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Twinned bis[tris(1,2-ethanediamine-κ^2N,N')-manganese(II)] pentaselenodiantimonate(III)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.009 Å
R factor = 0.030
wR factor = 0.075
Data-to-parameter ratio = 23.2

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

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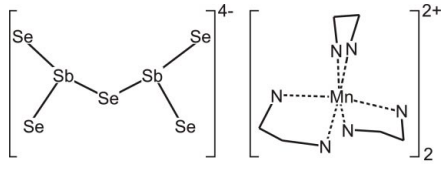
The crystal structure of the title compound, $[Mn(C_2H_8N_2)_3]_2[Sb_2Se_5]$, consists of discrete $[Mn(en)_3]^{2+}$ complex cations and $[Sb_2Se_5]^{4-}$ anions. There are two crystallographically independent cations and two crystallographically independent anions in the asymmetric unit. The cations occupy general positions whereas the anions are each located on a twofold axis. Between the Se atoms of the anions and the H atoms of the amino groups, short intermolecular distances are observed, which indicate hydrogen bonding.

Comment

The structure determination of the title compound, (I), was performed as part of a project on the synthesis of new thioantimonates or selenoantimonates under mild solvothermal conditions.



(I)

Recently, several new selenoantimonates were prepared by this route (Bensch *et al.*, 1997; Wendland *et al.*, 1998*a*,*b*; Wachhold & Sheldrick, 1995; Girard *et al.*, 1998). However, only in $[Fe(en)_3]_2[Sb_2Se_5]$ are discrete $[Sb_2Se_5]^{4-}$ anions found as in (I) (Chen *et al.*, 2000). The crystal structure of (I) consists of discrete $[Mn(en)_3]^{2+}$ (en = ethylenediamine) complex cations and $[Sb_2Se_5]^{4-}$ anions. Each Mn atom is surrounded by six N atoms of three en ligands within strongly distorted octahedra. There are two crystallographically independent cations in the asymmetric unit, both of which are located in general positions. The bond lengths involving Mn are between 2.238 (4) and 2.325 (5) Å, in good agreement with those found in other $[Mn(en)_3]^{2+}$ complex cations. The geometry of both crystallographically independent cations is comparable. There are two crystallographically independent $[Sb_2Se_5]^{4-}$ anions in the asymmetric unit and both are located on twofold axes. In both anions, the Sb—Se bond lengths to the bridging Se atoms are significantly longer than those to the terminal Se atoms. The geometry of the $[Sb_2Se_5]^{4-}$ anions is comparable to that in the analogous iron compound (Chen *et al.*, 2000). In the crystal structure, intermolecular Se···H contacts are found between the amino H atoms and the $[Sb_2Se_5]^{4-}$ anions which are shorter than the sum of the van der Waals radii, indicating hydrogen bonding.

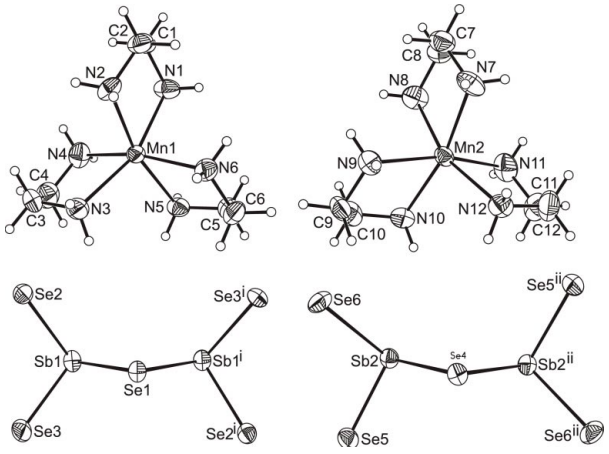


Figure 1
The structure of the anions and cations in the crystal structure of the title compound, with labelling and with displacement ellipsoids drawn at the 50% probability level [symmetry codes: (i) $-x+\frac{3}{2}, y, -z+\frac{3}{2}$; (ii) $-x+\frac{1}{2}, y, -z+\frac{3}{2}$].

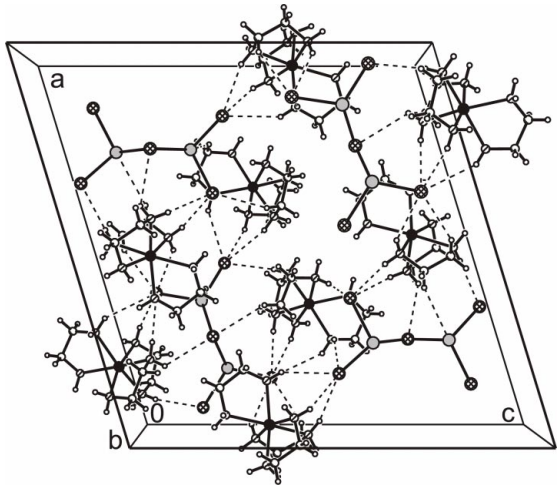


Figure 2
The crystal structure of the title compound, viewed along the crystallographic *b* axis. Short intermolecular Se···H contacts are shown as dashed lines.

Experimental

The title compound was prepared by the reaction of manganese(II) dichloride tetrahydrate, antimony(III) telluride and elemental Se (molar ratio 1:1:3) in 2 ml of 1,2-ethanediamine in a Teflon-lined steel autoclave at 433 K for 7 d. The product was filtered off, washed with ethanol and dried *in vacuo*. The product consists of a mixture of orange crystals of the title compound as the minor phase, $[Mn(en)_3][SbSe]_4$-enH as the major phase and a small amount of unreacted Sb_2Te_3.

Crystal data

$[Mn(C_2H_8N_2)_3]_2[Sb_2Se_5]$
M_r = 1108.80
Monoclinic, $P2/n$
a = 19.909 (3) Å
b = 9.0410 (10) Å
c = 20.015 (3) Å
β = 106.928 (17)°
V = 3446.5 (8) Å^3
Z = 4
D_x = 2.137 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7997 reflections
θ = 2–28°
μ = 7.57 mm^{-1}
T = 293 (2) K
Polyhedron, orange
0.11 × 0.08 × 0.06 mm

Data collection

Stoe IPDS diffractometer
φ scans
Absorption correction: numerical (*X-SHAPE*; Stoe & Cie, 1998)
T_{min} = 0.476, T_{max} = 0.625
29806 measured reflections
6991 independent reflections
6407 reflections with $I > 2\sigma(I)$
R_{int} = 0.055
θ_{max} = 27.0°
$h = -25 \rightarrow 25$
$k = -11 \rightarrow 11$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.075
S = 1.04
6991 reflections
301 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0493P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.96 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.00151 (18)

Table 1
Selected geometric parameters (Å, °).

Sb1—Se2	2.5113 (7)	Mn1—N6	2.297 (4)
Sb1—Se3	2.5121 (9)	Mn1—N2	2.298 (4)
Sb1—Se1	2.6077 (6)	Mn1—N4	2.325 (5)
Sb2—Se5	2.4920 (7)	Mn2—N8	2.257 (5)
Sb2—Se6	2.5131 (8)	Mn2—N11	2.279 (5)
Sb2—Se4	2.6309 (6)	Mn2—N9	2.280 (4)
Mn1—N1	2.238 (4)	Mn2—N12	2.281 (5)
Mn1—N3	2.251 (4)	Mn2—N7	2.284 (4)
Mn1—N5	2.266 (4)	Mn2—N10	2.296 (5)
Se2—Sb1—Se3	103.57 (3)	N3—Mn1—N4	77.04 (18)
Se2—Sb1—Se1	99.56 (2)	N5—Mn1—N4	90.06 (18)
Se3—Sb1—Se1	101.81 (2)	N6—Mn1—N4	166.94 (17)
Sb1^{i}—Se1—Sb1	95.53 (3)	N2—Mn1—N4	103.85 (18)
Se5—Sb2—Se6	102.94 (3)	N8—Mn2—N11	92.0 (2)
Se5—Sb2—Se4	102.48 (2)	N8—Mn2—N9	93.57 (19)
Se6—Sb2—Se4	101.93 (2)	N11—Mn2—N9	166.83 (19)
Sb2^{ii}—Se4—Sb2	91.56 (3)	N8—Mn2—N12	161.18 (18)
N1—Mn1—N3	160.19 (18)	N11—Mn2—N12	76.98 (18)
N1—Mn1—N5	97.39 (16)	N9—Mn2—N12	100.50 (16)
N3—Mn1—N5	97.59 (16)	N8—Mn2—N7	76.95 (17)
N1—Mn1—N6	94.70 (19)	N11—Mn2—N7	100.8 (2)
N3—Mn1—N6	101.18 (17)	N9—Mn2—N7	92.08 (17)
N5—Mn1—N6	77.29 (16)	N12—Mn2—N7	90.01 (17)
N1—Mn1—N2	76.64 (16)	N8—Mn2—N10	97.70 (17)
N3—Mn1—N2	91.74 (16)	N11—Mn2—N10	91.35 (19)
N5—Mn1—N2	164.73 (17)	N9—Mn2—N10	76.09 (16)
N6—Mn1—N2	89.08 (16)	N12—Mn2—N10	97.77 (17)
N1—Mn1—N4	90.1 (2)	N7—Mn2—N10	166.82 (18)

Symmetry codes: (i) $\frac{3}{2}-x, y, \frac{3}{2}-z$; (ii) $\frac{1}{2}-x, y, \frac{3}{2}-z$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H5N···Se2^{iii}	0.90	2.69	3.568 (4)	167
N5—H10N···Se5^{ii}	0.90	2.78	3.618 (5)	156
N6—H11N···Se6	0.90	2.73	3.623 (4)	170
N6—H12N···Se6^{iv}	0.90	2.79	3.619 (5)	155
N7—H14N···Se5^{v}	0.90	2.69	3.503 (5)	151
N9—H18N···Se3	0.90	2.76	3.634 (5)	165
N10—H19N···Se2^{i}	0.90	2.77	3.598 (5)	154
N12—H24N···Se5^{vi}	0.90	2.78	3.669 (4)	168

Symmetry codes: (i) $\frac{3}{2}-x, y, \frac{3}{2}-z$; (ii) $\frac{1}{2}-x, y, \frac{3}{2}-z$; (iii) $\frac{1}{2}-x, 1+y, \frac{3}{2}-z$; (iv) $-x, 1-y, 1-z$; (v) $1-x, -y, 1-z$; (vi) $1-x, 1-y, 1-z$.

The H atoms were positioned with idealized geometry and refined with fixed isotropic displacement parameters [U_{iso}(H) = 1.2U_{eq}(C/N)] using a riding model with C—H(methylene) = 0.97 Å and N—H = 0.90 Å. The crystal investigated was pseudo-merohedrally twinned

and was refined using the TWIN option in *SHELXL*97 and the matrix (001 010 100). The contributions of the two twin components refined to 0.2086 (6):0.7914(6). Refinement of the structure without the TWIN matrix gave significantly poorer reliability factors [$R1$ for 6407 $F_o > 4\sigma(F_o)$ = 0.137; $wR2$ for all data = 0.347; residual electron density = 5.05/−2.86 e Å^{-3}].

Data collection: *IPDS Program Package* (Stoe & Cie, 1998); cell refinement: *IPDS Program Package*; data reduction: *IPDS Program Package*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *CIFTAB* in *SHELXTL*.

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