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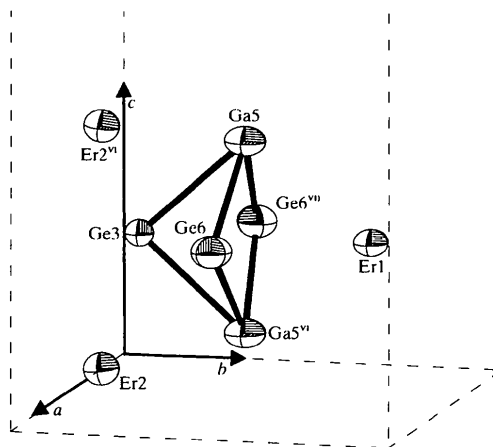Yuge, H., Mamada, A., Asai, M., Nishikiori, S. & Iwamoto, T. (1995). *J. Chem. Soc. Dalton Trans.* pp. 3195–3205.

Fig. 1. View of the asymmetric unit of $Er_3Ga_2Ge_3$. Displacement ellipsoids are shown at the 99% probability level.

The refinement of the occupancy factors of the Ga and Ge sites leads us to assume that the Ga5 and Ge6 sites are fully occupied by Ga and Ge atoms, respectively. The occupancy factor of the (Ge3,Ga4) site suggests a mixture of Ge and Ga atoms on this site. In spite of the close scattering factors of Ge and Ga atoms, the chemical formula deduced from the results of the refinements $(Er_{37.5}Ge_{34.9}Ga_{27.6})$ is in good agreement with that measured by microprobe analysis.

Trierbium digallide trigermanide

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Abstract

The synthesis and single crystal structure of $Er_3Ga_2Ge_3$ (analysed as $Er_3Ga_{2.21}Ge_{2.79}$) are reported. $Er_3Ga_2Ge_3$ is isotypic with Pu_3Pd_5.

Comment

The title compound, $Er_3Ga_2Ge_3$, is isotypic with Pu_3Pd_5 (Cromer, 1976). Until now, this structural type has been mainly reported for R_3In_5 and R_3Tl_5 compounds (R = lanthanide elements; Villars & Calvert, 1991). In the Er–Ga–Ge system investigated by us at 1173 K, $Er_3Ga_2Ge_3$ is in equilibrium with the Tm_3Ga_5-type compound $Er_3Ga_3Ge_2$ (Yatsenko *et al.*, 1983) and with the defect AlB_2-type compound $Er_{36}Ga_{17}Ge_{47}$. The Pu_3Pd_5 structure is closely related to the Tm_3Ga_5-type structure (Yatsenko *et al.*, 1983). The evolution of the structure as a function of the Ga content is not well understood.

Experimental

Crystals of the title compound were extracted from an $Er_{25}Ga_{20}Ge_{55}$ ingot annealed at 1173 K for one week. The microprobe analysis of the crystals gives the following composition: Er 37 (1), Ga 27 (1) and Ge 36 (1) atom%.

Crystal data

$Er_3Ga_{2.21}Ge_{2.79}$	Ag $K\alpha$ radiation
$M_r = 858.99$	$\lambda = 0.56090$ Å
Orthorhombic	Cell parameters from 176
$Cmcm$	reflections
$a = 9.2880\,(6)$ Å	$\theta = 0.64–23.58°$
$b = 7.4180\,(7)$ Å	$\mu = 32.069$ mm^{-1}
$c = 9.3830\,(4)$ Å	$T = 293\,(2)$ K
$V = 646.47\,(8)$ Å^3	Parallelepiped
$Z = 4$	$0.08 \times 0.07 \times 0.07$ mm
$D_x = 8.826$ Mg m^{-3}	Metallic grey
D_m not measured	

Data collection

Nonius KappaCCD diffractometer	358 reflections with $I > 2\sigma(I)$
Oscillations scan	$R_{int} = 0.063$
Absorption correction:	$\theta_{max} = 21.34°$
empirical (*SORTAV*;	$h = -11 \rightarrow 12$
Blessing, 1987)	$k = -9 \rightarrow 9$
$T_{min} = 0.090$, $T_{max} = 0.109$	$l = -12 \rightarrow 12$
3631 measured reflections	
423 independent reflections	

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.020$
$wR(F^2) = 0.054$
$S = 0.704$
423 reflections
32 parameters
$w = 1/[\sigma^2(F_o^2)]$
$(\Delta/\sigma)_{max} = 0.045$
$\Delta\rho_{max} = 1.64$ e Å^{-3}
$\Delta\rho_{min} = -1.63$ e Å^{-3}

Extinction correction:
SHELXL97 (Sheldrick, 1997a)
Extinction coefficient:
0.0055 (3)
Scattering factors from
International Tables for Crystallography (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a^ia^j\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Er1	0	0.65375 (9)	1/4	0.0097 (2)
Er2	0.19927 (5)	0	0	0.0105 (2)
Ge3†	0	0.0408 (2)	1/4	0.0059 (15)
Ga4‡	0	0.0408 (2)	1/4	0.041 (12)
Ga5	0	0.32084 (17)	0.44813 (12)	0.0117 (3)
Ge6	0.20266 (12)	0.29135 (17)	1/4	0.0119 (3)

† Site occupancy = 0.787 (16).   ‡ Site occupancy = 0.213 (16).

Table 2. *Selected bond lengths (Å)*

Er1—Ga5^i	2.8387 (11)	Ge3—Ge6^{iii}	2.6454 (17)
Er1—Ga5^{ii}	2.8387 (11)	Ge3—Ga5	2.7878 (18)
Er1—Ga5^{iii}	2.871 (2)	Ge3—Ga5^{vi}	2.7878 (18)
Er1—Ge6^{iv}	2.9443 (12)	Ge3—Er1^{xv}	2.871 (2)
Er1—Ge6^v	2.9443 (12)	Ge3—Er2^x	3.0033 (4)
Er1—Ga5^{vi}	3.0910 (13)	Ge3—Er2^{xvi}	3.0033 (4)
Er1—Ga5	3.0910 (13)	Ga5—Ge6^{vii}	2.6546 (12)
Er1—Ge6^{vii}	3.2817 (13)	Ga5—Ge6	2.6546 (12)
Er1—Ge6	3.2817 (13)	Ga5—Ga5^i	2.831 (2)
Er2—Ge6^{viii}	2.9543 (8)	Ga5—Er1^i	2.8387 (11)
Er2—Ge6^{ix}	2.9543 (8)	Ga5—Er2^{xvi}	3.0540 (10)
Er2—Ge3^x	3.0033 (4)	Ga5—Er2^{vi}	3.0540 (10)
Er2—Ge3	3.0033 (4)	Ga5—Er2^{xvii}	3.1313 (7)
Er2—Ga5^{xi}	3.0540 (10)	Ga5—Er2^{xviii}	3.1313 (7)
Er2—Ga5^{vi}	3.0540 (10)	Ge6—Ga5^{vi}	2.6546 (12)
Er2—Ga5^{xii}	3.1313 (7)	Ge6—Er1^{xix}	2.9443 (12)
Er2—Ga5^{xiii}	3.1313 (7)	Ge6—Er2^{xvii}	2.9543 (8)
Er2—Ge6^{xiv}	3.1897 (9)	Ge6—Er2^{viii}	2.9543 (8)
Er2—Ge6	3.1897 (9)	Ge6—Er2^{vi}	3.1897 (9)
Ge3—Ge6	2.6454 (17)		

Symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $-x, 1-y, z-\frac{1}{2}$; (iii) $x, 1+y, z$; (iv) $\frac{1}{2}-x, \frac{1}{2}+y, z$; (v) $x-\frac{1}{2}, \frac{1}{2}+y, z$; (vi) $x, y, \frac{1}{2}-z$; (vii) $-x, y, z$; (viii) $\frac{1}{2}-x, \frac{1}{2}-y, -z$; (ix) $\frac{1}{2}-x, y-\frac{1}{2}, z$; (x) $-x, -y, -z$; (xi) $-x, -y, z-\frac{1}{2}$; (xii) $\frac{1}{2}-x, \frac{1}{2}-y, z-\frac{1}{2}$; (xiii) $\frac{1}{2}+x, y-\frac{1}{2}, \frac{1}{2}-z$; (xiv) $x, -y, -z$; (xv) $x, y-1, z$; (xvi) $-x, -y, \frac{1}{2}+z$; (xvii) $\frac{1}{2}-x, \frac{1}{2}-y, \frac{1}{2}+z$; (xviii) $x-\frac{1}{2}, \frac{1}{2}+y, \frac{1}{2}-z$; (xix) $\frac{1}{2}+x, y-\frac{1}{2}, z$.

In the final electron-density difference map, both the minimum (-1.63 e Å^{-3} at 0.5,0.1456,0.347) and the maximum (1.64 e Å^{-3} at 0.4378,0.0552,0.5) peaks occur approximately 0.93 Å from the heavy Er1 atom. They are due to the irregular crystal shape and the empirical absorption correction method (*SORTAV*; Blessing, 1987). The *SHELXL97* (Sheldrick, 1997a) recommended weighting scheme ($a = 0, b = 0$) does not permit one to obtain a goodness-of-fit value close to 1.0. The low value of the least-squares goodness-of-fit is due to the data reduction procedure (*DENZO* and *SCALEPACK*; Otwinowski & Minor, 1997), which overestimates (by a factor of approximately 1.3) the standard deviation of the measured intensities.

Data collection: *COLLECT* (Nonius, 1998). Cell refinement: *COLLECT*. Data reduction: *DENZO* and *SCALEPACK*. Pro-

gram(s) used to solve structure: *SHELXS97* (Sheldrick, 1997b). Program(s) used to refine structure: *SHELXL97*. Molecular graphics: *ATOMS for Windows* (Dowty, 1995). Software used to prepare material for publication: *SHELXS97*.

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The solid solution of composition K$_2$PdBr$_{2.24}$Cl$_{1.76}$

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Abstract

The structure of K$_2$PdBr$_{2.24}$Cl$_{1.76}$, dipotassium bromochloropalladate, an isotype of K$_2$PdCl$_4$ and K$_2$PdBr$_4$, has been determined in the centrosymmetric space group $P4/mmm$. The tetragonal cell contains one Pd atom. The [PdX$_4$]$^{2-}$ ions (X = Cl, Br) are square planar, with Pd—X bond lengths of 2.3934 (9) Å.