SHORT-FORMAT PAPERS

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Cadmium Antimonide, CdSb

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Abstract. $M_r = 234{\cdot}16$, orthorhombic, *Pbca*, $a = 6{\cdot}469\,(1)$, $b = 8{\cdot}251\,(2)$, $c = 8{\cdot}522\,(2)$ Å, $V = 454{\cdot}87$ Å^3, $Z = 8$, $D_x = 6{\cdot}84$ Mg m^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71073$ Å, $\mu = 29{\cdot}6$ mm^{-1}, $F(000) = 792$, room temperature. The structure was refined to $R = 0{\cdot}043$, $wR = 0{\cdot}031$ for 394 absorption-corrected observed reflections. The results confirm the essential features of the structure as determined from photographic data by Almin [*Acta Chem. Scand.* (1948), **2**, 400–407]. The Cd—Cd distance, however, is significantly longer than previously found and has the same value as the mean Cd—Cd distance in metallic cadmium (3·136 Å).

Experimental. Single crystals of CdSb could be isolated from a regulus, obtained by melting a 1 : 1 mixture of Cd and Sb in an evacuated sealed quartz ampoule, and subsequent annealing at 725 K for 11 d, 705 K for 6 d, and 685 K for 10 d. Guinier patterns showed the product to be single phase. A silvery needle-shaped crystal fragment (approximately 0·04 × 0·04 × 0·22 mm) was used for data collection on an Enraf–Nonius CAD-4 diffractometer (Mo $K\alpha$, graphite monochromator). Lattice parameters were refined from 2θ values of 25 reflections in the range 8·5–30°. Intensities measured for $2 \le \theta \le 25°$; ω-scan technique (scan width $1{\cdot}0° + 0{\cdot}4°\text{tg}\theta$). Three standard reflections indicated no loss of intensity during data collection. Merging of the 1714 collected intensities [$(\sin\theta)_{\text{max}}/\lambda = 0{\cdot}595$ Å^{-1}; $-7 \le h \le 7$, $-9 \le k \le 9$, $0 \le l \le 10$] gave 394 unique reflections ($R_{\text{int}} = 0{\cdot}022$) with $F > 3\sigma(F)$, which were considered as observed and used for all calculations (program system *SDP*; Enraf–Nonius, 1985).

The structure was solved by standard direct methods. In least-squares refinement $|F|$ magnitudes were used to refine atomic coordinates, occupation factors and isotropic temperature factors. Convergence was obtained after a few cycles with s.o.f.'s for Cd [0·998 (4)] and Sb [1·003 (4)], corresponding to a stoichiometry CdSb within one standard deviation. At this stage ($R_{\text{iso}} = 0{\cdot}051$), the s.o.f.'s were fixed again at 100% and a numerical correction for absorption (program *DIFABS*; Walker & Stuart, 1983) was applied before performing the final anisotropic refinement. Final $R = 0{\cdot}043$, $wR = 0{\cdot}031$, $w^{-1} = \sigma^2(F)$, $(\Delta/\sigma)_{\text{max}} < 0{\cdot}001$ in final refinement cycle, 19 parameters, $S = 0{\cdot}536$. Max. features in the final $\Delta\rho$ map $\pm 1{\cdot}9$ e Å^{-3}. Atomic scattering factors and f', f'' values were set by the *SDP* program. Bond distances and angles were calculated using the program *SADIAN* (Baur & Wenninger, 1969). Final atomic coordinates and equivalent isotropic temperature factors are given in Table 1, derived bond distances in Table 2.[†]

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2)

$$B_{\text{eq}} = \tfrac{1}{3}(B_{11} + B_{22} + B_{33}).$$

	x	y	z	B_{eq}
Cd	0·5503 (1)	0·6238 (1)	0·63426 (8)	1·91 (1)
Sb	0·13980 (9)	0·07390 (9)	0·10390 (7)	1·20 (1)

Table 2. *Interatomic distances* (Å)

Cd—Sb	2·793 (1)	Cd—Sb	3·127 (1)
—Sb	2·824 (1)	—Cd	3·136 (1)
—Sb	2·882 (1)	Sb—Sb	2·810 (1)

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[†] Lists of structure factors, anisotropic thermal parameters and bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51306 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Related literature. The present investigation confirms the essential features of the CdSb structure as determined from photographic data by Almin (1948). The most important difference is the Cd—Cd distance, which is significantly longer than previously found and has the same value [3·136 (1) Å] as the mean Cd—Cd distance in metallic cadmium (Donohue, 1974). This excludes the possibility of substantial covalent interactions between the Cd atoms, in spite of the fact that the Cd atoms can be grouped into pairs in the structure. The Sb—Sb distance [2·810 (1) Å] agrees well with that in other diantimonides (Donaldson, Kjekshus, Nicholson & Rakke, 1975). The description of the CdSb-type structure {also adopted by ZnSb (Almin, 1948) and the high-pressure phases ZnAs and CdAs (Clark & Range, 1975, 1976)} as a heavily distorted diamond structure (Almin, 1948) is rather arbitrary. It should better be viewed as a binary variant of Si-III (Kasper & Richards, 1964), which in turn is an example of a dense sphere packing in space group $Ia3$ (Fischer, 1973). The group–subgroup relations between this sphere packing and the CdSb-type structure have already been discussed (Clark & Range, 1976).

For the stable and metastable phase diagram of the Cd–Sb system see Hansen (1958). Belash & Ponyatovskii (1974) reported a high-pressure decomposition of CdSb into Cd and Sb at 3·5 GPa which is unlikely from the phase diagram and could not be verified in our own high-pressure experiments.

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(Trifluoroacetato)(triphenylphosphine)gold(I)

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Abstract. $[Au(C_2F_3O_2)\{(C_6H_5)_3P\}]$, $M_r = 572\cdot27$, orthorhombic, $P2_12_12_1$, $a = 11\cdot771$ (2), $b = 11\cdot906$ (3), $c = 14\cdot169$ (3) Å, $V = 1986\cdot0$ (7) Å^3, $Z = 4$, $D_x = 1\cdot914$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 75\cdot12$ cm^{-1}, $F(000) = 1088$. Final $R = 0\cdot036$ for 1309 absorption-corrected reflections. Not isostructural with Ph_3AuCl, Ph_3AuBr, and $Ph_3Au(O_2CCH_3)$. The Au—O bond length is 2·107 (9) Å with Au—P equal to 2·208 (4) Å.

Experimental. The title compound was prepared by the reaction of Ph_3PAuCl with AgO_2CCF_3 in anhydrous toluene. Crystals were grown from anhydrous petroleum ether. A colorless crystal 0·30 × 0·2 × 0·2 mm was used in this study. The X-ray data were measured using a Nicolet $R3m$ diffractometer with Mo $K\alpha$ radiation and a graphite monochromator. Cell constants were determined using 25 reflections in the 2θ range 11–22°. A total of 2045 reflections in the 2θ range 3–50° (h 0 to 14, k 0 to 15, l 0 to 17) was measured. Two standard reflections were measured after every 98 reflections and were used to correct small variations (±2·5% or ±1·6σ) in their intensities. The data were corrected for absorption using the ψ-scan

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