

Structure of Er<sub>5</sub>Sb<sub>3</sub>

BY G. BORZONE

Istituto di Chimica Generale, Università di Genova, Viale Benedetto XV 3, 16132 Genova, Italy

AND M. L. FORNASINI

Istituto di Chimica Fisica, Università di Genova, Corso Europa 26, 16132 Genova, Italy

(Received 2 April 1990; accepted 10 May 1990)

**Abstract.** Pentaerbium triantimonide, *oP*32,  $M_r = 1201.55$ , orthorhombic, *Pnma*,  $a = 11.662$  (1),  $b = 9.136$  (1),  $c = 8.007$  (1) Å,  $V = 853.1$  (2) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 9.36$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $\mu = 59.8$  mm<sup>-1</sup>,  $F(000) = 1972$ ,  $T = 293$  K,  $R = 0.040$  for 732 reflections with  $F_o > 2\sigma(F_o)$ . The title compound is isotypic with  $\beta\text{-Yb}_5\text{Sb}_3$  and around the Sb atoms two coordination polyhedra with erbium at the vertices are found: a CN 9 tricapped trigonal prism and a CN 8 dodecahedron.

**Experimental.** The metals used were erbium and antimony with nominal purities of 99.9 and 99.99 wt% respectively. Samples (weighing 1–2 g), having nominal compositions near to 37.5 at.% Sb, were prepared from the weighed amounts of the two elements enclosed in small tantalum crucibles closed by welding under an argon atmosphere. They were melted in an induction furnace and then cooled. Metallographic examination was carried out on different samples: homogeneous samples were observed for the 5:3 composition.

Single-crystal and powder X-ray diffraction techniques were used to identify the phase and determine the structure. Intensities were collected from a prismatic crystal elongated in the *b* direction with dimensions 0.02 × 0.03 × 0.10 mm, on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo *K*α radiation. Cell parameters were obtained from 25 diffractometer-measured reflections with  $18 < \theta < 22^\circ$ . 1034 independent reflections were recorded in the  $\omega$ - $\theta$  scan mode up to  $\sin\theta/\lambda = 0.65$  Å<sup>-1</sup>,  $0 \leq h \leq 15$ ,  $0 \leq k \leq 11$ ,  $0 \leq l \leq 10$ . Six orientation and one intensity reference reflections were checked every 100 reflections and every hour respectively and showed no significant variation. A semi-empirical absorption correction was applied by azimuthal scans of two top reflections, with a ratio of 1.6 between maximum and minimum transmission factors. 302 reflections with  $F_o < 2\sigma(F_o)$  were considered as unobserved. The atomic coordinates of  $\beta\text{-Yb}_5\text{Sb}_3$  (Brunton & Steinfink, 1971) were used as

Table 1. Atomic coordinates and isotropic equivalent thermal parameters (Å<sup>2</sup> × 10<sup>2</sup>) for Er<sub>5</sub>Sb<sub>3</sub>

E.s.d.'s are in parentheses.  $U_{\text{eq}} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$ .

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$
Er1	8( <i>d</i> ) 0.0666 (1)	0.0575 (1)	0.1906 (1)	0.77 (3)
Er2	4( <i>c</i> ) 0.0043 (1)	0.25	0.5255 (2)	0.69 (4)
Er3	4( <i>c</i> ) 0.2280 (1)	0.25	0.8258 (2)	0.80 (4)
Er4	4( <i>c</i> ) 0.2914 (1)	0.25	0.3444 (2)	0.81 (4)
Sb1	8( <i>d</i> ) 0.3261 (1)	0.0094 (2)	0.0646 (2)	0.72 (4)
Sb2	4( <i>c</i> ) 0.4767 (2)	0.25	0.5948 (3)	0.73 (5)

Table 2. Interatomic distances (Å) in Er<sub>5</sub>Sb<sub>3</sub>, up to  $d/\sum r = 1.12$ 

E.s.d.'s are less than 0.01 Å.

Er1—Sb2	2.96	Er3—Sb2	3.00
—Sb2	3.07	—2Sb1	3.13
—Sb1	3.22	—2Sb1	3.22
—Er2	3.29	—Er2	3.43
—Sb1	3.30	—Sb2	3.44
—Er4	3.39	—Er2	3.55
—Sb1	3.45	—2Er1	3.85
—Er1	3.52	—2Er1	3.89
—Er1	3.58	—Er4	3.92
—Er4	3.67		
—Er2	3.71	Er4—Sb2	2.95
—Er3	3.85	—2Sb1	3.16
—Er3	3.89	—2Sb1	3.26
		—2Er1	3.39
Er2—Sb2	3.06	—Er2	3.65
—2Sb1	3.10	—2Er1	3.67
—2Sb1	3.11	—Er2	3.86
—2Er1	3.29	—Er3	3.92
—Er3	3.43		
—Er3	3.55	Sb1—Er2	3.10
—Er4	3.65	—Er2	3.11
—2Er1	3.71	—Er3	3.13
—Er4	3.86	—Er4	3.16
		—Er1	3.22
Sb2—Er4	2.95	—Er3	3.22
—2Er1	2.96	—Er4	3.26
—Er3	3.00	—Er1	3.30
—Er2	3.06	—Er1	3.45
—2Er1	3.07		
—Er3	3.44		

starting values in the refinement. Anisotropic refinement on *F* of 44 parameters led to  $R = 0.040$ ,  $wR = 0.037$  with  $w = [\sigma^2(F_o) + 0.000251F_o^2]^{-1}$ ,  $S = 1.06$ ,  $(\Delta/\sigma)_{\text{max}} = 0.001$ , max. and min. heights in final

difference Fourier synthesis 3.6 and  $-4.3 \text{ e } \text{Å}^{-3}$ . An extinction correction was applied according to the equation  $F_c' = F_c(1 - 2.5 \times 10^{-8} F_c^2 / \sin \theta)$ . Atomic scattering factors and anomalous-dispersion correction were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Programs used: *SHELX76* (Sheldrick, 1976) for structure refinement and *STRUCTURE TIDY* (Gelato & Parthé, 1987) for structure standardization. Table 1 reports atomic coordinates and isotropic equivalent thermal parameters, while interatomic distances up to  $d/\sum r = 1.12$  ( $\sum r$  is the sum of the metallic radii for CN 12) are given in Table 2.\* From the powder photographs (Fe  $K\alpha$  radiation) of the same samples the following unit-cell dimensions were obtained:  $a = 11.664$  (4),  $b = 9.138$  (4),  $c = 8.010$  (4) Å, in good agreement with those obtained by single-crystal diffraction.

**Related literature.** In  $\text{Er}_5\text{Sb}_3$  two coordination polyhedra are found around the Sb atoms: a CN 9

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53081 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

truncated trigonal prism and a CN 8 dodecahedron (or bisdisphenoid) with only Er at the vertices as no Sb—Sb contacts occur. The  $\beta\text{-Yb}_5\text{Sb}_3$  structure is adopted by several compounds formed by alkaline earths, rare earths or transition metals (e.g. Ti, V, Cr) with P, As, Sb and Bi (Villars & Calvert, 1985). Another phase,  $\text{Y}_5\text{Bi}_3$  (Wang, Gabe, Calvert & Taylor, 1976), crystallizing in the same space group *Pnma*, has lattice constants very similar to those of  $\beta\text{-Yb}_5\text{Sb}_3$  but with the  $a$  and  $c$  periods interchanged so that these two compounds cannot be considered as isotypic although their structures are very closely related. The similarity and the description in the same group is made possible only by the particular values of the coordinates in the two structures.

#### References

- BRUNTON, G. D. & STEINFINK, H. (1971). *Inorg. Chem.* **10**, 2301–2303.  
 GELATO, L. M. & PARTHÉ, E. (1987). *J. Appl. Cryst.* **20**, 139–143.  
 SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.  
 VILLARS, P. & CALVERT, L. D. (1985). *Pearson's Handbook of Crystallographic Data for Intermetallic Phases*. Metals Park: American Society for Metals.  
 WANG, Y., GABE, E. J., CALVERT, L. D. & TAYLOR, J. B. (1976). *Acta Cryst.* **B32**, 1440–1445.

*Acta Cryst.* (1990). **C46**, 2457–2458

## Structure of [1,2-Bis(diphenylphosphino)ethane]di(phenylthio)palladium(II)

BY GUOWEI WEI AND HANQIN LIU\*

*Fujian Institute of Research on the Structure of Matter and Fuzhou Laboratory of Structural Chemistry, Academia Sinica, Fuzhou, Fujian 350002, People's Republic of China*

(Received 11 April 1990; accepted 30 May 1990)

**Abstract.**  $[\text{Pd}(\text{C}_6\text{H}_5\text{S})_2\{(\text{C}_6\text{H}_5)_2\text{PCH}_2\text{CH}_2\text{P}(\text{C}_6\text{H}_5)_2\}]$ ,  $M_r = 723.17$ , monoclinic,  $C2/c$ ,  $a = 14.320$  (2),  $b = 16.383$  (2),  $c = 14.295$  (2) Å,  $\beta = 93.41$  (2)°,  $V = 3347.7$  (9) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.435 \text{ g cm}^{-3}$ , Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å,  $\mu = 7.856 \text{ cm}^{-1}$ ,  $F(000) = 1480$ ,  $T = 296$  (1) K,  $R = 0.0269$  for 3084 reflections with  $I > 3\sigma(I)$ . The molecule possesses a twofold axis passing through Pd and the midpoints of S—S and P—P with these five atoms being essentially coplanar.

**Experimental.** The title compound was prepared by reaction of  $\text{PdCl}_2$ ,  $\text{NaSPh}$ , 1,2-bis(diphenylphosphino)ethane (dppe), and elemental selenium in

DMF solution, and a crystal suitable for the X-ray analysis was grown in a mixed solution of DMF and acetone. Details of the sample preparation and the crystal growth are similar to those for the Co compounds (Wei, Liu, Huang, Huang & Kang, 1989). This synthetic method differs from that of Hayter & Humiec (1964).

Diffraction intensities were collected from a red prismatic crystal  $0.20 \times 0.22 \times 0.30$  mm, in the  $\omega$ - $2\theta$  scan mode on an Enraf-Nonius CAD-4 diffractometer using graphite-monochromated Mo  $K\alpha$  radiation. 25 reflections ( $10 < \theta < 15^\circ$ ) were used for measuring lattice parameters. 4437 unique reflections measured in the index range  $0 < h < 19$ ,  $0 < k < 22$ ,  $-19 < l < 19$ ,  $2\theta_{\text{max}} = 30^\circ$ . The intensities of three standard reflections monitored periodically

\* To whom correspondence should be addressed.