

$w = 1/[\sigma^2(F) + (0.02F)^2 + 1.00]$ (Killean & Lawrence, 1969)
 $(\Delta/\sigma)_{\max} = 0.002$

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

	x	y	z	U_{eq}
Hg1	0.2518 (2)	0.02260 (6)	0.36781 (7)	0.0248 (3)
Hg2	0.7070 (2)	0.18874 (7)	0.22898 (7)	0.0282 (3)
Se1	0.2260 (4)	0.1344 (2)	0.6136 (2)	0.0234 (9)
Se2	0.2659 (4)	0.0675 (2)	0.0934 (2)	0.0269 (9)
O1	0.268 (3)	0.007 (1)	0.606 (1)	0.033 (8)
O2	-0.105 (3)	0.141 (1)	0.623 (1)	0.024 (6)
O3	0.269 (3)	0.162 (1)	0.471 (1)	0.032 (8)
O4	0.292 (4)	-0.034 (1)	0.177 (1)	0.037 (9)
O5	0.586 (3)	0.108 (1)	0.083 (1)	0.029 (8)
O6	0.174 (3)	0.150 (1)	0.197 (1)	0.043 (9)
O7	0.772 (4)	0.261 (1)	0.387 (1)	0.045 (9)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Hg1—O1	2.751 (15)	Hg2—O6	2.742 (16)
Hg1—O1 ⁱ	2.671 (14)	Hg2—O6 ⁱⁱ	2.447 (16)
Hg1—O1 ⁱⁱⁱ	2.459 (14)	Hg2—O7	2.072 (16)
Hg1—O2 ⁱ	2.294 (13)	Se1—O1	1.704 (15)
Hg1—O3	2.199 (15)	Se1—O2	1.675 (14)
Hg1—O4	2.332 (15)	Se1—O3	1.696 (15)
Hg1—O6	2.617 (17)	Se2—O4	1.663 (16)
Hg2—O1 ⁱⁱⁱ	3.219 (15)	Se2—O5	1.705 (14)
Hg2—O2 ⁱⁱⁱ	2.757 (13)	Se2—O6	1.699 (17)
Hg2—O5	2.064 (15)		
O1—Se1—O2	100.1 (7)	O4—Se2—O5	103.8 (9)
O1—Se1—O3	98.3 (7)	O4—Se2—O6	97.9 (8)
O2—Se1—O3	102.4 (7)	O5—Se2—O6	97.1 (8)

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

The data were corrected for decay. As the absorption is high, both ψ -scan (North, Phillips & Mathews, 1968) and spherical absorption corrections were applied (*International Tables for X-ray Crystallography*, 1967, Vol. II, Table 5.3.6B) with $T_{\max} = 0.995$ and $T_{\min} = 0.705$ for the ψ -scan correction, and $T_{\max} = 0.069$ and $T_{\min} = 0.042$ for the spherical correction. The maximum and minimum electron densities in the final difference Fourier map are located within 1 \AA of Hg atoms.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN PROCESS* (Fair, 1990). Program(s) used to solve structure: *MULTAN* (Main *et al.*, 1982). Program(s) used to refine structure: *MolEN LSFM*. Molecular graphics: *ORTEP II* (Johnson, 1976). Software used to prepare material for publication: *MolEN*.

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: BR1119). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). **C52**, 1072–1074

Amminemercury(II) Selenite

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Abstract

The title compound {amminemercury(II) trioxoselenate(IV)}, [Hg(NH₃)]SeO₃ has a structure comprising corrugated edge-sharing chains of HgNO₅ octahedra and SeO₃ pyramids. The chains are apparently held in position by hydrogen bonds. The octahedra are 2 + 4 distorted. The selenite group exhibits one significantly differing Se—O bond length involving an O atom coordinated to three Hg^{II} ions.

Comment

We have reported the structures of three different mercury(II) selenites, HgSeO₃, Hg₃(HSeO₃)₂(SeO₃)₂ and Hg₂(SeO₃)₂·H₂O, previously (Koskenlinna & Valkonen, 1995a,b, 1996). These compounds contain Hg^{II} ions coordinated by six, six and seven, and five and seven O

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atoms, respectively. All of the polyhedra have two short Hg—O bonds with bond angles approaching 180°. Thus, all Hg^{II} ions are coordinated with 2 + 3–5 bond schemes, as is usual in Hg^{II} polyhedra (Dubler, Beck, Linowsky & Jameson, 1981). We report here the preparation of a mercury(II) selenite containing an ammonia group, and compare the structure of this compound with those determined previously.

The structure of the title compound comprises corrugated chains of HgNO₅ octahedra and SeO₃ pyramids parallel to the *b* axis. The SeO₃ pyramids share edges with two adjacent octahedra, which in turn share an edge with an adjacent octahedron. The Se^{IV}-ion lone pairs protrude into the spaces between the chains. The chains are apparently held in position by three hydrogen bonds (judged by distance): N···O2(*x* + 1, *y* – 1, *z*) [2.873 (9) Å], N···O2(*x* + 1, *y*, *z*) [2.857 (9) Å] and N···O3(2 – *x*, 2 – *y*, 1 – *z*) [2.894 (9) Å]. Thus, the N atom lies in one chain, the two O2 atoms lie in a second chain and O3 lies in a third chain. In (NH₄)₂Se₂O₅, the N···O distances involved in hydrogen bonding are between 2.782 (4) and 2.954 (4) Å (Makarova, Muradyan, Vinogradova & Simonov, 1990). Other N···O distances in the title compound are longer than 3.3 Å.

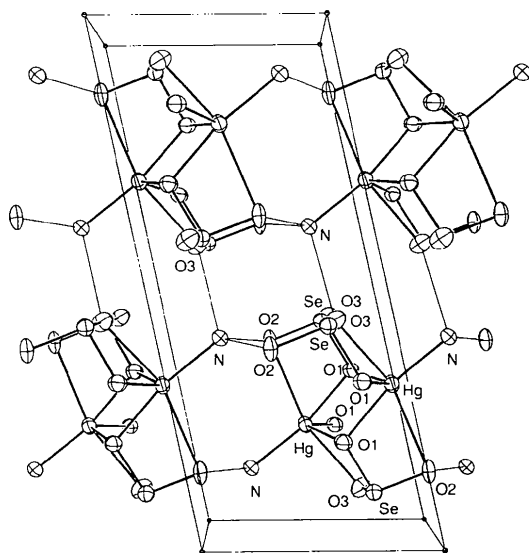


Fig. 1. An ORTEP (Johnson, 1976) drawing of the unit cell with displacement ellipsoids shown at the 50% probability level. The *a* axis is horizontal and *c* axis is angled slightly from the vertical. Thin lines indicate possible hydrogen bonds.

The coordination polyhedron of the Hg^{II} ion is a 2 + 4 distorted octahedron formed by five O atoms and a N atom; the O atoms are contributed by three selenite groups. The polyhedron has two short bonds, Hg—N [2.099 (6) Å] and Hg—O1 [2.129 (5) Å], with an N—Hg—O1 angle of 169.2 (3)°.

An ammine ligand coordinated to an Hg^{II} ion is observed in (NH₃HgN₂S)₂ and [Hg(NH₃)₂I₂]S₄N₄, where the corresponding Hg—N distances are 2.47 (2) and 2.30 (2) Å (Martan & Weiss, 1984). The Hg—N bond in the title compound is markedly shorter. However, in (NH₃HgN₂S)₂, the two other Hg—N bonds arising from the N₂S ligand are 2.092 (2) and 2.098 (24) Å, which are comparable with that in the title compound. The strong hydrogen bonds proposed for [Hg(NH₃)]SeO₃, as indicated by the N···O distances (Brown & Altermatt, 1985), might cause polarization of the ammine ligand and result in this short Hg—N distance.

The four long Hg—O bonds in [Hg(NH₃)]SeO₃ range from 2.496 (6) to 2.809 (6) Å. In the octahedra in HgSeO₃ and Hg₃(HSeO₃)₂(SeO₃)₂, the four long bonds vary from 2.593 (10) to 2.641 (11) Å and from 2.398 (9) to 2.561 (7) Å, respectively. Three of the four long bonds in the title compound [ranging from 2.496 (6) to 2.700 (7) Å] lie within the range observed for the other two selenites, but the longest bond is significantly longer and is close to the limit of what would usually be considered to be a bonding distance.

Each selenite group bridges three Hg^{II} ions. O1 is bonded to three Hg^{II} ions and the other two O atoms are each bonded to one Hg^{II} ion. The selenite group is markedly distorted, with the longest Se—O bond [1.740 (6) Å] involving the O atom which is coordinated to three Hg^{II} ions. The other two bonds [1.657 (5) and 1.678 (6) Å] lie in the normal range for Se—O bonds. The distortion is also expressed in the O—Se—O bond angles, which range from 98.6 (3) to 103.9 (3)°.

Experimental

The title compound was prepared from a suspension of HgO in aqueous selenious acid, which was adjusted to a pH of 8–9 by the addition of excess aqueous ammonia. The compound crystallized at 310–330 K within a few weeks. The presence of ammonia in the structure was confirmed by IR analysis.

Crystal data

[Hg(NH₃)]SeO₃

M_r = 344.58

Monoclinic

*P*2₁/*n*

a = 6.456 (1) Å

b = 4.969 (1) Å

c = 14.658 (2) Å

β = 101.82 (1)°

V = 460.3 (1) Å³

Z = 4

D_x = 4.97 Mg m^{–3}

D_m not measured

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 25 reflections

θ = 16–22°

μ = 41.145 mm^{–1}

T = 294 K

Prism

0.15 × 0.10 × 0.05 mm

Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer

1129 observed reflections
[*I* > 3σ(*I*)]

$\omega/2\theta$ scans	$R_{\text{int}} = 0.052$
Absorption correction:	$\theta_{\text{max}} = 29.98^\circ$
ψ scan (North, Phillips & Mathews, 1968) and spherical	$h = -9 \rightarrow 9$
$T_{\text{min}} = 0.021$, $T_{\text{max}} = 0.091$	$k = 0 \rightarrow 6$
2951 measured reflections	$l = -20 \rightarrow 20$
1480 independent reflections	2 standard reflections
	frequency: 120 min
	intensity decay: 2.1%

Refinement

Refinement on F	$\Delta\rho_{\text{max}} = 3.38 \text{ e } \text{\AA}^{-3}$
$R = 0.029$	$\Delta\rho_{\text{min}} = -1.41 \text{ e } \text{\AA}^{-3}$
$wR = 0.036$	Extinction correction:
$S = 1.192$	isotropic (Zachariasen, 1963)
1129 reflections	Extinction coefficient:
56 parameters	0.65×10^{-6}
H atoms not located	Atomic scattering factors
$w = 1/[\sigma^2(F) + (0.02F)^2 + 1.00]$ (Killean & Lawrence, 1969)	from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV, Tables 2.2B and 2.3.1)
$(\Delta/\sigma)_{\text{max}} = 0.001$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
Hg	0.95584 (4)	0.46626 (6)	0.29788 (2)	0.0232 (1)
Se	0.7220 (1)	1.0075 (1)	0.41717 (5)	0.0230 (3)
O1	0.8099 (9)	0.9399 (11)	0.3150 (4)	0.026 (2)
O2	0.4640 (9)	0.9825 (12)	0.3757 (5)	0.029 (3)
O3	0.7659 (9)	1.3404 (12)	0.4238 (4)	0.033 (3)
N	1.2516 (10)	0.4844 (13)	0.3896 (5)	0.024 (3)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Hg—O1 ¹	2.809 (6)	Hg—N	2.099 (6)
Hg—O1	2.566 (6)	Se—O1	1.740 (6)
Hg—O1 ⁱⁱ	2.129 (5)	Se—O2	1.657 (5)
Hg—O2 ⁱⁱ	2.700 (7)	Se—O3	1.678 (6)
Hg—O3 ¹	2.496 (6)		
O1—Se—O2	98.6 (3)	O2—Se—O3	103.9 (3)
O1—Se—O3	99.2 (3)		

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

As the absorption is high, both ψ -scan (North, Phillips & Mathews, 1968) and spherical absorption corrections were applied (*International Tables for X-ray Crystallography*, 1967, Vol. II, Table 5.3.6B), with $T_{\text{max}} = 0.993$ and $T_{\text{min}} = 0.321$ for the ψ -scan correction, and $T_{\text{max}} = 0.092$ and $T_{\text{min}} = 0.065$ for the spherical correction. The maximum and minimum electron densities in the final difference Fourier map are *ca* 0.8 \AA from Hg.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN PROCESS* (Fair, 1990). Program(s) used to solve structure: direct methods (*MULTAN*; Main *et al.*, 1982). Program(s) used to refine structure: *MolEN LSFM*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *MolEN*.

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: BR1136). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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