Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Sodium ytterbium carbonate difluoride, $NaYb(CO_3)F_2$

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(O–C) = 0.012 Å
R factor = 0.030
wR factor = 0.077
Data-to-parameter ratio = 13.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound was obtained by microwave-assisted hydrothermal synthesis at 463 K. It is isostructural with the mineral horváthite, $NaY(CO_3)F_2$. The structure is built up from (010) infinite $[NaYbCO_3]^{2+}$ layers interspersed by fluoride ions. All the atoms except F have site symmetry *m*.

Received 24 April 2006
Accepted 28 April 2006

Comment

In a study of the YbF_3–Na_2CO_3–H_2O system at T = 463 K, five phases were found (Ben Ali *et al.*, 2002): the title compound, $NaYb(CO_3)F_2$, (I), together with $Na_2Yb(CO_3)_2F$, $Na_3Yb(CO_3)_2F_2$, $Na_5Yb(CO_3)_4\cdot 2H_2O$ (Awaleh *et al.*, 2002) and $Yb(CO_3)(OH,F)\cdot xH_2O$.

Compound (I), synthesized at low [Na]/[Yb] ratios (< 8), is isostructural with horváthite, $NaY(CO_3)F_2$, a mineral from Mont Saint-Hilaire, Quebec (Grice *et al.*, 1997). Compound (I) contains (010) infinite $[NaYbCO_3]^{2+}$ layers separated by fluoride anions along the *b* axis (Figs. 1 and 2). The sodium and yttrium cations are seven- and eight-coordinated with mean Na—(O,F) and Yb—(O,F) distances of 2.408 and 2.305 Å, respectively (Table 1). The carbonate groups are constrained by symmetry to be perfectly flat (Grice *et al.*, 1994). The C—C distance between two successive layers in (I), 3.67 Å, is close to that observed in the yttrium analogue (3.68 Å).

According to the Krivovichev description (Krivovichev *et al.*, 1997), the fluoride anions in (I), surrounded by two sodium and two ytterbium cations, form distorted $[FNa_2Yb_2]$ tetrahedra. Two tetrahedra share two Yb atoms in order to form a $[F_2Na_{4/2}Yb_{4/2}]$ dimer. These $[F_2Na_2Yb_2]$ dimers are connected by sodium vertices and build the three-dimensional network shown in Fig. 3.

On heating, $NaYb(CO_3)F_2$ exhibits a one-step weight loss which occurs in the range 570–720 K. It is attributed to the loss of one mole of CO_2 per mole of $NaYb(CO_3)F_2$ (observed/

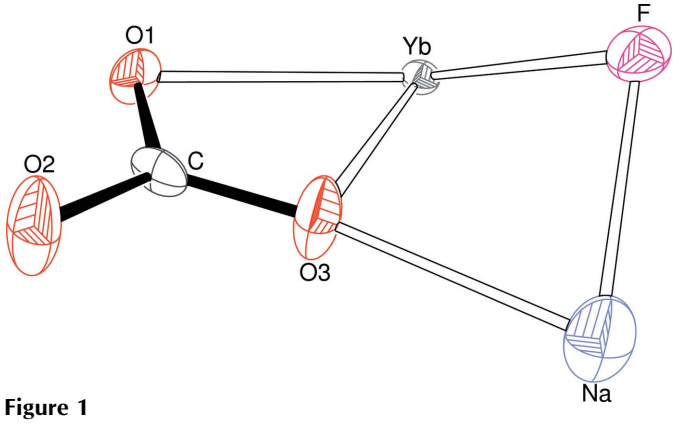

Figure 1
The asymmetric unit of (I), showing 70% displacement ellipsoids.

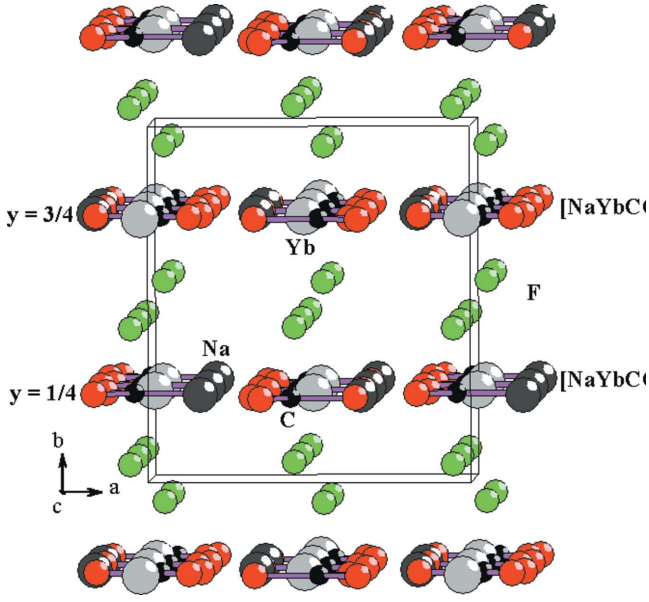

Figure 2
$[NaYbCO_3]^{2+}$ layers separated by fluoride anions along the *b* axis in (I).

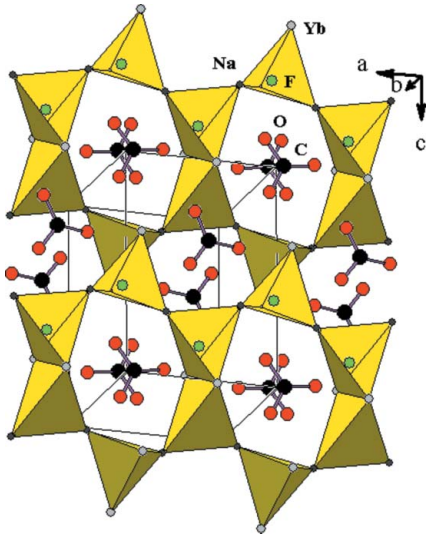

Figure 3
The three-dimensional network of vertex- and edge-linked fluorine-centred tetrahedra in (I).

calculated = 15.0/14.9%). The decomposition products are YbOF, NaF and CO_2.

Experimental

The title compound was synthesized in a CEM microwave oven (MDS 2100) and Teflon-lined autoclaves from a mixture of Na_2CO_3 and YbF_3 in the molar ratio range 2 < [Na]/[Yb] < 7 and $[YbF_3] > 0.2$ *M*. Reaction conditions were: $T = 463$ K, $P = 11 \times 10^5$ Pa, $V = 10$ ml and $t = 1$ h. The resulting crystals of (I) were washed with water and acetone and dried in air. The density of (I) was measured with an AccuPyc 1330 V3.03 pycnometer. Thermal analysis was performed with a DTA–TGA TA-Instrument 2960 (heating rate 10 K min^{-1}, argon atmosphere) in the temperature range 298–1073 K.

Crystal data

$NaYb(CO_3)F_2$	$D_x = 4.973$ Mg m^{-3}
$M_r = 294.04$	$D_m = 4.95(1)$ Mg m^{-3}
Orthorhombic, *Pnma*	D_m measured by pycnometery
$a = 6.243$ (1) Å	Mo $K\alpha$ radiation
$b = 6.892$ (2) Å	$\mu = 23.86$ mm^{-1}
$c = 9.127$ (2) Å	$T = 298$ (2) K
$V = 392.71$ (16) $Å^3$	Parallelepiped, colorless
$Z = 4$	0.15 × 0.10 × 0.04 mm

Data collection

Siemens AED2 diffractometer	609 independent reflections
$2\theta/\omega$ scans	523 reflections with $I > 2\sigma(I)$
Absorption correction: Gaussian (*SHELX76*; Sheldrick, 1976)	$\theta_{max} = 30.0°$
$T_{min} = 0.07$, $T_{max} = 0.39$	3 standard reflections
609 measured reflections	frequency: 120 min
	intensity decay: 15%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.046P)^2 + 2.2577P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.030$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.077$	$\Delta\rho_{max} = 2.15$ e $Å^{-3}$
$S = 1.13$	$\Delta\rho_{min} = -2.32$ e $Å^{-3}$
609 reflections	
46 parameters	

Table 1
Selected bond lengths (Å).

Na—F	2.336 (6)	Yb—O2^{vii}	2.227 (8)
Na—F^{i}	2.336 (6)	Yb—O1^{viii}	2.314 (7)
Na—O3	2.363 (9)	Yb—F^{i}	2.326 (4)
Na—F^{ii}	2.367 (6)	Yb—F	2.326 (4)
Na—F^{iii}	2.367 (6)	Yb—O3	2.327 (7)
Na—O2^{iv}	2.395 (10)	Yb—O1	2.439 (7)
Na—O3^{iv}	2.587 (9)	C—O2	1.261 (11)
Yb—F^{v}	2.191 (5)	C—O3	1.282 (12)
Yb—F^{vi}	2.191 (5)	C—O1	1.287 (11)

Symmetry codes: (i) $x, -y+\frac{1}{2}, z$; (ii) $x+\frac{1}{2}, y, -z+\frac{1}{2}$; (iii) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+\frac{1}{2}$; (iv) $x-\frac{1}{2}, y, -z+\frac{1}{2}$; (v) $-x, -y+1, -z+1$; (vi) $-x, y-\frac{1}{2}, -z+1$; (vii) $x-1, y, z$; (viii) $x-\frac{1}{2}, y, -z+\frac{3}{2}$.

The maximum residual electron-density peak is located 1.03 Å from Yb and the deepest hole is 0.97 Å from yb.

Data collection: *STADI4* (Stoe & Cie, 1998); cell refinement: *STADI4*; data reduction: *X-RED32* (Stoe & Cie, 1998); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *enCIFer* (Version 1.0; Allen *et al.*, 2004).

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