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## Crystal architecture of $R_2\text{SnS}_5$ ( $R = \text{Pr, Nd, Gd}$ and $\text{Tb}$ ): crystal structure relationships in chalcogenides

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The crystal structure of the  $R_2\text{SnS}_5$  ( $R = \text{Pr, Nd, Gd}$  and  $\text{Tb}$ ) compounds has been investigated using X-ray single-crystal diffraction. Crystal architecture and structural relationships among  $\text{U}_3\text{S}_5$ ,  $\text{Y}_2\text{HfS}_5$ ,  $R_2\text{SnS}_5$  compounds are discussed and a structural origin is determined. It is shown that the complex architecture of the crystal structure of  $\text{Eu}_5\text{Sn}_3\text{S}_{12}$  is a result of interweaving of the simple crystal structures. The location of the copper ions in the non-stoichiometric compound  $\text{Y}_2\text{Cu}_{0.20}\text{Sn}_{0.95}\text{S}_5$  is proposed on the basis of comparative analysis of the  $R\text{—S}$  interatomic distances in the  $R_2\text{SnS}_5$  series of compounds.

### 1. Introduction

The synthesis of compounds with increasingly complex compositions, such as ternary, quaternary *etc.*, has become a principal direction in modern material sciences (Eliseev & Kuzmichyeva, 1990; Mitchell & Ibers, 2002). Among the multicomponent systems an important place belongs to the complex rare-earth chalcogenides. The rare-earth chalcogenides have been intensively studied over recent years owing to their specific thermal, electrical and optical properties, which for example make them prospective materials in the field of IR and nonlinear optics. Therefore, the synthesis and investigation of the crystal structures of complex chalcogenides are important steps in the search for new materials.

The formation of the compounds  $R_2\text{SnS}_5$  ( $R = \text{La, Ce, Pr, Nd, Sm, Gd, Tb}$  and  $\text{Dy}$ ;  $\text{La}_2\text{SnS}_5$  structure type, space group  $Pb\bar{a}m$ ) was reported by Jaulmes (1974), Guittard *et al.* (1976) and Julien-Pouzoi & Jaulmes (1979). However, a complete crystal structure determination has only been performed for  $\text{La}_2\text{SnS}_5$  and  $\text{Sm}_2\text{SnS}_5$ . The  $\text{Eu—Sn—S}$  system is more complicated and four crystalline products can be isolated. The crystal structures of  $\text{Eu}_2\text{SnS}_5$  ( $\text{Eu}_2\text{SnS}_5$  structure type, space group  $Pb\bar{a}m$ ; Jaulmes *et al.*, 1982),  $\text{Eu}_5\text{Sn}_3\text{S}_{12}$  ( $\text{Eu}_5\text{Sn}_3\text{S}_{12}$  structure type, space group  $Pmc2_1$ ; Jaulmes & Julien-Pouzoi, 1977*a*),  $\text{Eu}_3\text{Sn}_2\text{S}_7$  ( $\text{Eu}_3\text{Sn}_2\text{S}_7$  structure type, space group  $Pb\bar{a}m$ ; Jaulmes & Julien-Pouzoi, 1977*b*) and  $\text{Eu}_2\text{SnS}_4$  ( $\text{Eu}_2\text{SnS}_4$  structure type, space group  $Pnma$ ; Pocha *et al.*, 2003) have been determined.

This paper presents part of our systematic investigation of the rare-earth chalcogenides. Results of the single-crystal structure reinvestigation of ternary  $R_2\text{SnS}_5$  ( $R = \text{Pr, Nd, Gd}$  and  $\text{Tb}$ ) compounds are reported. Crystal structure analysis of the  $R_2\text{SnS}_5$  compounds and structural relations between the compounds of the  $R\text{—Sn—S}$  systems are discussed. Also, structural aspects connected to a location of the copper ions in the non-stoichiometric  $\text{Y}_2\text{Cu}_{0.20}\text{Sn}_{0.95}\text{S}_5$  are considered.

**Table 1**  
Crystal data and structure refinement details for  $R_2\text{SnS}_5$  ( $R = \text{Pr, Nd, Gd and Tb}$ ).

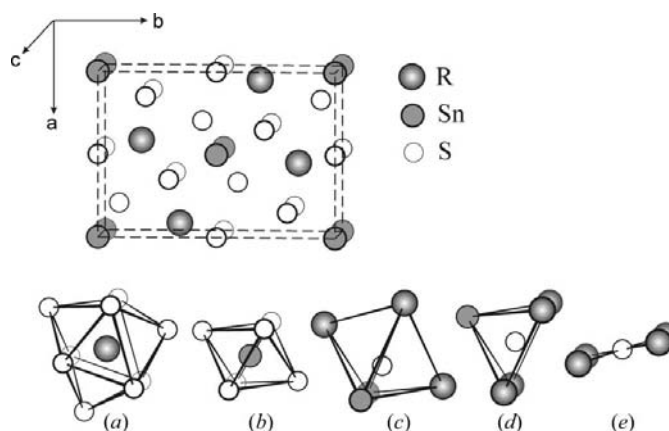
	$\text{Pr}_2\text{SnS}_5$	$\text{Nd}_2\text{SnS}_5$	$\text{Gd}_2\text{SnS}_5$	$\text{Tb}_2\text{SnS}_5$
Crystal data				
Chemical formula	$\text{Pr}_2\text{S}_5\text{Sn}$	$\text{Nd}_2\text{S}_5\text{Sn}$	$\text{Gd}_2\text{S}_5\text{Sn}$	$\text{S}_5\text{SnTb}_2$
$M_r$	560.81	567.47	593.49	596.83
Cell setting, space group	Orthorhombic, $Pbam$	Orthorhombic, $Pbam$	Orthorhombic, $Pbam$	Orthorhombic, $Pbam$
Temperature (K)	293 (2)	293 (2)	293 (2)	293 (2)
$a, b, c$ (Å)	7.8195 (9), 11.2145 (14), 3.9462 (5)	7.7721 (14), 11.218 (2), 3.9272 (9)	7.7330 (17), 11.290 (2), 3.8217 (9)	7.717 (2), 11.2460 (15), 3.8056 (8)
$V$ (Å <sup>3</sup> )	346.05 (7)	342.41 (12)	333.66 (13)	330.27 (13)
$Z$	2	2	2	2
$D_x$ (Mg m <sup>-3</sup> )	5.382	5.504	5.907	6.002
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
$\mu$ (mm <sup>-1</sup> )	18.82	19.96	24.80	26.38
Crystal form, colour	Prism, dark red	Prism, black	Prism, black	Prism, black
Crystal size (mm)	0.09 × 0.08 × 0.06	0.22 × 0.21 × 0.19	0.10 × 0.03 × 0.02	0.11 × 0.06 × 0.03
Data collection				
Diffractometer	Kuma KM-4 with area CCD detector	Kuma KM-4 with area CCD detector	Kuma KM-4 with area CCD detector	Kuma KM-4 with area CCD detector
Data collection method	$\omega$ scan	$\omega$ scan	$\omega$ scan	$\omega$ scan
Absorption correction	Numerical	Numerical	Numerical	Numerical
$T_{\min}$	0.239	0.123	0.242	0.155
$T_{\max}$	0.458	0.258	0.546	0.422
No. of measured, independent and observed reflections	4036, 452, 424	3642, 402, 392	3712, 401, 348	4139, 467, 436
Criterion for observed reflections	$I > 2\sigma(I)$	$I > 2\sigma(I)$	$I > 2\sigma(I)$	$I > 2\sigma(I)$
$R_{\text{int}}$	0.041	0.094	0.062	0.042
$\theta_{\max}$ (°)	27.5	26.7	26.7	28.3
Refinement				
Refinement on	$F^2$	$F^2$	$F^2$	$F^2$
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.013, 0.025, 1.12	0.026, 0.067, 1.09	0.018, 0.027, 0.87	0.016, 0.028, 1.04
No. of reflections	452	402	401	467
No. of parameters	28	27	28	28
Weighting scheme	$w = 1/[\sigma^2(F_o^2) + (0.0102P)^2]$ , where $P = (F_o^2 + 2F_c^2)/3$	$w = 1/[\sigma^2(F_o^2) + (0.030P)^2 + 3.9487P]$ , where $P = (F_o^2 + 2F_c^2)/3$	$w = 1/[\sigma^2(F_o^2) + (0.0053P)^2]$ , where $P = (F_o^2 + 2F_c^2)/3$	$w = 1/[\sigma^2(F_o^2) + (0.0124P)^2]$ , where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$	0.001	< 0.0001	< 0.0001	< 0.0001
$\Delta\rho_{\max}, \Delta\rho_{\min}$ (e Å <sup>-3</sup> )	0.64, -0.70	1.33, -1.83	0.77, -0.96	0.95, -1.05
Extinction method	SHELXL	None	SHELXL	SHELXL
Extinction coefficient	0.0093 (4)	–	0.0222 (6)	0.0076 (3)

Computer programs: *CrysAlis* (Oxford Diffraction, 2007), *SHELXS97* and *SHELXL97* (Sheldrick, 2008).

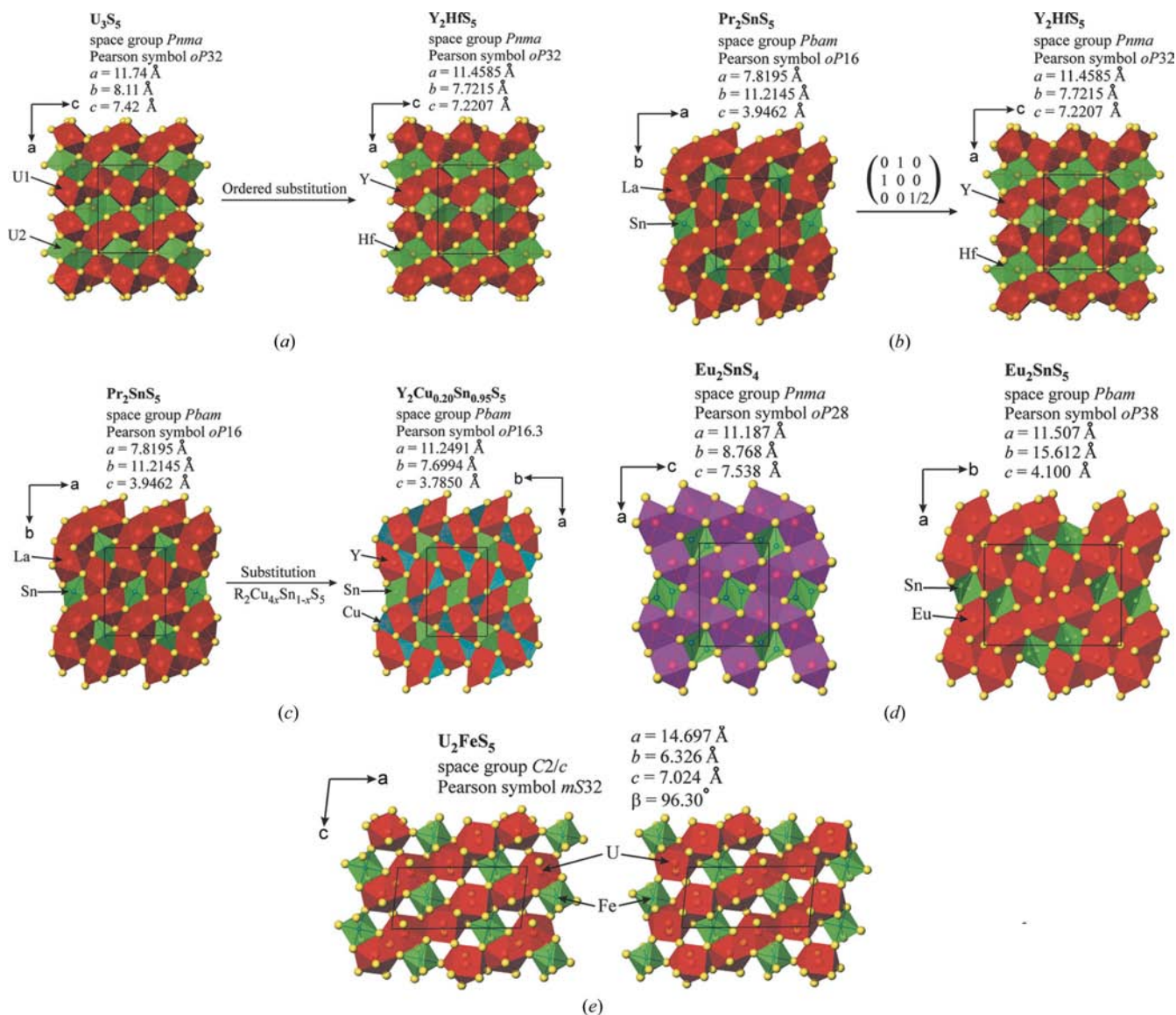
## 2. Experimental

The samples were prepared by sintering the elemental constituents, of purity better than 99.9 wt%, in the atomic ratio  $R:\text{Sn}:\text{S} = 2:1:5$  in evacuated quartz ampoules in a tube furnace. The ampoules were heated at a rate of 30 K h<sup>-1</sup> to a maximum temperature of 1420 K and kept at this temperature for 4 h. Afterwards they were cooled slowly (10 K h<sup>-1</sup>) to 870 K and annealed at this temperature for 200 h. After annealing the ampoules the samples were quenched in cold water.

Diffraction-quality single crystals for the crystal structure determination were selected from the samples. X-ray diffraction measurements were carried out on a Kuma Diffraction KM-4 four-circle single-crystal diffractometer using graphite-monochromated Mo  $K\alpha$  radiation ( $\lambda = 0.71073$  Å) and equipped with a CCD detector. The raw data were treated using the *CrysAlis* data reduction program (Oxford Diffraction, 2007), taking into account an absorption correction. A



**Figure 1**  
The unit cell and the coordination polyhedra of the (a) R, (b) Sn, (c) S1, (d) S2 and (e) S3 atoms in the structure of  $R_2\text{SnS}_5$ .



**Figure 2** Comparison of the crystal structures of  $U_3S_5$ ,  $Y_2HfS_5$ ,  $R_2Sn_5$  ( $R = La, Ce, Pr, Nd, Sm, Gd, Tb$  and  $Dy$ ),  $Y_2Cu_{0.20}Sn_{0.95}S_5$ ,  $Eu_2Sn_5S_4$  and  $Eu_2Sn_5S_5$ .

numerical absorption correction was applied using the *X-SHAPE* program (Stoe & Cie, 1998). The intensities of the reflections were corrected for Lorentz and polarization effects. The crystal structures were solved by Patterson methods (Sheldrick, 2008) and refined by the full-matrix least-squares method using the *SHELXL97* program (Sheldrick, 2008).

### 3. Results and discussion

#### 3.1. Relationships between crystal structures of $U_3S_5$ , $Y_2HfS_5$ , $R_2Sn_5$ and $Y_2Cu_{0.20}Sn_{0.95}S_5$

The formation of  $R_2Sn_5$  ( $R = Pr, Nd, Gd$  and  $Tb$ ) was confirmed during the investigation of the phase relations in the respective  $R_2S_3-SnS_2$  systems. X-ray single-crystal diffraction was used for the investigation of the crystal structure of these compounds. The crystal data and the structure refinement details are summarized in Table 1, and the refined

atomic coordinates and the anisotropic displacement parameters have been deposited.<sup>1</sup> The positions of all the atoms are fully occupied. Generally, the results of the single-crystal investigation of  $R_2Sn_5$  ( $R = Pr, Nd, Gd$  and  $Tb$ ) are in good agreement with the data reported previously (Julien-Pouzou & Jaulmes, 1979).

The relevant interatomic distances and coordination numbers of the atoms are listed in Table 2, and related coordination polyhedra for each atom are shown in Fig. 1. The coordination sphere for the lanthanide and Sn atoms can be described as tricapped trigonal prismatic [ $RS_1S_2S_3S_4$ ] and octahedral [ $SnS_4S_2$ ], respectively. Lanthanide trigonal prisms dominate in the structure. They are connected to each other by faces along the  $[110]$  and  $[\bar{1}\bar{1}0]$  directions and

<sup>1</sup> Supplementary data for this paper are available from the IUCr electronic archives (Reference: BS5058). Services for accessing these data are described at the back of the journal.

**Table 2**

Interatomic distances ( $\delta$ , Å) and coordination numbers (CN) of the atoms  $R$  and Sn atoms in the structures of  $R_2\text{SnS}_5$  ( $R = \text{Pr, Nd, Gd}$  and  $\text{Tb}$ ).

	$\text{Pr}_2\text{SnS}_5$	$\text{Nd}_2\text{SnS}_5$	$\text{Gd}_2\text{SnS}_5$	$\text{Tb}_2\text{SnS}_5$	CN
$R-2\text{S3}$	2.7876 (3)	2.7770 (5)	2.7444 (5)	2.7328 (4)	9 (8†)
$R-2\text{S2}$	2.9775 (7)	2.9618 (16)	2.9082 (15)	2.8948 (10)	–
$R-2\text{S2}$	2.9892 (8)	2.9779 (17)	2.9704 (15)	2.9653 (12)	–
$R-1\text{S1}$	3.0211 (10)	2.993 (2)	2.871 (2)	2.8381 (12)	–
$R-1\text{S1}$	3.1618 (10)	3.110 (2)	2.968 (2)	2.9359 (15)	–
$R-1\text{S1}$	3.351 (2)	3.409 (3)	3.720 (3)†	3.756 (2)†	–
$\text{Sn}-2\text{S2}$	2.5433 (9)	2.549 (2)	2.568 (2)	2.5686 (12)	6
$\text{Sn}-4\text{S1}$	2.5879 (6)	2.5782 (14)	2.5472 (13)	2.5445 (9)	–

† Owing to the long  $R-S1$  distance, the third S1 atom is not included in the coordination sphere.

translated along the  $c$  axis (Fig. 2). Additionally, tin octahedra are connected to each other by edges forming a chain along the  $c$  axis. Thus, it appears that a one-dimensional Sn–S sublattice is embedded in the three-dimensional Ln–S lattice.

All of the interatomic distances are close to the sum of the respective ionic radii (Wiberg, 1995). However, the ionic radius for gadolinium and terbium ions is smaller than for praseodymium and neodymium ions. Therefore, one  $R-S1$  distance for  $\text{Gd}_2\text{SnS}_5$  and  $\text{Tb}_2\text{SnS}_5$  is too long to consider as a noteworthy interatomic (bonding) interaction (Table 2). Thus, bicapped trigonal prismatic surroundings with a coordination number (CN) = 8 for Gd and Tb [ $\text{RS1}_2\text{S2}_4\text{S3}_2$ ] is probably the best description.

The similarity of the crystal structures of  $R_2\text{SnS}_5$  ( $R = \text{La, Ce, Pr, Nd, Sm, Gd, Tb}$  and  $\text{Dy}$ ) and  $\text{U}_3\text{S}_5$ ,  $\text{Y}_2\text{HfS}_5$ ,  $\text{Eu}_2\text{SnS}_4$ ,  $\text{Eu}_2\text{SnS}_5$ ,  $\text{Y}_2\text{Cu}_{0.20}\text{Sn}_{0.95}\text{S}_5$ ,  $\text{U}_2\text{SnS}_5$  is described below. When the  $\text{U}_3\text{S}_5$  structure is considered as a basic structure, two different U atoms must be located in the lattice, *i.e.* three- and four-valent uranium. A simple substitution, each of them with an isovalent ion, can facilitate the production of new compounds. For example, it can be clearly seen that  $\text{Y}_2\text{HfS}_5$  adopts the crystal structure and symmetry of  $\text{U}_3\text{S}_5$  (Potel *et al.*, 1972; see Fig. 2*a*). Three- and four-valent uranium ions correspond to three-valent yttrium and four-valent hafnium ions in  $\text{Y}_2\text{HfS}_5$ , respectively (Jeitschko & Donohue, 1975).

The unit cell of  $\text{Y}_2\text{HfS}_5$  can be obtained by doubling of the unit cell of  $R_2\text{SnS}_5$  in the  $c$  direction (Fig. 2*b*). Of course, this operation involves respective changes in symmetry elements and the space group  $Pnma$  is replaced with  $Pbam$ .

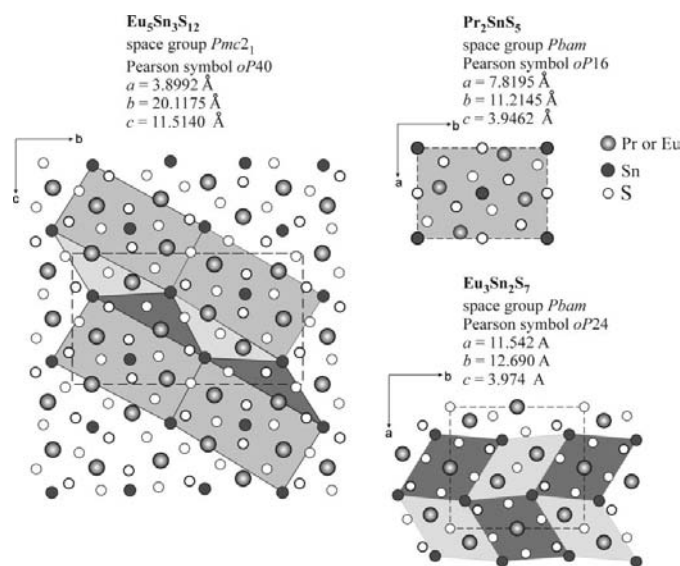
During the investigation of the phase relations in the quasi-ternary  $\text{Y}_2\text{S}_3\text{–Cu}_2\text{S–SnS}_2$  system (Shemet *et al.*, 2006), the formation of the non-stoichiometric compound  $\text{Y}_2\text{Cu}_{0.20}\text{Sn}_{0.95}\text{S}_5$  was observed, which crystallizes in the space group  $Pbam$ . The crystal structure of this compound is closely related to the  $R_2\text{SnS}_5$  structure and it can be obtained from a hypothetical ‘ $\text{Y}_2\text{SnS}_5$ ’ structure ( $\text{La}_2\text{SnS}_5$  structure type) by the substitution of four-valent Sn by monovalent Cu. However, the Sn atoms have octahedral surroundings, but because of the small size of the copper ion an octahedral coordination is not observed for Cu rare-earth chalcogenides. The Cu atoms in  $\text{Y}_2\text{Cu}_{0.20}\text{Sn}_{0.95}\text{S}_5$  are located in separate positions with tetrahedral coordination (Fig. 2*c*). The stabi-

lization provided by the presence of a small amount of Cu in  $\text{Y}_2\text{Cu}_{0.20}\text{Sn}_{0.95}\text{S}_5$  allows the formation of this type of compound in the  $\text{Y}_2\text{S}_3\text{–Cu}_2\text{S–SnS}_2$  system. When a series of  $R_2\text{SnS}_5$  compounds are compared a significant increase of the one  $R-S$  distance is observed when the ionic radius of the  $R$  ion decreases. Since the smaller ionic radius of  $\text{Y}^{3+}$  involves a subsequent increase of the  $R-S$  distance, a simple substitution of the lanthanide ion by  $\text{Y}^{3+}$  allows the incorporation of a dopant ( $\text{Cu}^+$  ions).

### 3.2. Crystal architecture of complex compounds

A similar trigonal prismatic arrangement of four-valent U, Hf and Sn is observed for the  $\text{U}_3\text{S}_5$ ,  $\text{Y}_2\text{HfS}_5$ ,  $\text{Pr}_2\text{SnS}_5$  and  $\text{Y}_2\text{Cu}_{0.20}\text{Sn}_{0.95}\text{S}_5$  compounds described above. However, for  $\text{Eu}_2\text{SnS}_4$  seven S ions are located around the Eu ion on the apical positions of a mono-capped trigonal prism. The arrangement of the trigonal prisms along the  $b$  axis in  $\text{Eu}_2\text{SnS}_4$  (Fig. 2*d*; Pocha *et al.*, 2003) is similar to that along the  $c$  axis for  $R_2\text{SnS}_5$  compounds and along the  $b$  axis for  $\text{U}_3\text{S}_5$  ( $\text{Y}_2\text{HfS}_5$ ). Blocks of paired tri-capped trigonal prisms in the structures of  $\text{Eu}_2\text{SnS}_5$  (Fig. 2*d*; Jaulmes *et al.*, 1982) and  $R_2\text{SnS}_5$  ( $\text{La}_2\text{SnS}_5$  structure type) are similar within local space, but overall the structural fragments of  $\text{Eu}_2\text{SnS}_5$  and  $R_2\text{SnS}_5$  ( $\text{La}_2\text{SnS}_5$  structure type) are different.

In the monoclinic structure of  $\text{U}_2\text{FeS}_5$  (Noël *et al.*, 1976) the U trigonal prisms and Fe octahedra create two distinguishable layers in the  $ac$  plane and they are similar to those in  $R_2\text{SnS}_5$  ( $\text{La}_2\text{SnS}_5$  structure type, Fig. 2*e*). However, these two layers are shifted relative to each other according to the symmetry operations in the  $C2/c$  space group. In the orthorhombic structures of  $R_2\text{SnS}_5$  the adjacent layers are located exactly one above the other. Therefore, the crystal structures of  $\text{U}_2\text{SnS}_5$  and  $R_2\text{SnS}_5$  ( $\text{La}_2\text{SnS}_5$  structure type) are similar in

**Figure 3**

The distorted fragments of  $R_2\text{SnS}_5$  ( $\text{La}_2\text{SnS}_5$  structure type) and  $\text{Eu}_3\text{Sn}_2\text{S}_7$  in the structure of  $\text{Eu}_5\text{Sn}_3\text{S}_{12}$ . Similar fragments are marked by similar colours.

local space, but overall they have a different architecture which is reflected in a different set of the symmetry elements.

The unit cell of  $\text{Eu}_5\text{Sn}_3\text{S}_{12}$  (Jaulmes & Julien-Pouzol, 1977a) can be completely constructed by the interweaving of the distorted fragments of  $R_2\text{SnS}_5$  ( $\text{La}_2\text{SnS}_5$  structure type) and  $\text{Eu}_3\text{Sn}_2\text{S}_7$  (Jaulmes & Julien-Pouzol, 1977b; Fig. 3). Since di- and trivalent europium ions are present in the crystal structure of ternary  $\text{Eu}_5\text{Sn}_3\text{S}_{12}$ , the ordered substitutions of divalent Eu by divalent Pb and trivalent Eu by trivalent R (rare-earth element) are possible. The successful formation of quaternary compounds was reported recently for  $\text{Eu}_4\text{LuSn}_3\text{S}_{12}$  (Jakubcová *et al.*, 2007) and  $R_2\text{Pb}_3\text{Sn}_3\text{S}_{12}$  ( $R = \text{Y, La, Ce, Pr, Nd, Sm, Gd, Tb, Dy, Ho, Er}$  and  $\text{Tm}$ ; Marchuk *et al.*, 2007; Gulay *et al.*, 2008).

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