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Key indicators

Single-crystal X-ray study
 T = 293 K
 Mean $\sigma(\text{Sc-N}) = 0.008 \text{ \AA}$
 R factor = 0.040
 wR factor = 0.096
 Data-to-parameter ratio = 37.3

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

Diamminehexabromodiscandium(III)

The title compound, $[\text{Sc}_2\text{Br}_6(\text{NH}_3)_2]$, was obtained by the reaction of ammonium bromide, $(\text{NH}_4)\text{Br}$, with scandium metal in a sealed tantalum metal container. The crystal structure contains zigzag chains of edge-connected $[\text{Sc}(\text{NH}_3)\text{Br}_5]$ octahedra according to the formulation $[\text{Sc}(\text{NH}_3)_{1/1}\text{Br}_{1/1}\text{Br}_{4/2}]$. $\text{Sc}(\text{NH}_3)\text{Br}_3$ is isotypic with $\text{Sc}(\text{NH}_3)\text{Cl}_3$.

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Comment

Non-noble metals may be oxidized by an H atom of the ammonium ion and incorporated in complexes such as ammonium salts or ammoniates (Meyer, 1994). The reaction of ammonium bromide, $(\text{NH}_4)\text{Br}$, with scandium yielded $(\text{NH}_4)_2[\text{Sc}(\text{NH}_3)\text{Br}_5]$ (Böhmer & Meyer, 2001) with the erythrosiderite-type structure. A closer inspection of this reaction has now resulted in the isolation of single crystals of the mono- and diammoniates of scandium(III) bromide, $\text{Sc}(\text{NH}_3)\text{Br}_3$ and $\text{Sc}(\text{NH}_3)_2\text{Br}_3$. Both compounds crystallize isostructurally with the respective chlorides, $\text{Sc}(\text{NH}_3)\text{Cl}_3$ and $\text{Sc}(\text{NH}_3)_2\text{Cl}_3$ (Meyer & Klein, 2002). The crystal structure of the monoammoniate, $\text{Sc}(\text{NH}_3)\text{Br}_3$, is presented here.

In $\text{Sc}(\text{NH}_3)\text{Br}_3$, the Sc^{3+} cation is surrounded octahedrally by five bromide ions and one ammonia molecule. The Sc–N distances [2.252 (7) and 2.244 (7) Å] are very similar to the corresponding distances in $\text{Sc}(\text{NH}_3)\text{Cl}_3$. The Sc–Br distances are between 2.532 (2) and 2.720 (2) Å, with an average of 2.648 (2) Å. Considering the difference between the ionic radii of Cl^- and Br^- of about 0.15 Å, this value compares very well with the mean Sc–Cl distance in $\text{Sc}(\text{NH}_3)\text{Cl}_3$ of 2.499 Å.

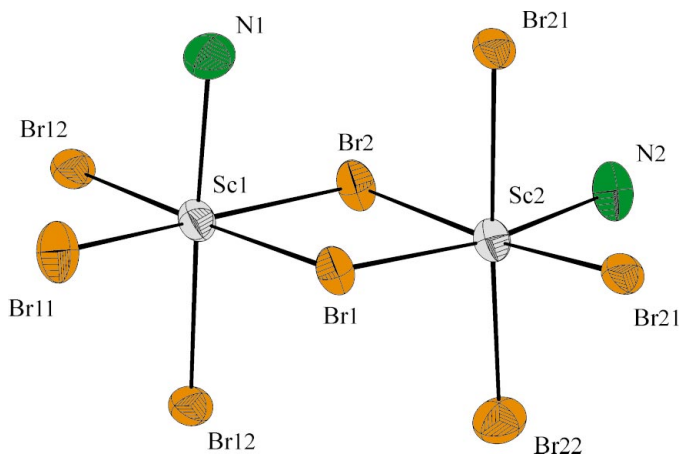


Figure 1

One dimer of octahedra in the structure of $\text{Sc}(\text{NH}_3)\text{Br}_3$, showing the atom-numbering scheme and 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

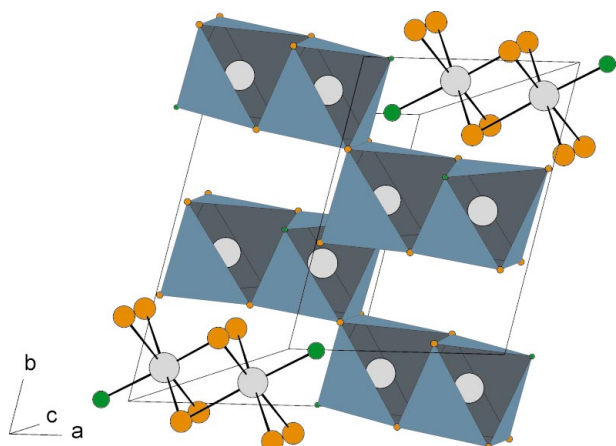


Figure 2
A perspective view of the crystal structure of $\text{Sc}(\text{NH}_3)\text{Br}_3$ in a polyhedral representation, showing the zigzag chains of edge-connected dimers.

The $[\text{Sc}(\text{NH}_3)\text{Br}_5]$ octahedra in $\text{Sc}(\text{NH}_3)\text{Br}_3$ are connected via two common bromide edges, in accordance with the formulation $[\text{Sc}(\text{NH}_3)_{1/1}\text{Br}_{1/1}\text{Br}_{4/2}]$, to form a zigzag chain (Fig. 1). These chains are arranged as shown in Fig. 2 and they appear to be held together by van der Waals and hydrogen bonding.

Experimental

Ammonium bromide, $(\text{NH}_4)\text{Br}$ (5 mmol, 490 mg), and scandium metal (2 mmol, 90 mg) were placed in a tantalum container, which was then sealed by helium arc welding and jacketed by a silica ampoule. The reaction mixture was heated to 623 K for 100 h. Colourless single crystals of $\text{Sc}(\text{NH}_3)\text{Br}_3$ were selected under a microscope in an argon-filled dry box.

Crystal data

$[\text{Sc}_2\text{Br}_6(\text{NH}_3)_2]$	$Z = 2$
$M_r = 603.45$	$D_x = 3.192 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 7.392(2) \text{ \AA}$	Cell parameters from 3629 reflections
$b = 9.578(2) \text{ \AA}$	$\theta = 1.3\text{--}29.8^\circ$
$c = 9.551(2) \text{ \AA}$	$\mu = 20.12 \text{ mm}^{-1}$
$\alpha = 73.07(2)^\circ$	$T = 293(2) \text{ K}$
$\beta = 80.04(2)^\circ$	Column, colourless
$\gamma = 77.82(2)^\circ$	$0.25 \times 0.10 \times 0.10 \text{ mm}$
$V = 627.8(3) \text{ \AA}^3$	

Data collection

Stoe IPDS II diffractometer	9270 measured reflections
ω and φ scans	3470 independent reflections
Absorption correction: numerical;	1750 reflections with $I > 2\sigma(I)$
the absorption correction (<i>X-RED</i> ;	$R_{\text{int}} = 0.072$
Stoe & Cie, 2001) was performed	$\theta_{\text{max}} = 29.6^\circ$
after optimizing the crystal shape	$h = -10 \rightarrow 10$
using <i>X-SHAPE</i> (Stoe & Cie, 1999)	$k = -13 \rightarrow 13$
$T_{\text{min}} = 0.052$, $T_{\text{max}} = 0.155$	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.043P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.040$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.096$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 0.79$	$\Delta\rho_{\text{max}} = 1.06 \text{ e \AA}^{-3}$
3470 reflections	$\Delta\rho_{\text{min}} = -1.19 \text{ e \AA}^{-3}$
93 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	(Sheldrick, 1997)
	Extinction coefficient: 0.0021 (3)

Table 1

Selected geometric parameters (\AA , $^\circ$).

Sc1—N1	2.252 (7)	Br1—Sc2	2.6769 (18)
Sc1—Br11	2.5316 (18)	Br2—Sc2	2.6542 (18)
Sc1—Br1	2.6548 (18)	Sc2—N2	2.244 (7)
Sc1—Br12	2.6555 (19)	Sc2—Br22	2.5293 (18)
Sc1—Br2	2.7197 (17)	Sc2—Br21	2.6477 (18)
Br12—Sc1 ⁱ	2.6779 (17)	Sc2—Br21 ⁱⁱ	2.7190 (18)
N1—Sc1—Br11	93.8 (2)	Sc2—Br2—Sc1	94.33 (6)
N1—Sc1—Br1	92.4 (2)	N2—Sc2—Br22	92.8 (2)
Br11—Sc1—Br1	91.10 (6)	N2—Sc2—Br21	92.6 (2)
N1—Sc1—Br12	87.8 (2)	Br22—Sc2—Br21	92.49 (6)
Br11—Sc1—Br12	94.50 (6)	N2—Sc2—Br2	88.7 (2)
Br1—Sc1—Br12	174.38 (6)	Br22—Sc2—Br2	94.32 (6)
N1—Sc1—Br12 ⁱ	170.1 (2)	Br21—Sc2—Br2	172.99 (6)
Br11—Sc1—Br12 ⁱ	94.49 (6)	N2—Sc2—Br1	171.1 (2)
Br1—Sc1—Br12 ⁱ	92.78 (6)	Br22—Sc2—Br1	94.51 (6)
Br12—Sc1—Br12 ⁱ	86.30 (5)	Br21—Sc2—Br1	92.25 (6)
N1—Sc1—Br2	84.9 (2)	Br2—Sc2—Br1	85.59 (5)
Br11—Sc1—Br2	175.57 (7)	N2—Sc2—Br21 ⁱⁱⁱ	86.6 (2)
Br1—Sc1—Br2	84.73 (5)	Br22—Sc2—Br21 ⁱⁱⁱ	176.44 (7)
Br12—Sc1—Br2	89.69 (6)	Br21—Sc2—Br21 ⁱⁱⁱ	84.05 (5)
Br12 ⁱ —Sc1—Br2	87.21 (5)	Br2—Sc2—Br21 ⁱⁱⁱ	89.16 (6)
Sc1—Br12—Sc1 ⁱ	93.70 (5)	Br1—Sc2—Br21 ⁱⁱⁱ	86.49 (5)
Sc1—Br1—Sc2	95.32 (6)	Sc2—Br21—Sc2 ⁱⁱⁱ	95.95 (5)

Symmetry codes: (i) $-x, 2 - y, 1 - z$; (ii) $1 - x, 1 - y, 2 - z$.

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
N1—H1B \cdots Br11 ⁱ	0.89	2.64	3.532 (8)	175
N1—H1C \cdots Br22 ⁱⁱ	0.89	2.64	3.520 (8)	170
N2—H2B \cdots Br22 ⁱⁱⁱ	0.89	2.68	3.556 (8)	171
N2—H2C \cdots Br11 ^{iv}	0.89	2.66	3.551 (7)	174

Symmetry codes: (iii) $-x, 2 - y, 2 - z$; (iv) $x, 1 + y, z$; (v) $1 - x, 1 - y, 1 - z$; (vi) $1 + x, y - 1, z$.

The H atoms were placed in idealized positions and constrained to ride on their parent atoms, with N—H distances of 0.890 \AA . A common $U_{\text{iso}}(\text{H})$ value was refined for all six H atoms. The highest peak is located 1.30 \AA from Br21 and the deepest hole 0.85 \AA from Br2.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1996); software used to prepare material for publication: *SHELXL97*.

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