Crystal structure of hexapotassium pentaplumbide, K_6Pb_5

T. F. Fässler* and C. Kronseder

ETH Zürich, Laboratorium für Anorganische Chemie, Universitätstraße 6, CH-8092 Zürich, Switzerland

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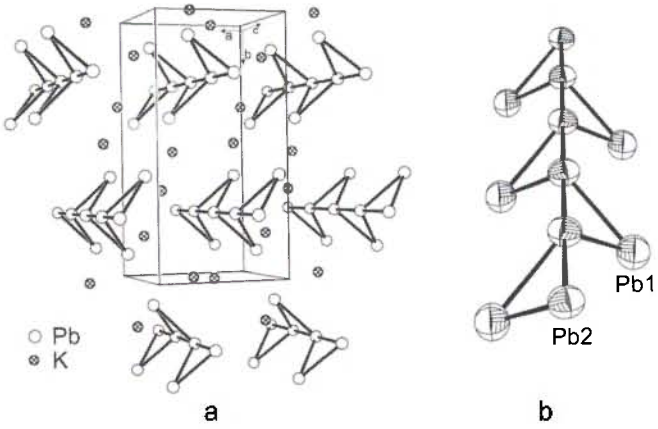

Abstract

$K_8Pb_{6.67}$, orthorhombic, *Pbcm* (No. 57), $a = 6.720(1)$ Å, $b = 13.389(3)$ Å, $c = 6.520(1)$ Å, $V = 586.6$ Å^3, $Z = 1$, $R_{gt}(F) = 0.038$, $wR(F^2) = 0.103$, $T = 293$ K.

Source of material

Single crystals of K_6Pb_5 were obtained from a mixture of the elements of the ratio K : Pb = 6 : 25. The mixture was heated in a welded Nb ampoule to 973 K for 5 hours and cooled within 4 hours to room temperature. Needle-shaped single crystals of K_6Pb_5 can be separated from elemental Pb. From stoichiometric mixtures of the elements and equivalent experimental conditions, KPb was identified as the only product.

Discussion

K_6Pb_5 is a new phase in the system K-Pb [1] and crystallizes in the structure of K_2SnBi [2] with Pb on Sn and Bi positions, respectively. Pb atoms on the latter site (Pb1) refine to an occupancy of 0.62. During final refinement cycles the occupancy was fixed to 2/3 resulting in the composition K_6Pb_5. There are no indications for superstructure reflections. In the crystal structure of K_6Pb_5 the Pb2 atoms form a linear chain, with a Pb–Pb distance of 3.260 Å. The atoms are bridged with Pb1. The Pb1–Pb2 distance is 3.019 Å. The resulting folded zigzag chain of Pb atoms (Figure 1b) has the following bond angles: Pb2–Pb1–Pb2 65.56° and Pb1–Pb2–Pb1 112.00°. The Pb1–Pb2 distance is relatively short, but is comparable to distances observed between the atoms of Pb_4 tetrahedra in CsPb (3.087 Å) [3].

Table 1. Data collection and handling.

Crystal:	grey needle, size 0.12 × 0.20 × 0.48 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	488.92 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS, 127 exposures, $\Delta\varphi = 1.5°$
$2\theta_{max}$:	56.34°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4914, 753
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 707
$N(param)_{refined}$:	25
Programs:	SHELXS-97 [4], SHELXL-97 [5]

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

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pb(1)	4*d*	2/3	0.8355(2)	0.10784(7)	1/4	0.0335(5)	0.0336(5)	0.0445(6)	0.0000(4)	0	0
Pb(2)	4*c*		0.58496(8)	1/4	0	0.0247(4)	0.0330(4)	0.0271(4)	0	0	0.0023(2)
K(1)	4*d*		0.0982(5)	0.3388(3)	1/4	0.033(2)	0.035(2)	0.049(2)	–0.005(1)	0	0
K(2)	4*d*		0.3364(6)	0.0136(3)	1/4	0.033(2)	0.046(2)	0.055(2)	–0.001(2)	0	0

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References

1. Fässler, T. F.; Hoffmann, S.: Valence compounds at the border to intermetallics: alkli and alkaline earth metal stannides and plumbides. Z. Kristallogr. submitted for publication.
2. Asbrand, M.; Eisenmann, B.: Crystal structure of dipotassium bismutidostannate, K_2SnBi. Z. Kristallogr. **198** (1992) 285.
3. Carrillo-Cabrera, W.; Somer, M.; Peters, K.; von Schnering, H. G.: Single-crystal structure refinement of CsPb. VIth European Conference on Solid State Chemistry, Zürich 1997, p. 122.
4. Sheldrick, G. M.: SHELXS-97. Program for the solution of crystal structures. University of Göttingen, Germany 1997.
5. Sheldrick, G. M.: SHELXL-97. Program for refining crystal structures. University of Göttingen, Germany 1997.

* Correspondence author (e-mail: faessler@inorg.chem.ethz.ch)