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Scandium(III) oxide bromide, ScOBr

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(Sc–O) = 0.002 Å
R factor = 0.018
wR factor = 0.042
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.

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Single crystals of scandium oxide bromide, ScOBr, were obtained as a by-product of the reaction of scandium tribromide, $ScBr_3$, and scandium metal in a sealed tantalum container. ScOBr crystallizes with the FeOCl structure type. $Br_{1/2}Sc_{1/2}OSc_{1/2}Br_{1/2}$ slabs are stacked in the direction of the c axis. Sc^{3+} is surrounded in a distorted octahedral fashion according to the Niggli notation $ScO_{4/4}Br_{2/2}$.

Comment

Rare-earth oxide halides, *MOX*, often appear as by-products in reactions when impure anhydrous rare-earth trihalides, MX_3, are used as starting materials. These impurities are easily generated when the so-called ammonium halide route is used improperly (Meyer, 1983; Meyer & Staffel, 1986; Meyer *et al.*, 1987). In conproportionation reactions of rare-earth halides with their metals (*e.g.* $ScBr_3$ + Sc) or when the halides are reduced with alkali metals, the oxide–halides *MOX* often appear as (a few) single crystals that are easy to separate (Meyer & Schleid, 1986). In special cases, even the rare earths M_2O_3 crystallize from appropriate melts (Schleid & Meyer, 1989) when oxygen is present that might also originate from the reaction containers made of materials such as silica or of refractory metals (tantalum, for example).

Scandium oxide bromide, ScOBr, was obtained as a by-product from a reaction of nominally pure scandium tribromide, $ScBr_3$, obtained *via* the ammonium bromide route (Meyer *et al.*, 1987) with scandium metal in a tantalum container at 1123 K. It crystallizes with the FeOCl type of structure (Goldsztaub, 1934, 1935). In ScOBr, the Sc^{3+} ions are surrounded in a distorted octahedral fashion with Sc—O distances of 2.0807 (12) and 2.1464 (18) Å (two times each) and two Sc—Br distances of 2.6480 (9) Å (Fig. 1). The corresponding angles deviate by as much as 15° from the ideal octahedral angles. According to the Niggli notation $ScO_{4/4}Br_{2/2}$, the oxide ions have the rather high coordination number of four and form layers which are topped by bromide layers that contain half the number of bromide ions. All possible octahedral voids between these $Br_{1/2}OBr_{1/2}$ slabs are occupied by Sc^{3+} ions. These slabs are stacked in the direction of the c axis (Fig. 2).

Experimental

ScOBr was obtained as a by-product (approximately 30%) from the reaction of scandium powder (0.012 g) (Chempur, 99,9%) and nominally pure $ScBr_3$ (0.150 g) in an He-arc welded tantalum container jacketed with a silica ampoule. This ensemble was heated to 1123 K for 10 d, after which it was cooled to ambient temperature by turning off the power of the furnace. The reaction container was opened in an argon-filled dry box (M. Braun, Garching, Germany),

and crystals were selected under a microscope and mounted in thin-walled glass capillaries.

Crystal data

ScOBr
M_r = 140.87
Orthorhombic, *Pmmn*
a = 3.5505 (10) Å
b = 3.9543 (16) Å
c = 8.700 (3) Å
V = 122.15 (7) Å^3
Z = 2
D_x = 3.830 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1655 reflections
θ = 2.3–29.8°
μ = 18.98 mm^{-1}
T = 293 (2) K
Plate, brown–orange
0.20 × 0.10 × 0.05 mm

Data collection

Stoe IPDS-II diffractometer
ω and φ scans
Absorption correction: numerical (*X-SHAPE*; Stoe & Cie, 1999)
T_{min} = 0.058, T_{max} = 0.160
1117 measured reflections
176 independent reflections
170 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 26.9°
$h = -3 \rightarrow 4$
$k = -5 \rightarrow 5$
$l = -10 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.018
$wR(F^2)$ = 0.042
S = 1.12
176 reflections
14 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0248P)^2 + 0.075P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.63 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.063 (8)

Table 1
Selected geometric parameters (Å, °).

Sc—O^{i}	2.0807 (12)	Sc—Sc^{v}	3.2404 (11)
Sc—O^{ii}	2.1464 (18)	Sc—Sc^{iii}	3.5505 (10)
Sc—Br^{iii}	2.6480 (9)		
O^{i}—Sc—O	143.70 (16)	O^{ii}—Sc—Br^{iii}	166.30 (8)

Symmetry codes: (i) $x, y+1, z$; (ii) $-x, -y, -z+1$; (iii) $x-1, y, z$; (v) $-x, -y+1, -z+1$.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-RED* (Stoe & Cie, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *SHELXL97*.

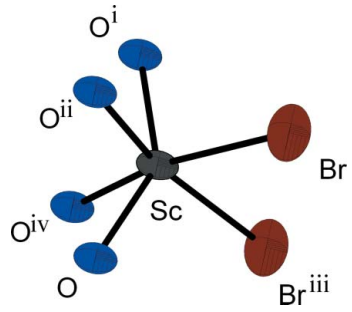

Figure 1
The distorted octahedral surrounding of Sc^{3+} with displacement ellipsoids at the 50% probability level. [Symmetry codes: (i) $x, 1+y, z$; (ii) $-x, -y, 1-z$; (iii) $x-1, y, z$; (iv) $-1-x, -y, 1-z$.]

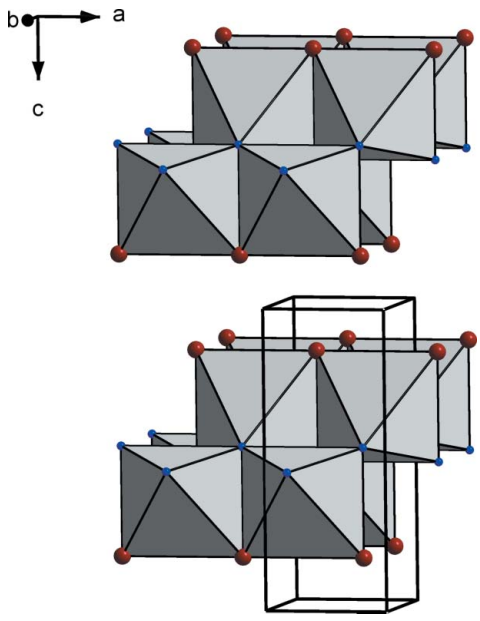

Figure 2
The crystal structure of ScOBr, showing the stacking of the $Br_{1/2}Sc_{1/2}OSc_{1/2}Br_{1/2}$ slabs in the direction of the c axis. O atoms are blue, Br atoms are red.

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