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Crystal chemistry of thorium nitrates and chromates

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ABSTRACT

The structures and infrared spectra of six novel thorium compounds are reported. $Th(NO_3)_2(OH)_2(H_2O)_2$ (**1**) crystallizes in space group *C*2/*c*, $a=14.050(1)$, $b=8.992(7)$, $c=5.954(5)$ Å, $\beta=101.014(2)^\circ$. $K_2Th(NO_3)_6$ (**2**), *P*-3, $a=13.606(1)$, $c=6.641(6)$ Å. $(C_{12}H_{28}N)_2Th(NO_3)_6$ (**3**), *P*2$_1$/*c*, $a=14.643(4)$, $b=15.772(5)$, $c=22.316(5)$ Å, $\beta=131.01(1)^\circ$. $KTh(NO_3)_5(H_2O)_2$ (**4**), *P*2$_1$/*c*, $a=10.070(8)$, $b=12.731(9)$, $c=13.231(8)$ Å, $\beta=128.647(4)^\circ$. $Th(CrO_4)_2(H_2O)_2$ (**5**), *P*2$_1$/*n*, $a=12.731(1)$, $b=9.469(8)$, $c=12.972(1)$ Å, $\beta=91.793(2)^\circ$. $K_2Th_3(CrO_4)_7(H_2O)_{10}$ (**6**), *Ama*2, $a=19.302(8)$, $b=15.580(6)$, $c=11.318(6)$ Å. The coordination polyhedra about Th in these structures are diverse. Th is coordinated by 9 O atoms in **5** and **6**, seven of which are from monodentate (CrO_4) tetrahedra and two are (H_2O). The Th in compound **1** is coordinated by ten O atoms, four of which are O atoms of two bidentate (NO_3) triangles and six of which are (OH) and (H_2O). In compounds **2**, **3** and **4** the Th is coordinate by 12 O atoms. In **2** and **3** there are six bidentate (NO_3) triangles, and in **4** ten of the O atoms are part of five bidentate (NO_3) triangles and the others are (H_2O) groups. The structural units of these compounds consist of a chain of thorium and nitrate polyhedra (**1**), isolated thorium hexanitrate clusters (**2**, **3**), an isolated thorium pentanitrate dihydrate cluster (**4**), and a sheet (**6**) and framework (**5**) of thorium and chromate polyhedra. These structures illustrate the complexity inherent in the crystal chemistry of Th.

1. Introduction

Despite its potential importance in a nuclear fuel cycle and in managing nuclear waste, the crystal chemistry of Th is understudied relative to main-group elements. Th-232, which occurs in the tetravalent oxidation state, has a long half-life and thus occurs in nature. It is used as an analogue for the redox-active tetravalent actinides, although Th is larger than any of those cations and the extent to which the crystal chemical analogy holds is unclear [1]. As part of our ongoing research of actinide materials and inspired by several recent studies of Th compounds [1–12], we have synthesized and characterized four novel Th nitrates and two Th chromates.

The structures of six thorium nitrates have previously been reported. $MgTh(NO_3)_6(H_2O)_8$ [13] contains Th cations that are coordinated by six bidentate (NO_3) groups, and the resulting clusters are isolated from each other and the $Mg(H_2O)_6$ octahedra, with linkages only through H bonds. The compound $(NH_4)_2[Th(NO_3)_6]$ [14] contains an identical Th hexanitrate cluster linked through $(NH_4)^+$ cations. Both $Th(NO_3)_4(H_2O)_4$ [15] and $Th(NO_3)_4(H_2O)_5$ [16] contain Th cations coordinated by four bidentate (NO_3) groups and three (H_2O) groups, giving a coordination number of 11. Linkage between the clusters is through H bonds. The compound $[Th_2(OH)_2(NO_3)_6(H_2O)_6](H_2O)$ contains a dimer of Th polyhedra [8]. Each Th cation is coordinated by three bidentate (NO_3) groups, three (H_2O) groups, and two (OH) groups that form the shared edge of the dimer. The dimers of polyhedra are linked only through H bonds. $[Th_2(OH)_2(NO_3)_4(H_2O)_8](NO_3)_2$ also contains of dimer of Th polyhedra [8], but in this case each Th atom is coordinated by two bidentate (NO_3) groups, four (H_2O) groups, and two $(OH)^-$ groups that form an edge that is shared between the polyhedra.

The structures of only three thorium chromates have been described. $Th(OH)_2CrO_4(H_2O)$ contains Th that is coordinated by four monodentate (CrO_4) tetrahedra and four (OH) groups. These polyhedra share edges defined by two (OH) groups, forming chains that are cross-linked into a framework structure through the (CrO_4) tetrahedra. The compound $Th(OH)_2(CrO_4)(H_2O)$ [17] contains two different Th sites, one of which is coordinated by seven O atoms of monodentate (CrO_4) tetrahedra and one (H_2O) group. The other site is coordinated by eight monodentate (CrO_4) tetrahedra and an (H_2O) group. The Th polyhedra and (CrO_4) tetrahedra are linked into a framework. The framework structure of the mixed oxyanion compound $Th(CrO_4)(IO_3)_2$ contains nine-coordinate Th [18].

2. Experimental

2.1. Synthesis

Caution! Thorium-232 is radioactive with a half-life of 14.1×10^9 years. Appropriate precautions should be followed when working with radioactive materials.

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$Th(NO_3)_4 \times H_2O$ (Johnson Matthey), $K_2Cr_2O_7$ (Fisher), $Na_2Cr_2O_7$ (Fisher), and $Gd(NO_3)_3 \cdot 6\,H_2O$ (Acros Organics) were used as-received. Distilled and Millipore-filtered water with a resistance of 18.2 MΩ cm was used in all reactions. In the case of the thorium-chromate experiments, a thorium nitrate stock solution was prepared by adding 0.783 g of $Th(NO_3)_4 \times H_2O$ to 20.6 mL H_2O in the Teflon liner of a 125-mL digestion vessel. The pH of the solution was adjusted to 4 by the addition of 1 M NaOH. The reaction vessel was sealed and heated at 65 °C for 24 h. The solution was cooled to room temperature in air and was subsequently employed in further synthesis experiments.

$Th(NO_3)_2(OH)_2(H_2O)_2$ (1): $Th(NO_3)_4$ (0.602 g) was added to 5 mL H_2O. The resulting solution was brought to a boil and then 3 mL of 1.0 M LiOH was added drop-wise. The total solution volume was then reduced to 2 mL by boiling. The resulting solution was cooled and left standing open in air to allow evaporation. Crystals were harvested once they formed, prior to the solution entirely evaporating.

$K_2Th(NO_3)_6$ (2): $Th(NO_3)_4$ (0.597 g) was added to 5 mL of H_2O that was then heated to a boil, at which time 3 mL of 0.1 M KOH was added drop-wise. The total volume was reduced to 2 mL by boiling. The resulting solution was cooled to room temperature and was left open to air to evaporate. Crystals were harvested as soon as they formed.

$(C_{12}H_{28}N)_2Th(NO_3)_6$ (3): 5 mL of a 50 mM $Th(NO_3)_4$ aqueous solution was combined with 0.179 g of tetrapropylammonium chloride in a 23 mL Teflon-lined reaction vessel that was subsequently heated at 150 °C for 4 days. Once cooled, the resulting solution, which did not contain crystals, was transferred to a glass vial that was capped. Crystals were harvested as soon as they were present.

$KTh(NO_3)_5(H_2O)_2$ (4): 0.598 g of $Th(NO_3)_4$ was added to 5 mL of H_2O and the resulting solution was heated to a boil. While boiling, 3 mL of 0.25 M KOH was added drop-wise. Continued boiling reduced the solution volume to 2 mL, at which time it was cooled and left open to air to allow evaporation. Crystals were harvested as soon as they formed.

$Th(CrO_4)_2(H_2O)_2$ (5): 0.250 mL of the Th nitrate stock solution was mixed with 0.100 mL of 1 M $K_2Cr_2O_7$ aqueous solution in a 5 mL polypropylene vial that was subsequently capped. The vial was heated contained in a 125-mL Teflon-lined acid digestion vessel at 100 °C for 24 h in a preheated convection oven. Twenty-five milliliter of H_2O was placed in the 125 mL liner to provide counter-pressure during heating. The product consisted of a low yield of orange crystals in an orange-colored solution. The products were transferred to a glass vial and capped for later analysis.

$Th_3(CrO_4)_7(OH)_2(H_2O)_8$ (6): 0.100 mL of the Th nitrate stock solution was added to a combination of 0.100 mL gadolinium nitrate and 0.100 mL of aqueous 0.1 M $K_2Cr_2O_7$. The solution was placed in a 5 mL polypropylene vial and heated in a 125-mL Teflon-lined acid digestion vessel at 100 °C for 24 h in a preheated convection oven. Twenty-five milliliter of H_2O was placed in the 125 mL liner to provide counter-pressure. The product consisted of orange crystals in an orange-colored solution. The products were transferred to a glass vial for further analysis.

2.2. *Crystallographic studies*

X-ray diffraction data were collected using a Bruker PLATFORM Three-circle single crystal diffractometer equipped with an APEX CCD detector and Mo*K*α radiation. Crystals were mounted on cryoloops in oil and data were collected at 110 K. Lorentz, polarization, and background corrections were performed using the program SAINT [19]. Empirical absorption corrections for each crystal were done using XPREP [19]. Structure solutions (direct methods) and least squares refinement on the basis of F^2 were done using SHELXTL Version 6.12 [19]. All structure determinations and refinements were straightforward and included anisotropic displacement parameters for all non-H atoms. With the exception of compound **1**, H atom positions were not located, as is typical of X-ray diffraction studies of actinide compounds. Miscellaneous crystallographic data for the compounds are provided in Tables 1 and 2. Atomic coordinates, displacement parameters, and selected interatomic distances are in the supporting information.

Table 1
Crystallographic data for compounds **1**, **2**, **3**, and **4**.

	1 $Th(NO_3)_2(OH)_2(H_2O)_2$	**2** $K_2Th(NO_3)_6$	**3** $(C_{12}H_{28}N)_2Th(NO_3)_6$	**4** $KTh(NO_3)_5(H_2O)_2$
a (Å)	14.050(1)	13.606(1)	14.643(4)	10.070(8)
b (Å)	8.992(7)	13.606(1)	15.772(5)	12.731(9)
c (Å)	5.954(5)	6.641(6)	22.316(5)	13.231(8)
β (deg.)	101.014(2)	90	131.008(1)	128.647(4)
V (Å^3)	738.31(10)	1064.71(16)	3889.2	1324.71(16)
Space group	*C*2/*c*	*P*-3	*P*2$_1$/*c*	*P*2$_1$/*c*
Z	4	3	4	4
F(000)	760	942	1736	1120
μ (mm^{-1})	20.24	11.22	3.91	11.69
D_{calc} (g/cm^3)	3.833	3.192	1.572	3.075
Crystal size (mm)	0.15 × 0.10 × 0.10	0.10 × 0.075 × 0.075	0.10 × 0.10 × 0.075	0.10 × 0.075 × 0.075
Radiation	Mo*K*α	Mo*K*α	Mo*K*α	Mo*K*α
T (K)	110	110	110	110
Frame width (deg.)	0.3	0.3	0.3	0.3
Count time per frame (s)	10	10	10	10
2θ range	2–69.00	2–69.00	2–70.09	2–69.00
Reflections collected	7441	21935	71386	26553
Independent reflections	1541	2999	15468	5497
Unique $F_o > 4s_F$	1486	2306	10321	4806
*R*1 (%)	2.19	2.29	3.79	2.53
*wR*2 (%)	4.3	4.83	9.46	4.83
S	0.985	0.711	0.942	0.984

$R1 = \sum||F_o|-|F_c||/\sum|F_o|.Rw2(F_o^2) = [\sum[w(F_o^2-F_c^2)^2]/\sum wF_o^4]^{1/2}$.

Table 2
Crystallographic data for **5** and **6** structures.

	5 $Th(CrO_4)_2(H_2O)_2$	**6** $K_2Th_3(CrO_4)_7(H_2O)_{10}$
a (Å)	12.731(1)	19.302(8)
b (Å)	9.469(8)	15.580(6)
c (Å)	12.972(1)	11.318(6)
β (deg.)	91.793(2)	90
V (Å^3)	1562.9(2)	3403.6
Space group	$P2_1/n$	$Ama2$
Z	8	4
F(000)	1744	3119
μ (mm^{-1})	21.75	15.56
D_{calc} (g/cm^3)	4.216	3.408
Crystal size (mm)	0.050 × 0.10 × 0.150	0.050 × 0.075 × 0.075
Radiation	Mo$K\alpha$	Mo$K\alpha$
T (K)	110	110
Frame width (deg.)	0.3	0.3
Count time per frame (s)	10	10
2θ range	2–69.03	2–69.00
Reflections collected	31553	34263
Independent reflections	6494	7184
Unique $F_o > 4s_F$	5550	6633
$R1$ (%)	6.3	3.34
$wR2$ (%)	17.71	8.05
S	1.079	1.042

$R1=\sum||F_o|-|F_c||/\sum|F_o|.Rw2(F_o^2)=\left[\sum[w(F_o^2-F_c^2)^2]/\sum wF_o^4\right]^{1/2}$.

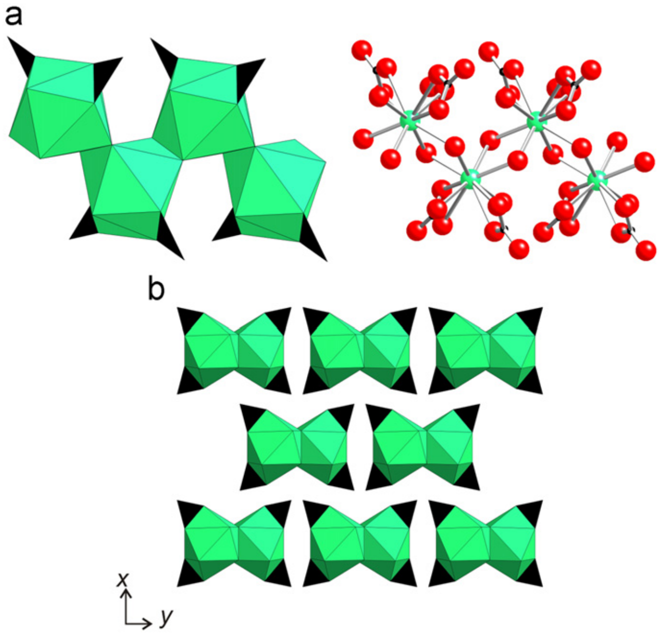

Fig. 1. Polyhedral and ball-and-stick representations of the structure of compound **1**. (a) The chain of Th polyhedra and nitrate triangles extending along [001]. (b) Projection along [001] showing the packing of the chains of polyhedra. Th polyhedra: green; Nitrate triangles: black. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

2.3. Infrared spectroscopy

Infrared spectra were collected for crushed single crystals of each compound using a diamond attenuated total reflectance objective mounted on a SensIR spectrometer. Data were collected from 650 to 4000 cm^{-1} for each compound. The IR spectrum of each compound is shown in the supporting information. For compound **1,** nitrate stretches are present at 1460, 1283, 1041, 806, 823, and 741 cm^{-1}. A band at 608 cm^{-1} is attributed to Th–O linkages. The mode at 1625 arises from H_2O bending, and H bonds associated with OH and H_2O give rise to modes at ~3400 cm^{-1}. The IR spectrum of **2** contains stretches resulting from nitrate are at 1553, 1482, 1251, 1024, 802, 741, and 707 cm^{-1}. The band at 577 cm^{-1} is assigned to the Th polyhedra. The IR spectrum of **3** has stretches representing nitrate are at 742, 809, 967, 1026, 1276, and 1496 cm^{-1}, whereas the C–C peaks are at 2883 and 2973 cm^{-1}. The IR spectrum of **4** shows hydroxide and water vibrations located at 3359 and 1618 cm^{-1}. Nitrate bands are at 1478, 1277, 1251, 1022, 804, 744, and 705 cm^{-1}. Stretches related to thorium polyhedra are present at 649 and 558 cm^{-1}. The IR spectrum of compound **5** shows H_2O modes located at 3200 and 1580 (H_2O) cm^{-1}. Modes at 957, 893, and 841 cm^{-1} are due to chromate, and the mode at 719 cm^{-1} is assigned to the Th polyhedra. The IR of compound **6** has H_2O modes in the 3200 region and at 1597 cm^{-1}. Modes associated with chromate are at 956 and 885 cm^{-1}. The mode at 756 cm^{-1} is attributed to the Th polyhedra.

3. Results and discussion

Compound **1** contains a single symmetrically unique Th site that is coordinated by two bidentate (NO_3) groups, two (H_2O) groups, and four (OH) groups (Fig. 1). Bond lengths to the (OH) HH groups are 2.325(2) and 2.374(2) Å, to the (H_2O) groups are 2.573(2) Å, and to the O atoms of the (NO_3) groups are 2.630(2) and 2.882(2) Å. The (NO_3) groups are located on the same side of the polyhedron with their planes in opposing directions. Nitrate bond lengths are 1.218(3) Å for the non-bridging O atoms and range from 1.264(3) to 1.275(4) Å for the O atoms that are bonded to Th. The (OH) groups define two edges on the opposite sides of the polyhedron, and these (OH) groups are shared between polyhedra, resulting in a zigzag chain of polyhedra that extends along [001] (Fig. 1a). The Th–Th separations along the chain length are 3.92 Å. Adjacent chains are connected into the extended structure only through H bonds. H atom positions determined from the X-ray data indicate that the H bonds link the chains by extending from the (OH) groups of a given chain to O atoms of (NO_3) groups on adjacent chains.

Compounds **2** and **3** contain Th cations that are coordinated by six bidentate nitrate groups (Fig. 2a,b, respectively). The Th–O bond lengths in these clusters range from 2.535(2) to 2.581(2) Å, which are considerably shorter than the Th–O bond distances to (NO_3) groups found in compound **1**. In the anhydrous structure of **2** the Th hexanitrate clusters are linked through bonds to K cations only (Fig. 2c). The K cations are coordinated by eight O atoms, resulting in a one-sided coordination polyhedron (Fig. 2c). The K cations delimit empty channels that extend along [001], with a K–K separation of 9.2 Å. The compound is isostructural with $K_2Ce(NO_3)_6$ [20].

In compound **3,** Th hexanitrate groups are connected by tetrapropylammonium cations and the linkages between the clusters are through H bonding (Fig. 2d). Note that the arrangement of (NO_3) triangles of the Th polyhedra differ in compounds **2** and **3**. This difference presumably arises from the steric constraints associated with the tetrapropylammonium cations.

The structure of compound **4** is illustrated in Fig. 3. It contains one symmetrically unique Th cation that is coordinated by 12 O atoms, of which ten oxygen atoms are of five bidentate (NO_3) groups and two are part of (H_2O) groups. The Th pentanitrate clusters are linked into the extended structure though bonds to interstitial K cations. The Th–O bond lengths to the O atoms of (H_2O) groups are 2.453(2) and 2.551(2) Å, whereas the Th–O bond lengths to the (NO_3) groups range from 2.531(2) to 2.661(2) Å. The nitrate groups are located in a plane bisecting the Th

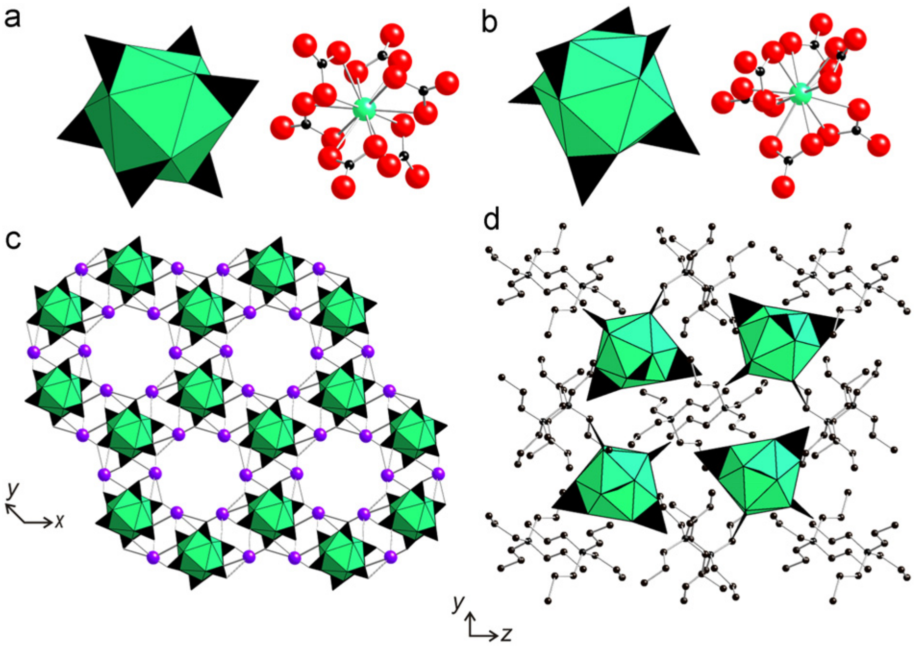

Fig. 2. Polyhedral and ball-and-stick representations of the structure of compounds **2** and **3**. (a) The cluster of Th and nitrate triangles that occurs in **2**. (b) The cluster of Th and nitrate triangles that occurs in **3**. (c) Projection of the structure of **2** along [001] showing the packing of the clusters of polyhedra and the channels extending along [001]. (d) Projection of the structure of **3** along [100]. Th polyhedra: green; Nitrate triangles: black; K atoms: blue spheres; N and C atoms: black spheres. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

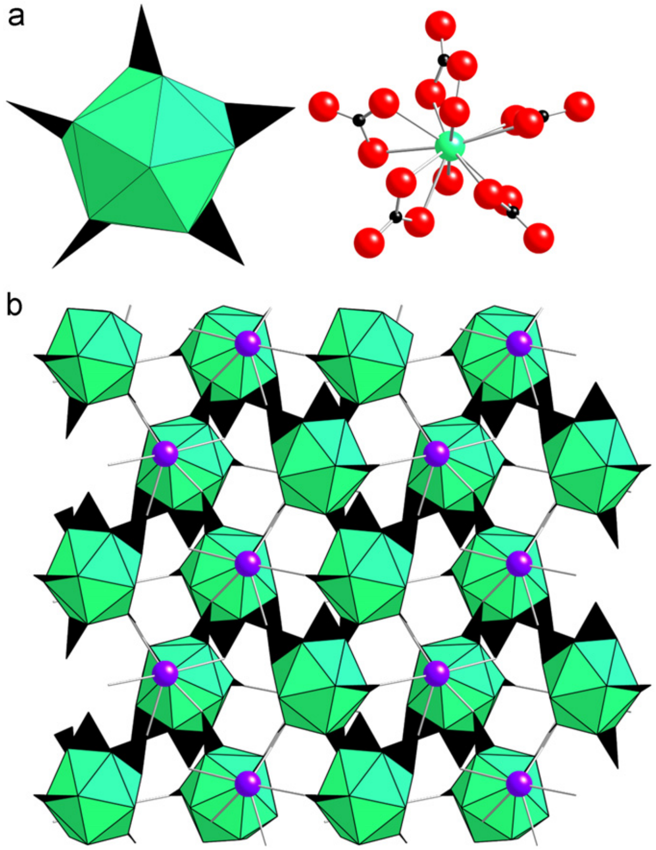

Fig. 3. Polyhedral and ball-and-stick representations of the structure of compound **4**. (a) The cluster of Th and nitrate triangles that occurs in **4**. (b) Projection of the structure of **4** along [100] showing the packing of the clusters of polyhedra. Th polyhedra: green; Nitrate triangles: black; K atoms: blue spheres. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

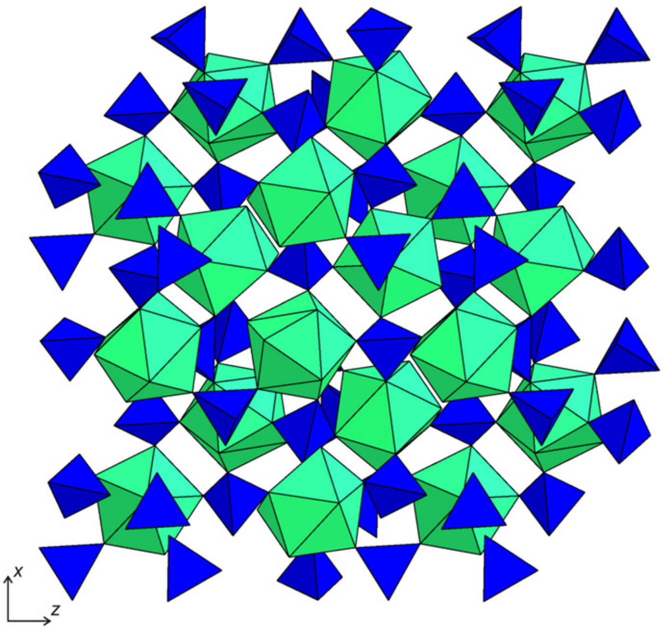

Fig.4. Polyhedral representation of the framework structure of compound **5** projected along [010]. Th polyhedra: green; Chromate tetrahedra: blue. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

polyhedron. Compound **4** is isostructural with several light rare earth nitrates [21]. La, Ce, Pr, and Nd also form potassium pentanitrate dihydrate compounds, but these differ from **4** in that they contain trivalent metals. Compound **4** presents the first example of a Th pentanitrate compound with two waters in the Th polyhedron.

The structure of **5** is a three-dimensional framework of Th polyhedra and (CrO_4) tetrahedra (Fig. 4). There are two

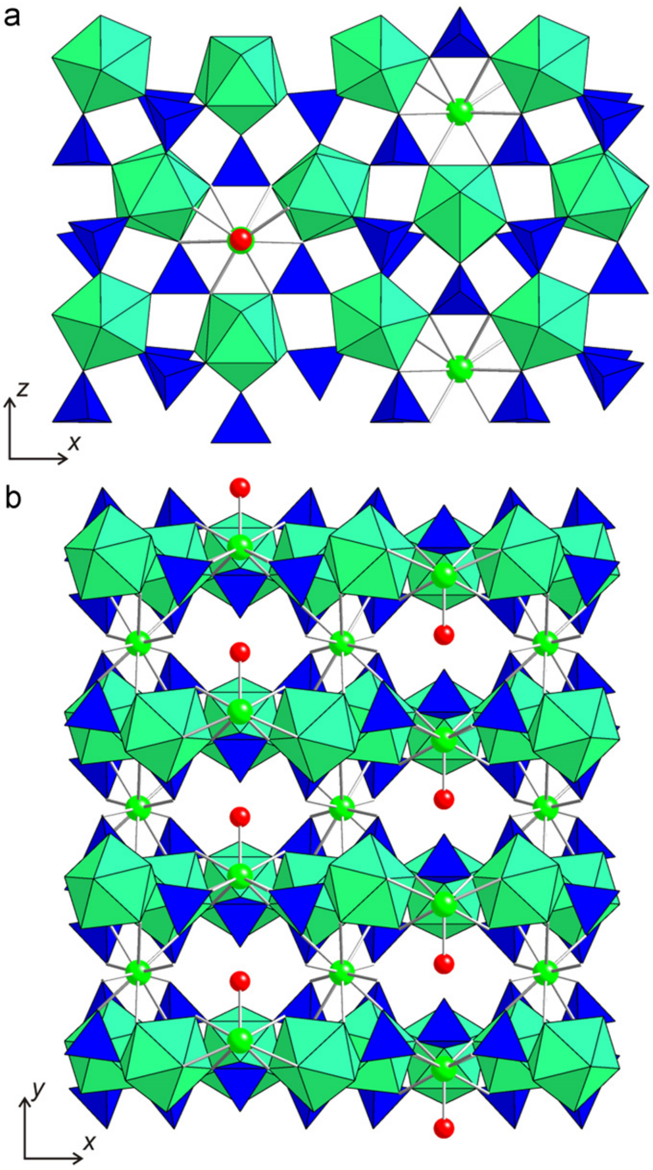

Fig. 5. Polyhedral representation of the sheet structure of compound **6**. (a) The sheet of Th and Cr polyhedra. (b) Projection of the extended structure along [001]. Th polyhedra: green; Chromate tetrahedra: blue; K atoms: green spheres; O atoms: red spheres. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

crystallographically distinct Th atoms, each of which are present as (ThO_9) tricapped trigonal prisms. Each Th center is bound to seven monodentate (CrO_4) tetrahedra and two (H_2O) groups. There are four crystallographically distinct (CrO_4) tetrahedra, all of which share verticies with Th polyhedra. The Cr(1) and Cr(2) tetrahedra bridge between four Th polyhedra and Cr(3) and Cr(4) bridge between three Th polyhedra. The Th–O bond distances range from 2.344(9) to 2.608(10) Å. Chromate bond distances range from 1.627(9) to 1.683(9) Å. The compound $Th(OH)_2(CrO_4)(H_2O)$ [17] contains one (ThO_8) polyhedron and one (ThO_9) polyhedron, but otherwise is similar to **5** in that it consists of a framework of Th polyhedra and (CrO_4) tetrahedra.

The structure of **6** contains sheets of Th polyhedra and (CrO_4) tetrahedra (Fig. 5). The Th cations occur as ThO_9 tricapped trigonal prisms consisting of seven monodentate (CrO_4) tetrahedra and two (H_2O) groups. There are two crystallographically distinct Th atoms and four independent Cr atom locations. Each (CrO_4) tetrahedron is linked to three Th polyhedra by vertex sharing. The sheet is one Th-polyhedron wide and contains channels defined by three Th polyhedra and three (CrO_4) tetrahedra. The K1 cation is located in the resulting six-sided cavity within the sheet. K2 cations and (H_2O) molecules are located in the interlayer and provide linkages between the sheets. Th–O distances range from 2.362(6) to 2.663(5) Å. Cr–O distances range from 1.552(13) to 1.688(5) Å.

The structures of the compounds reported herein demonstrate the complexity that arises in Th crystal chemistry that is largely due to the variability of the Th coordination polyhedra. These compounds exhibit Th that is coordinated by nine to 12 O atoms, and the configurations of the oxyanions coordinating Th can differ, presumably owing to steric constraints of the longer-range structure.

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Appendix A. Supplementary Material

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jssc.2010.04.042.

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