Tableau 2. *Paramètres géométriques* (Å, °)

Mg(1)—O(1)	2,121 (3)	Mg(3)—O(7^{iv})	2,031 (3)
Mg(1)—O(2^{i})	2,131 (3)	Mg(3)—O(7^{vii})	2,031 (3)
Mg(1)—O(3^{ii})	1,988 (3)	Na(1)—O(1^{viii})	2,587 (4)
Mg(1)—O(4^{iii})	2,046 (4)	Na(1)—O(1^{iii})	2,534 (3)
Mg(1)—O(6^{iii})	2,047 (3)	Na(1)—O(2^{iv})	2,627 (3)
Mg(1)—O(8^{iv})	2,278 (4)	Na(1)—O(2)	2,551 (3)
Mg(2)—O(1^{iii})	2,047 (3)	Na(1)—O(3^{ix})	2,845 (3)
Mg(2)—O(3)	2,038 (4)	Na(1)—O(4^{ix})	2,539 (4)
Mg(2)—O(5^{iii})	1,900 (4)	Na(1)—O(7^{iii})	2,427 (4)
Mg(2)—O(6)	2,078 (3)	Na(1)—O(8^{iii})	2,398 (3)
Mg(2)—O(8^{iii})	2,066 (3)	P(1)—O(3^{x})	1,555 (3)
Mg(3)—O(2^{iii})	2,112 (3)	P(1)—O(4^{x})	1,551 (3)
Mg(3)—O(2^{v})	2,112 (3)	P(1)—O(6^{iii})	1,528 (2)
Mg(3)—O(4^{i})	2,218 (3)	P(1)—O(7^{iii})	1,517 (4)
Mg(3)—O(4^{vi})	2,218 (3)		
O(3^{x})—P(1)—O(4^{x})	107,1 (2)	O(2^{iii})—P(2)—O(5^{iii})	109,9 (2)
O(3^{x})—P(1)—O(6^{iii})	108,5 (2)	O(2^{iii})—P(2)—O(8^{iv})	112,0 (1)
O(3^{x})—P(1)—O(7^{iii})	107,9 (2)	O(5^{iii})—P(2)—O(8^{iv})	114,2 (2)
O(4^{x})—P(1)—O(6^{iii})	110,7 (2)	O(1)—P(2)—O(2^{iii})	106,9 (2)
O(4^{x})—P(1)—O(7^{iii})	110,2 (2)	O(1)—P(2)—O(5^{iii})	108,8 (2)
O(6^{iii})—P(1)—O(7^{iii})	112,3 (2)	O(1)—P(2)—O(8^{iv})	104,8 (2)
O(1)—P(2)—O(2^{iii})	106,9 (2)	O(2^{iii})—P(2)—O(5^{iii})	109,9 (2)
O(1)—P(2)—O(5^{iii})	108,8 (2)	O(2^{iii})—P(2)—O(8^{iv})	112,0 (1)
O(1)—P(2)—O(8^{iv})	104,8 (2)	O(5^{iii})—P(2)—O(8^{iv})	114,2 (2)

Codes de symétrie: (i) $x-1, y, z-1$; (ii) $1-x, 1-y, -z$; (iii) $1-x, 1-y, 1-z$; (iv) $x, y, z-1$; (v) $x-1, y-1, z-1$; (vi) $1-x, -y, 1-z$; (vii) $-x, -y, 1-z$; (viii) $1+x, y, z$; (ix) $2-x, 1-y, 1-z$; (x) $x, 1+y, z$.

La collecte des données, l'affinement de constantes de la maille et la réduction des données: *Rigaku AFC/MSC Diffractometer Control Program* (Rigaku Corporation, 1991). La synthèse de Patterson et de Fourier: *RSSFR-5*, *UNICS* (Sakurai, 1971). L'affinement par moindres carrés à matrice complètes: *RSFLS-4*, *UNICS*. Les distances interatomiques et les angles: *CCPC* (Kawamura & Kawahara, 1980). Le graphique: *ORTEP* (Johnson, 1971); *ATOMS* (Dowty, 1992). L'impression des tableaux de F_o et de σF_o: *LISTHKL* (Yamakawa & Kawahara, 1992).

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Les listes des facteurs de structure et des facteurs d'agitation thermique anisotrope ont été déposées aux dépôt d'archives de la British Library Document Supply Centre (Supplementary Publication No. SUP 71797: 11 pp.). On peut en obtenir des copies en s'adressant à: The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre. [Référence de CIF: DU1066]

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SrZrSi$_2$O$_7$

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Abstract

Rietveld refinement of strontium zirconium disilicate, $SrZrSi_2O_7$, using neutron powder diffraction data [$\lambda = 2.57167$ (3) Å, $F(000) = 252.45$, $\mu R = 0.11$, 295 contributing reflections] resulted in $R_{wp} = 2.72\%$. The structure can be described as alternate layers, containing the ZrO_6 octahedra and formed by the Si_2O_7 groups, stacked parallel to [001]. Elongated cages are formed for Sr, which coordinates to give distorted SrO_8 dodecahedra. The Si_2O_7 groups are in a nearly eclipsed conformation. The title compound is isostructural with the high-temperature form of $NaFeP_2O_7$.

Comment

The silicates of strontium are of little or no geothermal interest and, therefore, have not been studied very carefully. Knowledge of strontium silicates, however, has become increasingly important as they may play a role in nuclear-safety studies. When, during very severe nuclear accidents, the reactor core has melted through the reactor vessel, the formation of strontium silicates may occur. The abundant presence of zirconium from the fuel cladding and silica in the concrete may lead to the formation of $SrZrSi_2O_7$.

$SrZrSi_2O_7$ is found in the pseudo-binary section $SrSiO_3$–$ZrSiO_4$. The existence of $SrZrSi_2O_7$ was first reported by Ghanbari-Ahari & Brett (1988), who

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studied phase relations in the ternary system SrO–SiO_2–ZrO_2. Their unindexed *d* values, also obtained by X-ray powder diffraction, agree nicely with ours. In order to understand better the chemical activity of this compound in complex systems such as SrO–SiO_2–ZrO_2, its crystallographic structure has been studied.

The agreement between the observed and the calculated profiles of the data is shown in Fig. 1. The structure can be described (Gabelica-Robert, Goreaud, Labbe & Raveau, 1982) as a cage structure built up from corner-sharing ZrO_6 octahedra and Si_2O_7 groups (Fig. 2). The Si_2O_7 group is formed from slightly distorted SiO_4 groups having one common O atom. The two tetrahedra are in a nearly eclipsed conformation. The Sr ions have irregular eight coordination.

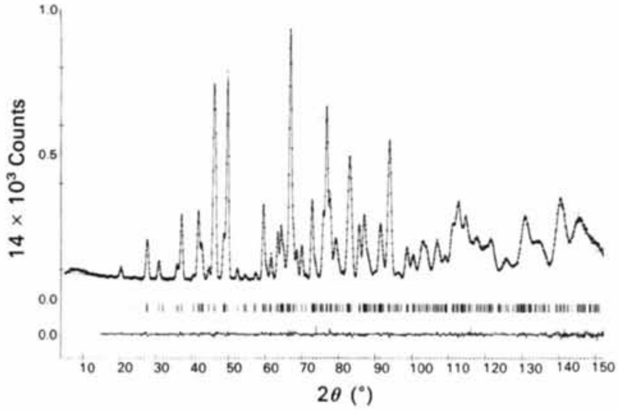

Fig. 1. Observed (dots) and calculated (solid line) neutron diffraction profile of $SrZrSi_2O_7$ at 295 K; the positions of nuclear lines and *I*(obs.) − *I*(calc.) are indicated.

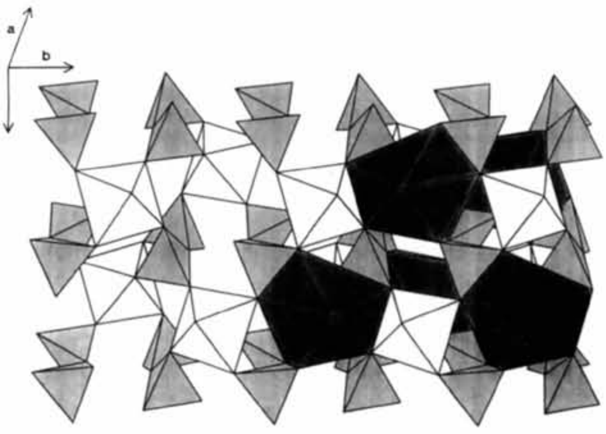

Fig. 2. A drawing of the polyhedra in the $SrZrSi_2O_7$ structure. In the left-hand part, the irregular SrO_8 dodecahedrons are omitted to show the cages and the characteristic linkage between the Si_2O_7 groups and the ZrO_6 octahedra.

The disilicates $A_2Si_2O_7$ (*A* = lanthanide, Sc, Y) have received some attention in the structural literature. For small *A* (*A* = Sc, Tb, Tm), the thortveitite structure has been found in which Si_2O_7 groups exist in a staggered conformation and *A* has six coordination. The structure changes as the radius of *A* increases (Table 3) and the bridge angle Si—O—Si decreases. The coordination of *A* also increases (Smolin & Shepelev, 1970). The $Ca_2P_2O_7$ structure type has been reported (Felsche & Hirsiger, 1969) at low temperature for *A* = La, Pr, Nd and Sm. Mixed disilicates *AA′* are less well known; only $CaZrSi_2O_7$, which has a deformed thortveitite ($Sc_2Si_2O_7$) structure (Roelofsen-Ahland & Peterson, 1989), has been studied. The Si—O—Si angle in this compound has decreased to 147.4°. In the title compound, the bridge angle is further decreased to 133.8° (Table 3). Replacing Zr at the octahedral site by the much smaller V atom, as in $SrVSi_2O_7$ (Takeuchi & Joswig, 1967), causes the structure with Si_2O_7 groups to become a chain structure built up of Si_4O_{12} groups.

Experimental

$SrZrSi_2O_7$(s) was prepared in two steps. In the first step $SrCO_3$(s) (Cerac) was dissolved slowly in HNO_3 and mixed with alcohol and a calculated amount of TEOS(l) (tetraethyl orthosilicate, Merck). After precipitation with ammonia, the sample was dried in an oven (353 K) and decomposed in purified oxygen in a gold boat at 1073 K (Ueno, Hayashi, Okada & Otsuka, 1990). In the second step, a calculated stoichiometric amount of ZrO_2(s) (Aldrich, <200 p.p.m. Hf) was added to the mixture, and then the sample was heated in a platinum boat in a purified argon atmosphere. After each heating, the sample was ground in an alundum mortar and analysed by X-ray diffraction. This procedure was repeated at gradually higher temperatures up to 1673 K until the sample was phase pure. In the final heating, the sample was heated in a gold boat in purified oxygen at 973 K to compensate for possible oxygen loss during the previous heatings.

Crystal data

$SrZrSi_2O_7$
M_r = 347.011
Monoclinic
$P2_1/c$
a = 7.7617 (9) Å
b = 8.0713 (10) Å
c = 10.0559 (11) Å
β = 111.90 (1)°
V = 584.51 (7) Å^3
Z = 4

D_x = 3.943 (1) Mg m^{-3}
D_m = 3.928 (22) Mg m^{-3}
Cu $K\alpha_1$ radiation
λ = 1.540598 Å
Cell parameters from 33 reflections
θ = 14.4–46.4°
T = 295 K
White powder

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

	x	*y*	*z*	*B*
Sr	0.2825 (4)	0.4817 (4)	0.2945 (3)	0.72 (10)
Zr	0.2597 (5)	0.0110 (4)	0.2460 (4)	0.48 (7)
Si(1)	0.0650 (8)	0.2484 (7)	0.4577 (5)	0.22 (15)
Si(2)	0.6737 (7)	0.2136 (7)	0.4605 (5)	0.65 (17)
O(1)	0.8675 (5)	0.1539 (5)	0.4390 (4)	0.54 (12)

O(2)	0.1918 (5)	0.2689 (5)	0.6250 (4)	0.88 (10)
O(3)	0.0115 (5)	0.4214 (4)	0.3695 (4)	0.79 (12)
O(4)	0.1698 (5)	0.1344 (5)	0.3809 (4)	0.57 (14)
O(5)	0.5257 (6)	0.0915 (5)	0.3492 (4)	0.72 (11)
O(6)	0.6943 (5)	0.2050 (5)	0.6239 (4)	0.06 (12)
O(7)	0.6331 (6)	0.4044 (4)	0.4064 (4)	0.08 (11)

Table 2. *Atomic distances* (Å) *and angles* (°)

Si(1) tetrahedron			
Si—O(1^{i})	1.660 (7)	Si—O(3)	1.625 (6)
Si—O(2)	1.610 (6)	Si—O(4)	1.605 (7)
O(1^{i})—Si—O(2)	110.2 (4)	O(2)—Si—O(4)	109.9 (4)
O(1^{i})—Si—O(3)	107.3 (4)	O(3)—Si—O(4)	107.7 (4)
O(1^{i})—Si—O(4)	107.1 (4)	Si—O(1^{i})—Si(2^{i})	133.8 (4)
O(2)—Si—O(3)	114.5 (4)		
Si(2) tetrahedron			
Si—O(1)	1.669 (7)	Si—O(6)	1.592 (7)
Si—O(5)	1.609 (6)	Si—O(7)	1.627 (6)
O(1)—Si—O(5)	100.2 (4)	O(5)—Si—O(6)	117.5 (4)
O(1)—Si—O(6)	111.9 (3)	O(5)—Si—O(7)	110.5 (3)
O(1)—Si—O(7)	107.6 (4)	O(6)—Si—O(7)	108.6 (4)
Zr octahedra			
Zr—O(2^{ii})	2.108 (5)	Zr—O(5)	2.042 (5)
Zr—O(3^{iii})	2.118 (5)	Zr—O(6^{iv})	2.131 (5)
Zr—O(4)	2.007 (6)	Zr—O(7^{v})	2.176 (7)
O(2^{ii})—Zr—O(3^{iii})	89.3 (2)	O(3^{iii})—Zr—O(7^{v})	92.4 (2)
O(2^{ii})—Zr—O(4)	84.0 (2)	O(4)—Zr—O(5)	92.2 (2)
O(2^{ii})—Zr—O(5)	90.7 (2)	O(4)—Zr—O(6^{iv})	90.8 (2)
O(2^{ii})—Zr—O(6^{iv})	174.0 (3)	O(4)—Zr—O(7^{v})	173.5 (3)
O(2^{ii})—Zr—O(7^{v})	90.4 (2)	O(5)—Zr—O(6^{iv})	92.5 (2)
O(3^{iii})—Zr—O(4)	90.9 (2)	O(5)—Zr—O(7^{v})	84.5 (2)
O(3^{iii})—Zr—O(5)	176.9 (3)	O(6^{iv})—Zr—O(7^{v})	95.0 (2)
O(3^{iii})—Zr—O(6^{iv})	87.8 (2)		
Sr dodecahedron			
Sr—O(1^{vi})	2.602 (5)	Sr—O(5^{vi})	2.588 (6)
Sr—O(2^{ii})	2.571 (5)	Sr—O(6^{vii})	2.647 (5)
Sr—O(3)	2.533 (6)	Sr—O(7)	2.605 (5)
Sr—O(4)	3.157 (5)	Sr—O(7^{vii})	2.976 (5)

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

Table 3. *Comparison of interatomic distances* (Å) *and angles* (°) *in* $A_2Si_2O_7$ *and* $AA'Si_2O_7$

	Coordination of *A*	Terminal Si—O distance	Bridge Si—O distance	Si—O—Si angle
$Yb_2Si_2O_7$^{a}	6	1.62 (2)	1.63 (1)	180
$Er_2Si_2O_7$^{a}	6	1.62 (1)	1.63 (1)	180
$Gd_2Si_2O_7$^{a}	7	1.61 (1)	1.67 (2)	158.7 (7)
$Nd_2Si_2O_7$^{a}	8	1.63 (1)	1.61 (2)	132.6 (7)
$CaZrSi_2O_7$^{b}	6,8	1.622 (7)	1.643 (3)	147.4 (5)
$SrZrSi_2O_7$^{c}	6,8	1.613 (6)	1.665 (7)	133.8 (4)

References: (*a*) Smolin & Shepelav (1970); (*b*) Roelofsen-Ahland & Peterson (1989); (*c*) this study.

The density of $SrZrSi_2O_7$ was measured with double-distilled carbon tetrachloride (CCl_4, Merck) in a pycnometer with a contents of *ca* 25 cm^3. Between 2 and 5 g of the compound were used per experiment to reduce the temperature dependency. Afterwards, the $SrZrSi_2O_7$ was checked by X-ray diffraction but no change in structure could be observed.

The X-ray powder diffraction pattern was obtained with a Philips PW1130/90 generator and a Delft Instruments Guinier-de Wolff camera using Cu $K\alpha_1$ radiation with λ = 1.540598 Å. A mixture of silicon and tungsten was used as an internal standard. Si from NBS (now designated NIST), standard reference material (SRM) 640a with a_0 = 5.430825 (36) Å (Hubbard, 1983), and W (Schuchardt) with a_0 = 3.16540 (9) Å (Parrish, 1960), converted to the wavelength mentioned above. All diffraction lines could be indexed with the ITO method (Visser, 1969) yielding a figure of merit F_{20} = 38 (0.008, 41). The absent reflections in the X-ray diffraction pattern indicate the space group $P2_1/c$. The lattice parameters and intensity distribution suggest a strong similarity with the high-temperature structure of $NaFeP_2O_7$ (Gabelica-Robert, Goreaud, Labbe & Raveau, 1982).

Because no single crystals could be obtained, the Rietveld (1969) method was used for refinement of neutron powder diffraction data, gathered with the Petten High-Flux reactor: $5 < 2\theta < 152.6°$ in steps of 0.1°. Neutrons of λ = 2.57167 (3) Å were obtained using the beam reflected from (111) planes of a single crystal of copper and reducing the λ/n contamination to less than 0.1% by means of a pyrolytic graphite filter. Soller slits with a horizontal divergence of 30′ were placed between the reactor and the monochromator and in front of the four ^{3}He counters. The sample holder (φ = 10 mm) consisted of a V tube filled with Cd slices and closed with Cu plugs fitted with 'O' rings. No precautions were taken to avoid preferred orientation. The neutron powder diffraction results were analysed with the program *DBW*3.2, version 8804 (Wiles & Young, 1981). The structure of high-temperature $NaFeP_2O_7$ (Gabelica-Robert, Goreaud, Labbe & Raveau, 1982) was used as a trial model. Coherent scattering lengths: Sr 7.02, Zr 7.16, Si 4.149, O 5.805 fm (Koester, Rauch, Herkens & Schroeder, 1981). 61 parameters were used in the refinement: a scale factor, three half-width parameters defining the Gaussian-like shape of the reflections, six background parameters, the counter zero error, the unit-cell parameters, the atomic positional parameters, isotropic displacement parameters and a scale factor for the V sample holder. The largest correlation-matrix element for structural parameters was 0.46 with $\Delta/c < 0.3$. In the final cycle, the *R* values obtained were R_p = 2.05, R_{wp} = 2.72, $R_{\rm exp}$ = 2.08%, S = 1.31, D-wD = 1.52 and $\chi^2_{\rm red}$ = 2.03.

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Lists of structure factors and X-ray powder diffraction data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71764 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1069]

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Cadmium Selenite–Water (4/3) and Two Polymorphic Forms of Cadmium Selenite

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Abstract

Cadmium selenite has two polymorphic forms: $CdSeO_3$(*oP*20) and $CdSeO_3$(*mP*40). In the orthorhombic form the coordination polyhedron around cadmium is an octahedron and in the monoclinic form it is a trigonal prism. In $4CdSeO_3.3H_2O$ the coordination polyhedra are distorted between octahedra and trigonal prisms.

Comment

Like other transition metals, cadmium forms several selenites. The earliest reports as compiled in *Gmelin's Handbuch der Anorganische Chemie* (1925) describe the compounds $CdSeO_3$, $2CdSeO_3.3H_2O$, $3CdSeO_3.H_2SeO_3$, $2CdSeO_3.H_2SeO_3.H_2O$, $CdSe_2O_5$ and $Cd(NH_3)SeO_3$. Markovskii & Sapozhnikov (1961), Gospodinov & Bogdanov (1983) and Micka, Uchytilova & Ebert (1984) have reported two different modifications for anhydrous cadmium selenite. Thermal, IR and analytical data are also available in these publications.

The crystal structures of cadmium selenites are almost unknown. The only published structure is that of cadmium hydrogen selenite 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), $4CdSeO_3.3H_2O$, $CdSe_2O_5$, $Cd_3(HSeO_3)_2(SeO_3)_2$, $(NH_4)Cd(SeO_3)_2$ and $Cd(NH_3)SeO_3$. The structures of the first three compounds are discussed in this paper and the other four structures will be reported shortly. Pearson notation is used for phase nomenclature (Leigh, 1990).

$CdSeO_3$(*oP*20) is isomorphous with the selenites and tellurites of Mg, Mn, Co, Ni, Cu and Zn (Kohn, Inoue, Horie & Akimoto, 1976). All three compounds contain a three-dimensional network of cadmium-oxygen polyhedra. In $CdSeO_3$(*oP*20) the CdO_6 octahedra share only corners, but in the other compounds edge-sharing occurs as well. In $CdSeO_3$(*mP*40), CdO_6 trigonal prisms and SeO_3 tetrahedra share common edges and in $4CdSeO_3.3H_2O$ the Cd(1)O_6 octahedra and SeO_3 tetrahedra share common edges. The three structures are shown in Figs. 1–3.

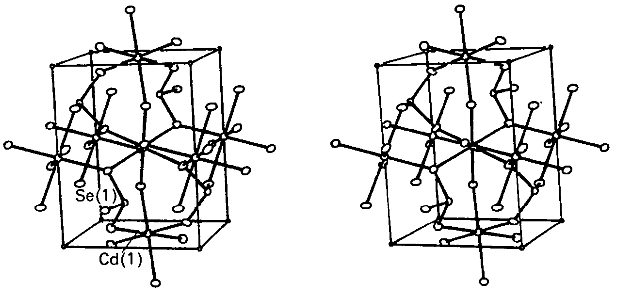

Fig. 1. Stereoscopic *ORTEP*II drawing of the unit cell of $CdSeO_3$(*oP*20) showing 75% probability displacement ellipsoids. The *a* axis is horizontal and the *b* axis is vertical.

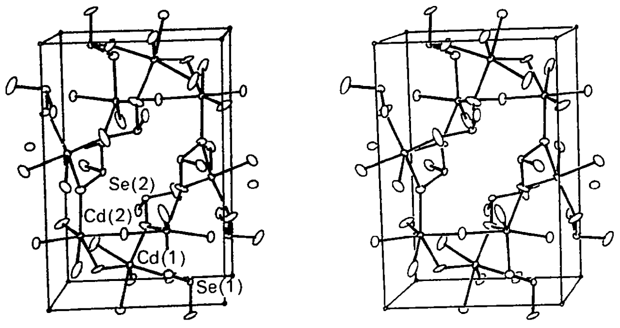

Fig. 2. A stereoscopic *ORTEP*II drawing of the unit cell of $CdSeO_3$(*mP*40) showing 75% probability displacement ellipsoids. The *c* axis is horizontal and the *b* axis is vertical.

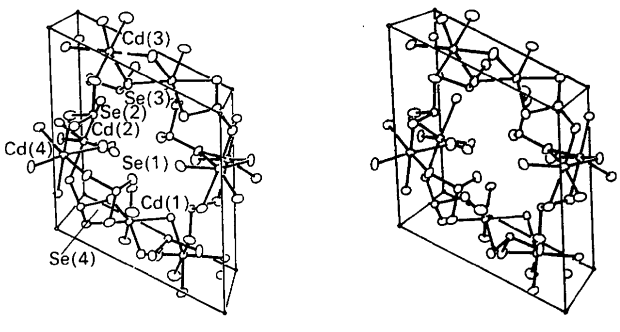

Fig. 3. A stereoscopic *ORTEP*II drawing of the unit cell of $4CdSeO_3.3H_2O$ showing 75% probability displacement ellipsoids. The *a* axis is tilted slightly from the horizonal and the *c* axis is vertical.

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