Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Ru_3Sn_7, a re-investigation

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(Ru–Ru) = 0.002 Å
R factor = 0.030
wR factor = 0.045
Data-to-parameter ratio = 10.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 21 August 2001
Accepted 28 August 2001
Online 11 September 2001

Triruthenium heptatin, Ru_3Sn_7, is an example of a body-centered cubic structure that can be described with two interpenetrating similar networks of slightly distorted square-antiprism Ru–Sn polyhedra. Each network can be modeled as dimers of face-sharing Ru–Sn square-antiprism polyhedra connected to each other with the square face. These barrel-like dimers make up one of the two networks. The barrels are connected to each other by the edge of the outer square face. The two interpenetrating networks are connected *via* the central corner Sn atoms of each dimer.

Comment

The title compound was synthesized as part of a reinvestigation of the Ru–Sn binary system. Earlier reports (Nial, 1947) of this compound were based on visually estimated intensities from powder data and only approximate structural parameters were determined. Ru_3Sn_7 is an example of an electron-rich compound (Häussermann *et al.*, 1998) in the Ru/Sn system. The structure is built of square-face-sharing dimers of square antiprisms of Ru–Sn polyhedra (Fig. 1). These barrel-like dimers are, in turn, connected *via* edges of the outer square faces, occupied by Sn1, to each other giving one of the two component networks. The quadratic faces of six barrels make up an empty cube at the corner of the unit cell. The two equivalent interpenetrating frameworks (Fig. 2) are related by the *I*-centering condition and are connected to each other with the corner-shared Sn2 atom. The valence electron concentration (VEC) of the title compound is 52 electrons per formula unit. Several isomorphous T_3E_7 compounds (T is a transition metal and E is an electron-rich main group element) are

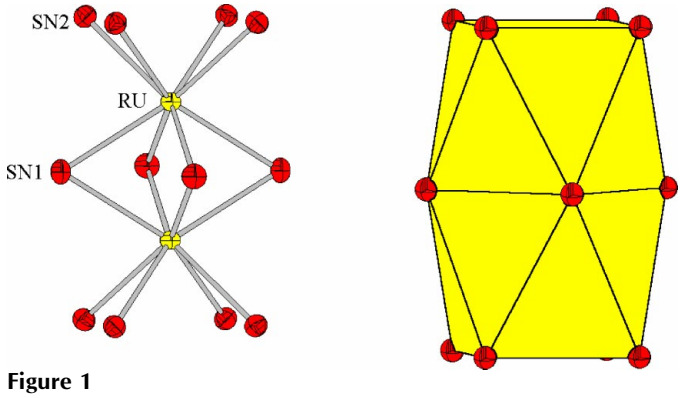

Figure 1
The dimer unit of two quadratic antiprism Ru-Sn polyhedra. Ru is shown in yellow and Sn red. The displacement ellipsoids in the left picture are shown at the 90% probability level and in the right picture, a polyhedral representation is shown.

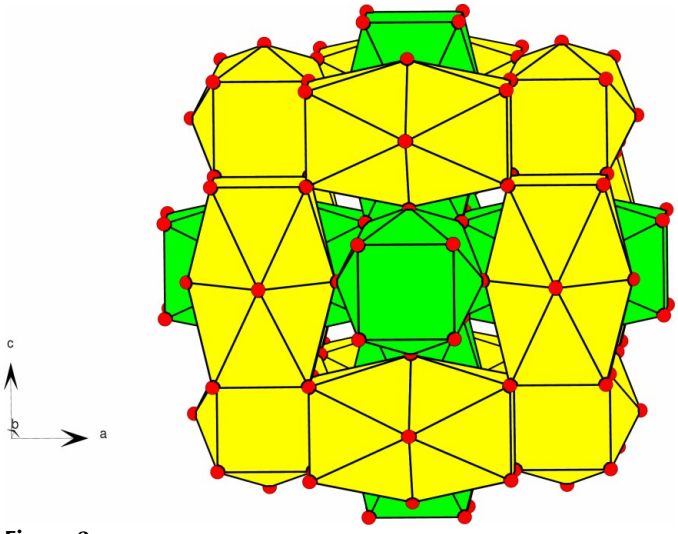

Figure 2
Perspective view of slightly more than the unit-cell content, with Sn atoms shown as red circles and the two different interpenetrating networks as either yellow or green polyhedra.

known with VEC ranging from 51 to 55. The improved accuracy of parameters of the structural model may imply slight changes to the interpretation of the bonding situation in similar compounds. Doping of the title compound, both with neighboring transition metals and electron-rich main group elements will be attempted in future experiments in order to elucidate the possibilities for affecting electronic properties.

Experimental

The title compound was crystallized from a solid-state reaction between a 1:10 mixture of Ru and Sn. The mixture was heated in an evacuated quartz ampoule at 973 K for 24 h and left to cool to room temperature over a period of approximately 1 h. Small crystals of the title compound were found in a matrix of Sn. The excess Sn was removed by treatment with NaOH aqueous solution, 2 mol dm^{-3}.

Crystal data

Ru_3Sn_7
M_r = 1134.04
Cubic, $Im\bar{3}m$
a = 9.3532 (19) Å
V = 818.2 (3) Å^3
Z = 4
D_x = 9.206 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 984 reflections
θ = 1.6–26.0°
μ = 26.25 mm^{-1}
T = 293 (2) K
Prism, dark grey
0.12 × 0.11 × 0.10 mm

Data collection

Stoe IPDS diffractometer
Area-detector (φ) scans
Absorption correction: numerical (*X-RED*; Stoe & Cie, 1997)
T_{min} = 0.045, T_{max} = 0.083
1394 measured reflections
102 independent reflections
97 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 25.9°
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 9$
$l = -11 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.045
S = 2.51
102 reflections
10 parameters
$w = 1/[\sigma^2(F_o^2) + (0.01P)^2$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.83 e Å^{-3}
$\Delta\rho_{min}$ = −2.17 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0022 (2)

Table 1
Selected bond lengths (Å).

Ru—Sn2	2.7393 (11)	Sn2—Sn2^{iii}	2.8804 (19)
Ru—Sn1^{i}	2.7506 (9)	Sn2—Sn2^{iv}	3.0136 (12)
Ru—Ru^{ii}	2.897 (3)		

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

The maximum residual electron density was located at (0.1229, 0, 0.1229), 1.59 Å from Sn2, and the minimum residual density was located at (0, 0, 0), 2.61 Å from Sn2. None of these residual densities were interpreted as anything other than artefacts, perhaps occurring due to the high symmetry of these positions. The lattice parameter from the single-crystal experiment was checked by powder diffraction methods with a Guinier–Hägg camera using Si as internal standard for the 2θ scale, giving a value of the unit-cell edge a = 9.3533 (4) Å, well in accordance with the value estimated from the IPDS measurements.

Data collection: *EXPOSE* in *IPDS* (Stoe & Cie, 1997); cell refinement: *CELL* in *IPDS*; data reduction: *X-RED* (Stoe & Cie, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Bergerhoff, 1996).

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