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Syntheses and crystal structures of two novel alkaline uranyl chromates $A_2(UO_2)(CrO_4)_2$ (A=Rb, Cs) with bidentate coordination mode of uranyl ions by chromate anions

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ABSTRACT

Single crystals of $Cs_2(UO_2)(CrO_4)_2$ and $Rb_2(UO_2)(CrO_4)_2$ were prepared by solid state reactions. The structures are based upon the $[(UO_2)(CrO_4)_2]^{2-}$ chains. Within the chains, UrO_5 pentagonal bipyramids (Ur=uranyl) form Ur_2O_8 dimers, which are linked via CrO_4 tetrahedra into one-dimensional chains. The CrO_4 tetrahedra coordinate uranyl ions in both mono- and bidentate fashion, which is unusual for uranyl chromates. The bidentate coordination has a strong influence upon geometrical parameters of both U and Cr coordination polyhedra. The conformation of the chains in **1** and **2** is different due to the different size of the Cs^+ and Rb^+ cations.

1. Introduction

A large amount of structural and chemical data has been accummulated recently on uranium minerals and compounds due to their high importance for nuclear waste management and understanding secondary processes of alteration of spent nuclear fuel (SNF) [1–3]. Uranyl chromates are of special interest, since potassium chromate solution is used as both coolant and corrosion inhibitor for some SNF rod arrays made from Mg–Be alloys [4,5]. In addition, uranyl chromates are less studied than other uranyl oxosalts, due to the tendency of Cr^{6+} to reduction in high-temperature hydrothermal environment [6]. Most of known uranyl chromates are hydrous [7–15], with only few anhydrous compounds reported to date [16–19]. However, general trends in the structural chemistry of known uranyl chromates are the same as for other uranyl oxysalts containing tetrahedral anions formed by hexavalent elements (Mo, S, Se) [20]. In their structures, uranyl ions form UO_7 pentagonal bipyramids that share common vertices with CrO_4 tetrahedral oxoanions in a monodentate fashion to form extended polymerized structures. It should be noted that uranyl sulphates are a bit out of tune with this principle. In contrast to uranyl selenates and molybdates, where only monodentate coordination mode is observed, there are many uranyl sulfates with bidentate coordination of uranyl cations by sulfate anions [21–28]. This type of coordination has also been reported for uranyl sulfate solutions [29–31]. The first structure of uranyl chromate with a bidentate coordination mode had recently been reported by our group [32]. Here we report on the syntheses and structures of the new compounds $Cs_2(UO_2)(CrO_4)_2$ (**1**) and $Rb_2(UO_2)(CrO_4)_2$ (**2**), low-temperature phases in the ANO_3 (A=Cs, Rb)–CrO_3–$(UO_2)(NO_3)_2$ system that have been obtained by the solid-state reaction method in the frame of our ongoing research of the crystal chemistry in this system.

2. Experimental

2.1. Synthesis

Single crystals of **1** were grown by mixing $CsNO_3$ (Vekton, 99.5%), CrO_3 (Vekton, 98%) and $(UO_2)(NO_3)_2 \cdot 6H_2O$ (Vekton, 99.7%) in the ratio of 2:3:1. The produced mixture was loaded into a platinum crucible and kept at 270 °C for 10 h in air, followed by cooling to 100 °C with a cooling rate of 3 °C/h and then cooling down to room temperature with a rate of 7 °C/h. The product consisted of yellow–green isometric crystals of **1** in the mass of amorphous Cr_2O_3. Orange–green transparent crystals of **2** were produced by the same procedure, when $RbNO_3$ was used instead of $CsNO_3$. The electron–microprobe analyses (LINK AN-10000 EDS system) were performed. The averaged of seven points (for each compound) gave the empirical formulas calculated on the basis of

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10 total oxygen atoms: $Cs_{2.01}(U_{0.98}O_2)(Cr_{0.96}O_4)_2$ and $Rb_{1.98}(U_{0.97}O_2)(Cr_{0.98}O_4)_2$.

2.2. Single crystal X-ray study

Yellow–green crystals of **1** and **2** were mounted on a thin glass fibers for X-ray diffraction analysis. More than a hemisphere of X-ray diffraction data with frame widths of 0.3° in ω, and with 30 s spent counting for each frame were collected at room temperature using a Bruker three-circle Smart *APEX* II X-ray diffractometer operated with MoK_α radiation at 50 kV and 40 mA. The data were integrated and corrected for absorption using an empirical ellipsoidal model using the Bruker programs *APEX* and *XPREP*. The observed systematic absences were consistent with space group $P-1$ in both compounds. The structures were solved by direct methods and refined to $R_1=0.023$ (**1**) and $R_1=0.030$ on the basis of F^2 for all unique data. The *SHELX* program package was used for all structural calculations. Technical details of the data acquisition as well as some refinement results for the title compounds are summarized in Table 1. The atomic coordinates and displacement parameters are given in Tables 2 and 3, and selected bond lengths in Table 4. Further details of the crystal structure investigation are available from the Fachinformationszentrum Karlsruhe, D-76344 Eggenstein-Leopoldshafen, Germany, on quoting the depository numbers CSD-424125 (for $Rb_2(UO_2)(CrO_4)_2$) or CSD-424126 (for $Cs_2(UO_2)(CrO_4)_2$), the names of the authors and the citation of the paper.

Table 1
Crystallographic data and refinement parameters for **1** and **2**.

	1	2
Crystal size (mm^3)	0.11 × 0.09 × 0.08	0.14 × 0.10 × 0.09
Space group	$P-1$	
a (Å)	7.829(6)	8.052(5)
b (Å)	8.588(6)	10.362(6)
c (Å)	9.796(7)	13.707(8)
α (°)	74.73(1)	102.93(1)
β (°)	71.58(1)	106.89(1)
γ (°)	74.18(1)	94.54(1)
V (Å^3)	589.8(7)	1053.8(11)
μ (mm^{-1})	21.617	26.566
D_{calc} (g/cm^3)	4.324	4.242
Radiation wavelength (Å)	0.71073 (MoK_α)	
θ-range (°)	2.23–27.99	1.61–28.00
Total ref.	6773	12,213
Unique ref.	2832	5,095
Unique $\lvert Fo \rvert \geq 4\sigma_F$	2526	4,109
R_{int}	0.034	0.064
R_1	0.023	0.030
R_1 (all data)	0.027	0.040
GoF	0.962	0.944
$\rho_{max,min}$ (e·Å^{-3})	+1.257/−1.031	+1.981/−2.387

3. Results

The structure of **1** contains one symmetrically independent U^{6+} cation, two Cr^{6+} and two Cs^+ cations. U atom is strongly bonded to two O atoms and further coordinated by five atoms of O arranged at the equatorial vertices of UrO_5 (Ur=uranyl) pentagonal bipyramids. Average Ur–O_{eq} (O_{eq}=equatorial O atom) bond lengths are in the range from 2.246 to 2.449 Å. Each of the two Cr atoms is tetrahedrally coordinated by four O atoms. CrO_4 tetrahedra are distorted with the Cr–O bond lengths varying from 1.593 to 1.726 Å. Cs atoms are coordinated by eight O atoms each. In comparison to **1**, the structure of **2**[10] has twice more cations sites, which essentially follow coordination features observed in the structure of **1**. The Ur–O_{eq} bonds are in the range of 2.253–2.439 Å and Cr–O bonds vary from 1.574 to 1.709 Å. The structures of **1** and **2** are based upon one-dimensional uranyl chromate chains with the composition $[(UO_2)(CrO_4)_2]^{2-}$ (Figs. 1, 2). Within the chain, two adjacent UrO_5 pentagonal bipyramids share two equatorial O atoms to form Ur_2O_8 dimers. Each bipyramid is also coordinated by a CrO_4 tetrahedron in a bidentate fashion. The resulting $[Ur_2O_4(CrO_4)_2]$ complexes are further linked through additional CrO_4 tetrahedra (that coordinate uranyl cations in a monodentate fashion) into the $[(UO_2)(CrO_4)_2]^{2-}$ chains extended along [0 0 1] in **1** and along [1–11] in **2**. The alkali metal cations are located in between the chains providing three-dimensional integrity of the structures.

Both bidentate and monodentate coordination of uranyl cations by CrO_4 tetrahedral groups results in significant distortions of both U and Cr coordination polyhedra. In bidentate complexes, the U–Cr distances are in the range of 3.158–3.196 Å, which is in agreement with the results previously reported [33]. In order to describe structure distortions in **1** and **2**, the O atoms in the CrO_4 tetrahedra can be classified into three groups: (i) atoms involved in bidentate coordination (bidentate bridging, O_{bb}); (ii) atoms involved in monodentate coordination (monodentate bridging, O_{mb}); (iii) terminal atoms not bonded to U (terminal, O_t). Geometrical distortions induced by bidentate coordination of uranyl ion by chromate include the following particular effects: (a) the U–O_{bb} bonds are essentially longer (2.446–2.449 Å in **1** and 2.408–2.439 Å in **2**) than the average

Table 2
Atomic coordinates and displacement parameters (Å^2) for **1**.

Atom	x	y	z	U_{eq}	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
U(1)	0.46072(3)	0.57422(2)	0.19002(1)	0.01701(7)	0.0202(1)	0.0170(1)	0.0140(1)	−0.00352(7)	−0.00615(7)	−0.00178(7)
Cr(1)	0.27004(12)	0.7367(1)	0.54166(9)	0.0227(1)	0.0288(5)	0.0195(4)	0.0163(4)	−0.0039(3)	−0.0081(3)	0.0034(4)
Cr(2)	0.75688(12)	0.1792(1)	0.03551(9)	0.0239(1)	0.0259(5)	0.0206(4)	0.0240(5)	−0.0060(3)	−0.0128(4)	0.0061(4)
Cs(1)	0.23951(5)	0.12992(4)	0.22698(4)	0.03117(10)	0.0353(2)	0.0268(1)	0.0312(2)	−0.0056(1)	−0.0065(1)	−0.0088(1)
Cs(2)	0.15631(5)	0.27676(5)	0.66260(4)	0.03685(11)	0.0341(2)	0.0348(2)	0.0449(2)	−0.0040(1)	−0.0193(1)	−0.0053(1)
O(1)	0.5973(5)	0.3616(4)	0.0519(4)	0.0281(9)	0.035(2)	0.0231(19)	0.024(2)	−0.010(1)	−0.015(1)	0.011(1)
O(2)	0.6516(5)	0.6708(5)	0.1001(4)	0.0338(9)	0.031(2)	0.045(3)	0.030(2)	−0.009(1)	−0.002(1)	−0.017(1)
O(3)	0.2296(5)	0.8228(5)	0.1376(4)	0.0312(9)	0.039(2)	0.029(2)	0.025(2)	−0.014(1)	−0.012(1)	0.005(1)
O(4)	0.4022(6)	0.7286(5)	0.3695(4)	0.0320(9)	0.044(3)	0.036(2)	0.020(2)	−0.010(1)	−0.005(1)	−0.013(1)
O(5)	0.2661(5)	0.4787(5)	0.2793(4)	0.0354(10)	0.034(2)	0.030(2)	0.041(2)	−0.010(1)	0.001(1)	−0.016(1)
O(6)	0.9500(6)	0.1848(6)	0.0539(5)	0.0523(13)	0.031(3)	0.066(3)	0.070(3)	−0.032(3)	−0.027(2)	0.008(2)
O(7)	0.6749(7)	0.0264(6)	0.1467(5)	0.0524(13)	0.055(3)	0.033(3)	0.055(3)	0.002(2)	−0.008(2)	−0.002(2)
O(8)	0.0849(6)	0.6705(7)	0.5651(6)	0.0595(14)	0.032(3)	0.076(4)	0.073(4)	−0.035(3)	0.002(2)	−0.017(3)
O(9)	0.2097(7)	0.9235(5)	0.5645(5)	0.0503(13)	0.080(4)	0.027(2)	0.037(3)	−0.0138(19)	−0.013(2)	0.003(2)
O(10)	0.6170(7)	0.3858(6)	0.3387(5)	0.0669(18)	0.092(4)	0.064(4)	0.032(3)	−0.018(2)	−0.041(3)	0.042(3)

Table 3
Atomic coordinates and displacement parameters (Å^2) for **2**.

Atom	*x*	*y*	*z*	U_{eq}	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
U(1)	0.82666(3)	0.10352(2)	0.42149(2)	0.01710(8)	0.0217(1)	0.0161(1)	0.0158(1)	0.0058(1)	0.0072(1)	0.00704(9)
U(2)	0.60806(3)	0.34896(2)	0.05462(2)	0.01648(8)	0.0203(1)	0.0172(1)	0.0174(1)	0.00867(11)	0.01029(10)	0.00689(9)
Rb(1)	0.05921(9)	0.49790(8)	0.33328(6)	0.03412(18)	0.0339(4)	0.0363(5)	0.0310(5)	0.0074(3)	0.0107(3)	0.0010(3)
Rb(2)	0.62847(9)	−0.2368(1)	0.50885(7)	0.03407(19)	0.0288(3)	0.0468(5)	0.0370(5)	0.0216(4)	0.0174(3)	0.0079(3)
Rb(3)	0.94788(9)	0.69142(8)	0.02597(7)	0.03469(19)	0.0311(4)	0.0314(4)	0.0470(5)	0.0107(4)	0.0182(3)	0.0125(3)
Rb(4)	0.37327(11)	0.04093(8)	0.14425(7)	0.0383(2)	0.0586(5)	0.0248(4)	0.0303(5)	0.0040(3)	0.0174(4)	−0.0041(3)
Cr(1)	0.88489(15)	0.1016(1)	0.15670(9)	0.0223(2)	0.0326(6)	0.0180(6)	0.0203(6)	0.0072(5)	0.0113(5)	0.0095(5)
Cr(2)	0.60614(14)	0.3832(1)	0.34371(9)	0.0188(2)	0.0252(5)	0.0195(6)	0.0175(6)	0.0088(5)	0.0112(5)	0.0086(4)
Cr(3)	0.60809(15)	0.3632(1)	−0.1731(1)	0.0217(2)	0.0344(6)	0.0205(6)	0.0159(6)	0.0072(5)	0.0129(5)	0.0119(5)
Cr(4)	0.87566(16)	0.1447(1)	0.6646(1)	0.0286(3)	0.0409(7)	0.0355(7)	0.0182(7)	0.0098(6)	0.0156(5)	0.0247(6)
O(1)	0.7542(6)	0.2159(5)	0.5758(4)	0.0274(11)	0.040(3)	0.030(3)	0.020(3)	0.008(2)	0.016(2)	0.022(2)
O(2)	0.6568(7)	0.2441(5)	−0.1081(4)	0.0283(12)	0.049(3)	0.020(3)	0.021(3)	0.006(2)	0.015(2)	0.017(2)
O(3)	0.6090(6)	0.2284(5)	0.3625(4)	0.0252(11)	0.026(2)	0.026(3)	0.030(3)	0.015(2)	0.010(2)	0.011(2)
O(4)	0.8234(6)	0.4446(5)	0.0934(4)	0.0297(12)	0.024(2)	0.030(3)	0.028(3)	0.003(2)	0.003(2)	−0.001(2)
O(5)	0.5238(7)	0.4610(5)	−0.0870(4)	0.0287(12)	0.046(3)	0.034(3)	0.022(3)	0.015(2)	0.023(2)	0.026(2)
O(6)	0.7220(7)	0.1577(5)	0.0745(4)	0.0356(13)	0.059(4)	0.028(3)	0.021(3)	0.007(2)	0.012(3)	0.019(3)
O(7)	0.9967(6)	0.2413(5)	0.4508(4)	0.0291(12)	0.026(2)	0.025(3)	0.035(3)	0.006(2)	0.010(2)	0.000(2)
O(8)	0.8063(7)	0.0578(5)	0.2478(4)	0.0313(12)	0.043(3)	0.039(3)	0.017(3)	0.010(2)	0.015(2)	0.015(2)
O(9)	0.3961(7)	0.2489(6)	0.0135(5)	0.0407(15)	0.028(3)	0.039(4)	0.057(4)	0.017(3)	0.014(3)	0.001(2)
O(10)	0.9679(7)	0.0450(5)	0.5852(4)	0.0337(13)	0.053(3)	0.039(3)	0.015(3)	0.006(2)	0.013(2)	0.034(3)
O(11)	0.6554(7)	−0.0299(5)	0.3948(4)	0.0331(13)	0.050(3)	0.016(3)	0.031(3)	0.005(2)	0.013(3)	−0.003(2)
O(12)	0.9366(7)	−0.0236(5)	0.0892(5)	0.0357(13)	0.052(3)	0.026(3)	0.040(4)	0.009(3)	0.029(3)	0.017(2)
O(13)	0.4253(7)	0.4275(6)	0.3522(5)	0.0437(16)	0.035(3)	0.044(4)	0.072(5)	0.031(3)	0.031(3)	0.022(3)
O(14)	0.6322(9)	0.3782(6)	0.2263(5)	0.0511(17)	0.103(5)	0.046(4)	0.029(4)	0.022(3)	0.043(4)	0.037(4)
O(15)	0.7664(8)	0.4856(6)	0.4342(5)	0.0482(16)	0.053(4)	0.032(4)	0.043(4)	−0.002(3)	0.002(3)	−0.003(3)
O(16)	0.7851(7)	0.4441(6)	−0.1741(5)	0.0464(16)	0.044(3)	0.062(4)	0.059(5)	0.040(4)	0.034(3)	0.022(3)
O(17)	0.4714(9)	0.3105(6)	−0.2872(5)	0.0559(18)	0.091(5)	0.039(4)	0.020(4)	0.001(3)	−0.005(3)	0.005(3)
O(18)	0.0517(8)	0.2158(6)	0.2103(6)	0.0537(18)	0.050(4)	0.040(4)	0.064(5)	0.002(3)	0.020(3)	−0.010(3)
O(19)	0.0171(9)	0.2536(7)	0.7566(5)	0.060(2)	0.080(5)	0.054(4)	0.024(4)	−0.008(3)	−0.008(3)	0.029(4)
O(20)	0.7611(9)	0.0581(8)	0.7109(6)	0.069(2)	0.076(5)	0.102(6)	0.079(6)	0.069(5)	0.057(4)	0.050(4)

Table 4
Selected bond lengths in the structures of **1** and **2**.

1				**2**			
U(1)–O(2)	1.776(4)	Cr(1)–O(9)		U(1)–O(11)	1.768(5)	Cr(1)–O(12)	1.577(5)
U(1)–O(5)	1.795(4)	Cr(1)–O(8)		U(1)–O(7)	1.785(5)	Cr(1)–O(18)	1.590(6)
U(1)–O(10)	2.246(4)	Cr(1)–O(10)		U(1)–O(8)	2.275(5)	Cr(1)–O(8)	1.684(5)
U(1)–O(4)	2.334(4)	Cr(1)–O(4)		U(1)–O(3)	2.325(5)	Cr(1)–O(6)	1.697(5)
U(1)–O(1)	2.373(4)			U(1)–O(10)	2.355(5)		
U(1)–O(1)	2.446(4)	Cr(1)–O(6)		U(1)–O(1)	2.428(5)	Cr(2)–O(13)	1.589(5)
U(1)–O(3)	2.449(4)	Cr(1)–O(7)		U(1)–O(10)	2.436(5)	Cr(2)–O(15)	1.610(6)
		Cr(1)–O(3)				Cr(2)–O(14)	1.672(6)
		Cr(1)–O(1)		U(2)–O(9)	1.790(5)	Cr(2)–O(3)	1.681(5)
				U(2)–O(4)	1.797(5)		
				U(2)–O(14)	2.253(6)	Cr(3)–O(17)	1.574(6)
				U(2)–O(6)	2.286(5)	Cr(3)–O(16)	1.600(5)
				U(2)–O(5)	2.327(5)	Cr(3)–O(2)	1.685(5)
				U(2)–O(2)	2.408(5)	Cr(3)–O(5)	1.709(5)
				U(2)–O(5)	2.439(5)		
						Cr(4)–O(19)	1.575(7)
						Cr(4)–O(20)	1.589(6)
						Cr(4)–O(1)	1.675(5)
						Cr(4)–O(10)	1.701(5)

'standard' U–O bond in a UO_7 pentagonal bipyramid [33] (2.37 Å); (b) the O_{bb}–U–O_{bb} valence angles are smaller (65.2° in **1** and 63.0–63.6° in **2**) than the expected 'ideal' value of 72°; (c) the Cr–O_{bb} bond lengths are longer (1.670–1.726 Å in **1** and 1.675–1.709 Å in **2**) than the average value of 1.647 Å observed for uranyl chromates [34]; (d) the O_{bb}–Cr–O_{bb} bond valence angles shrink (98.1° in **1** and 97.9–98.8° in **2**) with respect to the ideal tetrahedron value of 109.5°. For chromate tetrahedra involved in a monodentate bridging, the Cr–O_{mb} bonds (1.661–1.684 Å in **1** and 1.672–1.697 Å in **2** are essentially longer than the Cr–O_t bonds (1.598–1.627 Å in **1** and 1.577–1.610 Å in **2**).

Though topologically similar, uranyl chromate chains in the structures of **1** and **2** possess different conformations (Fig. 3), which is most probably the result of different size of the Cs^+ and Rb^+ ions. A similar situation has been observed for uranyl molybdate chains in the structures of $A_2[(UO_2)O(MoO_4)_2]$ (A=Na, K, Rb) [35,36]. Conformation in the uranyl chromate chains observed in **1** and **2** results from the possibility of CrO_4 tetrahedra within the chain to rotate around the O_{bb}–O_{bb} or O_{mb}–O_{mb} edges, respectively, without changing the overall topology of the chains. Fig. 3(a) and (b) show black-and-white connectivity graphs of the $[(UO_2)(CrO_4)_2]^{2-}$ chains with the U–Cr distances written above

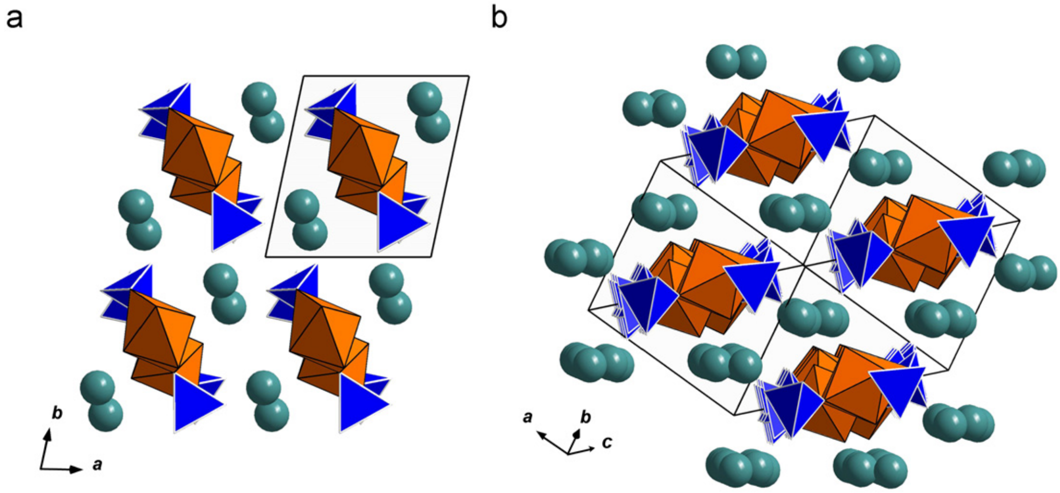

Fig. 1. General projections of the crystal structures of **1** (a) and **2** (b). (Legend: Cs, Rb=teal, UO_7=orange, CrO_4=blue). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

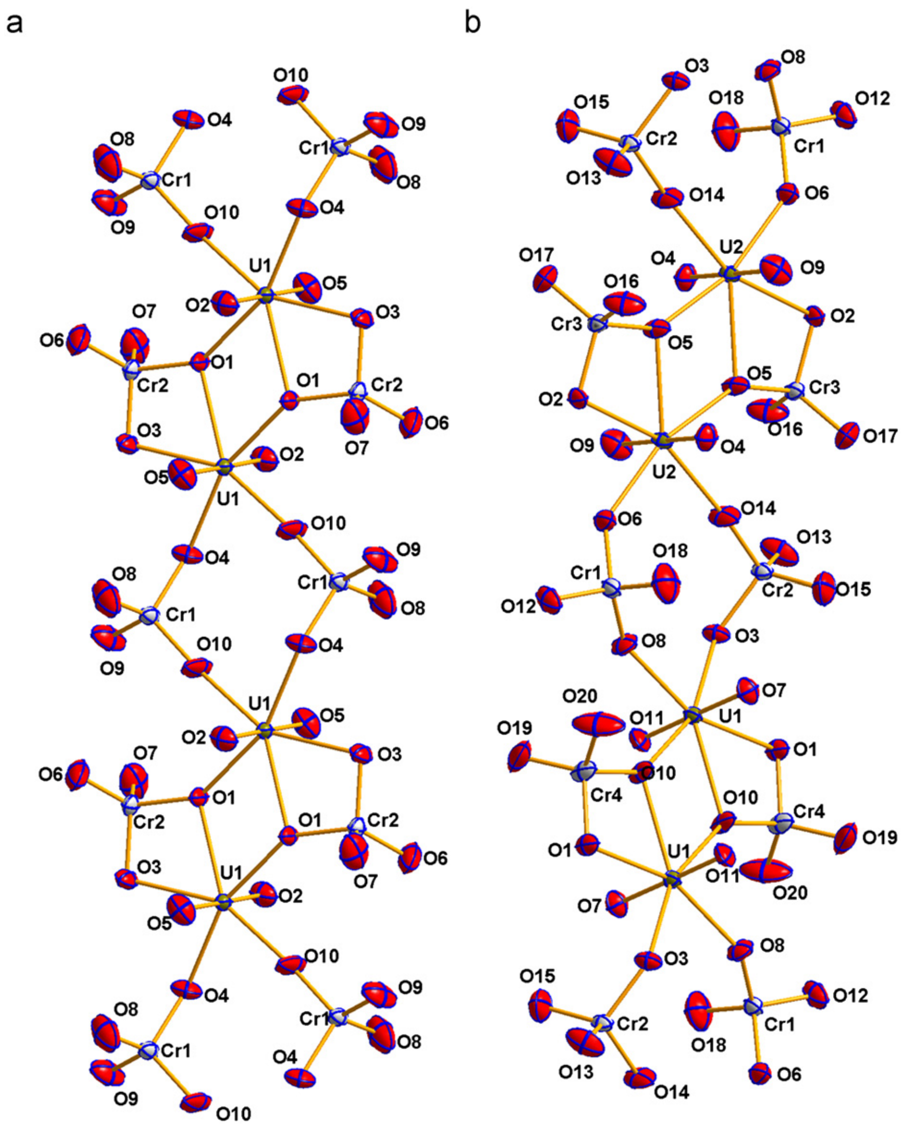

Fig. 2. ORTEP representation of the $[(UO_2)(CrO_4)_2]^{2-}$ chains in the crystal structures of **1**(a) and **2**(b). Ellipsoids are drawn at 50% probability.

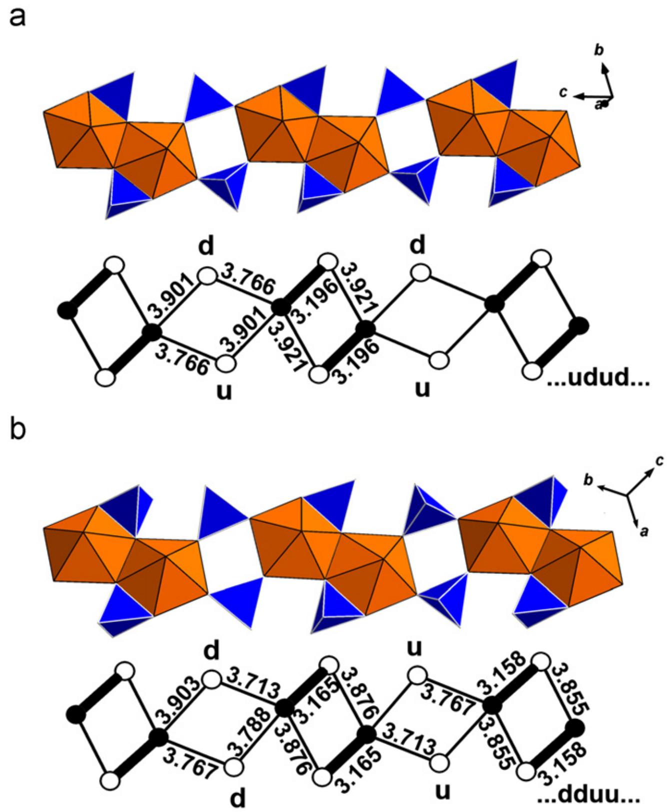

Fig. 3. Conformation of uranyl chromate chains in the structures of **1** and **2**: polyhedral representation of the chains (**1**(a), **2**(b)) and their description using graphs. The U–Cr distances are written near the corresponding interpolyhedral links. See text for details.

the edges linking adjacent U (black) and Cr (white) vertices. Note that single and bold edges correspond to mono- and bidentate linkage modes, respectively. The **u** and **d** symbols near the white vertices with monodentate linkages only indicate orientation of the chromate tetrahedra relative to the plane of the chain. Thus the conformation types of uranyl chromate chains in **1** and **2** can be described as (**u**)(**d**) and as (**ud**)(**ud**), respectively. The chain conformation in **2** is more complex than that in **1**, which results in the observed doubling of the number of symmetrically independent atoms in the structure. It is of interest that the chains topologically identical to those observed in **1** and **2** have been reported in the structure of $Cs_3[NpO_2(SO_4)_2](H_2O)_2$ [37].

In conclusion, we reported on the syntheses and structures of two first uranyl chromates containing chains with bidentate coordination mode of uranyl cations by CrO_4 tetrahedra.

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