Crystal structure of tripotassium tetraselenidophosphate, K_3PSe_4

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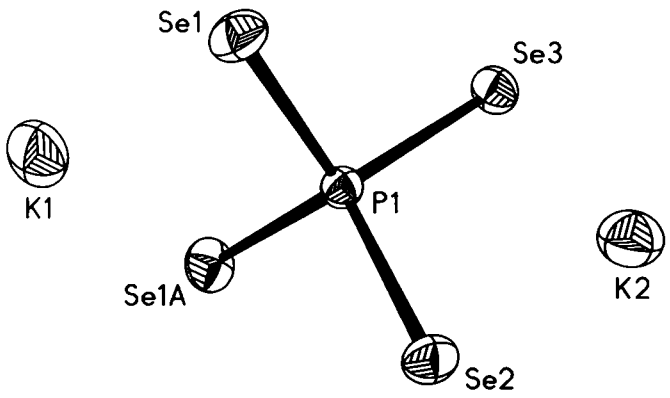

Abstract

K_3PSe_4, orthorhombic, *Pnma* (no. 62), $a = 9.393(4)$ Å, $b = 10.954(5)$ Å, $c = 9.578(4)$ Å, $V = 985.5$ Å^3, $Z = 4$, $R_{gt}(F) = 0.029$, $wR_{ref}(F^2) = 0.063$, $T = 571$ K.

Source of material

Crystals of K_3PSe_4 were formed in a molten chalcogenide flux reaction of 14 mg P, 107 mg Se, and 158.3 mg K_2Se_2. The reactants were combined in a fused silica ampoule in an inert atmosphere glovebox, sealed under vacuum, and heated to 500 °C at a rate of 30 K/hour. After 150 hours of heating, the ampoule was cooled at 3 K/hour to room temperature. Dimethylformamide was added to dissolve remaining potassium selenide flux, resulting in well-formed block-like crystals of K_3PSe_4.

Discussion

This compound is isostructural to K_3PS_4 [1]. The structure consists of isolated tetrahedra of PSe_4 surrounded by K cations.

Table 1. Data collection and handling.

Crystal:	transparent green block, size 0.20 × 0.20 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	162.36 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	56.56°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8714, 1278
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 900
$N(param)_{refined}$:	44
Program:	SHELXTL [2]

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}
Se(1)	8*d*	0.16168(5)	0.41608(4)	0.46283(5)	0.0322(3)	0.0254(3)	0.0343(3)	0.0060(2)	−0.0042(2)	0.0062(2)
Se(3)	4*c*	0.48781(6)	¼	0.41529(7)	0.0223(3)	0.0331(4)	0.0243(4)	0	0.0047(3)	0
Se(2)	4*c*	0.30896(7)	¼	0.74941(6)	0.0307(4)	0.0328(4)	0.0183(3)	0	0.0000(3)	0
K(1)	4*c*	−0.1457(2)	¼	0.4128(2)	0.0376(9)	0.067(1)	0.0354(9)	0	0.0088(7)	0
P(1)	4*c*	0.2788(2)	¼	0.5218(2)	0.0190(8)	0.0200(8)	0.0183(8)	0	−0.0004(6)	0
K(2)	8*d*	0.5536(1)	0.4553(1)	0.6985(1)	0.0392(6)	0.0290(6)	0.0344(6)	−0.0026(5)	0.0003(5)	0.0039(5)

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References

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