Table 5. *Selected geometric parameters (Å, °) for (I) at 150 K*

Co1—N1	1.990 (2)	N4—O2	1.235 (3)
Co1—N2	1.9623 (19)	Si1—F1	1.6769 (18)
Co1—N3	1.9629 (17)	Si1—F2	1.6877 (13)
Co1—N4	1.920 (2)	Si1—F3	1.6890 (19)
N4—O1	1.240 (3)	Si1—F4	1.6899 (13)
Co1—N4—O1	121.29 (19)	O1—N4—O2	118.9 (2)
Co1—N4—O2	119.81 (19)	F1—Si1—F2	91.43 (7)
N1—Co1—N2	91.47 (7)	F1—Si1—F3	178.43 (10)
N1—Co1—N3	88.68 (7)	F1—Si1—F4	89.90 (7)
N1—Co1—N4	178.86 (10)	F2—Si1—F2^{i}	90.77 (10)
N2—Co1—N2^{i}	86.63 (14)	F2—Si1—F3	89.67 (7)
N2—Co1—N3	90.38 (9)	F2—Si1—F4	89.89 (7)
N2—Co1—N3^{i}	177.01 (9)	F2—Si1—F4^{i}	178.50 (8)
N2—Co1—N4	89.36 (8)	F3—Si1—F4	88.98 (7)
N3—Co1—N3^{i}	92.60 (12)	F4^{i}—Si1—F4	89.42 (10)
N3—Co1—N4	90.53 (7)		

Symmetry code: (i) $x, \frac{1}{2} - y, z$.

Table 6. *Hydrogen-bonding geometry (Å, °) for (I) at 150 K*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···F4	0.89	2.54	3.076 (2)	120
N1—H1*A*···F4^{i}	0.89	2.19	3.076 (2)	169
N1—H1*C*···F4^{ii}	0.89	2.03	2.8897 (14)	161
N2—H2*B*···F2^{ii}	0.86	2.36	3.024 (2)	134
N2—H2*C*···F2^{iii}	0.86	2.28	3.088 (2)	158
N2—H2*A*···F3^{ii}	0.86	2.19	2.899 (2)	140
N3—H3*B*···F1	0.87	2.34	3.201 (2)	172
N3—H3*A*···F2^{iv}	0.87	2.13	2.986 (2)	169
N3—H3*B*···F4	0.87	2.34	2.936 (2)	126
N3—H3*C*···F4^{ii}	0.87	2.41	3.171 (2)	148
N1—H1*B*···O1^{v}	0.89	2.32	2.983 (3)	131
N2—H2*B*···O1^{v}	0.86	2.52	3.295 (3)	1450
N2—H2*A*···O2^{vi}	0.86	2.54	3.254 (3)	141

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

H atoms were located from a difference electron-density map. The positions of the H atoms were refined as rotating ammine groups with fixed Co—N—H angles and free N—H bond lengths.

For both compounds, data collection: *SMART* (Siemens, 1994*a*); cell refinement: *SAINT* (Siemens, 1994*b*); data reduction: *SAINT*; program(s) used to solve structures: *SHELXS*97 (Sheldrick, 1997*b*); program(s) used to refine structures: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Siemens, 1994*c*); software used to prepare material for publication: *SHELXL*97.

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Dirubidium *catena*-poly[dichloroargentate(I)-μ-chloro]

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Abstract

The crystal structure of the title compound, Rb_2AgCl_3, consists of polymeric $[AgCl_3]^{2-}$ anions composed of corner-sharing slightly distorted $AgCl_4$ tetrahedra, with Rb^+ ions situated between the chains.

Comment

Compounds of the type R_2MX_3 (R is a monovalent cation), such as K_2AgI_3 (Brink & Kroese, 1952), K_2CuCl_3 (Brink & MacGillavry, 1949) and $(NH_4)_2CuX_3$ (X = Cl, Br; Brink & van Arkel, 1952), have been studied previously and found to be of three closely related structural types (Brink Shoemaker, 1973). Kildea *et al.* (1986) determined the structure of $(NH_4)_2[AgI_3]_{\infty/\infty}\cdot H_2O$, which is also of the same general structural type, and found that a water molecule could be incorporated without essentially changing the structure. The crystal structure of Rb_2AgI_3 has been refined and the polarized Raman spectrum reported (Brown *et al.*, 1977).

Rb_2AgCl_3 was crystallized during attempts to synthesize other compounds containing haloargentate(I) anions. In contrast to the more common simple iodoargentate(I) structures of the type R_2MX_3, the only other chloroargentate(I) hitherto reported is the caesium salt of $[AgCl_3]^{2-}$ (Brink & MacGillavry, 1949).

Rb_2AgCl_3 is isostructural with K_2AgI_3, Rb_2AgI_3 and $(NH_4)_2AgI_3$. The most prominent feature of the structure is the polymeric anion chain formed by chloride-linked corner-sharing $AgCl_4$ tetrahedra. There are four such chains parallel to the *b* axis in the unit cell, the Ag—Cl distances within the chains being in the range 2.587 (5)–2.602 (4) Å. The tetrahedra are slightly distorted, with a small Cl1—Ag1—Cl2 angle and a large Cl3—Ag1—Cl3 angle. Both Rb^+ ions are situated between the chains, each with seven neighbouring chloride ions. Rb1 is surrounded by four chlorides from one chain and three from another (4:3), while Rb2 has a 3:2:1:1 configuration involving four chains. The Rb1—Cl and Rb2—Cl distances are in the ranges 3.305 (3)–3.456 (4) and 3.245 (3)–3.527 (5) Å, respectively.

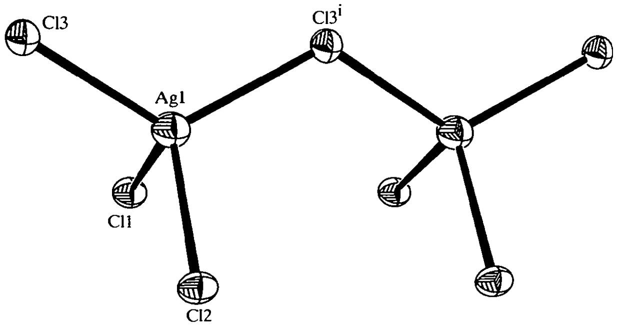

Fig. 1. Part of the $[AgCl_3]^{2-}$ chain shown with 50% probability displacement ellipsoids. [Symmetry code: (i) *x*, 1 + *y*, *z*.]

Experimental

The title compound was prepared from AgCl (0.014 g, 0.1 mmol, Aldrich 99.999% Gold Label) added to a warm solution of RbCl (0.012 g, 0.1 mmol, Merck 99.5%) in dimethylformamide (2 ml). The solution was stirred in the dark for 30 min after which it was filtered and left to evaporate at ambient temperature. Colourless needles (m.p. 565–567 K) formed after 2 d.

Crystal data

Rb_2AgCl_3
$M_r = 385.16$
Orthorhombic
Pnma
$a = 9.205\,(3)$ Å
$b = 4.4822\,(10)$ Å
$c = 17.8738\,(13)$ Å
$V = 737.5\,(3)$ Å^3
$Z = 4$
$D_x = 3.469$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 11.2–18.5°$
$\mu = 16.811$ mm^{-1}
$T = 293\,(2)$ K
Needle
0.20 × 0.15 × 0.10 mm
Colourless

Data collection

Rigaku AFC-6*R* diffractometer
$\omega–2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.096$, $T_{max} = 0.186$
741 measured reflections
741 independent reflections

416 reflections with $I > 2\sigma(I)$
$\theta_{max} = 24.96°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 5$
$l = -21 \rightarrow 0$
3 standard reflections every 150 reflections intensity decay: 0.99%

Refinement

Refinement on F^2
$R(F) = 0.035$
$wR(F^2) = 0.094$
$S = 1.046$
741 reflections
43 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0287P)^2]$ where $P = [2F_c^2 + \max(F_o^2,0)]/3$

$(\Delta/\sigma)_{max} = 0.011$
$\Delta\rho_{max} = 0.979$ e Å^{-3}
$\Delta\rho_{min} = -2.11$ e Å^{-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}
Ag1	0.37873 (15)	1/4	0.63107 (8)	0.0327 (4)
Rb1	0.07860 (18)	−1/4	0.71280 (9)	0.0258 (4)
Rb2	0.76095 (19)	1/4	0.54384 (9)	0.0264 (4)
Cl1	0.1216 (5)	1/4	0.5727 (2)	0.0270 (10)
Cl2	0.3153 (5)	1/4	0.7729 (2)	0.0259 (10)
Cl3	0.5066 (5)	−1/4	0.5974 (2)	0.0266 (10)

Table 2. *Selected geometric parameters (Å, °)*

Ag1—Cl1	2.587 (5)	Ag1—Cl3	2.602 (2)
Ag1—Cl2	2.602 (4)		
Cl1—Ag1—Cl2	100.82 (14)	Cl2—Ag1—Cl3	109.08 (10)
Cl1—Ag1—Cl3	108.71 (11)	Cl3^i—Ag1—Cl3	118.93 (17)

Symmetry code: (i) *x*, 1 + *y*, *z*.

Data collection was carried out on a crystalline fragment cut from a needle-shaped crystal. Cutting the crystals resulted in irregular fragments with faces which were not well defined and could not be indexed reliably. An analytical correction for absorption effects was therefore precluded and an empirical ψ-scan correction was used instead. The large minimum in the final difference map is approximately 1.9 Å from Cl3 and 2.7 Å from Ag1.

Data collection: *TEXSAN for Windows* (Molecular Structure Corporation, 1997). Cell refinement: *TEXSAN for Windows*. Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993). Program(s) used to refine structure: *SHELXL97*

(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

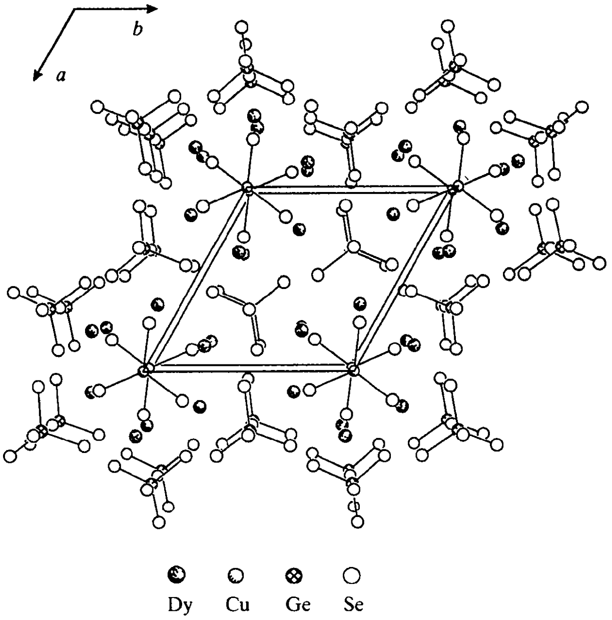

Fig. 1. The structure of $Dy_3CuGeSe_7$ viewed down [001]. For the sake of clarity, Dy—Se bonds are not shown.

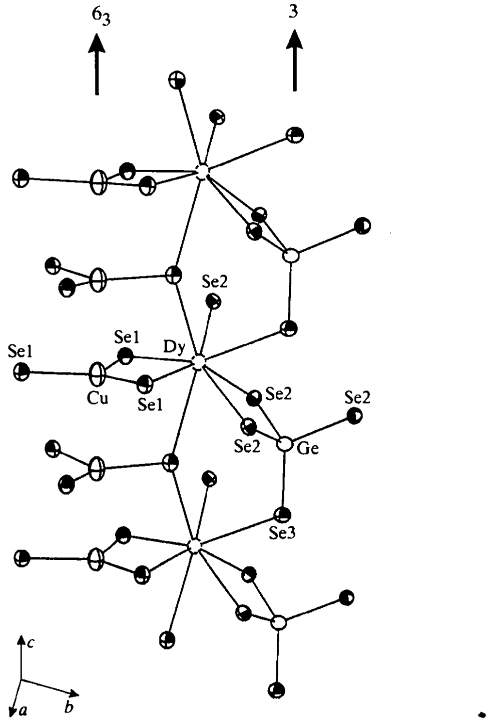

Fig. 2. Partial view of the structure of $Dy_3CuGeSe_7$, shown with 90% probability displacement ellipsoids.

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