$Z = 2$
$D_x = 3.363$ Mg m^{-3}

Thin plate
$0.28 \times 0.10 \times 0.03$ mm
Dark red

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: empirical (*SHELX76*; Sheldrick, 1976)
$T_{\min} = 0.425$, $T_{\max} = 0.732$
1935 measured reflections
437 independent observed reflections

1837 observed reflections $[I > 3\sigma(I)]$
$R_{\rm int} = 0.026$
$\theta_{\max} = 30°$
$h = 0 \rightarrow 15$
$k = -15 \rightarrow 0$
$l = 0 \rightarrow 11$
3 standard reflections monitored every 60 reflections intensity variation: <3%

Refinement

Refinement on F
$R = 0.0181$
$wR = 0.0227$
$S = 0.912$
437 reflections
27 parameters
$w = 1.7761[\sigma^2(F) + 0.00066F^2]$

$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.58$ e Å^{-3}
$\Delta\rho_{\min} = -1.04$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from Cromer & Mann (1968)

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

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

		x	y	z	$U_{\rm eq}$
In	2(*b*)	1/3	2/3	0.20500	0.0184 (4)
Te(1)	6(*c*)	0.19953 (2)	2*x*	0.0985 (2)	0.0212 (1)
Te(2)	2(*b*)	1/3	2/3	0.5505 (2)	0.0343 (4)
K(1)	6(*c*)	0.1245 (1)	2*x*	0.5186 (3)	0.0324 (8)
K(2)	6(*c*)	0.5209 (1)	2*x*	0.3190 (3)	0.0331 (6)
Cl	2(*a*)	0	0	0.7768 (5)	0.026 (1)

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

In—Te(1)	× 3	2.752 (1)	K(2)—Te(1)	× 2	3.539 (2)
In—Te(2)		2.768 (2)	K(2)—Te(2)		3.571 (2)
K(1)—Cl		3.110 (3)	K(2)—Te(1)	× 2	3.718 (2)
K(1)—Cl		3.194 (3)	K(2)—Te(2)		4.110 (2)
K(1)—Te(1)	× 2	3.472 (2)	Cl—K(1)	× 3	3.110 (3)
K(1)—Te(1)		3.673 (3)	Cl—K(1)	× 3	3.194 (3)
K(1)—Te(2)		4.092 (2)			
Te(1)—In—Te(1)		110.83 (3)	Te(1)—In—Te(2)		108.07 (3)

Statistical tests on intensities were in agreement with a non-centrosymmetric structure and space group $P6_3mc$ (No. 186). The structure was solved using Patterson synthesis and refinements were carried out using the *SHELX76* program (Sheldrick, 1976).

Lists of structure factors and anisotropic displacement parameters have been deposited with the IUCr (Reference: DU1081). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Scandium Fluorocarbonate, $Ba_3Sc(CO_3)F_7$

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Abstract

Single crystals of barium scandium fluorocarbonate, $Ba_3Sc(CO_3)F_7$, were obtained by hydrothermal growth at high temperature and high pressure. The structure, solved from single-crystal X-ray diffraction data, comprises Ba and Sc polyhedra which form connected layers parallel to (001).

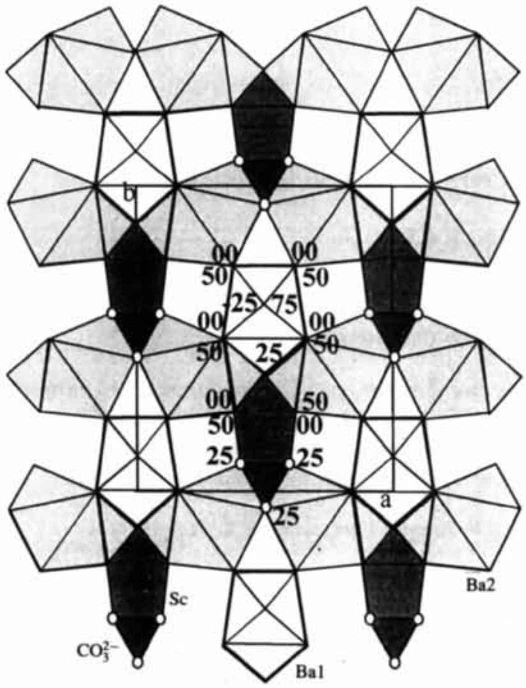

Fig. 1. [001] projection of a layer of Sc and Ba polyhedra at $z = \frac{1}{4}$ in $Ba_3Sc(CO_3)F_7$.

Comment

Several rare earth and 3*d* transition metal fluorocarbonates have been produced recently by hydrothermal growth (T = 1000 K, P = 200 MPa; Mercier & Leblanc, 1993*a*,*b*). Under the same conditions, a mixture of $BaCO_3$ and ScF_3 led to $Ba_3Sc(CO_3)F_7$. Ba atoms adopt two types of coordination; Ba1 is located at the centre of a tricapped pseudo-cube, $Ba1F_{11}$, while Ba2 is surrounded by seven F atoms with two O atoms at greater distances. Each Sc atom occupies the centre of a monocapped triangular prism, ScF_5O_2. All these polyhedra form infinite layers at $z = \frac{1}{4}$ and $z = \frac{3}{4}$ related by the centre of symmetry at (0,0,0) (Fig. 1). Each Sc polyhedron shares vertices along *b* with one Ba1 and two Ba2 polyhedra; along *a*, they share F-atom edges with two Ba2 polyhedra. Each ScF_5O_2 polyhedron is also linked to four Ba2 and two Ba1 polyhedra of neighbouring layers. Carbonate groups lie in the *ab* plane; each shares edges with one Sc polyhedron and two Ba2 polyhedra (Fig. 1).

Experimental

$Ba_3Sc(CO_3)F_7$ crystals were prepared by hydrothermal growth. A mixture of $BaCO_3$ and ScF_3 in a 1/1 ratio, inserted in a sealed platinum tube, was heated at 1000 K for 24 h (P = 200 MPa). The sample was then cooled to room temperature at 20 K h^{-1}. The resulting material was extracted with water and washed with acetone. Small colourless crystals of $Ba_3Sc(CO_3)F_7$, obtained as a by-product together with $Ba_3Al_2F_9$, were present. A parallelepipedic crystal was chosen for X-ray analysis by optical examination and its quality tested with Laue photography.

Crystal data

$Ba_3Sc(CO_3)F_7$
M_r = 649.91
Orthorhombic
Cmcm
a = 11.519 (3) Å
b = 13.456 (3) Å
c = 5.9740 (10) Å
V = 926.0 (4) Å^3
Z = 4
D_x = 4.662 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 34 reflections
θ = 14.0–16.0°
μ = 13.392 mm^{-1}
T = 293 (2) K
Block
0.2 × 0.1 × 0.05 mm
Colourless

Data collection

Stoe Siemens AED four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: Gaussian integration
T_{min} = 0.36, T_{max} = 0.56
1271 measured reflections
1110 independent reflections
956 observed reflections [$I > 3\sigma(I)$]

R_{int} = 0.019
θ_{max} = 34.93°
$h = 0 \rightarrow 18$
$k = 0 \rightarrow 21$
$l = 0 \rightarrow 9$
3 standard reflections
frequency: 60 min
intensity variation: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0248
$wR(F^2)$ = 0.0659
S = 0.466
954 reflections
48 parameters
$w = 1/[\sigma^2(F_o^2) + (0.1014P)^2 + 64.3346P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.566
$\Delta\rho_{max}$ = 1.483 e Å^{-3}
$\Delta\rho_{min}$ = −2.932 e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1994)
Extinction coefficient: 0.0109 (4)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Ba1	0	0.09440 (3)	1/4	0.00965 (11)
Ba2	0.22319 (2)	0.37986 (2)	1/4	0.01132 (11)
Sc	0	−0.26928 (9)	1/4	0.0085 (2)
F1	−0.1610 (3)	0	0	0.0141 (5)
F2	0.6207 (2)	0.7386 (2)	−0.0052 (4)	0.0148 (4)
F3	0	−0.1149 (3)	1/4	0.0167 (9)
O1	0	0.4416 (5)	1/4	0.0245 (13)
O2	−0.0952 (4)	0.5867 (3)	1/4	0.0256 (10)
C	0	0.5348 (5)	1/4	0.0165 (13)

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

Ba1—F1	2.699 (2)	Ba2—F2^{vii}	2.708 (2)
Ba1—F1^{i}	2.699 (2)	Ba2—F2^{xi}	2.813 (2)
Ba1—F1^{ii}	2.699 (2)	Ba2—F2^{xii}	2.813 (2)
Ba1—F1^{iii}	2.699 (2)	Ba2—O2^{xiii}	3.150 (4)
Ba1—F3	2.817 (5)	Ba2—F3^{x}	3.1893 (9)
Ba1—F2^{iv}	2.832 (2)	Sc—F2^{xiv}	2.060 (2)
Ba1—F2^{v}	2.832 (2)	Sc—F2^{xv}	2.060 (2)
Ba1—F2^{vi}	2.832 (2)	Sc—F2^{xvi}	2.060 (2)
Ba1—F2^{vii}	2.832 (2)	Sc—F2^{xvii}	2.060 (2)
Ba1—F3^{viii}	2.9998 (7)	Sc—F3	2.077 (5)
Ba1—F3^{iii}	2.9998 (6)	Sc—O2^{xviii}	2.227 (5)
Ba2—F1^{ix}	2.574 (2)	Sc—O2^{xix}	2.227 (5)
Ba2—F1^{x}	2.574 (2)	O1—C	1.254 (9)
Ba2—O1	2.702 (2)	O2—C	1.299 (6)
Ba2—F2^{v}	2.708 (2)	C—O2^{xiii}	1.300 (6)
O1—C—O2	122.5 (3)	O2—C—O2^{xiii}	115.0 (7)
O1—C—O2^{xiii}	122.5 (3)		

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

Data collection: *DIF*4 (Stoe & Cie, 1988*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990), option *PATT*. Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1994). Molecular graphics: *STRUPLO*90 (Fischer, Le Lirzin, Kassner & Rüdinger, 1991).

The authors thank Dr R. Retoux, Université du Maine, for his help in X-ray data collection.

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: PA1123). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A New Rare Earth Fluorocarbonate, $Na_2Eu(CO_3)F_3$

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Abstract

The structure of sodium europium fluorocarbonate, $Na_2Eu(CO_3)F_3$, obtained by hydrothermal growth, has been determined by single-crystal X-ray diffraction. The orthorhombic structure comprises EuO_3F_6 polyhedra linked by triangular faces and edges. Infinite EuO_2F_3 sheets in the *ab* plane are connected by the carbonate groups and Na atoms.

Comment

At high temperature (T = 1000 K), the study of the Na_2CO_3–LnF_3 system by hydrothermal growth leads only to $Na_3Ln_2(CO_3)_4F$ phases (Ln = La, Pr) (Mercier & Leblanc, 1993). At lower temperature, a new structure type, $Na_2Eu(CO_3)F_3$, is found. In the title compound, the cations adopt classical coordination numbers. Atoms Na1 and Na2 occupy the centres of NaO_4F_2 and NaO_2F_4 polyhedra, respectively. Each Eu atom is surrounded by three O atoms and six F atoms which form a tricapped triangular prism. It must be noted that the valence-bond analysis, as proposed by Brown (1982), is satisfied for all atoms. The EuO_3F_6 polyhedra are connected through the triangular faces formed by the F atoms and form infinite chains along *a*. These chains are linked together by O···O edges in order to build infinite $EuF_{6/2}O_{2/2}O$ sheets in the *ab* plane (Fig. 1). These sheets are shifted one from another along **c** and linked by Na atoms. One O atom of a carbonate group bonds solely to Na atoms. Only Na2 atoms are shown in Fig. 1, at the centre of pseudo-hexagonal cavities.

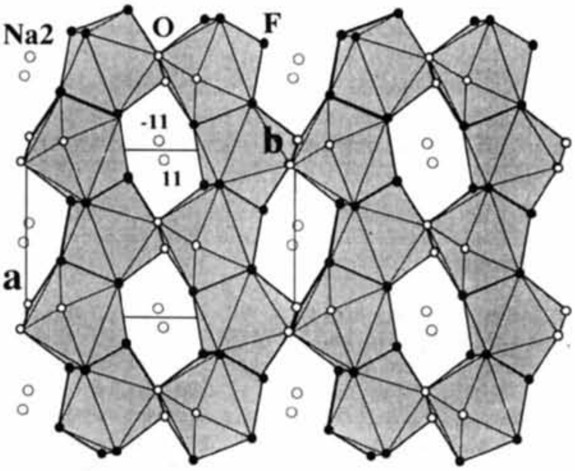

Fig. 1. Part of the structure of $Na_2Eu(CO_3)F_3$ showing a layer of EuO_3F_6 polyhedra in the *ab* plane.

Experimental

A mixture of Na_2CO_3 and EuF_3 in a 3/1 ratio under hydrothermal conditions (T = 650 K, P = 130 MPa) for 48 h leads to a new phase, $Na_2Eu(CO_3)F_3$. A parallelepipedic crystal was chosen for X-ray analysis by optical examination and its quality was tested with Laue photography.

Crystal data

$Na_2Eu(CO_3)F_3$
M_r = 314.94
Orthorhombic
Pbca
a = 6.596 (4) Å
b = 10.774 (4) Å
c = 14.090 (10) Å
V = 1001.3 (10) Å^3
Z = 8
D_x = 4.178 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 38 reflections
θ = 15.20–15.75°
μ = 12.713 mm^{-1}
T = 293 (2) K
Block
0.2 × 0.15 × 0.15 mm
Colourless

Data collection

Stoe Siemens AED four-circle diffractometer
$\omega/2\theta$ scans

1666 observed reflections [$I > 3\sigma(I)$]
$R_{\rm int}$ = 0.028

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