Crystal Chemistry of Uranyl Halides Containing Mixed $(UO_2)(X_mO_n)_5$ Bipyramids (X = Cl, Br): Synthesis and Crystal Structure of $Cs_2(UO_2)(NO_3)Cl_3$

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Single crystals of $Cs_2(UO_2)(NO_3)Cl_3$ were prepared by a hydrothermal method at 205 °C. The crystal structure has been solved by Direct Methods: monoclinic, $P2_1/n$, a = 10.3748(13), b = 9.4683(13), c = 12.5535(16) Å, β = 110.280(2)°, V = 1156.7(3) Å^3, R_1 = 0.029. In the structure, strongly bonded linear uranyl cations UO_2^{2+} are equatorially coordinated by two O and three Cl atoms to form $(UO_2)Cl_3O_2$ pentagonal bipyramids. Each bipyramid shares its O–O edge with an adjacent $(NO_3)^-$ anion to form finite clusters with the chemical composition $[(UO_2)(NO_3)Cl_3]^{2-}$. The Cs^+ cations provide three-dimensional connectivity of the structure by forming Cs–O and Cs–Cl contacts to the uranyl nitrate chloride complexes. Related structures of mixed-ligand uranyl halides are compared.

Key words: Uranyl, Chloride, Crystal Structure, Crystal Chemistry, Nitrate

Introduction

Within the past decade great attention was paid to the crystal chemistry of inorganic uranyl oxo-compounds due to their environmental and technological importance [1], whereas inorganic uranyl halides received relatively little focus, despite their interest from the viewpoint of nuclear technology and radioactive waste management [2]. An understanding of the chemistry and structures of alteration of uranyl phases is important for predicting the long-term behavior of radioactive wastes in a geological repository, as some of the alteration products may incorporate radionuclides into their structures. Herein we report on the synthesis and crystal structure of the new cesium uranyl nitrate chloride that is based upon finite $[(UO_2)Cl_3(NO_3)]^{2-}$ units and compare the results with related structures of uranyl oxohalides.

Experimental Section

Synthesis

Single crystals of $Cs_2(UO_2)(NO_3)Cl_3$ were prepared by a hydrothermal method from a mixture of CsCl (0.674 g) and $UO_2(NO_3)_2 \cdot 6H_2O$ (0.450 g) with 10 mL of H_2O. The pH of the mixture was adjusted to 10 by addition of NaOH. The reactants were placed in a 23 mL teflon-lined Parr reaction vessel and heated to 205 °C for 96 h. The products were isolated

Table 1. Crystal structure data for $Cs_2(UO_2)(NO_3)Cl_3$.

Formula	$Cl_3Cs_2NO_5U$
M_r	704.21
Crystal size, mm^3	$0.10 \times 0.08 \times 0.11$
Space group	$P2_1/n$
a, Å	10.3748(13)
b, Å	9.4683(13)
c, Å	12.5535(16)
β, deg	110.280(2)
V, Å^3	1156.7(3)
Z	4
D_{calcd}, g cm^{-3}	4.04
μ (MoK_α), cm^{-1}	20.9
F(000), e	1199.8
θ range, deg	2.20 – 36.20
h,k,l ranges	$-15 \rightarrow 17$, $-15 \rightarrow 15$, $-20 \rightarrow 19$
Total / independ. refl. / R_{int}	20673 / 5314 / 0.0658
Reflections with $I \geq 2\sigma(I)$ / R_σ	3591 / 0.0652
Final indices R_1 / wR_2[a]	0.0287 / 0.0439
Indices R_1 / wR_2 (all data)[a]	0.0541 / 0.0480
Weighting coefficients a / b[b]	0.0056 / 0.00
Goodness-of-fit on F^2 [c]	1.083
Extinction coefficient	0.00029(4)
Largest diff. peak / hole, e Å^{-3}	1.17 / −1.38

[a] $R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$, $wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{1/2}$;
[b] $w = [\sigma^2(F_o^2) + (aP)^2 + bP]^{-1}$, where $P = (\text{Max}(F_o^2, 0) + 2F_c^2)/3$;
[c] $\text{GoF} = [\Sigma w(F_o^2 - F_c^2)^2/(n_{\text{obs}} - n_{\text{param}})]^{1/2}$.

Atom	Wyckoff site	x	y	z	U_{eq}
U(1)	4*e*	0.34041(1)	0.66185(2)	0.09198(1)	0.02196(5)
Cs(1)	4*e*	0.62394(3)	0.02075(3)	0.22259(2)	0.03233(7)
Cs(2)	4*e*	0.34263(3)	0.72862(3)	0.43962(2)	0.03825(8)
Cl(1)	4*e*	0.12280(11)	0.60180(14)	0.15262(10)	0.0386(3)
Cl(2)	4*e*	0.53013(11)	0.76698(13)	0.00807(9)	0.0346(2)
Cl(3)	4*e*	0.18342(13)	0.86717(14)	−0.03570(12)	0.0495(3)
O(1)	4*e*	0.4043(3)	0.7770(3)	0.2085(2)	0.0313(7)
O(2)	4*e*	0.2773(3)	0.5403(3)	−0.0191(2)	0.0349(7)
O(3)	4*e*	0.5594(3)	0.5280(3)	0.1725(3)	0.0326(7)
O(4)	4*e*	0.5976(3)	0.3492(3)	0.2881(3)	0.0416(8)
O(5)	4*e*	0.3984(3)	0.4532(4)	0.2277(3)	0.0396(8)
N(1)	4*e*	0.5206(4)	0.4400(4)	0.2320(3)	0.0295(8)

Table 2. Atomic coordinates and isotropic equivalent displacement parameters U_{eq} (Å^2) for $Cs_2(UO_2)(NO_3)Cl_3$.

Table 3. Selected bond lengths (Å) for $Cs_2(UO_2)(NO_3)Cl_3$ with estimated standard deviations in parentheses.

U(1)–O(2)	1.751(3)	Cs(2)–O(1)	3.213(3)
U(1)–O(1)	1.760(3)	Cs(2)–O(4)	3.338(3)
U(1)–O(3)	2.489(3)	Cs(2)–O(5)	3.398(3)
U(1)–O(5)	2.541(3)	Cs(2)–O(2)	3.480(3)
U(1)–Cl(1)	2.6817(11)	Cs(2)–Cl(3)	3.558(5)
U(1)–Cl(3)	2.6817(12)	Cs(2)–Cl(1)	3.576(7)
U(1)–Cl(2)	2.7225(11)	Cs(2)–Cl(2)	3.631(5)
		Cs(2)–Cl(3)	3.671(8)
Cs(1)–O(3)	3.088(3)		
Cs(1)–O(1)	3.205(3)	N(1)–O(3)	1.274(4)
Cs(1)–O(4)	3.253(3)	N(1)–O(4)	1.218(4)
Cs(1)–O(4)	3.357(3)	N(1)–O(5)	1.256(4)
Cs(1)–Cl(2)	3.4308(12)		
Cs(1)–Cl(2)	3.4865(12)		
Cs(1)–Cl(1)	3.5179(11)		

by filtration and washed with ultrapure water. The crystals of $Cs_2(UO_2)(NO_3)Cl_3$ occurred as lemon-yellow transparent needles. The residual solution had a yellowish tint, which indicates the presence of dissolved, non-reacted uranyl nitrate.

Qualitative electron microprobe analysis (LINK AN-10000 EDS system) indicated the absence of other elements with atomic number greater than 11 (Na), except U^{6+}, Cs^+ and Cl^-. Quantitative electron microprobe analysis was not performed taking into account the high volatility of Cl under an electron beam.

X-Ray structure determination

For the crystal structure analysis, a small single crystal of $Cs_2(UO_2)(NO_3)Cl_3$ was isolated by mechanical fragmentation and measured with a Bruker three-circle Smart APEX II X-ray diffractometer with graphite-monochromatized MoK_α radiation operated at 50 kV and 40 mA. More than a hemisphere of data was collected. According to the systematic extinctions, the space group $P2_1/n$ (no. 14) was derived. Structure solution and parameter refinement (full-matrix least-squares against F^2) was successfully performed in this group, using the SHELX-97 software suite [3] with anisotropic displacement parameters for all atoms. All relevant details of the data collection and evaluation are listed in Table 1. The final difference Fourier synthesis did not reveal any significant residual peaks. The positional parameters (Table 2) and the interatomic distances and angles (Table 3) are listed below. The anisotropic displacement parameters are available as Supporting Information (online only). See note at the end of the article for availability.

Further details of the crystal structure investigation may be obtained from Fachinformationszentrum Karlsruhe, 76344 Eggenstein-Leopoldshafen, Germany (fax: +49-7247-808-666; e-mail: crysdata@fiz-karlsruhe.de, http://www.fiz-informationsdienste.de/en/DB/icsd/depot_anforderung.html) on quoting the deposition number CSD-422362.

Results

The structure of $Cs_2(UO_2)(NO_3)Cl_3$ contains one independent U atom, which is bonded to two O atoms forming uranyl ions (*Ur*) with U–O_{Ur} bond lengths of 1.75 and 1.76 Å. The uranyl ion is coordinated by two O and three Cl atoms arranged at the equatorial vertices of a UrCl$_3$O$_2$ pentagonal bipyramid. Differences in size between chlorine and oxygen atoms contribute to the distortion of the polyhedron. The U–O_{eq} (*eq*: equatorial) bond lengths range from 2.49 to 2.59 Å, which is notably longer than the value of 2.37 Å obtained for "pure" oxygen uranyl pentagonal bipyramids [4]. This increment of the average bond length value is caused by the participation of O(3) and O(5) atoms in strongly bound NO_3 groups. The U–Cl_{eq} bond lengths vary from 2.68 to 2.72 Å. The structure contains two independent Cs sites each coordinated by eight anions to form Cs(1)Cl_3O_5 and Cs(2)Cl_4O_4 configurations. As it is typical for nitrates, the unique N site in the structure is in triangular-planar coordination, with an average N–O bond length of 1.25 Å.

The bond-valence sums for the atoms in the structure were calculated using parameters given by Burns *et al.* [5] for U^{6+}–O bonds and by Brese and

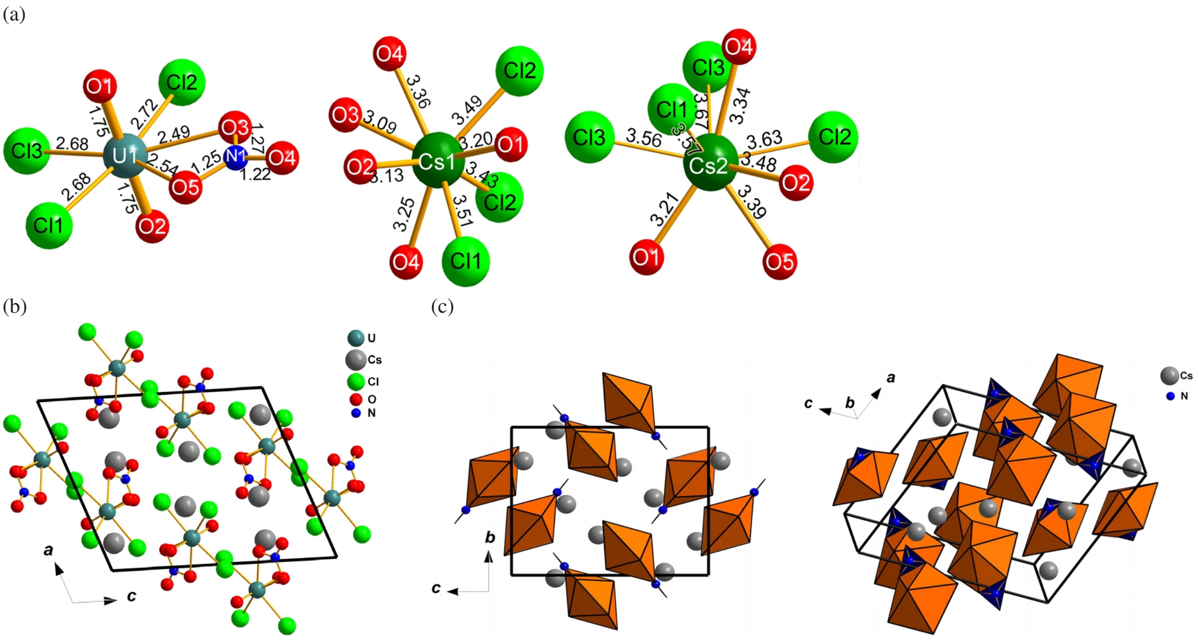

Fig. 1 (color online). (a) Coordination polyhedra of U^{6+} and Cs^{+} cations in the crystal structure of $Cs_2(UO_2)(NO_3)Cl_3$; (b) ball and stick representation of the crystal structure of $Cs_2(UO_2)(NO_3)Cl_3$; (c) general projections of the crystal structure of $Cs_2(UO_2)(NO_3)Cl_3$. $\mathit{Ur}O_2Cl_3$ polyhedra are drawn in orange.

Table 4. Bond-valence values $\Sigma_v{}^a$ for $Cs_2(UO_2)(NO_3)Cl_3$.

	Cs(1)	Cs(2)	U(1)	N(1)	$\Sigma_v a$
Cl(1)	0.14	0.12	0.55		0.81
Cl(2)	0.18 0.15	0.10	0.50		0.93
Cl(3)		0.13 0.10	0.55		0.78
O(1)	0.12	0.12	1.75		1.99
O(2)		0.06	1.78		1.84
O(3)	0.16		0.42	1.54	2.12
O(4)	0.11 0.08	0.09		1.77	2.05
O(5)		0.07	0.38	1.58	2.03
$\Sigma_v c$	0.94	0.79	5.93	4.90	

[a] Expressed in valence units (vu); $\Sigma_v a$ and $\Sigma_v c$ are the bond valence sums for anions and cations, respectively.

O'Keeffe [6] for U^{6+}–Cl, Cs^{+}–O, Cs^{+}–Cl and N^{5+}–O bonds. The results are summarized in Table 4. The bond-valence sums for all atoms are in agreement with their expected formal valencies.

The structure of $Cs_2(UO_2)(NO_3)Cl_3$ is based upon finite asymmetric units of composition $[(UO_2)(NO_3)Cl_3]^{2-}$ (see Fig. 2a). These units consist of $\mathit{Ur}Cl_3O_2$ pentagonal bipyramids with the two equatorial O atoms forming strong covalent bonds to the N atom, which results in sharing of one edge of the bipyramid with the NO_3 triangle (Fig. 1). Such units have never been observed before in purely inorganic compounds, but are known in the structures of $[C_2mim]_2[UO_2Cl_3(NO_3)]$ [7] and $[C_{14}H_{22}ON_2]_2[UO_2Cl_3(NO_3)]$ [8]. The structure of $Cs_2(UO_2)(NO_3)Cl_3$ as a whole consists of $[(UO_2)(NO_3)Cl_3]^{2-}$ units held together by cesium cations.

Discussion

The compound $Cs_2(UO_2)(NO_3)Cl_3$ reported herein complements the class of inorganic uranyl oxohalides containing mixed (oxygen-halogen) coordination in the equatorial planes of *Ur* cations. The $\mathit{Ur}X_2O_3$ (X = Cl, Br) isolated bipyramid with two chlorine anions in the equatorial plane (Fig. 2b) has previously been observed in $(UO_2)X_2(H_2O)_3$ (X = Cl, Br) [9, 10]. Tetragonal $\mathit{Ur}X_4$ (X = Cl, Br) bipyramids with the uranyl cation coordinated by four equatorial halide anions (Fig. 2c) have been found in the crystal structures of $A_2(UO_2)X_4$ (X = Cl, Br; A = Cs, Rb) [11–14].

Edge linking of uranyl bipyramids $\mathit{Ur}(X_mO_n)_5$ (X = Cl, Br) results in the formation of various condensed units as shown in Fig. 2. There are two types of dinuclear units (Figs. 2d, e), where bipyramids share either an O–O [as in the

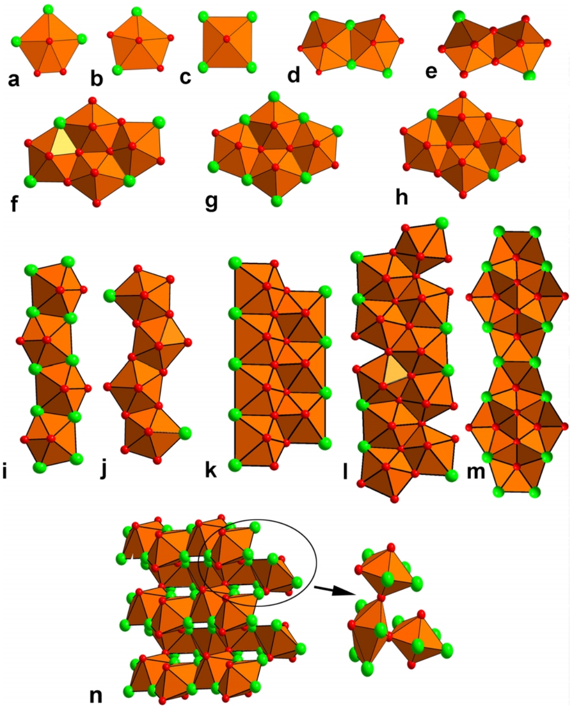

Fig. 2 (color online). Structural units based on $\mathit{Ur}X_mO_n$ (X = Cl, Br) bipyramids. $[\mathit{Ur}X_3O_2]^{5-}$, $[\mathit{Ur}X_2O_3]^{6-}$ and $[\mathit{Ur}X_4]^{2-}$ isolated bipyramids in $Cs_2(UO_2)(NO_3)Cl_3$ (a), $(UO_2)X_2(H_2O)_3$ (b) and $A_2(UO_2)X_4$ (A = Cs, Rb) (c); ${}^{0}_{\infty}[\mathit{Ur}_2X_4O_4]^{8-}$ and ${}^{0}_{\infty}[\mathit{Ur}_2X_2O_6]^{10-}$ dinuclear units in $(UO_2)(OH)Cl{\cdot}2H_2O$ (d) and $(UO_2)Br_2 \cdot 2H_2O$ (e); ${}^{0}_{\infty}[\mathit{Ur}_4X_4O_4]^{4-}$, ${}^{0}_{\infty}[\mathit{Ur}_4X_2O_{10}]^{14-}$ and ${}^{0}_{\infty}[\mathit{Ur}_4X_8O_4]^{8-}$ tetranuclear units in $K_2(UO_2)_4Cl_4O_2(OH)_2 \cdot 6H_2O$ (f), $(UO_2)_4O_2(OH)_2Cl_2(H_2O)_6 \cdot 4H_2O$ (g) and $Rb_4(UO_2)_4O_2Cl_8(H_2O)_2 \cdot 2H_2O$ (h); ${}^{1}_{\infty}[\mathit{Ur}Cl_2O]^{2-}$ and ${}^{1}_{\infty}[\mathit{Ur}_3ClO_8]^{11-}$ units in $(UO_2)Cl_2(H_2O)$ (i) and $A_7[(UO_2)_8(VO_4)_2]O_8Cl]$ (A = Rb, Cs) (j); ${}^{1}_{\infty}[\mathit{Ur}OCl]^{-}$ double chains in $Cs_{0.9}(UO_2)OCl_{0.9}$ (k); ${}^{1}_{\infty}[\mathit{Ur}_3Cl_2O_6]^{8-}$ and ${}^{1}_{\infty}[\mathit{Ur}_4Cl_6O_4]^{6-}$ complex chains in $Cs_2[(UO_2)_3Cl_2(IO_3)(OH)O_2] \cdot 2H_2O$ (l) and $Li(H_2O)_2[(UO_2)_2Cl_3OH_2O]$ (m); ${}^{3}_{\infty}[\mathit{Ur}Cl_2]$ framework in $(UO_2)Cl_2$ (n).

structure of $(UO_2)(OH)Cl{\cdot}2H_2O$ [15]] (Fig. 2d) or an X-X (X = Cl, Br) equatorial edge [as in $(UO_2)Br_2 \cdot 2H_2O$ [10]] (Fig. 2e). In the crystal structures of $(UO_2)_4O_2(OH)_2Cl_2(H_2O)_6 \cdot 4H_2O$ [16, 17], $Rb_4(UO_2)_4O_2Cl_8(H_2O)_2 \cdot 2H_2O$ and $K_2(UO_2)_4Cl_4O_2(OH)_2 \cdot 6H_2O$ [18, 19], four uranyl pentagonal bipyramids are linked *via* common edges into tetrameric units (Figs. 2f, g, h). It is noteworthy that the structure of $K_2(UO_2)_4Cl_4O_2(OH)_2 \cdot 6H_2O$ contains *Ur* cations in two compositionally different coordinations, $\mathit{Ur}Cl_2O_3$ and $\mathit{Ur}ClO_4$ (Fig. 2f), whereas the structures of $(UO_2)_4O_2(OH)_2Cl_2(H_2O)_6 \cdot 4H_2O$ and $Rb_4(UO_2)_4O_2Cl_8(H_2O)_2 \cdot 2H_2O$ contain uranyl pentagonal bipyramids with the compositions $\mathit{Ur}Cl_1O_4$ (Fig. 2g) and $\mathit{Ur}Cl_3O_2$ (Fig. 2h), respectively. In some cases, polycondenzation of $\mathit{Ur}(X_mO_n)_5$ (X = Cl, Br) coordination polyhedra results in the formation of higher-dimensional topologies. Infinite chains formed by sharing Cl–Cl edges between $\mathit{Ur}Cl_4O$ pentagonal bipyramids (Fig. 2i) are present in the crystal structure of $(UO_2)Cl_2(H_2O)$ [9, 20]. Similar chains, but with another and more complex alternation sequence of Cl atoms (Fig. 2j), are involved in the formation of layers in the structures of $A_7[(UO_2)_8(VO_4)_2]O_8Cl]$ (A = Rb, Cs) [21]. The structure of $Cs_{0.9}(UO_2)OCl_{0.9}$ [22] is based upon double chains formed by edge linkage of $\mathit{Ur}Cl_2O_3$ polyhedra (Fig. 2k). The structure of $Li(H_2O)_2[(UO_2)_2Cl_3OH_2O]$ is remarkable since, despite two different $\mathit{Ur}O_3Cl_2$ and $\mathit{Ur}OCl_4$ pentagonal coordination modes forming tetranuclear clusters (Fig. 2d), the sharing of bipyramids *via* common equatorial Cl–Cl edges gives rise to unusual 1-D units [23] (Fig. 2m). Another type of complex chains formed by linking $\mathit{Ur}ClO_4$ and $\mathit{Ur}Cl_2O_3$ polyhedra *via* common O–O edges has been observed in $Cs_2((UO_2)_3Cl_2(IO_3)(OH)O_2) \cdot 2H_2O$ [23] (Fig. 2l). Simple single chains depicted in Fig. 2i may share their common O atoms to form a 3*D* unit (Fig. 2n) observed in the crystal structure of UO_2Cl_2 [24]. Note that, in this case, O atoms of the uranyl cation are also involved in the aggregation of coordination polyhedra.

As it can be seen from the discussion above, most of the uranyl halides are hydrous species with 3-D linkage of the $\mathit{Ur}(X_mO_n)_5$ units provided by hydrogen bonding or interstitial alkali cations. Linking of uranyl bipyramids through their equatorial edges results in the dominance of 0-D and 1-D polyhedra within this class of compounds. This feature is in strong contrast with pure uranyl oxo-compounds, where corner-linking of uranyl bipyramids often results in the formation of sheets with unique and unprecedent diversity [5].

The uranyl nitrate chloride $Cs_2(UO_2)(NO_3)Cl_3$ described herein and its comparison with related uranyl compounds illustrate the remarkable structural diversity and variability of uranyl oxohalides. In general, linkage of $\mathit{Ur}(X_mO_n)_5$ polyhedra may proceed not only *via* edge- but also *via* corner-sharing. $Cs_2(UO_2)(NO_3)Cl_3$ is the first purely inorganic uranyl oxohalide containing triangular NO_3 groups strongly bonded to $\mathit{Ur}(X_mO_n)_5$ polyhedra (see also [7, 8]). Further systematic attempts of hydrothermal synthesis of new alkali uranyl halides using halides of the heavier alkali metals (K, Rb, Cs) halides in excess as starting materials may lead to the discovery of new structure types with potentially interesting applications.

Supporting information

The anisotropic displacement parameters of $Cs_2(UO_2)(NO_3)Cl_3$ are provided as Supporting Information online only (www.znaturforsch.com/ab/v66b/c66b.htm).

Acknowledgement

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Supporting Information

Table S1. Anisotropic displacement parameters U_{ij} (Å^2) for $Cs_2(UO_2)(NO_3)Cl_3$.

Table S1. Anisotropic displacement parameters U_{ij} (Å^2) for $Cs_2(UO_2)(NO_3)Cl_3$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
U(1)	0.02230(8)	0.02450(7)	0.02082(8)	−0.00082(6)	0.00971(6)	−0.00094(6)
Cs(1)	0.02905(15)	0.03815(16)	0.03063(15)	0.00092(12)	0.01142(12)	−0.00012(12)
Cs(2)	0.03468(16)	0.04832(19)	0.03446(16)	0.00118(13)	0.01542(13)	0.00116(13)
Cl(1)	0.0308(6)	0.0438(7)	0.0495(7)	0.0011(5)	0.0244(5)	0.0073(6)
Cl(2)	0.0346(6)	0.0401(6)	0.0358(6)	−0.0022(5)	0.0207(5)	0.0034(5)
Cl(3)	0.0410(7)	0.0510(8)	0.0607(8)	0.0141(6)	0.0229(6)	0.0276(7)
O(1)	0.0351(17)	0.0346(17)	0.0271(15)	−0.0075(14)	0.0148(14)	−0.0074(13)
O(2)	0.0290(17)	0.048(2)	0.0269(16)	−0.0020(14)	0.0086(14)	−0.0118(14)
O(3)	0.0250(15)	0.0367(18)	0.0379(17)	0.0023(13)	0.0133(14)	0.0082(14)
O(4)	0.0387(19)	0.0354(19)	0.043(2)	0.0098(15)	0.0038(16)	0.0098(16)
O(5)	0.0243(16)	0.046(2)	0.050(2)	0.0002(14)	0.0146(15)	0.0170(16)
N(1)	0.028(2)	0.028(2)	0.029(2)	0.0037(16)	0.0071(17)	0.0003(16)