Crystal structure of tetracesium decaselenidotetrasilicate, $Cs_4Si_4Se_{10}$

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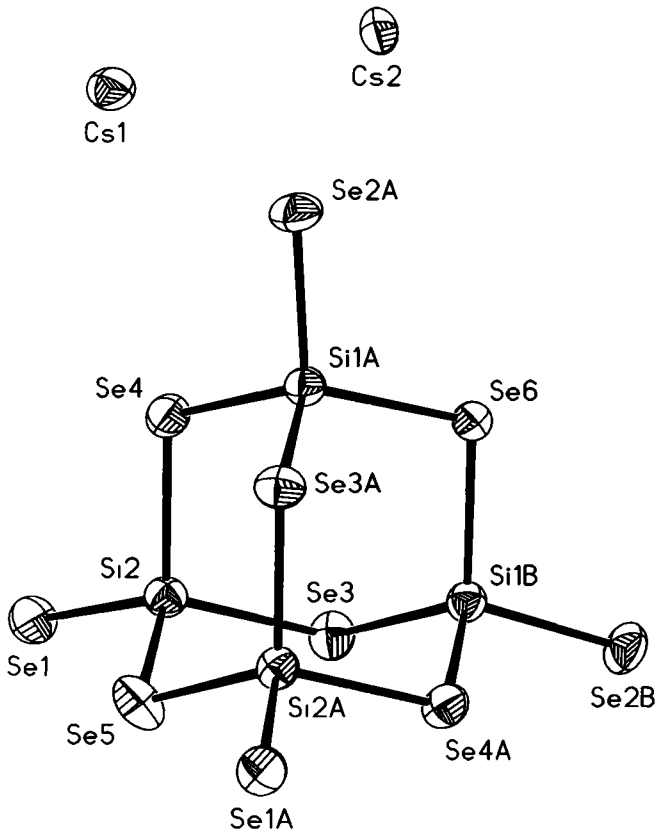

Abstract

$Cs_4Se_{10}Si_4$, monoclinic, $C12/c1$ (no. 15), $a = 16.164(2)$ Å, $b = 16.314(2)$ Å, $c = 9.723(1)$ Å, $\beta = 106.683(3)°$, $V = 2456.1$ Å^3, $Z = 4$, $R_{gt}(F) = 0.036$, $wR_{ref}(F^2) = 0.083$, $T = 298$ K.

Source of material

Crystals of $Cs_4Si_4Se_{10}$ were formed from a molten chalcogenide flux reaction of 127.12 mg Cs_2Se_2, 4.21 mg Si, and 71.06 mg Se. The reactants were combined in a fused silica ampoule in an inert atmosphere glovebox, sealed under vacuum, and heated to 750 °C at a rate of 30 K/hour. After 200 hours of heating, the ampoule was cooled at 3 K/hour to room temperature. Dimethylformamide was added to dissolve remaining cesium selenide flux, resulting in well-formed colorless crystals of $Cs_4Si_4Se_{10}$.

Discussion

$Cs_4Si_4Se_{10}$ crystallizes with adamantane-like $[Si_4Se_{10}]^{4-}$ units composed of four corner-shared $SiSe_4$ tetrahedra and Cs^+ cations for charge balancing the compound. $Cs_4Si_4Se_{10}$ is isostructural to $Na_4Ge_4Se_{10}$ [1], $Cs_4Ge_4S_{10}$ [2] and $K_4Ge_4Se_{10}$ [3,4].

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.03 × 0.05 × 0.08 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	208.90 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	56.56°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11334, 3012
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2115
$N(param)_{refined}$:	83
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}
Cs(1)	8*f*	0.15246(3)	0.13588(3)	0.37597(5)	0.0433(3)	0.0435(3)	0.0377(3)	0.0104(2)	0.0080(2)	−0.0012(2)
Cs(2)	8*f*	0.36471(3)	0.07139(3)	0.85764(5)	0.0408(3)	0.0472(3)	0.0399(3)	0.0005(2)	0.0153(2)	0.0103(2)
Se(1)	8*f*	0.20158(5)	0.05978(4)	0.04569(8)	0.0334(4)	0.0367(4)	0.0349(4)	0.0098(3)	0.0079(3)	0.0037(3)
Se(2)	8*f*	0.38508(5)	0.13056(4)	0.52056(8)	0.0413(5)	0.0408(4)	0.0351(4)	−0.0077(3)	0.0200(3)	0.0051(3)
Se(3)	8*f*	0.16885(4)	0.21614(4)	−0.23361(7)	0.0235(4)	0.0357(4)	0.0373(4)	0.0008(3)	0.0121(3)	0.0046(3)
Se(4)	8*f*	0.04227(5)	0.21696(4)	0.02981(7)	0.0342(4)	0.0350(4)	0.0288(4)	0.0088(3)	0.0116(3)	0.0041(3)
Se(5)	4*e*	0	0.05131(6)	−¼	0.0305(6)	0.0262(5)	0.0522(7)	0	0.0020(5)	0
Se(6)	4*e*	½	0.11960(5)	¼	0.0374(6)	0.0241(4)	0.0292(5)	0	0.0156(4)	0
Si(1)	8*f*	0.4370(1)	0.2051(1)	0.3791(2)	0.026(1)	0.0258(9)	0.0253(9)	−0.0015(7)	0.0088(8)	0.0001(7)
Si(2)	8*f*	0.1037(1)	0.1330(1)	−0.1034(2)	0.024(1)	0.0281(9)	0.029(1)	0.0027(7)	0.0090(8)	−0.0003(8)

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References

1. Eisenmann, B.; Hansa, J.: Crystal structure of tetrasodium decaselenotetragermanate, $Na_4[Ge_4Se_{10}]$. Z. Kristallogr. **205** (1993) 325-326.
2. Klepp, K. O.; Zeitlinger, M.: Crystal structure of tetracesium decasulfidotetragermanate, $Cs_4Ge_4S_{10}$. Z. Kristallogr. **215** (2000) 7-8.
3. Eisenmann, B.; Hansa, J.: Crystal structure of tetrapotassium decaselenidotetragermanate, $K_4[Ge_4Se_{10}]$. Z. Kristallogr. **206** (1993) 101-102.
4. Wachhold, M.; Kanatzidis, M. G.: Surfactant-Templated Inorganic Lamellar and Non-Lamellar Hybrid Phases Containing Adamantane $[Ge_4Se_{10}]^{4-}$ Anions. Chem. Mater. **12** (2000) 2914-2932.
5. Sheldrick, G. M.: SHELXTL. Structure Determination Software Suite. V. 6.10. Bruker AXS, Madison, Wisconsin, USA 2000.