Crystal structure of palladium selenide, PdSe

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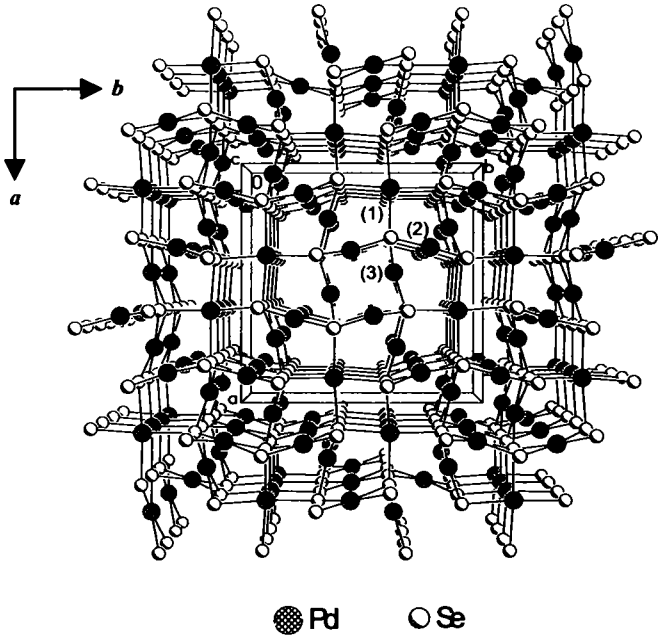

Abstract

PdSe, tetragonal, $P4_2/mbc$ (No. 135), $a = 11.5646(6)$ Å, $c = 6.9978(6)$ Å, $V = 935.9$ Å^3, $Z = 24$, $R_{gt}(F) = 0.034$, $wR_{ref}(F^2) = 0.093$, $T = 153$ K.

Source of material

Single crystals of PdSe were obtained in the reaction of Bi (0.0819 g, Alfa, 99.9%), Pd (0.1252 g, Johnson Matthey, 99.95%), and Se (0.0929 g, Alfa, 99.5%). The elements were thoroughly mixed and sealed in a carbon-coated fused-silica tube that was then evacuated to $5 \cdot 10^{-5}$ Torr. A computer-controlled furnace was used to heat the sample from 300 K to 923 K in 48 h. This temperature was maintained for 72 h and then the sample was quenched to room temperature in water. Black block-like single crystals of PdSe were found. Qualitative energy dispersive spectroscopy (EDS) analysis indicated the presence of Pd and Se but not of Bi.

Discussion

X-ray powder diffraction studies of PdSe [1,2] led to a tetragonal structure of the PdS type. The structure of PdS was reinvestigated and found to crystallize in the tetragonal space group $P4_2/m$ [3]. The study of $PdS_{1-x}Se_x$ mixed phases ($0 \le x \le 1$) [4] showed that the PdS-type mixed crystals are stable only in the ranges $0.0 \le x \le 0.2$ and $0.9 \le x \le 1$; for $0.2 < x < 0.9$ $PdS_{1-x}Se_x$ crystallizes in the space group $P4_2/mbc$. Here we report the first single-crystal study of the title compound, PdSe. This binary compound crystallizes in space group $P4_2/mbc$ and consequently is not isostructural with PdS. A perspective view of PdSe along [001] is shown in the figure. There are three crystallographically unique Pd atoms. Each of these sits in a slightly distorted square-planar environment. Each of the two crystallographically independent Se atoms is coordinated by a distorted tetrahedron of Pd atoms. The Pd—Se distances range from 2.4039(7) Å to 2.4683(8) Å, and are consistent with those observed in square-planar $PdSe_4$ environments.

Table 1. Data collection and handling.

Crystal:	black block, size 0.130 × 0.178 × 0.250 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	345.43 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, $\Delta\omega = 0.3°$
$2\theta_{max}$:	57.58°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7424, 639
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 629
$N(param)_{refined}$:	33
Program:	SHELXTL [5]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pd(1)	8*h*	0.04507(6)	0.13065(5)	0	0.0076(4)	0.0090(4)	0.0029(4)	0.0006(2)	0	0
Pd(2)	8*h*	0.23178(6)	0.35785(6)	0	0.0075(4)	0.0089(4)	0.0032(4)	0.0006(2)	0	0
Pd(3)	8*g*	0.11259(4)	*x*+1/2	1/4	0.0072(3)	U_{11}	0.0061(4)	–0.0002(2)	–0.0002(2)	$-U_{13}$
Se(1)	16*i*	0.17697(5)	0.11009(5)	0.27523(9)	0.0076(4)	0.0079(4)	0.0032(4)	0.0003(2)	0.0000(2)	0.0001(2)
Se(2)	8*g*	0.59667(6)	*x*+1/2	1/4	0.0078(3)	U_{11}	0.0046(5)	–0.0002(3)	0.0001(2)	$-U_{13}$

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