O1	0.7814 (2)	0.0406 (2)	−0.3014 (2)	0.0109 (3)
O2	0.6877 (2)	0.1309 (2)	0.1833 (2)	0.0122 (3)
O3	0.7135 (2)	0.3934 (2)	0.2846 (2)	0.0110 (3)
O4	0.7623 (2)	0.0184 (2)	−0.0121 (2)	0.0101 (3)
O5	1.0549 (2)	−0.0931 (2)	−0.1988 (2)	0.0105 (3)
O6	0.7375 (2)	0.1688 (2)	0.4751 (2)	0.0121 (3)
O7	0.5007 (2)	0.3587 (2)	0.4403 (2)	0.0087 (3)
O8	0.4451 (2)	0.2718 (2)	0.1578 (2)	0.0126 (3)
O9	0.7952 (2)	−0.2160 (2)	−0.1955 (2)	0.0124 (3)
O*W*1	0.4941 (2)	0.0534 (2)	0.3443 (2)	0.0203 (4)
O*W*2	0.6809 (3)	−0.1468 (2)	0.4654 (2)	0.0253 (4)
N	0.5103 (3)	0.1894 (3)	−0.1031 (3)	0.0210 (5)

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

Fe1—O6	1.940 (2)	Fe2—O7^{i}	2.162 (2)
Fe1—O2	1.951 (2)	Fe2—O7^{ii}	2.165 (2)
Fe1—O8	1.957 (2)	P1—O2	1.517 (2)
Fe1—O3	1.976 (2)	P1—O5^{iii}	1.543 (2)
Fe1—O*W*1	2.091 (2)	P1—O3^{ii}	1.543 (2)
Fe1—O7	2.153 (2)	P1—O4	1.551 (2)
Fe2—O9	1.936 (2)	P2—O6^{iv}	1.522 (2)
Fe2—O1	1.963 (2)	P2—O8^{i}	1.530 (2)
Fe2—O4	1.966 (2)	P2—O9^{v}	1.532 (2)
Fe2—O5	1.975 (2)	P2—O1	1.552 (2)
O6—Fe1—O2	93.64 (7)	O9—Fe2—O7^{i}	176.58 (7)
O6—Fe1—O8	170.68 (8)	O1—Fe2—O7^{i}	93.32 (7)
O2—Fe1—O8	87.94 (7)	O4—Fe2—O7^{i}	83.84 (7)
O6—Fe1—O3	93.83 (8)	O5—Fe2—O7^{i}	84.14 (7)
O2—Fe1—O3	88.33 (7)	O9—Fe2—O7^{ii}	91.49 (7)
O8—Fe1—O3	95.40 (8)	O1—Fe2—O7^{ii}	178.76 (7)
O6—Fe1—O*W*1	83.97 (8)	O4—Fe2—O7^{ii}	83.64 (7)
O2—Fe1—O*W*1	89.13 (9)	O5—Fe2—O7^{ii}	83.69 (7)
O8—Fe1—O*W*1	86.87 (8)	O7^{i}—Fe2—O7^{ii}	87.59 (7)
O3—Fe1—O*W*1	176.54 (8)	O2—P1—O5^{iii}	111.32 (10)
O6—Fe1—O7	91.34 (7)	O2—P1—O3^{ii}	109.68 (10)
O2—Fe1—O7	174.12 (7)	O5^{iii}—P1—O3^{ii}	109.48 (10)
O8—Fe1—O7	87.64 (7)	O2—P1—O4	107.09 (10)
O3—Fe1—O7	88.21 (7)	O5^{iii}—P1—O4	110.15 (10)
O*W*1—Fe1—O7	94.51 (8)	O3^{ii}—P1—O4	109.08 (10)
O9—Fe2—O1	87.65 (7)	O6^{iv}—P2—O8^{i}	108.20 (10)
O9—Fe2—O4	99.34 (8)	O6^{iv}—P2—O9^{v}	111.86 (10)
O1—Fe2—O4	95.62 (7)	O8^{i}—P2—O9^{v}	111.33 (11)
O9—Fe2—O5	92.49 (8)	O6^{iv}—P2—O1	108.04 (10)
O1—Fe2—O5	97.23 (7)	O8^{i}—P2—O1	109.98 (10)
O4—Fe2—O5	162.86 (7)	O9^{v}—P2—O1	107.37 (10)

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

Data collection and cell refinement: *DIF*4 (Stoe & Cie, 1988*a*). Data reduction: *REDU*4 (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990), option *TREF*. Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993).

The authors thank Dr R. Retoux, Université du Maine, for his help in data collection and microprobe analysis.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: DU1089). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Cadmium Diselenite and Cadmium Hydrogenselenite Selenite

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Abstract

Cadmium diselenite, $CdSe_2O_5$, consists of an octahedrally coordinated Cd atom and a diselenite group that acts as a bidentate ligand. Cadmium hydrogenselenite selenite, $Cd_3(HSeO_3)_2(SeO_3)_2$, has seven- and six-coordinated Cd atoms and two different selenite groups: hydrogenselenite and selenite.

Comment

The crystal structures of cadmium selenites are almost unknown. Before this year the only published structure was that of cadmium hydrogenselenite nitrate (Leskelä, Valkonen & Leskelä, 1984). We have now synthesized and solved the structures of seven different cadmium selenites: $CdSeO_3$(*oP*20), $CdSeO_3$(*mP*40), $4CdSeO_3.3H_2O$, $CdSe_2O_5$, $Cd_3(HSeO_3)_2(SeO_3)_2$, $(NH_4)_2Cd(SeO_3)_2$ and $Cd(NH_3)SeO_3$. [The Pearson notation is used for phase nomenclature: *oP*20 and *mP*40 (IUPAC, 1990)]. The structures of the first three of these compounds have been published recently (Valkonen, 1994), the next two are discussed in this paper and the final two structures will be reported shortly.

The structures of both of the title compounds comprise three-dimensional networks in which cadmium polyhedra are connected together. In $CdSe_2O_5$, the octahedra share edges only [O(2)—O(2)] but in $Cd_3(HSeO_3)_2(SeO_3)_2$ there are common edges and corners. Unit cells are shown in Figs. 1 and 2 together with the atomic numbering.

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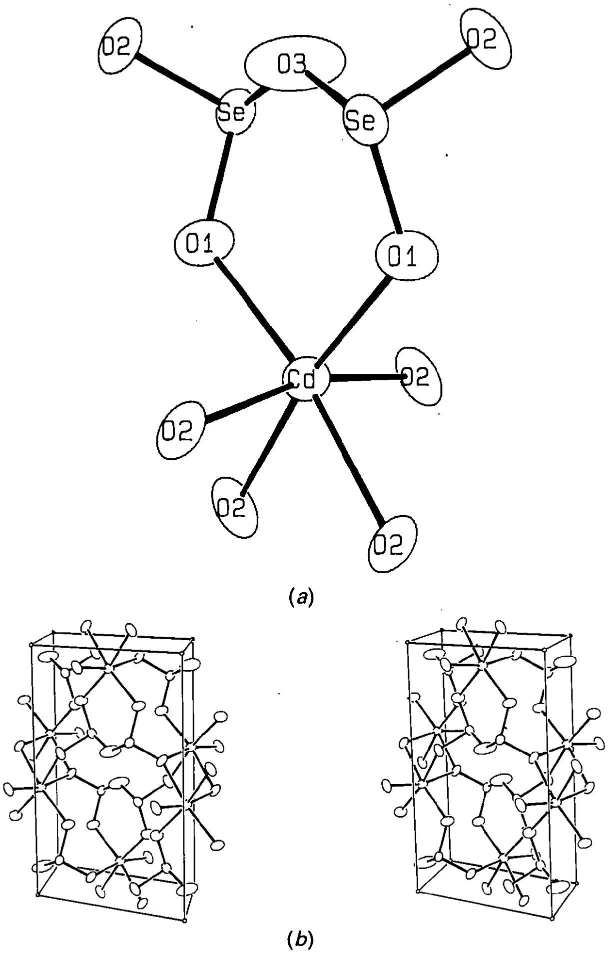
The diselenite group in $CdSe_2O_5$ has an Se—O—Se group in which the Se—O distance [1.801 (2) Å] is elongated relative to the other Se—O bonds. The bond angle Se—O(3)—Se [140.3 (3)°] is typical of diselenites. The diselenite group connects together five Cd atoms, acting as a bidentate ligand and bridge. The geometry around Cd is slightly distorted octahedral; the angles vary between 166.3 (1) and 171.2 (1)°, and 76.2 (1) and 98.0 (1)°.

$Cd_3(HSeO_3)_2(SeO_3)_2$ has two different anions: hydrogenselenite and selenite. Se(1) is seen to form the hydrogenselenite group because one Se—O distance, Se(1)—O(3), is elongated [1.750 (7) Å] relative to the other Se—O bonds. This stretching is caused by the H atom. O(3) is not coordinated to the Cd atom. Both hydrogenselenite and selenite act as bidentate and bridging ligands.

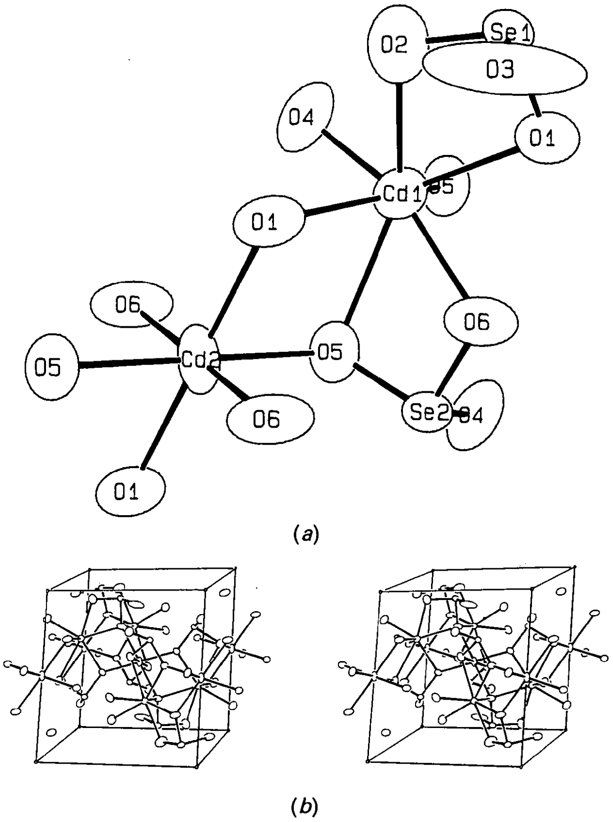
There are two crystallographically different Cd atoms in $Cd_3(HSeO_3)_2(SeO_3)_2$: Cd(1) is seven-coordinated and Cd(2) six-coordinated. The polyhedron around Cd(1) is irregular: one Cd—O distance is long [2.647 (5) Å] and the O—Cd(1)—O angles vary between 62.0 (2) and 151.7 (2)°. The geometry around Cd(2) is slightly distorted octahedral.



(*a*)

(*b*)

Fig. 1. *ORTEP* (Johnson, 1976) drawings of $CdSe_2O_5$ showing (*a*) the atomic numbering scheme and (*b*) a stereoview of the unit cell (*a* axis horizontal, *b* axis vertical). Probability ellipsoids are drawn at the 75% level.



(*a*)

(*b*)

Fig. 2. *ORTEP* (Johnson, 1976) drawings of $Cd_3(HSeO_3)_2(SeO_3)_2$ showing (*a*) the atomic numbering scheme and (*b*) a stereoview of the unit cell (*a* axis vertical, *b* axis horizontal). Probability ellipsoids are drawn at the 75% level.

Experimental

Both compounds were crystallized from an aqueous solution containing cadmium acetate and selenious acid. For cadmium diselenite, selenious acid was present in excess.

$CdSe_2O_5$

Crystal data

$CdSe_2O_5$
$M_r = 350.32$
Monoclinic
$C2/c$
$a = 8.024\ (3)$ Å
$b = 11.319\ (1)$ Å
$c = 6.020\ (1)$ Å
$\beta = 119.38\ (2)°$
$V = 476.5\ (2)$ Å^3
$Z = 4$
$D_x = 4.88$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 7.77–13.96°$
$\mu = 19.62$ mm^{-1}
$T = 298$ K
Prism
0.3 × 0.2 × 0.2 mm
White

Data collection

Enarf–Nonius CAD-4 diffractometer
$2\theta/\omega$ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.81$, $T_{\max} = 1.32$
1756 measured reflections
1756 independent reflections
1252 observed reflections [$I > 3\sigma(I)$]
$\theta_{\max} = 40°$
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 20$
$l = -10 \rightarrow 9$
3 standard reflections frequency: 30 min intensity variation: <2%

Refinement

Refinement on F
$R = 0.028$
$wR = 0.033$
$S = 2.28$
1252 reflections
39 parameters
Unit weights applied
$(\Delta/\sigma)_{\max} = 0.005$
$\Delta\rho_{\max} = 1.93$ e Å^{-3}
$\Delta\rho_{\min} = -1.74$ e Å^{-3}
Extinction correction: $|F_c| = |F_o|(1 + gI_c)$ (Stout & Jensen, 1968)
Extinction coefficient: $g = 1.8 \times 10^{-8}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* $CdSe_2O_5$

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Cd	0	0.58867 (3)	1/4	0.00284 (3)
Se	0.14441 (5)	0.14053 (3)	0.61859 (6)	0.00314 (3)
O(1)	0.1978 (4)	0.2774 (3)	0.7229 (6)	0.0045 (3)
O(2)	0.3396 (4)	0.0647 (3)	0.8175 (5)	0.0043 (3)
O(3)	0	0.0865 (5)	3/4	0.0111 (6)

Table 2. *Selected bond lengths* (Å) *for* $CdSe_2O_5$

Cd—O(1$^{\rm i}$)	2.258 (3)	Se—O(1)	1.647 (3)
Cd—O(2$^{\rm ii}$)	2.285 (3)	Se—O(2)	1.668 (3)
Cd—O(2$^{\rm iii}$)	2.313 (3)	Se—O(3)	1.801 (2)

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

$\mathbf{Cd_3(HSeO_3)_2(SeO_3)_2}$

Crystal data

$Cd_3(HSeO_3)_2(SeO_3)_2$
$M_r = 847.05$
Monoclinic
$P2_1/c$
$a = 9.405$ (1) Å
$b = 9.147$ (2) Å
$c = 7.284$ (1) Å
$\beta = 112.62$ (1)°
$V = 578.5$ (1) Å^3
$Z = 2$
$D_x = 4.86$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 7.88–13.66°$
$\mu = 17.97$ mm^{-1}
$T = 298$ K
Prism
0.3 × 0.2 × 0.2 mm
White

Data collection

Enraf–Nonius CAD-4 diffractometer
$2\theta/\omega$ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.89$, $T_{\max} = 1.21$
3743 measured reflections
3743 independent reflections
2579 observed reflections [$I > 3\sigma(I)$]
$\theta_{\max} = 40°$
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 16$
$l = -13 \rightarrow 12$
3 standard reflections frequency: 60 min intensity variation: <2%

Refinement

Refinement on F
$R = 0.035$
$wR = 0.046$
$S = 2.83$
2579 reflections
89 parameters
H atoms could not be located
Unit weights applied
$(\Delta/\sigma)_{\max} = 0.005$
$\Delta\rho_{\max} = 2.92$ e Å^{-3}
$\Delta\rho_{\min} = -2.43$ e Å^{-3}
Extinction correction: $|F_c| = |F_o|(1 + gI_c)$ (Stout & Jensen, 1968)
Extinction coefficient: $g = 1.13 \times 10^{-8}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* $Cd_3(HSeO_3)_2(SeO_3)_2$

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Cd(1)	0.65636 (4)	0.36405 (5)	0.12044 (6)	0.00235 (3)
Cd(2)	1/2	0	0	0.00240 (5)
Se(1)	0.89877 (6)	0.37730 (6)	0.60135 (8)	0.00204 (5)
Se(2)	0.70337 (6)	0.66291 (6)	−0.04285 (8)	0.00195 (4)
O(1)	0.7058 (5)	0.3598 (6)	0.4617 (6)	0.0034 (4)
O(2)	0.9133 (6)	0.2268 (6)	0.7356 (8)	0.0052 (6)
O(3)	0.8829 (6)	0.5171 (7)	0.7564 (9)	0.0083 (6)
O(4)	0.7848 (5)	0.5805 (5)	0.1799 (7)	0.0034 (5)
O(5)	0.5866 (5)	0.5184 (5)	−0.1621 (6)	0.0029 (4)
O(6)	0.5624 (5)	0.7672 (5)	−0.0163 (7)	0.0036 (4)

Table 4. *Selected bond lengths* (Å) *for* $Cd_3(HSeO_3)_2(SeO_3)_2$

Cd(1)—O(1)	2.347 (5)	Cd(2)—O(5$^{\rm iii}$)	2.282 (4)
Cd(1)—O(1$^{\rm i}$)	2.482 (5)	Cd(2)—O(6$^{\rm ii}$)	2.224 (5)
Cd(1)—O(2$^{\rm i}$)	2.382 (5)	Se(1)—O(1)	1.713 (4)
Cd(1)—O(4)	2.272 (5)	Se(1)—O(2)	1.663 (6)
Cd(1)—O(5)	2.371 (4)	Se(1)—O(3)	1.750 (7)
Cd(1)—O(5$^{\rm ii}$)	2.647 (5)	Se(2)—O(4)	1.683 (4)
Cd(1)—O(6$^{\rm ii}$)	2.247 (4)	Se(2)—O(5)	1.725 (4)
Cd(2)—O(1$^{\rm i}$)	2.425 (5)	Se(2)—O(6)	1.703 (5)

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

Data collection was carried out using CAD-4 software. Data reduction and other calculations were performed using the *MolEN* (Fair, 1990) package. Lorentz and polarization corrections were applied to the data. Cd and Se atoms were located by direct methods using the program *MULTAN* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). The figures were drawn with *ORTEP* (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: AB1079). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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$Sm(HSeO_3)(SeO_3).2H_2O$

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Abstract

The structure of samarium hydrogenselenite selenite dihydrate is characterized by layers parallel to the *xy* plane consisting of SmO_8 polyhedra linked by selenite and hydrogenselenite ions. A selenite ion links one Sm atom to three other Sm atoms while the hydrogenselenite ion acts as a monodentate bridging ligand to two Sm atoms. The layers are held together by hydrogen bonds and van der Waals forces. The coordination polyhedron of Sm is a distorted square antiprism.

Comment

Several different crystalline phases in the system Ln–SeO_2 (where Ln is a lanthanide) have been structurally characterized. The unit-cell parameters and powder diffraction data for a compound with the formula $LnH(SeO_3)_2.2.5H_2O$ have been reported previously (Immonen, Koskenlinna, Niinistö & Pakkanen, 1976). Several single-crystal structures of this type of compound, *e.g.* $PrH_3(SeO_3)_2(Se_2O_5)$ (Koskenlinna & Valkonen, 1977*a*), $Sc(HSeO_3)_3$ (Valkonen & Leskelä, 1978), $CeSe_2O_6$ (Delage, Carpy, H'Naïfi & Goursolle, 1986) and $LaHSe_2O_6$ (Morris, Harrison, Stucky & Cheetham, 1992), or with an alkali metal added to the system, *e.g.* $NaLa(SeO_3)_2$ (Morris, Hriljac & Cheetham, 1990), have been reported also. In addition, anions other than selenite can be incorporated in the structures as in the compounds $Y(Se_2O_5)NO_3.3H_2O$ (Valkonen & Ylinen, 1979) and $Er(SeO_3)(SeO_4).0.5H_2O$ (Morris, Wilkinson & Cheetham, 1992). The structure of the title compound has remained unsolved until now because of the severe twinning and multiple twinning of the crystals. The tendency for the crystals to be twinned may be the reason why the unit cell reported here differs from that reported earlier (Immonen, Koskenlinna, Niinistö & Pakkanen, 1976).

The title compound contains both selenite and hydrogenselenite ions. The bond distances in the selenite ion [1.680 (13)–1.717 (14) Å] are normal compared to those in other lanthanide selenites. The hydrogenselenite ion has one elongated Se—O bond [1.75 (2) Å] due to the bonded H atom. This bond is slightly shorter in the title compound than that in $LaHSe_2O_6$ (1.783 Å; Morris, Harrison, Stucky & Cheetham, 1992) or in $PrH_3(SeO_3)_2(Se_2O_5)$ (Koskenlinna & Valkonen, 1977*a*), in which they vary between 1.766 and 1.805 Å.

The hydrogenselenite ion acts as a bridge between two Sm atoms. The selenite ion bridges four Sm atoms through monodentate coordination by O6 to one Sm atom, bidentate coordination by O1 and O5 to a second Sm atom and the coordination of O1 and O5 to two other Sm atoms. Thus the three-coordinate O atoms O1 and O5 are bonded to Se1 and two Sm atoms. Each $OSeSm_2$ group is tetrahedral in shape with an O atom at the apex. The angles Sm–HO–Se vary between 100.2 (6) and 132.9 (7)° for O5 and 101.8 (7) and 136.4 (8)° for O1. Three-coordinate O atoms are not uncommon in selenite compounds and are found in the structures of $PrH_3(SeO_3)_2(Se_2O_5)$ (Koskenlinna & Valkonen, 1977*a*), $LaHSe_2O_6$ (Morris, Harrison, Stucky & Cheetham, 1992), $MnSeO_3.D_2O$ (Koskenlinna & Valkonen, 1977*b*) and Ag_2SeO_3 (Okkonen, Hiltunen, Koskenlinna & Niinistö, 1994). In $MnSeO_3.D_2O$ (Koskenlinna

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