Crystal structure of diindium digermanate, $In_2(Ge_2O_7)$

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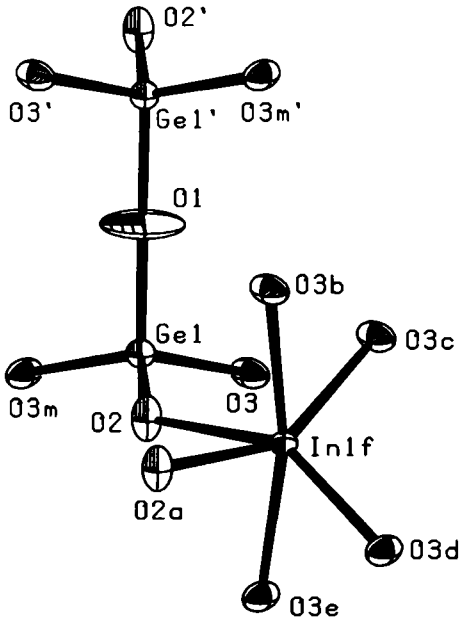

Abstract

$Ge_2In_2O_7$, monoclinic, $C12/m1$ (No. 12), $a = 6.647(2)$ Å, $b = 8.773(2)$ Å, $c = 4.918(1)$ Å, $\beta = 102.50(3)°$, $V = 280.0$ Å^3, $Z = 2$, $R_{gt}(F) = 0.016$, $wR_{ref}(F^2) = 0.039$, $T = 300$ K.

Source of material

Single crystals of indium digermanate were grown by chemical vapor transport of In_2O_3 and GeO_2 in a closed quartz ampoule. Chlorine (0.5 mmol) was used as transport agent and a mixture of In_2O_3 (1.5 mmol) and GeO_2 (3 mmol) as source materials. After one day of heating in a temperature gradient (1273 K → 1373 K), the transport was conducted for eight days in the inverse gradient. In a homogenous transport indium digermanate was deposited in the crystallization zone with a transport rate of 2 mg/h.

Discussion

The (average) crystal structure of $In_2Ge_2O_7$ is isotypic to that of $In_2Si_2O_7$ [1]. The Ge—O distances range from 170 pm to 175 pm and the In–O distances from 211 pm to 222 pm. The two longest half axes of the anisotropic displacement ellipsoid of the bridging oxygen atom O1 are larger than those in $In_2Si_2O_7$ by a factor of two. Therefore a structure model with a splitted O1 atom (two $O_{1/4}$ atoms in, one $O_{1/4}$ above and one $O_{1/4}$ below the mirror plane) was refined, too. This refinement was stable and converged to R values as low as in the average structure. As a result one obtains for the distance Ge—O1 172.4(4) pm and for the angle Ge–O1–Ge' 162(1)°. An analogous calculation for $In_2Si_2O_7$ yields for the distance Si-O1 161.6(3) pm and for the angle Si-O1-Si' 169(2)°. A recent discussion of a linear Si–O–Si bond is given in [2].

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.11 × 0.204 × 0.39 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	187.49 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS, 240 exposures, $\Delta\phi = 1.5°$
$2\theta_{max}$:	60.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2671, 426
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 418
$N(param)_{refined}$:	32
Programs:	SHELXL-93 [3], CIF2SX [4], PLATON [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}
In(1)	4*g*	0	0.30714(3)	0	0.0034(2)	0.0049(2)	0.0048(2)	0	0.00045(9)	0
Ge(1)	4*i*	0.23254(5)	0	0.40752(7)	0.0047(2)	0.0054(3)	0.0033(2)	0	–0.0001(1)	0
O(1)	2*c*	0	0	1/2	0.012(2)	0.047(4)	0.056(4)	0	0.023(2)	0
O(2)	4*i*	0.4084(4)	0	0.7282(5)	0.012(1)	0.006(2)	0.005(1)	0	–0.0017(9)	0
O(3)	8*j*	0.2391(3)	0.1658(3)	0.2121(4)	0.0062(7)	0.009(1)	0.0094(8)	0.0024(6)	0.0026(6)	0.0045(7)

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