(Sheldrick, 1997) included in the *WinGX* program system (Farrugia, 1998). Molecular graphics: *ORTEP*-3 (Farrugia, 1997).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BR1243). Services for accessing these data are described at the back of the journal.

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$Dy_3CuGeSe_7$

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Abstract

The structure of tridysprosium copper germanium heptaselenide has been determined from single-crystal X-ray data. This non-centrosymmetric structure (space group $P6_3$) is of the La_3CuSiS_7 structure type, comprising $CuSe_3$ triangles, $GeSe_4$ tetrahedra, and $DySe_8$ bicapped trigonal prisms.

Comment

La_3CuSiS_7 (Flahaut & Laruelle, 1970) was the first structure reported of the family of Ln_3MNQ_7 chalcogenides (Ln = La–Er, Y; M = Cu, Ag; N = Si–Sn; Q = S, Se) (Guittard *et al.*, 1968). These compounds crystallize in the non-centrosymmetric space group $P6_3$ and hence should exhibit piezoelectric and second-order non-linear optical behavior. La_3CuSiS_7 shows a very strong piezoelectric effect (Flahaut & Laruelle, 1970). The structure

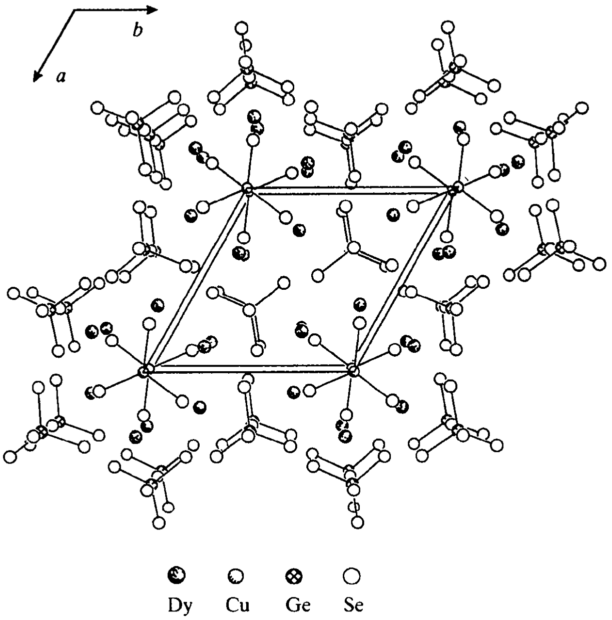

Fig. 1. The structure of $Dy_3CuGeSe_7$ viewed down [001]. For the sake of clarity, Dy—Se bonds are not shown.

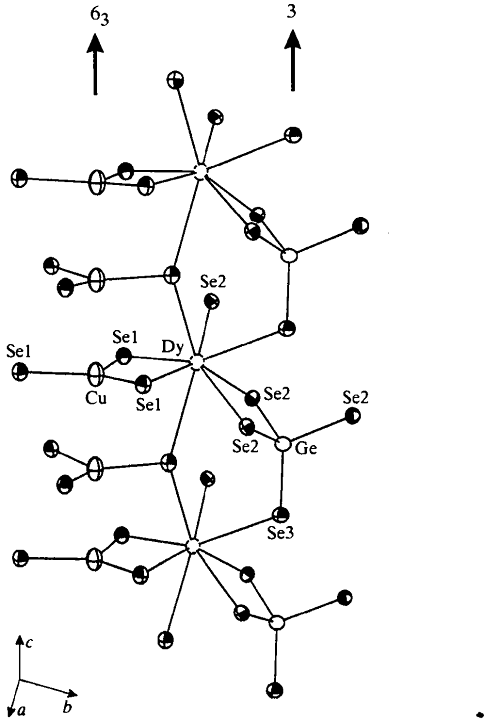

Fig. 2. Partial view of the structure of $Dy_3CuGeSe_7$, shown with 90% probability displacement ellipsoids.

of La_3AgGeS_7, a new member of the series, has been reported (Hwu *et al.*, 1995). Here, yet another new member of the series, namely $Dy_3CuGeSe_7$, is described.

The basic structure comprises three structural motifs, *i.e.* $DySe_8$ bicapped trigonal prisms, planar $CuSe_3$ triangles, and $GeSe_4$ tetrahedra, as shown in Figs. 1 and 2. Only $DySe_8$ trigonal prisms form corner-sharing chains along the *c* axis, whereas the isolated $CuSe_3$ triangles (6_3 axis through Cu) are staggered parallel to **c**, and $GeSe_4$ tetrahedra (3 axis through Ge) pack parallel to **c**. The $DySe_8$ units are connected to $CuSe_3$ and $GeSe_4$ units through Se1, and Se2, and Se3. A Cu atom is displaced 0.128 Å from the plane of the Se1/Se2/Se3 triangle; the Cu···Cu distance is 3.0161 (4) Å.

Experimental

Crystals of $Dy_3CuGeSe_7$ were obtained from an initial mixture of Dy (1.0 mmol, Aldrich, 40 mesh powder, 99.9%), Ge (1.0 mmol, Alfa Aesar, 100 mesh powder, 99.999%), Cu (1.0 mmol, Aldrich, powder, 99.999%), Se (4.0 mmol, Aldrich, 100 mesh powder, 99.5+%), and $DyCl_3$ (2.5 mmol, Aldrich, powder, 99.9%) as flux. The mixture was loaded under argon, sealed under 10^{-4} Torr in a fused-silica tube (1 Torr = 133.322 Pa), heated in a furnace to 1123 K at 1 K min^{-1}, kept at 1123 K for 70 h, cooled at a rate of 0.05 K min^{-1} to 573 K, and finally cooled to room temperature. The reaction mixture was washed with water and acetone. The major component, $Dy_3CuGeSe_7$, forms as hexagonal brown needles. Analysis of these crystals with an EDX-equipped Hitachi S-4500 SEM showed the presence of Dy, Cu, Ge and Se in the ratio 3:1:1:7.

Crystal data

$Dy_3CuGeSe_7$
$M_r = 1176.35$
Hexagonal
$P6_3$
$a = 10.2499 (9)$ Å
$c = 6.0322 (8)$ Å
$V = 548.84 (10)$ Å^3
$Z = 2$
$D_x = 7.118$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 2187 reflections
$\theta = 2.29$–$28.16°$
$\mu = 47.93$ mm^{-1}
$T = 153 (2)$ K
Needle
0.22 × 0.02 × 0.01 mm
Brown

Data collection

Bruker Smart 1000 CCD diffractometer
ω scans
Absorption correction: face-indexed numerical (Sheldrick, 1997*b*)
$T_{\min} = 0.111$, $T_{\max} = 0.672$
3631 measured reflections
492 independent reflections (plus 390 Friedel-related reflections)

831 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.038$
$\theta_{\max} = 28.16°$
$h = -13 \rightarrow 11$
$k = -13 \rightarrow 13$
$l = -7 \rightarrow 7$
Intensity decay: <2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.021$
$wR(F^2) = 0.053$
$S = 1.037$
882 reflections
38 parameters
$w = 1/[\sigma^2(F_o^2) + (0.03F_o^2)^2]$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.607$ e Å^{-3}
$\Delta\rho_{\min} = -1.279$ e Å^{-3}

Extinction correction: *SHELXL97*
Extinction coefficient: 0.0019 (2)
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = 0.08 (8)

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

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

	x	y	z	$U_{\rm eq}$
Dy	0.22051 (4)	0.35671 (4)	0.42074 (10)	0.00893 (13)
Cu	0	0	0.4737 (3)	0.0132 (5)
Ge	2/3	1/3	0.3361 (3)	0.0086 (4)
Se1	0.26309 (9)	0.10279 (9)	0.45248 (15)	0.0095 (2)
Se2	0.51895 (10)	0.42570 (10)	0.17352 (15)	0.00860 (19)
Se3	1/3	2/3	0.2213 (2)	0.0096 (3)

Table 2. *Selected distances* (Å)

Dy—Se1	2.8525 (9)	Dy—Se2	3.1493 (10)
Dy—Se1$^{\rm i}$	2.8982 (9)	Dy—Se1$^{\rm iv}$	3.3163 (12)
Dy—Se2$^{\rm ii}$	2.9112 (10)	Cu—Se1	2.3574 (8)
Dy—Se1$^{\rm iii}$	2.9475 (12)	Cu—Cu$^{\rm v}$	3.0161 (4)
Dy—Se2$^{\rm iv}$	2.9935 (10)	Ge—Se3$^{\rm ii}$	2.323 (2)
Dy—Se3	3.0340 (8)	Ge—Se2	2.3631 (11)

Symmetry codes: (i) $-y, x-y, z$; (ii) $1-x, 1-y, \frac{1}{2}+z$; (iii) $x-y, x, z-\frac{1}{2}$; (iv) $x-y, x, \frac{1}{2}+z$; (v) $-x, -y, z-\frac{1}{2}$.

The crystal-to-detector distance for data collection was 5.023 cm. Crystal decay was monitored by recollecting 50 initial frames at the end of data collection. Each exposure was 20 s and covered $-0.3°$ in ω. Data were collected in groups of 606 frames at φ settings of 0, 120, and 240°. Anisotropic displacement parameters were used for all atoms. All sites are fully occupied.

Data collection: *SMART* (Bruker, 1997). Cell refinement: *SMART*. Data reduction: *SAINT-Plus* (Bruker, 1997). Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1997*b*). Software used to prepare material for publication: *SHELXTL/PC*.

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