

$[Y(HSeO_3)(SeO_3)(H_2O)] \cdot H_2O$

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Key indicators

Single-crystal X-ray study
 $T = 120$ K
Mean $\sigma(\text{Se}-\text{O}) = 0.003$ Å
 R factor = 0.029
 wR factor = 0.053
Data-to-parameter ratio = 16.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, aqua(hydrogen trioxoselenato)(trioxoselenato)yttrium(III) monohydrate, which is isostructural with its samarium(III) and neodymium(III) analogues, contains YO_8 , SeO_3 and $HSeO_3$ coordination polyhedra, which fuse together by corner- and edge-sharing, resulting in a layered structure. A network of $O-H \cdots O$ hydrogen bonds helps to consolidate the crystal packing.

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Comment

The title compound, (I) (Fig. 1), is isostructural with its samarium (Koskenlinna *et al.*, 1994) and neodymium (de Pedro *et al.*, 1994) analogues.

Compound (I) contains both $(SeO_3)^{2-}$ selenite and $(HSeO_3)^-$ hydrogen selenite anions. The unobserved lone pair of electrons of the Se^{IV} species gives rise to the characteristic pyramidal shape of these oxo-anions. As seen previously (Koskenlinna *et al.*, 1994), the Se–OH vertex [1.745 (4) Å] in (I) is longer than the Se–O bonds [mean = 1.690 (15) Å] (Table 1). The Se atoms are displaced from the planes of their three attached oxygen atoms by 0.804 (2) and 0.814 (2) Å for Se1 and Se2, respectively. In terms of bond angles, the angle of the edge-sharing (to Y) O1–Se1–O2 grouping is significantly more acute [92.37 (16)°] than the other O–Se–O sets (mean = 100.4°).

The yttrium cation in (I) is surrounded by eight oxygen atoms, one of which (O7) is part of a water molecule, with a fairly narrow spread of distances [2.258 (3)–2.419 (3) Å];

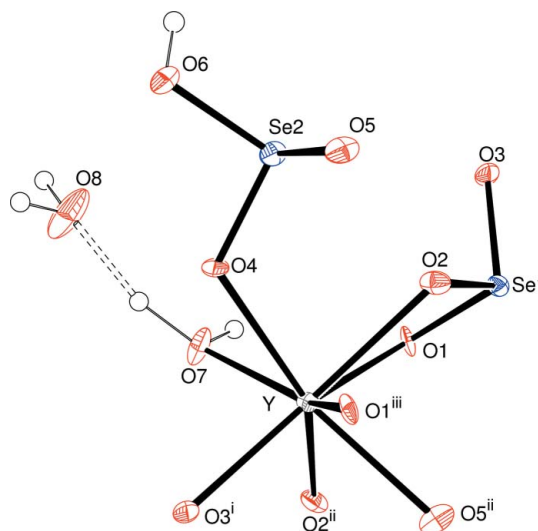


Figure 1

The asymmetric unit of (I) expanded to show the Y atom coordination (70% displacement ellipsoids; spheres of arbitrary radius for the H atoms). Symmetry codes as in Table 1.

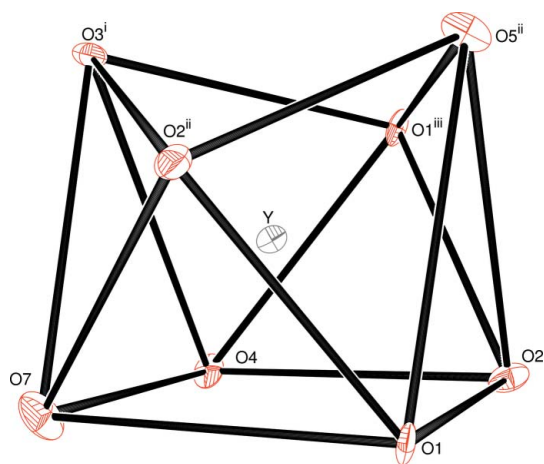


Figure 2
Detail of (I) showing the Y atom coordination with O...O contacts < 3.3 Å shown as lines (50% displacement ellipsoids). Symmetry codes as in Table 1.

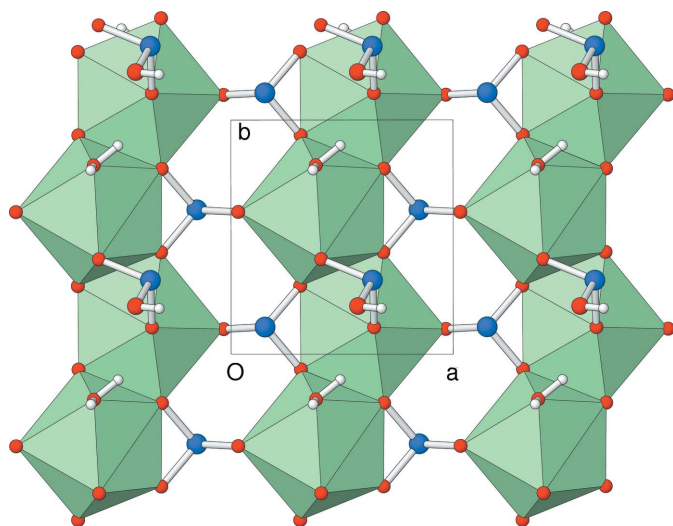


Figure 3
View down [001] of a layer in (I) in polyhedral representation, showing the [010] chains of edge-sharing YO₈ groups cross-linked by the Se1 atoms. Colour key: YO₈ groups green, Se atoms blue, O atoms red, H atoms grey.

mean = 2.36 (5) Å]. The next nearest O atom has a distance of Y—O4ⁱ = 3.872 (3) Å [symmetry code: (i) 1 - x, $\frac{1}{2}$ + y, $\frac{1}{2}$ - z]. The YO₈ grouping could be described as a highly distorted square antiprism (Fig. 2) or possibly as irregular. Atoms O1, O2, O4 and O7 conform well to a square [r.m.s. deviation from the mean plane = 0.041 Å; O1...O4 = 3.915 (5) Å and O2...O7 = 3.988 (5) Å], whereas the nominal O1ⁱⁱⁱ, O2ⁱⁱ, O3ⁱ and O5ⁱⁱ (see Table 1 for symmetry codes) square is grossly distorted [r.m.s. deviation from the mean plane = 0.399 Å; O1ⁱⁱⁱ...O2ⁱⁱ = 4.440 (5) Å and O3ⁱ...O5ⁱⁱ = 3.339 (5) Å]. The Y atom is displaced by 1.3249 (18) Å from the first plane, and 1.1900 (18) Å from the second. The interplanar dihedral angle is 1.8 (2)°. Atoms O3, O4 and O5 are bicoordinate to Y and Se (mean Y—O—Se = 124.2°), whilst O1 and O2 are tricoordinate to one Se and two Y atoms (bond angle sums = 343.5 and

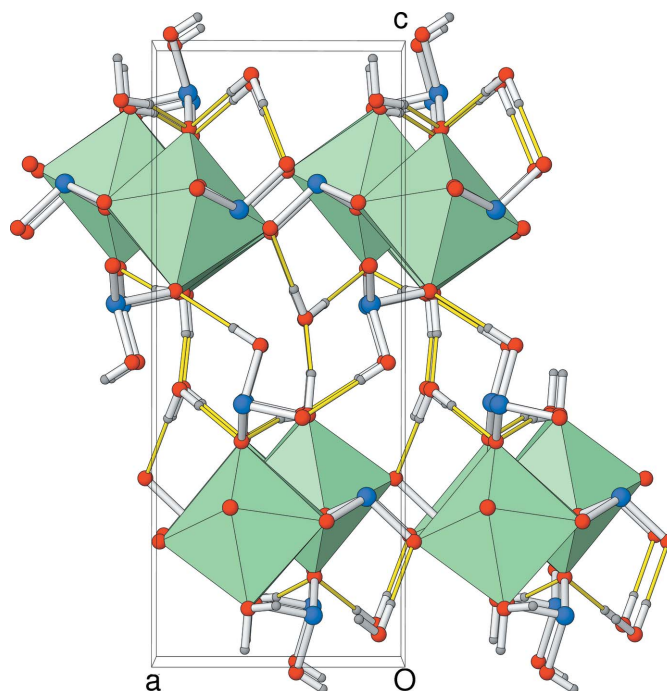


Figure 4
The packing in (I), viewed down [010]. Drawing convention as in Fig. 3, with the H...O portions of the hydrogen bonds highlighted in yellow.

349.0°, respectively). O6 is part of a terminal Se—OH vertex and O7 and O8 are parts of water molecules.

The polyhedral connectivity in (I) (Fig. 3) involves chains of YO₈ groups sharing edges, *via* O1 + O2ⁱⁱ and O1ⁱⁱⁱ + O2 pairs, to result in chains propagating along [010]. The relatively acute O1—Y—O2ⁱⁱ and O1ⁱⁱⁱ—Y—O2 bond angles of 67.81 (12) and 68.02 (11)° respectively, correlate with this polyhedron-fusing role. The Y...Yⁱⁱ separation within the chain is 3.9668 (5) Å. The Y/O chains are cross-linked in the [100] direction by the Se1O₃ groups, involving the edge-sharing motif noted above. Finally, the (HSe2O₃)⁻ groups decorate and reinforce the [010] Y/O chains, resulting in a structure with layered character.

The hydrogen-bonding scheme in (I) involves all the H atoms participating in O—H...O links (Table 2). The Y-bonded water molecule (O7) makes a hydrogen bond to an adjacent YO₈ group in the same sheet (*via* H2) and to the inter-sheet water molecule (*via* H1). The hydrogen selenite anion makes the only direct inter-sheet hydrogen bond (Fig. 4). As well as accepting a hydrogen bond, the non-coordinated water molecule (O8) makes two hydrogen bonds to the same adjacent sheet.

The average metal–oxygen distances in these isostructural phases are Y—O = 2.36 (5) Å, Sm—O = 2.42 Å and Nd—O = 2.45 Å. This pattern is exactly consistent with the differences in the eight-coordinate atomic radii (Shannon, 1976) of Y³⁺ (1.019 Å), Sm³⁺ (1.079 Å) and Nd³⁺ (1.109 Å).

Experimental

A mixture of YCl₃·6H₂O (0.83 g, 2.74 mmol), SeO₂ (0.5 g, 4.5 mmol) and water (10 ml) was sealed in a 23 ml Teflon-lined autoclave and

heated to 433 K for three days, followed by cooling to room temperature over a few hours. Product recovery by vacuum filtration and rinsing with water and acetone led to 0.173 g (16.6% based on Y) of tiny colourless bars and rods of (I).

Crystal data

[Y(HSeO₃)(SeO₃)(H₂O)]·H₂O
M_r = 379.87
 Orthorhombic, *P*2₁2₁2₁
a = 6.5485 (3) Å
b = 6.8987 (2) Å
c = 16.2022 (7) Å
V = 731.95 (5) Å³
Z = 4
D_x = 3.447 Mg m⁻³
 Mo *K*α radiation
 μ = 17.92 mm⁻¹
T = 120 (2) K
 Rod, colourless
 0.14 × 0.03 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
 ω and φ scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 1999)
T_{min} = 0.188, *T_{max}* = 0.716
 6897 measured reflections
 1667 independent reflections
 1523 reflections with *I* > 2σ(*I*)
R_{int} = 0.054
 θ_{max} = 27.5°

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.029
wR(*F*²) = 0.053
S = 1.05
 1667 reflections
 101 parameters
 H-atom parameters constrained
w = 1/[σ²(*F_o*²) + 0.7811*P*]
 where *P* = (*F_o*² + 2*F_c*²)/3
 (Δ/σ)_{max} < 0.001
 Δρ_{max} = 0.63 e Å⁻³
 Δρ_{min} = -0.72 e Å⁻³
 Absolute structure: Flack (1983),
 664 Friedel pairs
 Flack parameter: 0.646 (11)

Table 1

Selected geometric parameters (Å, °).

Y—O3 ⁱ	2.258 (3)	Y—O1	2.419 (3)
Y—O7	2.346 (3)	Se1—O3	1.680 (3)
Y—O5 ⁱⁱ	2.347 (3)	Se1—O1	1.701 (3)
Y—O4	2.367 (3)	Se1—O2	1.710 (3)
Y—O2 ⁱⁱ	2.372 (3)	Se2—O4	1.672 (3)
Y—O1 ⁱⁱⁱ	2.376 (4)	Se2—O5	1.689 (3)
Y—O2	2.402 (4)	Se2—O6	1.745 (4)
Se1—O1—Y ⁱⁱ	130.66 (18)	Y ⁱⁱⁱ —O2—Y	112.41 (13)
Se1—O1—Y	101.19 (15)	Se1—O3—Y ^{iv}	130.39 (19)
Y ⁱⁱ —O1—Y	111.66 (13)	Se2—O4—Y	117.18 (17)
Se1—O2—Y ⁱⁱⁱ	135.03 (18)	Se2—O5—Y ⁱⁱⁱ	124.96 (17)
Se1—O2—Y	101.58 (15)		

Symmetry codes: (i) *x* - 1, *y*, *z*; (ii) -*x* + 1, *y* + ½, -*z* + ½; (iii) -*x* + 1, *y* - ½, -*z* + ½; (iv) *x* + 1, *y*, *z*.

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O6—H1···O4 ^v	0.82	1.92	2.712 (5)	164
O7—H2···O5 ^{vi}	0.92	1.93	2.806 (5)	159
O7—H3···O8	0.86	1.82	2.644 (5)	163
O8—H4···O3 ^{vii}	0.94	1.93	2.874 (5)	179
O8—H5···O5 ^{viii}	0.93	2.03	2.964 (5)	179

Symmetry codes: (v) *x* + ½, -*y* + ½, -*z* + 1; (vi) *x*, *y* + 1, *z*; (vii) *x* - ½, -*y* + ½, -*z* + 1; (viii) *x* - ½, -*y* + ½, -*z* + 1.

The crystal studied was an inversion twin with volume fractions of 0.354 (11):0.646 (11) for the component reported in the tables and its enantiomer, respectively. All the H atoms were located in difference maps and refined as riding in their as-found relative positions, with *U*_{iso}(H) = 1.2*U*_{eq}(carrier).

Data collection: COLLECT (Nonius, 1998); cell refinement: SCALEPACK (Otwinowski & Minor, 1997); data reduction: SCALEPACK and DENZO (Otwinowski & Minor, 1997), and SORTAV (Blessing, 1995); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997) and ATOMS (Shape Software, 2005); software used to prepare material for publication: SHELXL97.

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