

Gerd Meyer,\* Nazife Cesur and  
Stephan Bremm

Institut für Anorganische Chemie, Universität zu  
Köln, Greinstrasse 6, D-50939 Köln, Germany

Correspondence e-mail:  
gerd.meyer@uni-koeln.de

#### Key indicators

Single-crystal X-ray study  
T = 293 K  
Mean  $\sigma(\text{Sc}-\text{N}) = 0.010 \text{ \AA}$   
R factor = 0.039  
wR factor = 0.081  
Data-to-parameter ratio = 26.5

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

## Tetraamminehexabromodiscandium(III)

The title compound,  $[\text{Sc}_2\text{Br}_6(\text{NH}_3)_4]$ , was obtained by the reaction of ammonium bromide,  $(\text{NH}_4)\text{Br}$ , with scandium metal in a sealed tantalum container. The crystal structure contains isolated dimers of bromide edge-connected  $[\text{Sc-mer}(\text{NH}_3)_3\text{Br}_3]$  and  $[\text{Sc}(\text{NH}_3)\text{Br}_5]$  octahedra.  $\text{Sc}(\text{NH}_3)_2\text{Br}_3$  is isotopic with  $\text{Sc}(\text{NH}_3)_2\text{Cl}_3$ .

Received 23 September 2003

Accepted 29 September 2003

Online 7 October 2003

#### Comment

Just like  $(\text{NH}_4)_2[\text{Sc}(\text{NH}_3)\text{Br}_5]$  (Böhmer & Meyer, 2001), the mono- and diammoniates of scandium(III) bromide,  $\text{Sc}(\text{NH}_3)\text{Br}_3$  and  $\text{Sc}(\text{NH}_3)_2\text{Br}_3$ , have been obtained by the reaction of ammonium bromide,  $(\text{NH}_4)\text{Br}$ , and scandium metal. 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 diammoniate,  $\text{Sc}(\text{NH}_3)_2\text{Br}_3$ , is presented here.

In  $\text{Sc}(\text{NH}_3)_2\text{Br}_3$ , atom Sc1 is surrounded by three ammonia and three bromide ligands, both in a meridional arrangement. Sc2 has only one ammonia ligand and is further surrounded by five bromide ligands. These two octahedra,  $[\text{Sc1}(\text{NH}_3)_3\text{Br}_1\text{Br}_{2/2}]$  and  $[\text{Sc2}(\text{NH}_3)\text{Br}_3\text{Br}_{2/2}]$ , are connected through a common bromide edge (Fig. 1). The Sc1–N distances [2.27 (1) Å (2 ×) and 2.28 (1) Å] are slightly longer than the Sc2–N distance of 2.21 (1) Å, which is very close to the Sc–N distance in both  $\text{Sc}(\text{NH}_3)\text{Br}_3$  (Meyer *et al.*, 2003) and  $\text{Sc}(\text{NH}_3)\text{Cl}_3$  (Meyer & Klein, 2002), where  $[\text{Sc}(\text{NH}_3)\text{X}_5]$  (X is Cl or Br) octahedra also occur. The Sc–Br distances are between 2.58 (1) and 2.62 (1) Å for the terminal ligands and between 2.65 (1) and 2.75 (1) Å for the edge-connecting ligands, all averaging to 2.64 Å, which is very close to the average Sc–Br distance of 2.65 Å in  $\text{Sc}(\text{NH}_3)\text{Br}_3$ . The unsymmetrical dimers, which may be considered as

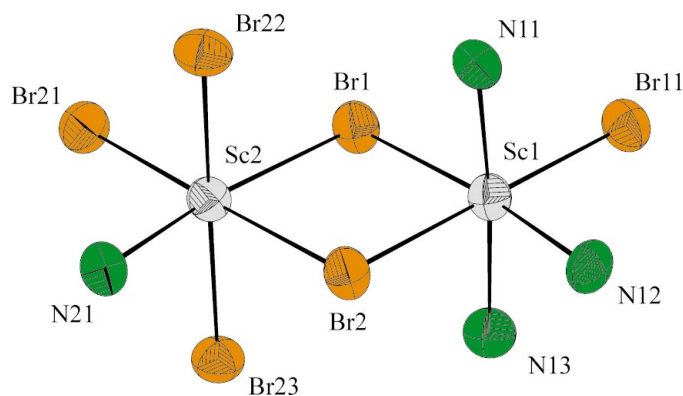
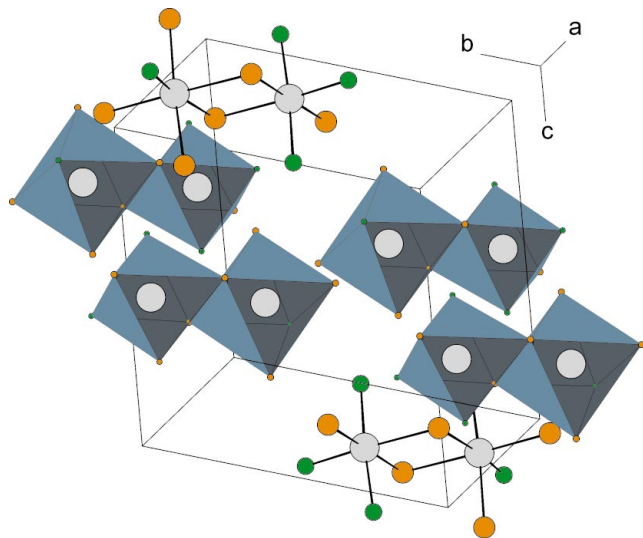


Figure 1

One dimer of octahedra in the structure of  $\text{Sc}(\text{NH}_3)_2\text{Br}_3$ , 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  $\text{Sc}(\text{NH}_3)_6\text{Br}_6$  in a polyhedral representation, showing the  $[\text{Sc}(\text{NH}_3)_3\text{Br}_1\text{Br}_{2/2}]^+ \cdot [\text{Sc}(\text{NH}_3)_3\text{Br}_3\text{Br}_{2/2}]^-$  dipoles.

$[\text{Sc}(\text{NH}_3)_3\text{Br}_1\text{Br}_{2/2}]^+ \cdot [\text{Sc}(\text{NH}_3)_3\text{Br}_3\text{Br}_{2/2}]^-$  dipoles, are arranged as shown in Fig. 2.

## Experimental

Ammonium bromide,  $(\text{NH}_4)\text{Br}$  (5 mmol, 490 mg), and scandium metal (3.6 mmol, 163 mg) were placed in a tantalum container, which was then sealed by helium arc welding, and jacketed with a silica ampoule. The reaction mixture was heated to 623 K for 100 h. Colourless single crystals of  $[\text{Sc}_2\text{Br}_6(\text{NH}_3)_4]$  were selected under a microscope in an argon-filled dry box.

### Crystal data

$[\text{Sc}_2\text{Br}_6(\text{NH}_3)_4]$	$Z = 2$
$M_r = 637.52$	$D_x = 2.827 \text{ Mg m}^{-3}$
Triclinic, $P1$	Mo $K\alpha$ radiation
$a = 7.211(2) \text{ \AA}$	Cell parameters from 2678 reflections
$b = 10.157(3) \text{ \AA}$	$\theta = 1.9\text{--}29.7^\circ$
$c = 11.350(3) \text{ \AA}$	$\mu = 16.88 \text{ mm}^{-1}$
$\alpha = 105.39(2)^\circ$	$T = 293(2) \text{ K}$
$\beta = 90.76(2)^\circ$	Column, colourless
$\gamma = 109.85(2)^\circ$	$0.2 \times 0.1 \times 0.1 \text{ mm}$
$V = 749.0(4) \text{ \AA}^3$	

### Data collection

Stoe IPDS II diffractometer	6953 measured reflections
$\omega$ and $\varphi$ scans	2938 independent reflections
Absorption correction: numerical; the absorption correction ( $X\text{-RED}$ ; Stoe & Cie, 2001) was performed after optimizing the crystal shape using $X\text{-SHAPE}$ (Stoe & Cie, 1999)	1237 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.100$ , $T_{\max} = 0.148$	$R_{\text{int}} = 0.077$
	$\theta_{\text{max}} = 26.0^\circ$
	$h = -8 \rightarrow 8$
	$k = -12 \rightarrow 12$
	$l = -13 \rightarrow 13$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0201P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.039$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.081$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 0.70$	$\Delta\rho_{\text{max}} = 0.66 \text{ e \AA}^{-3}$
2938 reflections	$\Delta\rho_{\text{min}} = -0.70 \text{ e \AA}^{-3}$
111 parameters	Extinction correction: $SHELXL97$ (Sheldrick, 1997)
H-atom parameters constrained	Extinction coefficient: 0.00190 (15)

**Table 1**

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

Br21—Sc2	2.575 (3)	Br23—Sc2	2.622 (3)
Br11—Sc1	2.538 (3)	Br22—Sc2	2.594 (3)
Br1—Sc1	2.654 (3)	Sc1—N11	2.269 (9)
Br1—Sc2	2.679 (3)	Sc1—N12	2.269 (9)
Br2—Sc1	2.679 (3)	Sc1—N13	2.284 (10)
Br2—Sc2	2.745 (3)	Sc2—N21	2.211 (10)
Sc1—Br1—Sc2	95.35 (9)	Br1—Sc1—Br2	86.61 (9)
Sc1—Br2—Sc2	93.25 (8)	N21—Sc2—Br21	95.2 (3)
N11—Sc1—N12	91.6 (4)	N21—Sc2—Br22	87.9 (3)
N11—Sc1—N13	175.3 (4)	Br21—Sc2—Br22	92.64 (9)
N12—Sc1—N13	90.5 (3)	N21—Sc2—Br23	87.8 (3)
N11—Sc1—Br11	91.6 (3)	Br21—Sc2—Br23	92.89 (8)
N12—Sc1—Br11	93.2 (3)	Br22—Sc2—Br23	173.27 (10)
N13—Sc1—Br11	92.4 (3)	N21—Sc2—Br1	171.6 (3)
N11—Sc1—Br1	89.0 (3)	Br21—Sc2—Br1	93.17 (9)
N12—Sc1—Br1	172.8 (3)	Br22—Sc2—Br1	92.17 (9)
N13—Sc1—Br1	88.4 (3)	Br23—Sc2—Br1	91.35 (9)
Br11—Sc1—Br1	93.95 (9)	N21—Sc2—Br2	86.8 (3)
N11—Sc1—Br2	88.2 (3)	Br21—Sc2—Br2	177.95 (11)
N12—Sc1—Br2	86.3 (3)	Br22—Sc2—Br2	87.55 (8)
N13—Sc1—Br2	87.7 (3)	Br23—Sc2—Br2	87.06 (8)
Br11—Sc1—Br2	179.43 (12)	Br1—Sc2—Br2	84.79 (9)

**Table 2**

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

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
N13—H13A $\cdots$ Br23 <sup>i</sup>	0.89	3.12	3.690 (10)	124.1
N13—H13B $\cdots$ Br21 <sup>ii</sup>	0.89	3.00	3.854 (11)	161.3
N13—H13C $\cdots$ Br23 <sup>iii</sup>	0.89	2.92	3.722 (9)	150.9
N11—H11A $\cdots$ Br22 <sup>iv</sup>	0.89	2.82	3.684 (9)	163.3
N11—H11B $\cdots$ Br22	0.89	3.13	3.846 (11)	139.4
N21—H21A $\cdots$ Br11 <sup>v</sup>	0.89	2.77	3.514 (10)	142.5
N21—H21B $\cdots$ Br21 <sup>vi</sup>	0.89	2.89	3.669 (10)	147.5
N21—H21C $\cdots$ Br21 <sup>vii</sup>	0.89	2.81	3.609 (10)	150.5
N12—H12A $\cdots$ Br21 <sup>ii</sup>	0.89	3.11	3.712 (10)	127.2
N12—H12B $\cdots$ Br23 <sup>i</sup>	0.89	2.79	3.671 (10)	169.7
N12—H12C $\cdots$ Br22 <sup>iv</sup>	0.89	2.80	3.678 (9)	168.2

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

The H atoms were placed in idealized positions and constrained to ride on their parent atoms, with N—H distances of 0.890  $\text{\AA}$ . For all H atoms, a common  $U_{\text{iso}}(\text{H})$  value was refined.

Data collection:  $X\text{-AREA}$  (Stoe & Cie, 2002); cell refinement:  $X\text{-AREA}$ ; data reduction:  $X\text{-AREA}$ ; 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:  $SHELXL97$ .

## References

- Altomare, A., Casciarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
- Böhmer, N. & Meyer, G. (2001). *Z. Kristallogr. Suppl.* **18**, 125.
- Brandenburg, K. (1996). *DIAMOND*. Release 1.0.3. University of Bonn, Germany.
- Meyer, G., Cesur, N. & Pantenburg, I. (2003). *Acta Cryst.* **E59**, i145–i146.
- Meyer, G. & Klein, D. (2002). *Z. Anorg. Allg. Chem.* **628**, 1447–1450.
- Sheldrick, G. M. (1997). *SHELXL97*. Release 97-2. University of Göttingen, Germany.

Stoe & Cie (1999). *X-SHAPE*. Version 1.06. Stoe & Cie, Darmstadt, Germany.  
Stoe & Cie (2001). *X-RED*. Version 1.22. Stoe & Cie, Darmstadt, Germany.

Stoe & Cie (2002). *X-AREA*. MainMenu Version 1.16. Stoe & Cie, Darmstadt, Germany.