chose the space group *I4/mmm* for Cs$_2$Au$_2$I$_6$. Fourteen reflections influenced by extinction (extinction factor < 0.90) were not included in the final refinement. In the final difference map, the largest positive and negative peaks are near the Au atoms.

Programs used were *UNICS*III (Sakurai & Kobayashi, 1979) and *SHELX*76 (Sheldrick, 1976) for the structure analysis, *RADIEL* (Coppens *et al.*, 1979) of *CHARGE SYSTEM* (Ito, Ooba, Tanaka, Sasaki & Haga, 1986) for the extinction correction, and *DABEX* (Toriumi & Ooba, 1985) of *CHARGE SYSTEM* for the absorption correction, on a HITAC M-880/310 at the Computer Centre of the University of Tokyo and a HITAC M-680H at the Computer Center, Institute for Molecular Science, Okazaki. *ORTEP*II (Johnson, 1976) was used for drawing the structure on a TOSHIBA SS-R590 personal computer.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: OA1010). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A New Acid Selenite: Mg(HSeO$_3$)$_2$

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Abstract

Magnesium bis[hydrogentrioxoselenate(IV)] crystallizes in a new structure type with Mg^{2+} and hydrogen-bonded dimers of trigonal pyramidal SeO$_2$OH$^-$ ions in an NaCl-like arrangement forming MgO$_6$ octahedra linked *via* O—Se—O bonds to create a three-dimensional network structure. The Mg—O and Se—O distances reflect the bonding schemes of the corresponding atoms. The hydrogen bonds in the (SeO$_2$OH$^-$)$_2$ dimers are strong, with $d(\mathrm{OH}\cdots\mathrm{O}) = 2.616\,(2)$ Å. They are joined through Se and Mg atoms to form layered hydrogen-bond systems parallel to (100).

Comment

As a part of our investigations of strong hydrogen bonds in acid selenites (Unterderweide, Engelen & Boldt, 1994; Engelen, Boldt, Unterderweide & Bäumer, 1995), single crystals of the hitherto unknown compound Mg(HSeO$_3$)$_2$ were prepared and the crystal structure was determined by means of X-ray diffraction measurements (Unterderweide, Boldt & Engelen, 1993; Boldt, 1994).

The Mg^{2+} ions, occupying the 2*a* positions of space group *P*2$_1$/*n*, are coordinated octahedrally by six O

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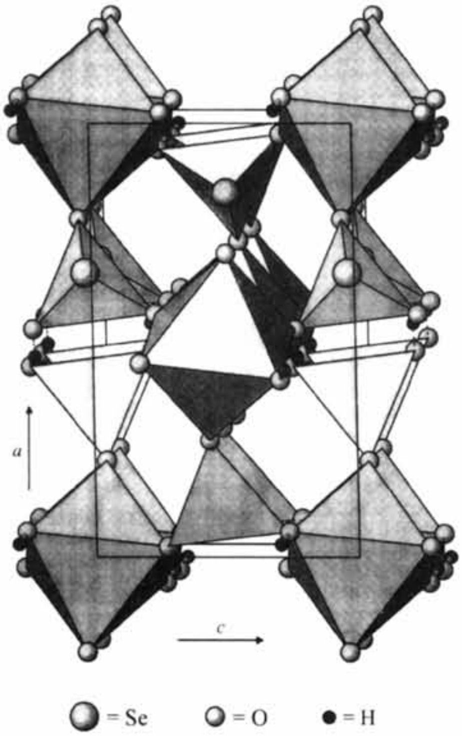

Fig. 1. The crystal structure of $\mathrm{Mg(HSeO_3)_2}$ viewed along [010]. The dashed lines indicate hydrogen bonds.

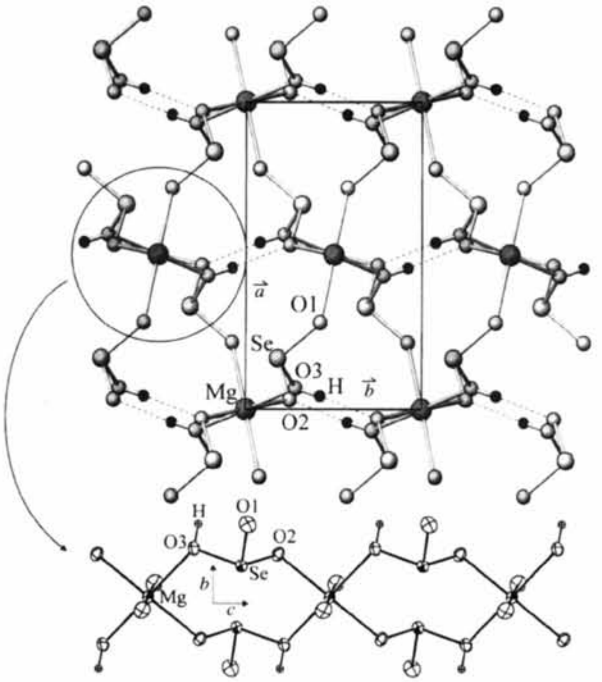

Fig. 2. The crystal structure of $\mathrm{Mg(HSeO_3)_2}$ projected along [001] showing the $[\mathrm{Mg(SeO_2OH)_4^{2-}}]^1_\infty$ chains, the $(\mathrm{SeO_2OH^-})_2$ dimers and the hydrogen-bond system (dashed lines). An *ORTEP*II (Johnson, 1971) plot of the $[\mathrm{Mg(SeO_2OH)_4^{2-}}]^1_\infty$ chains projected along [100] with displacement ellipsoids at the 48% probability level is also shown.

atoms, including two OH groups in *trans* positions (Fig. 1). The $\mathrm{MgO_6}$ octahedra are linked through the Se atoms of trigonal pyramidal hydrogen selenite ions of constitution $\mathrm{SeO_2OH^-}$ to form a three-dimensional network structure (Fig. 1). Within this network, $[\mathrm{Mg(SeO_2OH)_4}^{2-}]^1_\infty$ chains along [001] appear, which consist of $\mathrm{MgO_6}$ octahedra each being *cis* connected *via* Se—O bonds (Fig. 2). Between the chains there are strong, almost linear hydrogen bonds connecting the $\mathrm{SeO_2OH^-}$ ions to form cyclic dimers around the 2*d* positions (Fig. 2). Similar dimers are also found in other acid selenites of divalent metals (Boldt, 1994; Effenberger, 1985) or in ferroelectric $\mathrm{Li(H/D)_3(SeO_3)_2}$ (Liminga & Tellgren, 1982). The $\mathrm{Mg^{2+}}$ ions and the $(\mathrm{SeO_2OH^-})_2$ dimers exhibit an NaCl-like arrangement with the pseudo-cubic lattice vectors $\mathbf{a}' = \mathbf{a}$, $\mathbf{b}' = \mathbf{b} - \mathbf{c}$, $\mathbf{c}' = \mathbf{b} + \mathbf{c}$.

The coordination of the O atoms is either twofold bent by Se and Mg (O1) or trigonal by Se, Mg and H (O2 and O3). Due to the differences in the coordination of the O atoms and the anisotropic three-dimensional connection, the $\mathrm{MgO_6}$ octahedra and the $\mathrm{SeO_2OH^-}$ ions are distorted. The longer Mg—O and Se—O distances are those with the O atoms involved in the OH···O bond system (Table 2).

Experimental

The crystals were synthesized by slow evaporation of an aqueous solution of $\mathrm{H_2SeO_3}$ and $\mathrm{MgCO_3}$ at 370 K (Unterderweide, 1995).

Crystal data

$\mathrm{Mg(HSeO_3)_2}$
$M_r = 280.25$
Monoclinic
$P2_1/n$
$a = 9.091\ (1)$ Å
$b = 5.231\ (1)$ Å
$c = 5.570\ (1)$ Å
$\beta = 90.94\ (1)°$
$V = 264.85\ (8)$ Å^3
$Z = 2$
$D_x = 3.514$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 56 reflections
$\theta = 9.2–35.3°$
$\mu = 14.032$ mm^{-1}
$T = 293\ (2)$ K
Needle
$0.18 \times 0.10 \times 0.07$ mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968) and spherical (Bond, 1959)
$T_{\min} = 0.24$, $T_{\max} = 0.34$
2980 measured reflections
2180 independent reflections

1837 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.0160$
$\theta_{\max} = 44.93°$
$h = -18 \rightarrow 18$
$k = -1 \rightarrow 10$
$l = -1 \rightarrow 11$
3 standard reflections
frequency: 166.7 min
intensity decay: 1.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.056$
$S = 1.184$
2180 reflections
48 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.0136P)^2 + 0.4419P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.001$
$\Delta\rho_{\max} = 1.072$ e Å^{-3}
$\Delta\rho_{\min} = -0.804$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0038 (9)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)*

$U_{\rm iso}$ for H, $U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U^{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$ for all others.

	x	y	z	$U_{\rm eq}/U_{\rm iso}$
Mg	0	0	0	0.01306 (12)
Se	0.16547 (2)	0.18155 (3)	0.49075 (3)	0.01358 (4)
O1	0.28165 (13)	0.4209 (3)	0.4595 (3)	0.0225 (2)
O2	0.03231 (13)	0.2497 (3)	0.2852 (2)	0.0169 (2)
O3	0.06773 (15)	0.2828 (2)	0.7501 (2)	0.0187 (2)
H	0.038 (4)	0.425 (7)	0.725 (6)	0.041 (9)

Table 2. *Selected geometric parameters (Å, °)*

Mg—O1$^{\rm i}$	2.0366 (12)	Se—O1	1.6490 (13)
Mg—O1$^{\rm ii}$	2.0366 (12)	Se—O2	1.6906 (12)
Mg—O2	2.0738 (12)	Se—O3	1.7883 (12)
Mg—O2$^{\rm iii}$	2.0738 (12)	O3···O2$^{\rm vi}$	2.616 (2)
Mg—O3$^{\rm iv}$	2.1291 (13)	O3—H	0.80 (4)
Mg—O3$^{\rm v}$	2.1291 (13)	H···O2$^{\rm vi}$	1.82 (4)
O1—Se—O2	102.78 (7)	O2—Se—O3	97.17 (6)
O1—Se—O3	100.84 (7)	O3—H···O2$^{\rm vi}$	172 (4)

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

Lattice parameters were determined from X-ray Guinier powder photographs (Cu $K\alpha_1$ radiation, α-quartz as internal standard) using a Huber–Guinier system 600 and the refinement program *LSUCR* (Evans, Appleman & Handwerker, 1963). The atomic positions were obtained from a Patterson map (Se) and successive least-squares refinements and ΔF syntheses (Mg, O, H).

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *LSUCR*. Data reduction: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SDP* (B. A. Frenz & Associates Inc., 1988). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1971), *ATOMS* (Dowty, 1995). Software used to prepare material for publication: *SHELXL*93.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: BR1165). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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$SmMo_8O_{14}$

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Abstract

The crystal structure of samarium octamolybdate is characterized by layers of *cis*-edge-sharing bi-face-capped octahedral Mo_8 clusters parallel to the *bc* plane of the orthorhombic unit cell. The arrangement of the O atoms is derived from the close-packing layer sequence *ABAC*. The Mo—Mo distances range from 2.5959 (5) to 2.8413 (6) Å within the Mo_8 clusters, with a mean value of 2.731 Å. The shortest Mo—Mo distance between the Mo_8 clusters within the same layer is 3.0672 (5) Å, and between clusters of adjacent layers 3.6342 (5) Å. The Mo—O distances lie between 1.949 (3) and 2.163 (3) Å (mean value 2.050 Å). The Sm^{3+} ion lies away from the centre of a distorted cuboctahedron with Sm—O distances ranging from 2.316 (4) to 3.159 (3) Å.

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