Crystal structure of the (*REE*)-uranyl carbonate mineral kamotoite-(Y)

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ABSTRACT

Kamotoite-(Y) is a rare supergene product of uraninite hydration–oxidation weathering and its structure is unknown. Based on single-crystal X-ray diffraction data collected with high-redundancy using a microfocus source, kamotoite-(Y) is monoclinic, has space group $P2_1/n$, with a = 12.3525(5), b = 12.9432(5), c = 19.4409(7) Å, β = 99.857(3)°, V = 3069.8(2) Å^3 and Z = 4. Crystals are pervasively twinned (two-fold rotation around [0.75 0 0.75]), giving a strongly pseudo-orthorhombic diffraction pattern. The pseudo-orthorhombic pattern can be described with an orthorhombic super-cell (transformation matrix 0,1,0/1,0,1/3,0,1), approximately four times larger in volume then a true monoclinic unit cell. This unit-cell is the same as the cell given elsewhere for the structure of bijvoetite-(Y), another (*REE*)-containing uranyl carbonate. The successful structure solution and refinement (R = 0.044 for 6294 unique observed reflections), carried out using our choice of unit cell, as well as the superstructure refinement and comparison of the original structure data for bijvoetite-(Y) reveal that these two crystal structures are identical. The crystal structure of kamotoite-(Y) consists of electroneutral sheets of the bijvoetite-(Y) uranylanion topology and an interlayer with H_2O molecules not-coordinated directly to any metal cation. Despite determination of the kamotoite-(Y) structure and demonstration that bijvoetite-(Y) has the same structure, the identity of these two minerals cannot be proved without additional study of the holotype material.

Introduction

Uranyl carbonates are abundant products of hydration–oxidation weathering of uraninite, in the presence of solutions with dissolved CO_2 (from the atmosphere or juvenile). These solutions can also be derived by dissolution of gangue-carbonates on hydrothermal veins carrying uranium ores (Plášil, 2014). Due to the potentially high mobility of U in carbonate-bearing groundwaters (Langmuir, 1978) and thus its environmental impact, a good knowledge of the crystal chemistry and behaviour of uranyl carbonate minerals is of great importance. Kamotoite-(Y), originally described as kamotoïte-(Y) (Deliens and Piret, 1986), is one of the five uranyl carbonates containing rare-earth elements (*REE*) known from Nature as supergene alteration products of uraninite. Related minerals are bijvoetite-(Y) (Deliens and Piret, 1982), lepersonnite-(Nd) (Deliens and Piret, 1982), shabaite-(Nd) (Deliens and Piret, 1989) and astrocyanite-(Ce) (Deliens and Piret, 1990). Among them, only the bijvoetite-(Y) structure has been reported (Li *et al.*, 2000).

Here, a crystal structure determination of kamotoite-(Y) is provided along with a discussion of related species.

Occurrence

The specimen studied containing kamotoite-(Y) crystals originates from the Kamoto East Open Cut, Kamoto, Kolwezi district, Katanga, Democratic Republic of Congo, which is the type locality of this mineral (Deliens and Piret, 1986). Kamotoite-(Y)

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forms well-developed and terminated crystals on altered fine-grained uraninite along with becquerelite and fine-grained astrocyanite-(Ce). Individual prismatic crystals of bright yellow colour reach up to 4 mm long.

Single-crystal X-ray diffraction

A prismatic, large (54 μm × 40 μm × 10 μm), yellowish crystal of kamotoite-(Y) was selected under optical microscope for X-ray study using a Rigaku Oxford diffraction SuperNova diffractometer. The diffraction experiment was performed using MoKα radiation (λ = 0.71073 Å) from a microfocus X-ray tube collimated and monochromatized by mirror-optics and detected by an Atlas S2 CCD detector. The crystal studied is monoclinic, with

a = 12.3525(5), b = 12.9432(5), c = 19.4409(7) Å, β = 99.857(3)°, V = 3069.8(2) Å^3 and Z = 4 (Table 1). The collected diffraction dataset contained a high redundancy of measured reflections; with 74,490 reflections collected. Of these, 10,449 were unique and 6294 were classified as observed with criterion [I_{obs} > 3σ(I)]. Integration of the data, including corrections for background, polarization and Lorentz effects, were carried out with the *CrysAlis RED* program (Oxford Diffraction, 2006). The empirical absorption correction using spherical harmonics was applied in *SCALE3 ABSPACK* in *CrysAlis*, which led to a dataset with an R_{int} of 0.087. The structure of kamotoite-(Y) was solved by the charge-flipping algorithm using the *SHELXT* program (Sheldrick, 2015). The structure was then refined in the *Jana2006* program (Petříček *et al.*, 2014) based on F^2. The structure solution

TABLE 1. Summary of data collection conditions and refinement parameters for kamotoite-(Y).

Structural formula	$(Y_{1.38}Dy_{0.62})_{\Sigma 2.00}(H_2^{[3]}O)_8(UO_2)_4O_2(OH)_2(CO_3)_4(H_2^{[4]}O)_{4.54}$
Unit-cell parameters (from 10,679 reflections)	
a	12.3825(5) Å
b	12.9432(5) Å
c	19.4409(7) Å
β	99.857(3)°
V	3069.8(2) Å^3
Z	4
Space group	$P2_1/n$
$D_{calc.}$ [g.cm^{-3}]	3.912 (applicable for the above given formula without H atoms)
Temperature	295 K
Diffractometer	Rigaku Oxford Diffraction Supernova, Atlas S2 CCD
Source, wavelength	Microfocus X-ray tube, 0.71075 Å (MoKα)
Crystal dimensions	0.06 × 0.04 × 0.01 mm^3
Limiting θ angles	3.32–28.05°
Limiting Miller indices	$-16 \leq h < 16, -16 \leq k \leq 16, -25 \leq l \leq 25$
No. of reflections	74,940
No. of unique reflections	10,449
No. of observed reflections (criterion)	6294 [$I > 3\sigma(I)$]
Absorption coefficient (mm^{-1}), method	25.22, empirical
T_{min}/T_{max}	0.448/1
R_{int}	0.087
F_{000}	3116
Parameters refined, constraints, restraints	225, 36, 0
R, wR (obs)	0.0435, 0.0717
R, wR (all)	0.0839, 0.0959
Goof (obs, all)	1.40, 1.27
Weighting scheme	$1/(\sigma^2(I) + 0.0004I^2)$
$\Delta\rho_{min}$, $\Delta\rho_{max}$ (e Å^{-3})	−1.69, 2.85
Twin matrix	$\begin{pmatrix} 0.5 & 0 & 0.5 \\ 0 & -1 & 0 \\ 1.5 & 0 & 0.5 \end{pmatrix}$
Twin fractions	0.8641(3)/0.1359(3)

provided an incomplete structure sheet of Y- and U-polyhedra with some missing O and C atoms, which were later localized from the difference-Fourier maps. Despite completion of the sheet the difference-Fourier maps still contained a large number of peaks and the R_{obs} value remained high, ~9%. Moreover, inspection of the intensity distribution for the diffractions showed I_{calc} (from refinement) < I_{obs}. The systematic absences (or the presence, respectively) of reflections observed in the frames suggest on the one hand doubling of some of the cell parameters, though on the other hand, some absences are inconsistent with any kind of cell centration. This led us to discovering that the crystal used is twinned. The careful inspection of the diffraction frames (Fig. 1) and precession-like images (unwarp) revealed the contribution of the twin component. After introduction of the twin matrix into the refinement, the R values dropped significantly (~5%), returning reasonable values for twin fractions. The twin is represented by a rotation of 180° (two-fold axis) around [0.75 0 0.75]. In the final cycles the occupancy of the Y sites was refined to allow the presence of heavier Dy (as proxy for all lanthanides) at the sites, as indicated by the electron microprobe analysis. Other trivalent *REE*s were omitted due to the similar scattering curves. All heavy atoms, excluding O as well as C atoms (these were constrained to have the same values of anisotropic displacement parameters), were refined with anisotropic displacement parameters during the last cycles of the refinement, which converged smoothly to

$R = 0.0435$ for 6294 unique observed reflections (Table 1). Final atom coordinates and displacement parameters are listed in Table 2, selected interatomic distances are in Table 2, and the bond-valence sums (calculated with the *DIST* option in *Jana2006* following the procedure of Brown, 1981, 2002) are listed in Table 2. The original crystallographic information file (cif) has been deposited with the Principal Editor of *Mineralogical Magazine* and is available from http://www.minersoc.org/pages/e_journals/dep_mat_mm.html

Crystal structure

The structure of kamotoite-(Y) (Fig. 2) contains four independent U sites, two independent Y/Dy mixed sites, four C sites and 37 O sites (some of which are H_2O). Each U atom is coordinated by two O atoms at the distance of ~1.8 Å forming an approximately linear $(UO_2)^{2+}$ uranyl ion (designated *Ur*). Two uranyl ions are further coordinated by three O^{2-} and two $(OH)^-$ anions, arranged at the equatorial corners of pentagonal bipyramids, and two are coordinated by six O^{2-} anions, giving hexagonal bipyramids. In each case the bipyramids are capped by the O atoms of the uranyls (O_{Ur}). The <U–5Φ_{eq}> and <U–6Φ_{eq}> (Φ: O^{2-}, OH$^-$ or H_2O; eq: equatorial) bond lengths range from 2.20 to 2.44 Å and 2.25 to 2.54 Å, respectively, which are consistent with the values reported for well-refined structures of U^{6+} phases (Burns *et al.*, 1997; Burns, 2005). The structure contains four independent
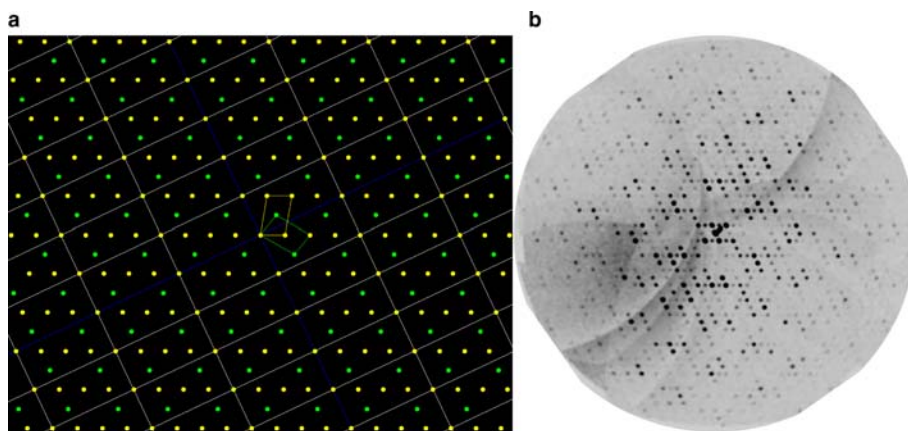

FIG. 1. Diffraction pattern of kamotoite-(Y). (*a*) Reciprocal space view of the two contributing twin domains; yellow = reflections of the larger domain, green = smaller domain. The grid reflects the settings of the super cell choice by Li *et al.* (2000). (*b*) Precession-like image of the *h*4 *l* layer of the diffraction pattern; the similarities with the reciprocal view given in the left are apparent.

TABLE 2. Atom positions and displacement parameters (U_{eq}, U_{iso}, in $Å^2$) for kamotoite-(Y).

Atom	BV	x/a	y/b	z/c	U_{eq}
U1	6.15(3)	−0.0598(3)	0.26873(3)	0.212187(18)	0.01605(12)
U2	6.38(4)	−0.02675(3)	0.28133(3)	0.414232(18)	0.01783(13)
U3	6.14(4)	0.25916(3)	0.28504(3)	0.535222(18)	0.01691(12)
U4	6.27(4)	0.22317(3)	0.29478(3)	0.332085(18)	0.01824(12)
Y1/Dy1[#]	3.11(2)/2.96(2)	0.50207(6)	0.30452(6)	0.24463(3)	0.0172(3)
Y2/Dy2[#]	3.19(2)/3.03(2)	0.70508(6)	0.18987(6)	0.50122(3)	0.0180(3)
C1	4.12(8)	0.7223(8)	0.2229(8)	0.6790(5)	0.0199(16)[$]
C2	4.07(8)	0.7502(8)	0.2451(8)	0.3335(5)	0.0199(16)[$]
C3	4.07(9)	0.4831(8)	0.2548(8)	0.0670(5)	0.0199(16)[$]
C4	4.03(8)	0.4508(8)	0.2692(8)	0.4144(5)	0.0199(16)[$]
O1	2.09(2)	0.0390(5)	0.3080(5)	0.3140(3)	0.0199(15)*
O2	2.15(4)	0.7734(6)	0.2373(5)	0.4003(4)	0.0214(17)*
O3	1.74(2)	−0.0007(6)	0.1480(5)	0.4079(4)	0.0290(18)*
O4	2.17(4)	0.8101(6)	0.2199(5)	0.7236(4)	0.0231(16)*
O5	2.21(5)	0.3650(6)	0.2891(5)	0.4414(3)	0.0234(16)*
O6	2.16(4)	0.6327(6)	0.2191(5)	0.7056(4)	0.0242(17)*
O7	2.00(5)	0.7204(6)	0.2315(5)	0.6150(4)	0.0283(18)*
O8	1.10(1)	−0.2410(6)	0.2339(5)	0.1557(4)	0.0199(16)*
O9	1.09(1)	0.4424(5)	0.2645(5)	0.5928(3)	0.0175(15)*
O10	1.70(2)	−0.0612(6)	0.4132(5)	0.4233(3)	0.0260(17)*
O11	1.64(2)	0.2530(6)	0.1473(5)	0.5252(3)	0.0265(17)*
O12	1.66(2)	−0.0416(6)	0.1338(5)	0.2302(3)	0.0250(17)*
O13	1.68(2)	0.2147(6)	0.1575(6)	0.3366(4)	0.0311(18)*
O14	2.07(5)	0.5432(7)	0.2518(5)	0.4469(4)	0.030(2)*
O15	1.70(2)	0.2715(6)	0.4203(5)	0.5501(3)	0.0265(17)*
O16	1.68(2)	−0.0844(6)	0.4015(5)	0.1908(3)	0.0259(17)*
O17	2.17(5)	0.5639(6)	0.2174(5)	0.0404(3)	0.0221(16)*
O18	1.88(4)	0.6568(6)	0.2226(5)	0.2995(4)	0.0245(17)*
O19	2.05(2)	0.1565(6)	0.3146(5)	0.4324(3)	0.0220(16)*
O20	1.65(2)	0.2409(6)	0.4316(5)	0.3258(4)	0.0277(17)*
O21	1.99(4)	0.4286(6)	0.2710(5)	0.3449(4)	0.0243(17)*
O22	0.35(1)	0.7160(6)	0.3749(6)	0.5074(4)	0.0306(18)*
O23	0.32(1)	0.4827(6)	0.1183(6)	0.2277(4)	0.0332(19)*
O24	2.21(5)	0.8298(6)	0.2766(5)	0.3044(4)	0.0248(17)*
O25	2.04(4)	0.3949(6)	0.2688(5)	0.0205(3)	0.0182(16)*
O26	0	0.8043(6)	0.5329(6)	0.3004(4)	0.039(2)*
O27	0	0.8900(7)	0.4519(6)	0.5954(4)	0.039(2)*
O28	2.05(5)	0.4876(6)	0.2757(6)	0.1295(4)	0.0284(18)*
O29	0.30(1)	0.6474(6)	0.4043(6)	0.2030(4)	0.0334(19)*
O30	0.29(1)	0.5632(7)	0.0863(6)	0.5445(4)	0.041(2)*
O31	0.32(1)	0.8054(7)	0.0375(6)	0.5503(4)	0.044(2)*
O32	0.34(1)	0.6559(7)	0.0550(7)	0.4160(4)	0.052(2)*
O33	0	0.5344(7)	0.4665(7)	0.5717(4)	0.053(2)*
O34	0.31(1)	0.5747(7)	0.4336(6)	0.3328(4)	0.042(2)*
O35	0	0.6260(8)	−0.0134(7)	0.6700(5)	0.063(3)*
O36	0.32(1)	0.4172(7)	0.4666(7)	0.2008(4)	0.052(2)*
O37[#]	0.02(1)	−0.0495(16)	−0.0370(14)	0.4427(10)	0.077(9)*

Atom	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
U1	0.00973(19)	0.0277(2)	0.00963(17)	−0.00054(17)	−0.00158(14)	0.00007(15)
U2	0.00923(19)	0.0338(3)	0.00965(18)	−0.00068(17)	−0.00067(14)	0.00070(16)
U3	0.01117(19)	0.0282(2)	0.00996(17)	−0.00040(17)	−0.00205(14)	0.00085(16)

(continued)

656

TABLE 2. (*contd*.)

Atom	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
U4	0.00870(19)	0.0350(3)	0.01025(17)	−0.00096(18)	−0.00064(14)	0.00002(17)
Y1/Dy1[#]	0.0108(4)	0.0293(5)	0.0108(4)	0.0000(3)	0.0002(3)	−0.0016(3)
Y2/Dy2[#]	0.0102(4)	0.0320(5)	0.0108(4)	−0.0024(3)	−0.0007(3)	0.0007(3)
C1	0.007(2)	0.040(3)	0.010(2)	0.003(2)	−0.0033(17)	0.002(2)

* Refined with isotropic displacement parameter; [$] atoms constrained to have the same anisotropic displacement parameters; [#]refined occupancies for Y1/Dy1, Y2/Dy2 and O37 sites are 0.699(4)/0.301(4), 0.684(4)/0.316(4) and 0.54(3), respectively.

CO_3 groups, with, as expected, distances of ~1.28 Å. There are two symmetrically independent M^{3+} sites that are occupied by different proportions of Y and Dy cations (Table 2). They are coordinated by four O^{2-} equatorial ligands of uranyl hexagonal bipyramids (the shared O ligands of the chelating CO_3 groups) within the sheets and H_2O groups with $<M^{3+}–\Phi>$ ranging from 2.25 to 2.48 Å. The distribution of Y and Dy over these two sites seems to be uniform; according to the results of site-scattering refinement, the Y content of both sites is ~70%.

The structure of kamotoite-(Y) is based upon the structural sheets of the bijvoetite topology (Burns, 2005) characteristic for the related uranyl carbonate mineral bijvoetite-(Y) (Li *et al.*, 2000) (see Discussion below). These sheets result from the sharing of equatorial edges of dimers of uranyl pentagonal and hexagonal bipyramids (described above). The dimers of hexagonal bipyramids are oriented perpendicular to the length of chains of uranyl polyhedra (approximately perpendicular to [101]) and are connected through the dimers of uranyl pentagonal bipyramids, which extend along the length of these chains. As such, each of the equatorial vertices of the pentagonal bipyramids are shared with other uranyl polyhedra, but two of the equatorial vertices of each hexagonal bipyramid are bonded to only one U^{6+} cation. Carbonate groups chelate the hexagonal
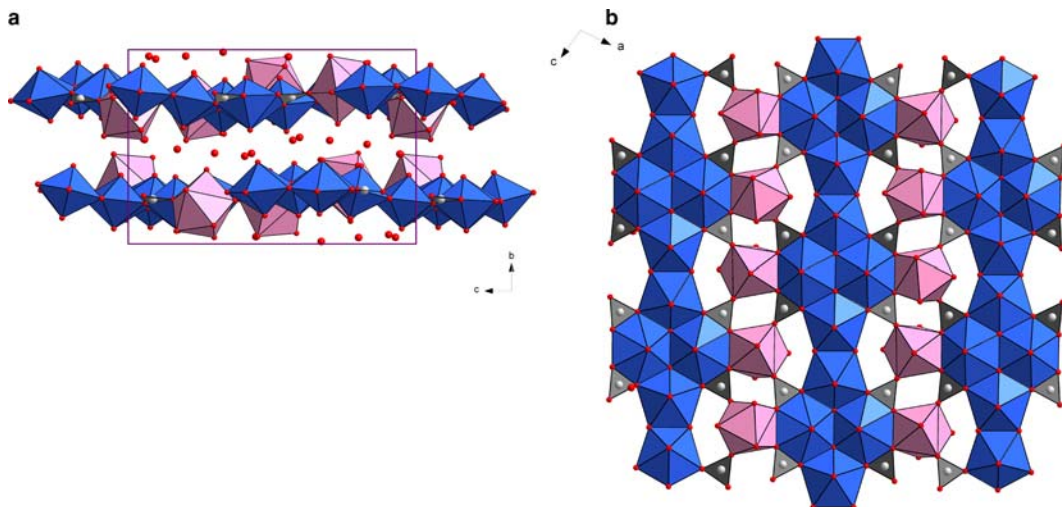

FIG. 2. Crystal structure of kamotoite-(Y). (*a*) Along [100]. Uranyl-carbonate-*REE*s sheets are stacked perpendicular to **b**; separated by an interlayer containing only molecular H_2O (represented by red coloured O atoms between the sheets). (*b*) A uranyl-carbonate-*REE* sheet of the bijvoetite-(Y) topology; blue = UO_7 and UO_8 bipyramids, pink = YO_8 polyhedra and black triangles = carbonate planar group. The OH groups are located at the shared edge between two pentagonal bipyramids of the tetramers of the UO-chains.

657

TABLE 3. Selected interatomic distances (in Å) for the structure of kamotoite-(Y).

U1–O12	1.788(7)	U2–O3	1.764(7)	U3–O11	1.794(7)
U1–O16	1.782(7)	U2–O10	1.775(7)	U3–O15	1.776(7)
U1–O1	2.202(6)	U2–O1	2.263(7)	U3–O5	2.422(7)
U1–O6[i]	2.415(7)	U2–O2[ii]	2.509(7)	U3–O8[iv]	2.356(7)
U1–O8	2.365(7)	U2–O17[iii]	2.517(6)	U3–O9	2.367(7)
U1–O9[i]	2.366(7)	U2–O19	2.277(7)	U3–O17[iii]	2.437(7)
U1–O24[ii]	2.435(8)	U2–O24[ii]	2.534(7)	U3–O19	2.212(6)
$<U1–O_{Ur}>$	1.785	U2–O25[iii]	2.513(7)	$<U3–O_{Ur}>$	1.785
$<U1–O_{eq}>$	2.357	$<U2–O_{Ur}>$	1.770	$<U3–O_{eq}>$	2.359
		$<U2–O_{eq}>$	2.436		
U4–O13	1.783(7)	C1–O4	1.270(11)	C3–O17	1.297(13)
U4–O20	1.791(7)	C1–O6	1.303(13)	C3–O25	1.306(11)
U4–O1	2.253(7)	C1–O7	1.246(12)	C3–O28	1.235(12)
U4–O4[i]	2.534(7)	$<C1–O>$	1.27	$<C3–O>$	1.28
U4–O5	2.516(7)				
U4–O6[i]	2.528(7)	C2–O2	1.285(12)	C4–O5	1.288(13)
U4–O19	2.258(7)	C2–O18	1.263(11)	C4–O14	1.229(13)
U4–O21	2.531(7)	C2–O24	1.283(13)	C4–O21	1.332(12)
$<U4–O_{Ur}>$	1.787	$<C2–O>$	1.28	$<C4–O>$	1.28
$<U4–O_{eq}>$	2.437				
Y1/Dy1–O4[i]	2.363(7)	Y2/Dy2–O2	2.348(7)		
Y1/Dy1–O18	2.288(7)	Y2/Dy2–O7	2.252(8)		
Y1/Dy1–O21	2.329(8)	Y2/Dy2–O14	2.248(8)		
Y1/Dy1–O23	2.439(7)	Y2/Dy2–O22	2.401(7)		
Y1/Dy1–O28	2.246(7)	Y2/Dy2–O25[iv]	2.377(7)		
Y1/Dy1–O29	2.461(8)	Y2/Dy2–O30	2.470(8)		
Y1/Dy1–O34	2.452(7)	Y2/Dy2–O31	2.436(8)		
Y1/Dy1–O36	2.435(8)	Y2/Dy2–O32	2.412(8)		
$<Y1/Dy1–O>$	2.377	$<Y2/Dy2–O>$	2.368		

Symmetry codes: (i) $x–1/2$, $–y+1/2$, $z–1/2$; (ii) $x–1$, y, z; (iii) $x–1/2$, $–y+1/2$, $z+1/2$; (iv) $x+1/2$, $–y+1/2$, $z+1/2$.

bipyramids, such that each bipyramid is linked to two carbonate triangles. Chains are linked into sheets through coordination-polyhedra about trivalent cations, which correspond to Y and Dy. Between the sheets in the interlayer, there are five O sites, which belong to molecular H_2O that is not coordinated to any metal cation directly; these are O26, O27, O31, O33 and O35 (Table 2). These H_2O participate in linkage of the adjacent *REE*-uranyl-carbonate sheets through a H-bond network. Within the sheets there are H_2O groups coordinated to the Y/Dy sites; Y1: O23, O29, O34, O36; Y2: O22, O30, O31, O32. While these H_2O groups linked to the M^{3+} sites can be described as 'transformer H_2O groups' containing [3]-coordinated O atoms (Hawthorne and Schindler, 2008; Schindler and Hawthorne, 2008), the H_2O in the interlayer are 'non-transformer H_2O groups', with [4]-coordinated O

atoms. The bond-valence acceptors within the sheets are undersaturated (in terms of bond-valence requirements) O_{Ur} atoms and some of the equatorial O atoms of the bipyramids shared with the CO_3 groups (Tables 2, 3).

The structural formula of kamotoite-(Y) obtained from the structure refinement is $(Y_{1.38}Dy_{0.62})_{\Sigma 2.00}(H_2^{[3]}O)_8[(UO_2)_4O_2(OH)_2(CO_3)_4](H_2^{[4]}O)_{4.54}$, $Z = 4$, with $D_{calc} = 3.91$ g cm^{-3}.

Discussion – identity of kamotoite-(Y) and bijvoetite-(Y)

Originally, kamotoite-(Y) was described (Deliens and Piret, 1986) with a monoclinic unit cell with the different settings (but with the same space group $P2_1/n$): $a = 21.22(1)$, $b = 12.93(1)$, $c = 12.39(1)$ Å, $\beta = 115.3(1)°$, $V = 3073$ Å^3 and $Z = 4$. This cell can be

transformed into our cell choice using the following matrix: (0,0,-1/0,1,0/1,0,1). We tested several crystals during the current study in order to find the best crystal for the structure determination and it should be noticed that the cell given in Table 1 was consistent for all of the checked crystals (within some range of experimental errors).

Kamotoite-(Y) is the second uranyl carbonate mineral containing *REE*s besides bijvoetite-(Y). Both minerals are based upon structural sheets of the same topology (Li *et al.*, 2000). There are several other similarities between these two minerals. Therefore, the questions about their close relationship should be examined. Kamotoite-(Y) and bijvoetite-(Y) are chemically closely related: the kamotoite-(Y) ideal formula is $Y_2[(UO_2)_4 O_2(OH)_2(CO_3)_4](H_2O)_{13}$, $Z = 4$ (this work); for bijvoetite-(Y) it is $Y_8[(UO_2)_{16}O_8(OH)_8(CO_3)_{16}] (H_2O)_{39}$ (Li *et al.*, 2000). Thus, the two minerals have identical Y:U:C ratios, but bijvoetite-(Y) has a slightly lower water content. Bijvoetite-(Y) has a unit cell, which is approximately four times larger in volume ($\sim 12{,}360$ Å^3): $a = 21.234(3)$, $b = 12.958(2)$, $c = 44.911(7)$ Å, $\beta = 90.00(2)°$, $Z = 4$. It is a monoclinic (pseudo-orthorhombic) cell in $B12_11$ (non-standard setting emphasizing the relationship to the twinned kamotoite-(Y) cell); this cell can be derived from that of kamotoite-(Y) by the transformation matrix (0,1,0/1,0,1/3,0,1). Because we were aware of the problems suggested in the structure determination of bijvoetite-(Y), we rechecked the reflection file for bijvoetite-(Y) used by Li *et al.* (2000) (provided to us by one of the authors – Peter Burns). Our model, presented here in the current paper, fits the data by Li *et al.* (2000) very well, converging smoothly to $R_{obs} \approx 5\%$ (with twin fractions 39:61). We conclude that the authors probably overinterpreted their data: they used a large number of unobserved reflections in their refinement, obtaining a poor fit, and inferred an incorrect twin law. Furthermore we notice that despite the distinct cell, $a = 21.234(3)$, $b = 12.958(2)$, $c = 24.839(3)$ Å, $\beta = 115.30(2)°$, deduced by Li *et al.* (2000) from the data that was probably affected by the twinning and wrong cell assignment, the two crystals investigated in the current study and by the study of Li *et al.* (2000) belong to the same mineral structure. Therefore, it is possible that these two minerals are identical.

For that reason, the identity of these two minerals should be questioned. The structure study conducted by Li *et al.* (2000) was not performed on a sample originating from the holotype specimen, but on a specimen from a private collection. (Note that a holotype specimen was deposited in the Museé Royal de l'Afrique Centrale in Tervuren, Belgium; see Deliens and Piret, 1982). To assess the existence of two distinct minerals or their identity, the crystal of bijvoetite-(Y) from the holotype specimen has to be investigated; however, this is beyond the scope of the present study.

Kamotoite-(Y) is another example of a uranyl carbonate mineral that contains a structure based upon the sheets of polyhedra of higher valence, besides bijvoetite-(Y) (see Discussion above for details), $Y_8[(UO_2)_{16}O_8(OH)_8(CO_3)_{16}](H_2O)_{39}$ (Li *et al.*, 2000), rutherfordine, $[(UO_2)(CO_3)]$ (Finch *et al.*, 1999), wyartite, $Ca[U^{5+}(UO_2)_2(CO_3)O_4(OH)]$ $(H_2O)_7$ (Burns and Finch, 1999), roubaultite, $[Cu_2(UO_2)_3(CO_3)_2O_2(OH)_2](H_2O)_4$ (Ginderow and Cesbron, 1985) and fontanite, $Ca[(UO_2)_3(CO_3)_2O_2]$ $(H_2O)_6$ (Hughes and Burns, 2003).

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