WinGX* (Version 1.70; Farrugia, 1999) and *enCIFer* (Version 1.2; Allen *et al.*, 2004).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN3136). Services for accessing these data are described at the back of the journal.

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Strontium tetrafluoridoborate and
barium tetrafluoridoborate

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In $\text{Sr}(\text{BF}_4)_2$, which is isomorphous with the previously published $\text{Ca}(\text{BF}_4)_2$, the metal atom possesses a coordination number of 8 with a square-antiprismatic environment. Each tetrafluoridoborate anion is bonded to four metal centers. In the barium derivative, the metal center, with symmetry $2m$, is surrounded by 14 F atoms. The B atom and two of the three independent F atoms occupy special positions with symmetry m . Each anion is connected to five Ba atoms. This structure differs significantly from an earlier published structure of $\text{Ba}(\text{BF}_4)_2$ [published as $\text{Ba}_2(\text{BF}_4)_4$; Lin, Cheng, Chen & Huang (1998). *Jiegon H a e*, **17**, 245]. The radial distribution functions for the present $\text{Ba}(\text{BF}_4)_2$ and earlier $\text{Ba}_2(\text{BF}_4)_4$ structures differ significantly.

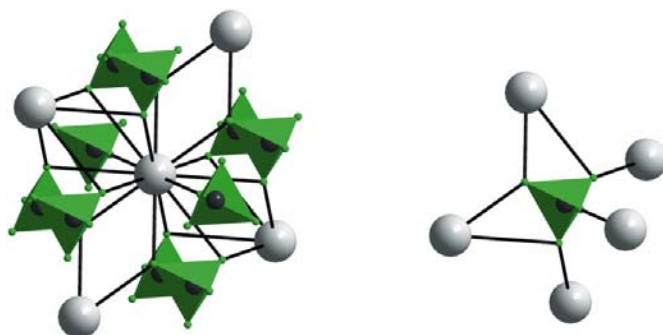
Comment

$\text{Sr}(\text{BF}_4)_2$ appears to be isomorphous with the previously reported compound $\text{Ca}(\text{BF}_4)_2$ (Jordan *et al.*, 1975). In this structure, the metal atom possesses a coordination number of 8 with a square-antiprismatic coordination polyhedron. The Sr—F distances lie in the narrow range 2.294 (5)–2.359 (4) Å compared with 2.33–2.401 Å in $\text{Ca}(\text{BF}_4)_2$. The metal center is bonded to eight BF_4^- units. In turn, each anion is connected to four Sr atoms. All four F atoms in each anion act as μ_2 -bridges between B and Sr atoms, resulting in very similar B—F bond lengths of 1.389 (9)–1.402 (9) Å.

Because of the much greater coordination ability of barium compared with strontium, the structure of $\text{Ba}(\text{BF}_4)_2$ appears to be more complicated. The Ba atom, located on special position $2a$ ($2m$), possesses coordination number 14 (or 10–4). Ten Ba—F bond lengths lie in the range 2.690 (5)–2.886 (2) Å, and there are four longer Ba—F distances of 3.361 (5) Å. Each Ba atom is surrounded by ten BF_4^- anions. The coordination polyhedron of barium shares two edges and two rectangular planes with four other metal centers. The shortest Ba···Ba distances are 4.763 (2) Å for two Ba atoms with a shared plane and 5.014 (1) Å for a pair of metal atoms connected by a shared edge (Fig. 1).

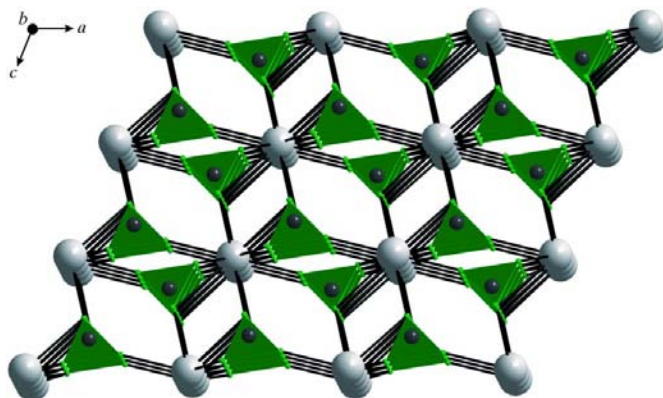
The B atom and two of the three crystallographically independent F atoms of each BF_4^- unit lie on mirror planes (4i Wyckoff positions). Each BF_4^- anion is bonded to five Ba atoms. The B—F distances clearly correlate with the bridging function of each F atom. The shortest B—F distance [B1—F2 = 1.369 (9) Å] is associated with the μ_2 -bridging function of atom F2. Two B1—F3 bond lengths of 1.393 (5) Å correspond to the μ_3 -bridging function of atom F3, which forms a shared corner between two Ba atoms. The longest B—F distance [B1—F1 = 1.420 (9) Å] corresponds to the location of atom F1 on an edge shared by two barium polyhedra. The bridging functions of both cations and anions result in the formation of a three-dimensional network (Fig. 2).

The results obtained for $\text{Ba}(\text{BF}_4)_2$ differ strongly from those of a previously published structure of $\text{Ba}(\text{BF}_4)_2$ [published as $\text{Ba}_2(\text{BF}_4)_4$ (Lin *et al.*, 1998); space group $P2_1n$, $a = 8.339$ (3) Å, $b = 16.530$ (7) Å, $c = 10.212$ (4) Å, $\beta = 106.64$ (3)° and $V = 1349$ (2) Å³], in which the Ba atom possesses the unusually low coordination number of 9 and the Ba—F distances are in the range 2.69 (2)–2.91 (3) Å. The radial distribution functions for the $\text{Ba}(\text{BF}_4)_2$ and $\text{Ba}_2(\text{BF}_4)_4$ structures are noticeably different.



figure

The coordination surrounding the Ba atom (left) and the BF_4^- anion (right) in the structure of $\text{Ba}(\text{BF}_4)_2$. Large gray spheres represent Ba atoms, the tetrahedra are BF_4^- units, and B atoms are shown as small dark spheres.



figure

The packing of the structure of $\text{Ba}(\text{BF}_4)_2$.

Experimental

Sr(BF₄)₂ was prepared by the reaction between SrF₂ (Alfa Aesar, 99.99%) and excess BF₃ (Union Carbide, 99.5%) in anhydrous HF (Fluka, purum) as solvent. When the reaction was complete, the solvent and excess BF₃ were removed on a vacuum line. Ba(BF₄)₂ was prepared in a similar way starting from BaF₂ (Alfa Aesar, 99.99%). The bulk samples were characterized by Raman spectroscopy [793, 533 and 353 cm⁻¹ for Sr(BF₄)₂; 783, 536 and 355 cm⁻¹ for Ba(BF₄)₂] and γ -ray powder diffraction. Samples for diffraction analysis were grown by crystallization of an M(BF₄)₂ (M = Sr and Ba) solution in anhydrous HF with a temperature gradient of 10 K in a fluorinated ethylene propylene crystallization vessel. Crystals were immersed in perfluorinated oil (ABCR, FO5960) in a dry-box, selected under a microscope and transferred into the cold nitrogen stream on the diffractometer.

Ba B

Crystal data

Ba(BF ₄) ₂	$V = 277.3 (2) \text{ \AA}^3$
$M_r = 310.96$	$Z = 2$
Monoclinic, $C2/m$	Mo $K\alpha$ radiation
$a = 12.513 (9) \text{ \AA}$	$\mu = 7.27 \text{ mm}^{-1}$
$b = 5.0135 (13) \text{ \AA}$	$T = 200 \text{ K}$
$c = 4.7628 (15) \text{ \AA}$	$0.1 \times 0.08 \times 0.06 \text{ mm}$
$\beta = 111.854 (4)^\circ$	

Data collection

Rigaku Mercury CCD diffractometer	596 measured reflections
Absorption correction: multi-scan (Blessing, 1995)	342 independent reflections
$T_{\min} = 0.471, T_{\max} = 0.650$	342 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.049$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$	32 parameters
$wR(F^2) = 0.070$	$\Delta\rho_{\max} = 1.14 \text{ e \AA}^{-3}$
$S = 1.19$	$\Delta\rho_{\min} = -1.11 \text{ e \AA}^{-3}$
342 reflections	

Sr B

Crystal data

Sr(BF ₄) ₂	$V = 1047.6 (13) \text{ \AA}^3$
$M_r = 261.24$	$Z = 8$
Orthorhombic, $Pbca$	Mo $K\alpha$ radiation
$a = 9.121 (7) \text{ \AA}$	$\mu = 10.41 \text{ mm}^{-1}$
$b = 8.714 (6) \text{ \AA}$	$T = 200 (2) \text{ K}$
$c = 13.180 (9) \text{ \AA}$	$0.07 \times 0.07 \times 0.05 \text{ mm}$

Data collection

Rigaku Mercury CCD (2 × 2 bin mode) diffractometer	4207 measured reflections
Absorption correction: multi-scan (Blessing, 1995)	1253 independent reflections
$T_{\min} = 0.486, T_{\max} = 0.595$	1132 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.016$

able

Selected geometric parameters ($\text{\AA}, ^\circ$) for Ba(BF₄)₂.

Ba1—F2 ⁱ	2.690 (5)	F1—B1	1.420 (9)
Ba1—F3 ⁱⁱ	2.765 (3)	F2—B1	1.369 (9)
Ba1—F1	2.886 (2)	F3—B1	1.393 (5)
Ba1—Ba1 ⁱⁱⁱ	4.7628 (15)		
F2—B1—F3	111.0 (4)	F2—B1—F1	109.7 (6)
F3—B1—F3 ^{iv}	110.4 (6)	F3—B1—F1	107.3 (4)

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

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$	100 parameters
$wR(F^2) = 0.153$	$\Delta\rho_{\max} = 2.11 \text{ e \AA}^{-3}$
$S = 1.09$	$\Delta\rho_{\min} = -1.48 \text{ e \AA}^{-3}$
1253 reflections	

The highest peaks of residual density were 0.02 e \AA^{-3} from atom Sr1 and 0.89 e \AA^{-3} from atom Ba1.

For both compounds, data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993) and *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAM ND* (Brandenburg, 1999); software used to prepare material for publication: *WinGX* (Version 1.70; Farrugia, 1999) and *enCIFer* (Version 1.2; Allen *et al.*, 2004).

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