Data collection

Powder diffractometer at the Petten High Flux Reactor
61 measured reflections
$2\theta_{\max} = 155°$

Refinement

$R_p = 0.0243$
$R_{wp} = 0.0310$
$S = 1.79$
61 reflections
27 parameters
$(\Delta/\sigma)_{\max} = 0.3$

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

	x	y	z	$U_{\rm iso}$
Sr	2/3	1/3	1/4	0.17 (8)
Ta1	1/3	2/3	1/4	0.20 (7)
Ta2	0.3570 (2)	0	0	0.14 (3)
O1	1/3	2/3	0.5347 (1)	0.22 (6)
O2	0.7532 (2)	0	0	0.53 (5)
O3	0.3701 (2)	0.4315 (2)	0.3454 (1)	0.47 (4)

Table 2. *Selected geometric parameters* (Å)

Sr—O1	× 2	2.655 (1)	Ta2—O2		2.478 (2)
Sr—O3	× 6	2.518 (1)	Ta2—O2	× 2	1.980 (1)
Ta1—O3	× 6	1.985 (1)	Ta2—O3	× 2	1.955 (1)
Ta2—O1	× 2	2.0598 (6)			

The neutron radiation was monochromated using the 111 reflection of a copper crystal and the λ/n contamination was reduced to less than 0.1% by means of a pyrolytic graphite filter. Data were collected in the range $5 < 2\theta < 155°$ in steps of 0.1°. 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. No precautions to avoid preferred orientation were taken. An absorption correction according to Weber (1967) was applied ($\mu r = 0.17$).

The neutron diffraction pattern was analyzed using the program *DBW*3.2, version 8804 (Wiles & Young, 1981). The structure of $CaTa_4O_{11}$ (Jahnberg, 1970) was used as a trial model. Coherent scattering lengths of 7.02, 6.91 and 5.803 fm for Sr, Ta and O, respectively, were used (Sears, 1992). The 27 parameters in the refinement comprised 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, an asymmetry parameter and a scale factor for the vanadium holder. The largest correlation matrix element for the structural parameters was 0.49.

A list of raw powder diffraction data has been deposited with the IUCr (Reference: CR1170). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Two New Scandium Phases: $ScSn_2$ and Sc_6Pb_5

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Abstract

The structures of two new phases in the scandium–tin and scandium–lead systems have been determined. The structure of $ScSn_2$ (Pearson symbol *tI*24) is an occupation variant of the $Zr_3Al_4Si_5$ and β-LiSn structures, closely related to the $ZrSi_2$ structure type. Sc_6Pb_5 (Pearson symbol *oI*44) is isotypic with Ti_6Ge_5 (or V_6Si_5). The shortest distances are Sn···Sn 2.954 (3) and Sc···Sn 3.030 (1) Å in $ScSn_2$, and Sc···Pb 2.858 (6), Sc···Sc and Pb···Pb 3.088 (1) Å in Sc_6Pb_5.

Comment

In the course of a recent investigation of the Sc–Sn and Sc–Pb phase diagrams (Palenzona & Manfrinetti, 1995) some new phases were identified. A complete structure determination has been undertaken for two, $ScSn_2$ and Sc_6Pb_5.

The structure of $ScSn_2$ can be compared with the structures of $Zr_3Al_4Si_5$ (Raman & Schubert, 1965) and β-LiSn (Blase & Cordier, 1990). All these phases belong to the same space group, with the same Wyckoff positions occupied and similar coordinates and lattice constants, so that they can be considered as occupation variants of the same structure. In $Zr_3Al_4Si_5$, all positions are statistically filled by two kinds of atoms (Al–Zr and Al–Si mixtures), while the other two compounds are ordered; the different stoichiometry in $ScSn_2$ is obtained by replacing the Li2 atoms [4(*b*)] in β-LiSn with Sn atoms.

The structure of $Zr_3Al_4Si_5$, together with the related structures of $ZrSi_2$ (Schachner, Nowotny & Kudielka, 1954), $HfGa_2$ and $ZrGa_2$ (Pötzschke & Schubert, 1962), have been described previously by Gladyshevskii (1971) and by Kripyakevich (1977). Likewise, $ScSn_2$ can

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be considered as a deformed superstructure of the Cu type, containing six face-centered cubic pseudocells stacked along the *c* axis. A similar sequence of layers is found in the orthorhombic structure of $ZrSi_2$, with three pseudocells stacked along the *b* axis. The close relationship between the two structures is shown in Fig. 1. It is evident that the $ScSn_2$ cell is formed by two $ZrSi_2$ cells, repeated by the glide plane at $z = 1/4$. In both structures, slabs of Sc (or Zr) trigonal prisms, centered by Sn (or Si) atoms, alternate with layers containing only Sn (or Si) atoms. While in $ZrSi_2$ the distortion of the pseudocells is so pronounced as to destroy the typical cuboctahedral coordination around the Si atoms and lower the coordination number to 8, in $ScSn_2$ all Sn atoms are surrounded by four other Sn atoms at a slightly larger distance, completing distorted cuboctahedra with coordination number 12.

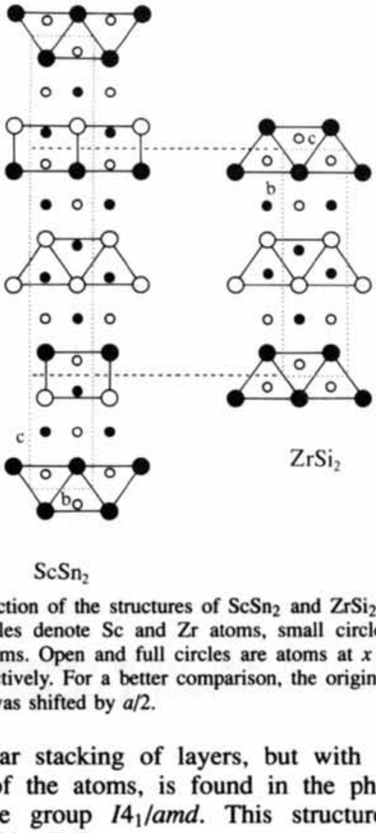

Fig. 1. Projection of the structures of $ScSn_2$ and $ZrSi_2$ along [100]. Large circles denote Sc and Zr atoms, small circles denote Sn and Si atoms. Open and full circles are atoms at $x = 0$ and $x = 1/2$, respectively. For a better comparison, the origin of the $ZrSi_2$ structure was shifted by *a*/2.

A similar stacking of layers, but with a different ordering of the atoms, is found in the phase $HfGa_2$ with space group $I4_1/amd$. This structure and the orthorhombic $ZrGa_2$ structure represent another pair formed by six and three pseudocells, respectively; again the cell of $HfGa_2$ comprises two cells of $ZrGa_2$, repeated by the glide plane. However, in these two structures all atoms have cuboctahedral coordination (coordination number 12).

The compound Sc_6Pb_5 crystallizes with the Ti_6Ge_5 (or V_6Si_5) structure type (Spinat, Fruchart & Herpin, 1970). This type and its ternary variants have been discussed by Parthé & Chabot (1984), and Chabot & Parthé (1985). The Sc_6Pb_5 structure is characterized by isolated columns of Sc tetragonal antiprisms centered by Pb atoms, and edge-connected columns of Pb octahedra centered by Sc atoms. All columns are parallel to the short axis of the cell (Fig. 2). The shortest distance is Sc···Pb 2.858 (6) Å, with Sc···Sc and Pb···Pb both 3.088 (1) Å. Distances and coordination polyhedra were determined using the program *SEXIE* (Rupp, Smith & Wong, 1991).

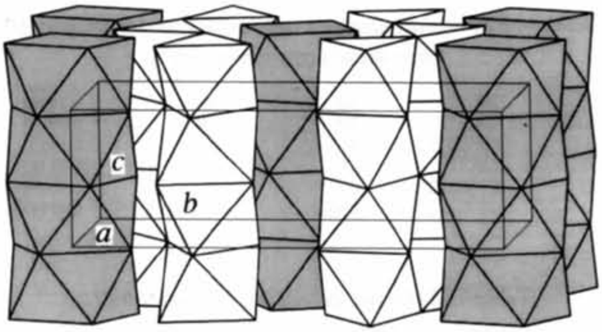

Fig. 2. Sc tetragonal antiprisms centered by Pb3 atoms and Pb octahedra centered by Sc3 atoms in the structure of Sc_6Pb_5.

Experimental

The two compounds were prepared according to the methods described by Palenzona & Manfrinetti (1995).

$ScSn_2$

Crystal data

$ScSn_2$
$M_r = 282.34$
Tetragonal
$I4_1/amd$
$a = 4.233$ (1) Å
$c = 31.039$ (9) Å
$V = 556.2$ (2) Å^3
$Z = 8$
$D_x = 6.74$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 24$–$33°$
$\mu = 19.8$ mm^{-1}
$T = 293$ (2) K
Plate
$0.19 \times 0.18 \times 0.04$ mm
Metallic

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968) and spherical
$T_{\min} = 0.030$, $T_{\max} = 0.239$
1489 measured reflections
375 independent reflections

253 observed reflections $[F_o > 4\sigma(F_o)]$
$R_{\rm int} = 0.10$
$\theta_{\max} = 35.0°$
$h = 0 \rightarrow 6$
$k = 0 \rightarrow 6$
$l = -50 \rightarrow 50$
2 standard reflections
frequency: 60 min
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.124$

Extinction correction: *SHELXL*93 (Sheldrick, 1993)

$S = 5.60$
375 reflections
14 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0010P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 8.0$ e Å^{-3}
$\Delta\rho_{\min} = -8.2$ e Å^{-3}

Extinction coefficient: 0.0019 (2)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* ScSn$_2$

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

	x	y	z	$U_{\rm eq}$
Sc	0	1/4	0.2016 (2)	0.0112 (8)
Sn1	0	1/4	0.0332 (1)	0.0112 (5)
Sn2	0	1/4	3/8	0.0127 (6)
Sn3	0	3/4	1/8	0.0126 (6)

Table 2. *Contact distances* (Å) *for* ScSn$_2$

Sc···Sn1	×4	3.030 (1)	Sn1···Sn2	×2	3.549 (3)
Sc···Sn2	×2	3.183 (5)	Sn1···Sn3	×2	3.549 (3)
Sc···Sn3	×2	3.183 (5)	Sn2···Sn3	×4	2.993 (1)
Sc···Sn1	×2	3.301 (5)	Sn2···Sc	×4	3.183 (5)
Sc···Sc	×2	3.675 (9)	Sn2···Sn1	×4	3.549 (3)
Sn1···Sn1	×2	2.954 (3)	Sn3···Sn2	×4	2.993 (1)
Sn1···Sc	×4	3.030 (1)	Sn3···Sc	×4	3.183 (5)
Sn1···Sc	×2	3.301 (5)	Sn3···Sn1	×4	3.549 (3)

Sc$_6$Pb$_5$

Crystal data

Sc$_6$Pb$_5$
$M_r = 1305.7$
Orthorhombic
Ibam
$a = 8.889$ (1) Å
$b = 19.274$ (2) Å
$c = 6.177$ (2) Å
$V = 1058.3$ (4) Å^3
$Z = 4$
$D_x = 8.19$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 20$–$25°$
$\mu = 82.7$ mm^{-1}
$T = 293$ (2) K
Prism
0.20 × 0.15 × 0.11 mm
Metallic

Data collection

Enraf–Nonius CAD-4 diffractometer
ω/θ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968) and spherical
$T_{\min} = 0.0026$, $T_{\max} = 0.0147$
1401 measured reflections
665 independent reflections

467 observed reflections $[F_o > 4\sigma(F_o)]$
$R_{\rm int} = 0.123$
$\theta_{\max} = 27.5°$
$h = 0 \rightarrow 11$
$k = -25 \rightarrow 25$
$l = 0 \rightarrow 8$
2 standard reflections frequency: 60 min intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.139$
$S = 1.05$
665 reflections
33 parameters
$w = 1/[\sigma^2(F_o^2) + (0.1160P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.013$

$\Delta\rho_{\max} = 4.7$ e Å^{-3}
$\Delta\rho_{\min} = -4.5$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* Sc$_6$Pb$_5$

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

	x	y	z	$U_{\rm eq}$
Sc1	0.1104 (9)	0.1426 (4)	0	0.022 (1)
Sc2	0.2335 (7)	0.4401 (3)	0	0.016 (1)
Sc3	0	0.3049 (3)	1/4	0.017 (1)
Pb1	0.3032 (2)	0.2936 (1)	0	0.0161 (4)
Pb2	0.3981 (2)	0.0754 (1)	0	0.0172 (4)
Pb3	0	0	1/4	0.0167 (5)

Table 4. *Contact distances* (Å) *for* Sc$_6$Pb$_5$

Sc1···Pb2		2.867 (8)	Sc3···Pb1	×2	3.114 (1)
Sc1···Pb1		2.995 (8)	Sc3···Sc1	×2	3.624 (8)
Sc1···Pb3	×2	3.302 (7)	Sc3···Sc2	×2	3.672 (8)
Sc1···Pb1		3.378 (8)	Pb1···Sc2		2.891 (7)
Sc1···Pb1	×2	3.412 (3)	Pb1···Sc1		2.995 (8)
Sc1···Sc3	×2	3.624 (8)	Pb1···Sc3	×2	3.008 (4)
Sc1···Sc1	×2	3.659 (8)	Pb1···Sc3	×2	3.114 (1)
Sc1···Sc2		3.71 (1)	Pb1···Sc1		3.378 (8)
Sc1···Sc2	×2	3.74 (1)	Pb1···Sc1	×2	3.412 (3)
Sc2···Pb2		2.858 (6)	Pb1···Pb1	×2	3.641 (2)
Sc2···Pb1		2.891 (7)	Pb2···Sc2		2.858 (7)
Sc2···Pb2		2.996 (7)	Pb2···Sc1		2.867 (8)
Sc2···Pb3	×2	3.054 (6)	Pb2···Sc3	×2	2.920 (5)
Sc2···Pb2	×2	3.316 (3)	Pb2···Sc2		2.996 (7)
Sc2···Sc3	×2	3.672 (8)	Pb2···Sc2	×2	3.316 (3)
Sc2···Sc1		3.71 (1)	Pb2···Pb2		3.425 (3)
Sc2···Sc1	×2	3.74 (1)	Pb2···Pb2	×2	3.581 (2)
Sc2···Sc2	×2	3.856 (7)	Pb3···Sc2	×4	3.054 (6)
Sc3···Pb2	×2	2.920 (5)	Pb3···Pb3	×2	3.088 (1)
Sc3···Pb1	×2	3.008 (4)	Pb3···Sc1	×4	3.302 (7)
Sc3···Sc3	×2	3.088 (1)			

For both compounds, data collection: Enraf–Nonius CAD-4 software; cell refinement: Enraf–Nonius CAD-4 software; data reduction: local software; program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993).

Financial support from the Italian National Research Council (CNR) is gratefully acknowledged.

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: NA1161). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Synthetic Dipotassium Zinc Disilicate

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Abstract

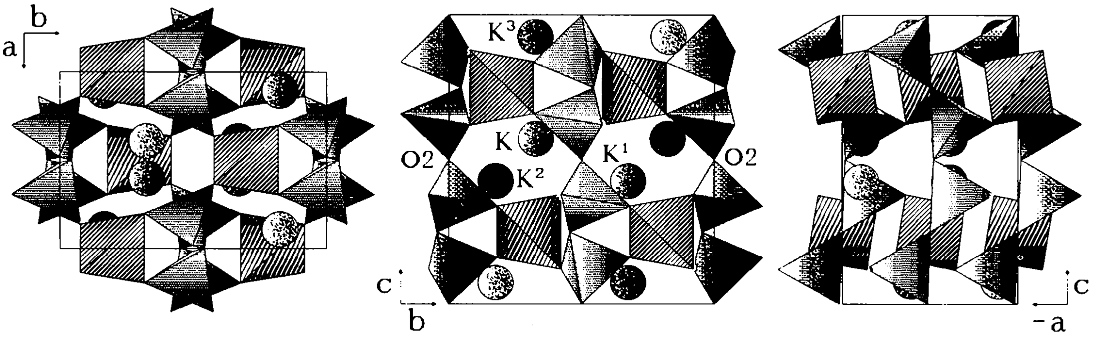
The structure of dipotassium zinc disilicate, $K_2ZnSi_2O_6$, comprises three-membered rings formed by two $[SiO_4]$ and one $[ZnO_4]$ tetrahedron, which are connected to form layers parallel to (001). Within the unit cell, one layer is connected by common O atoms to another layer, which is symmetry related to the first by a 2_1 axis along [001], thus forming a silicate *vierer* single chain with a large stretching factor of f_s = 96.8% and one unusually small Si—O—Si angle of 118.3 (2)°. The K atoms are coordinated by nine nearest O atoms and lie between the three-membered ring layers, forming a face-connected $[KO_3]$ network. The formula of this new potassium zinc silicate structure may be expressed, following the recommendations of the IUCr, as $K_4^{[9]}Zn_2^{[4t]}\{uB,1^1_\infty\}[^4Si_4O_{12}]$.

Comment

Recently, several new potassium zinc silicates have been synthesized (Dollase & Ross, 1993; Kawahara, Kohara, Konishi & Horiuchi, 1991; Kohara & Kawahara, 1990). Although several structures of zinc silicates, particularly the alkali and alkali earth zinc silicates, have been investigated (Hesse, Liebau & Boehm, 1977; Belekoneva, Egorov-Tismenko, Simonov & Belov, 1970; Plakhov & Belov, 1977; Hamilton & Finney, 1985), there are still too few known phases for a systematic description.

The new potassium zinc silicate described here was derived from work on the possibilities of substituting alkali and alkali earth cations for the water molecule in hemimorphite.

Projections of the structure of $K_2ZnSi_2O_6$ are shown in Fig. 1. The Zn and Si atoms are tetrahedrally coordinated by O atoms. Two $[SiO_4]$ and one $[ZnO_4]$ tetrahedron form a three-membered ring. The Si and Zn atoms of the three-membered ring are located on a plane roughly parallel to (101). The rings are condensed into a layer parallel to (001) such that seven-membered rings are created (Fig. 1*a*). Within the unit cell, one three-membered-ring layer is linked *via* the O2 atoms to another layer and the layers are symmetry related to each other by the 2_1 axis along [001]. Three additional and topologically different rings are formed about this connection: one seven-membered ring approximately parallel to (111) and two eight-membered rings approximately parallel to (010) and (110). The linkage of the two three-membered-ring layers within the unit cell creates very stretched silicate chains, with a large stretching factor of f_s = 96.8% (Fig. 1*c*).



(*a*) (*b*) (*c*)

Fig. 1. Projections of the structure of $K_4^{[9]}Zn_2^{[4t]}\{uB,1^1_\infty\}[^4Si_4O_{12}]$, showing the network of O-atom tetrahedra ($[ZnO_4]$ tetrahedra are striped, $[SiO_4]$ tetrahedra are dotted) and basic features such as the three-membered ring layers, the position of the K atoms within the plane of connection of two ring layers, and the very stretched *vierer* single chains parallel to **c**.