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Key indicators

Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{Pd-Pd}) = 0.002\text{ \AA}$
 R factor = 0.045
 wR factor = 0.101
Data-to-parameter ratio = 29.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The layered intermetallic compound LaPdSb_3 Received 24 February 2006
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The title compound, lanthanum palladium triantimony, adopts a new structure type and is an intermediate between the CeCrSb_3 and CeNiSb_3 structure types. Its structure consists of La atoms located above and below layers of nearly square buckled Sb nets ($\infty^2[\text{Sb}]$), and layers of highly distorted edge- and face-sharing PdSb_6 octahedra ($\infty^2[\text{PdSb}_2]$).

Comment

Compounds containing lanthanides, transition metals and antimony (Ln-T-Sb) are of interest, particularly because of their attractive physical properties and the rich variety of geometric networks, such as Sb zigzag chains, Sb_2 pairs and Sb_4 squares (Kauzlarich, 1996; Papoian & Hoffmann, 2000; Mills *et al.*, 2002; Sologub & Salamakha, 2003). Investigation of the LnTSb_3 phases (Macaluso *et al.*, 2004; Thomas *et al.* 2004) by systematic substitutions of the lanthanide and transition metals has led to the discovery of LaPdSb_3 and CePdSb_3 , the latter of which has recently been studied by Thamizhavel *et al.* (2005). We report here the results of a structural investigation of LaPdSb_3 using single-crystal X-ray diffraction.

The structure of LaPdSb_3 is shown in Fig. 1. It consists of La atoms positioned between layers of nearly square nets of Sb atoms ($\infty^2[\text{Sb}]$) and layers of slightly distorted face- and edge-sharing Pd-centered octahedra ($\infty^2[\text{PdSb}_2]$). The nearly square

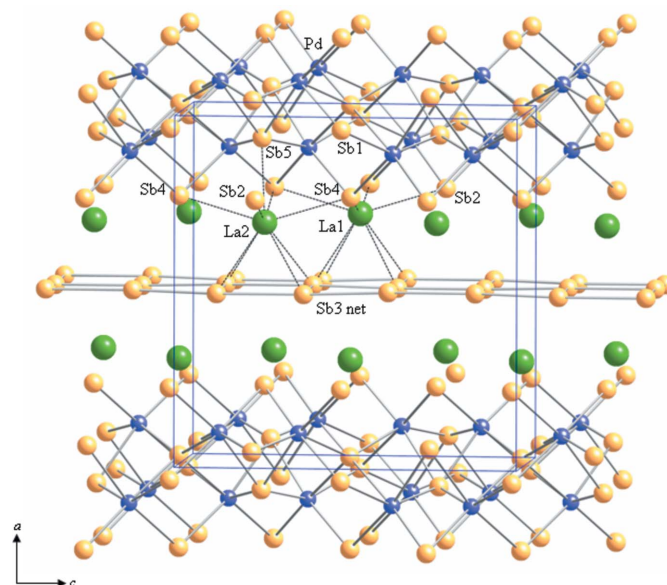


Figure 1

The structure of LaPdSb_3 , viewed down $[010]$. Green spheres denote La atoms, blue spheres Pd atoms and yellow spheres Sb atoms. The mono-capped square anti-prismatic and square anti-prismatic environments of the La1 and La2 atoms, respectively, are shown as dashed lines.

∞^2 [Sb] nets lie in the *bc* plane and are composed of Sb3 atoms which are equally separated along [010] but alternate between long and short separations along [001]. The Sb3—Sb3—Sb3 angles within the ∞^2 [Sb] nets deviate from 90°.

The Pd atoms in LaPdSb₃ are coordinated by six Sb atoms, forming distorted Pd(Sb1)_{2/2}(Sb2)_{1/2}(Sb4)_{1/2}(Sb5)_{2/2} octahedra that are edge-sharing in [010], whereas they are face-sharing, with every other octahedron sharing edges, in [001]. The Pd—Sb distances are comparable with the 2.737 Å Pd—Sb separations found in the binary compound PdSb (NiAs-type) (Pratt *et al.*, 1968). The Pd-centered octahedra of LaPdSb₃ are highly distorted, with Sb—Pd—Sb angles as acute as 73.87 (3)°. Some Pd···Pd interactions are also observed between the face-sharing octahedra along [001]. The distances are 2.895 (2) Å, slightly longer but comparable with the distances of 2.751 Å in Pd metal (Donohue, 1974). The short Pd···Pd distances contribute to the distortions within the ∞^2 [PdSb₂] layers.

The La atoms are located both above and below the ∞^2 [Sb] nets in a checkered fashion. The La1 atoms adopt an eight-coordinate square anti-prismatic geometry similar to that of the Ln atoms found in some LnTSb₂ compounds (Pankevich *et al.*, 1983; Brylak *et al.*, 1995; Ferguson *et al.*, 1996; Wollesen *et al.*, 1996; Mills & Mar, 2001). On the other hand, the La2 atoms in LaPdSb₃ adopt a nine-coordinate mono-capped square anti-prismatic geometry similar to the Ln environments in LnSb₂, LnIn_{1-x}Sb₂, CeCrSb₃ and CeNiSb₃ (Wang & Steinfink, 1967; Brylak & Jeitschko, 1995; Ferguson *et al.*, 1999; Macaluso *et al.*, 2004).

Experimental

Single crystals of LaPdSb₃ were synthesized by combining ingots of La (99.9% purity, Alfa Aesar), Pd powder (99.999% purity, Alfa Aesar) and Sb shot (99.9999% purity, Alfa Aesar) into alumina crucibles in a 1:1:20 (Ln:Pd:Sb) molar ratio. Each crucible was sealed into an evacuated 10 cm fused-silica tube. The samples were heated to 1423 K for 10 h, then cooled at 5 K h⁻¹ to 943 K, at which temperature they were removed from the furnace. Upon removal of the samples, the excess Sb flux was removed by centrifugation. After cooling to room temperature, the resulting silver block-shaped crystals, with dimensions of up to 1 × 2 × 2 mm, were mechanically extracted. The LnPdSb₃ (Ln = La, Ce) compounds show little, if any, surface degradation as a result of exposure to air and moisture for extended periods. Sample homogeneity was determined from multiple lattice parameter determinations of single crystals as well as by powder X-ray diffraction.

Crystal data

LaPdSb₃
M_r = 610.56
 Orthorhombic, *Pbcm*
a = 12.9210 (4) Å
b = 6.3450 (9) Å
c = 12.5030 (9) Å
V = 1025.04 (17) Å³
Z = 8
D_x = 7.913 Mg m⁻³

Mo *K*α radiation
 Cell parameters from 1722 reflections
 θ = 1.0–30.0°
 μ = 27.02 mm⁻¹
T = 298 (2) K
 Block-shaped fragment, silver
 0.10 × 0.10 × 0.08 mm

Data collection

Nonius KappaCCD area-detector diffractometer
 ω scans with κ offsets
 Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor 1997)
 T_{\min} = 0.092, T_{\max} = 0.12
 2790 measured reflections

1557 independent reflections
 1016 reflections with $I > 2\sigma(I)$
 R_{int} = 0.063
 θ_{\max} = 30.0°
 h = -18 → 18
 k = -8 → 8
 l = -17 → 17

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)]$ = 0.045
 $wR(F^2)$ = 0.101
 S = 1.08
 1557 reflections
 52 parameters

$w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 2.98 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -3.60 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

La1—Sb2 ⁱ	3.2850 (5)	Pd1—Sb1 ^{ix}	2.6779 (12)
La1—Sb4 ⁱⁱ	3.2918 (6)	Pd1—Sb5 ^x	2.6840 (14)
La1—Sb3 ⁱⁱⁱ	3.3787 (11)	Pd1—Sb4 ^{xi}	2.7097 (13)
La1—Sb3 ⁱ	3.3936 (11)	Pd1—Sb1 ^{xii}	2.7113 (11)
La1—Sb1	3.5629 (15)	Pd1—Sb2 ^{vii}	2.7888 (14)
La1—Pd1 ^{iv}	3.6026 (12)	Pd1—Sb5 ^{vii}	2.8320 (15)
La2—Sb4 ^v	3.2774 (5)	Pd1—Pd1 ^{viii}	2.895 (2)
La2—Sb2 ^{vi}	3.2915 (17)	Sb1—Sb1 ^{xiii}	3.2384 (7)
La2—Sb5 ^{vii}	3.3170 (15)	Sb2—Sb5 ^{xiv}	3.0052 (17)
La2—Sb3 ^{viii}	3.3443 (12)	Sb3—Sb3 ^{xv}	3.0535 (16)
La2—Sb3 ^{vii}	3.3576 (12)	Sb3—Sb3 ^{vii}	3.1735 (13)
La2—Pd1 ^{viii}	3.3598 (13)	Sb3—Sb3 ^{viii}	3.2032 (16)
La2—Sb2 ^{vii}	3.4252 (17)		
Sb1 ^{ix} —Pd1—Sb1 ^{xii}	73.87 (3)	Sb4 ^{xi} —Pd1—Sb5 ^{vii}	91.79 (4)
Sb1 ^{ix} —Pd1—Sb2 ^{vii}	168.35 (5)	Sb3 ^{xv} —Sb3—Sb3 ^{vii}	86.95 (3)
Sb1 ^{ix} —Pd1—Sb5 ^{vii}	78.72 (3)	Sb3 ^{xv} —Sb3—Sb3 ^{vi}	92.98 (3)
Sb5 ^x —Pd1—Sb5 ^{vii}	80.15 (3)		

Symmetry codes: (i) $x, -y + \frac{1}{2}, -z + 1$; (ii) $-x + 1, -y, -z$; (iii) $-x + 1, y - \frac{1}{2}, z - 1$; (iv) $-x + 1, y + \frac{1}{2}, z - 1$; (v) $x, y, -z + \frac{1}{2}$; (vi) $-x + 1, y + \frac{1}{2}, z$; (vii) $-x + 1, y - \frac{1}{2}, z$; (viii) $x, y, -z + \frac{3}{2}$; (ix) $x - 1, y, z + 1$; (x) $x - 1, y - 1, z$; (xi) $x, y, z + 1$; (xii) $-x + 1, -y, -z + 1$; (xiii) $-x + 2, -y, -z$; (xiv) $x, y - 1, z$; (xv) $-x + 1, -y + 1, -z + 2$.

As a check for partial occupancy within the structure, the atomic occupancy parameters were refined in separate sets of least-squares refinements. No significant deviations in the atomic occupancy parameters were found. The largest peak and deepest hole are located 0.99 Å and 1.56 Å, respectively, from Sb2.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *CRYSTALMAKER* (Palmer, 2003); software used to prepare material for publication: *SHELXL97* and *enCIFer* (Allen *et al.*, 2004).

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