

New Uranyl Succinate-Containing Polymers: Synthesis and Structure

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Abstract—Two uranyl succinate polymers, $\text{NH}_4[\text{UO}_2(\text{Suc})(\text{HSuc})]\cdot\text{H}_2\text{O}$ (**I**) and $[\text{UO}_2(\text{Suc})(\text{H}_2\text{O})_2]$ (**II**), where Suc = succinate ion ($\text{C}_4\text{H}_4\text{O}_4^{2-}$), were synthesized and studied by X-ray diffraction, IR spectroscopy, and thermography (CCDC nos. 2202634 and 2202635, respectively). The main structural units in the crystals of **I** are infinite zigzag chains $[\text{UO}_2(\text{Suc})(\text{HSuc})]^-$, corresponding to the $\text{AQ}^{02}\text{B}^{01}$ crystal chemical formula ($\text{A} = \text{UO}_2^{2+}$, $\text{Q}^{02} = \text{C}_4\text{H}_4\text{O}_4^{2-}$, $\text{B}^{01} = \text{C}_4\text{H}_5\text{O}_4$). It was established that in structure **II**, the electrically neutral $[\text{UO}_2(\text{Suc})(\text{H}_2\text{O})_2]$ chains correspond to the AQ^{02}M^1 crystal chemical formula ($\text{A} = \text{UO}_2^{2+}$, $\text{Q}^{02} = \text{C}_4\text{H}_4\text{O}_4^{2-}$, $\text{M}^1 = \text{H}_2\text{O}$). The intermolecular interactions in structures **I** and **II** were analyzed using the method of molecular Voronoi–Dirichlet polyhedra.

Keywords: uranyl complexes, succinate ions, crystal structure, Voronoi–Dirichlet polyhedral

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INTRODUCTION

The complex formation of U(VI) with succinic acid in aqueous solutions is actively studied by various experimental (in particular, spectroscopic and diffraction) and theoretical (density functional theory) methods. It was found that the $\text{Suc} : \text{U(VI)}$ ratio (Suc = succinate ion $\text{C}_4\text{H}_4\text{O}_4^{2-}$) in the structurally characterized succinate-containing complexes is usually 0.5, 1, 1.5, or 2 [1–10], although a complex with $\text{Suc} : \text{U(VI)} = 3$ was also reported [11]. It is known that succinic acid is a member of the homologous series of dicarboxylic acids. Owing to the presence of two carboxyl groups, succinate ions bind simultaneously to two to four U(VI) atoms [12]; therefore, uranyl succinates are a special case of coordination polymers, which have been actively studied in recent years [13–15]. In characterized uranyl succinates, 1D, 2D, or 3D structural groups with U(VI) coordination number of 7 or 8 can be found. One more factor responsible for the diversity of polymer structures is the flexibility of succinate ions, which can exist in the gauche (ϕ^1) or transoid (ϕ^3) conformations [16]. This paper describes the synthesis and structural study of new uranyl coordination polymers: ammonium succinate-hydrogen succinate monohydrate $\text{NH}_4[\text{UO}_2(\text{Suc})(\text{HSuc})]\cdot\text{H}_2\text{O}$ (**I**) and uranyl succinate dihydrate $\text{UO}_2(\text{Suc})\cdot 2\text{H}_2\text{O}$ (**II**).

EXPERIMENTAL

All chemicals used in the study were at least of analytical grade.

Synthesis of the crystals of $\text{NH}_4[\text{UO}_2(\text{Suc})(\text{HSuc})]\cdot\text{H}_2\text{O}$ (I**).** Succinic acid (2.305 mmol, 0.272 g) and ammonium fluoride (2.892 mmol, 0.107 g) were added successively to an aqueous solution of uranyl nitrate hexahydrate (0.578 mmol, 0.290 g), and the mixture was stirred until the compounds completely dissolved (pH 2). The initial molar ratio of the reactants was 1 : 4 : 5. The resulting solution was left for crystallization at room temperature. After 4–6 days, yellow crystals formed. The yield was 65.8%.

For $\text{C}_8\text{H}_{15}\text{O}_{11}\text{NU}$

Anal. calcd., % U, 44.15

Found, % U, 43.9

IR spectrum of **I** (ν , cm^{-1}): 3597 m, 3435 m, $\nu(\text{H}_2\text{O})$, $\nu(\text{NH})$; 3170 br.m, $\nu(\text{CH}_2)$; 1704 w, $\nu(\text{COOH})$; 1633 w, $\delta(\text{H}_2\text{O})$; 1535 s, $\nu_{\text{as}}(\text{COO})$; 1453 s, 1404 m, $\nu_{\text{s}}(\text{COO})$; 1298 w, 1283 w, 1230 w, $\nu(\text{CO})$, $\delta(\text{CH}_2)$; 1189 w, 1163 w, $\nu(\text{CCC})$, $\delta(\text{CH}_2)$, $\omega(\text{CH}_2)$; 931 s, $\nu_{\text{as}}(\text{UO}_2)$; 685 m, $\gamma(\text{COO})$.

Synthesis of crystals of $[\text{UO}_2(\text{Suc})(\text{H}_2\text{O})_2]$ (II**).** Acetamide (0.21 g, 2.8 mmol) and uranium(VI) oxide (0.2 g, 0.7 mmol) were added successively to a solution of succinic acid (0.33 g, 2.8 mmol in 10 mL of water) heated to 60°C. The resulting suspension was heated with magnetic stirring until the oxide completely dis-

solved (pH 3). The initial molar ratio of the reactants was 4 : 4 : 1. After 3–4 days, yellow crystals precipitated from the solution. The yield was 78.2%.

For $C_4H_8O_8U$

Anal. calcd., %	U, 56.40
Found, %	U, 56.7

IR spectrum of **II** (ν , cm^{-1}): 3389 br.m, 3219 m, $\nu(H_2O)$; 2947 w, $\nu(CH_2)$; 1629 w, $\delta(H_2O)$; 1513 s, $\nu_{as}(COO)$; 1462 s, 1406 w, $\nu_s(COO)$; 1392 m, $\nu_s(COO)$, $\delta(CH_2)$, $\omega(CH_2)$; 1310 m, $\nu(CO)$, $\delta(CH_2)$; 1183 w, $\nu(CCC)$, $\delta(CH_2)$, $\omega(CH_2)$; 970 w, $\nu(CC)$; 937 s, $\nu_{as}(UO_2)$; 901 w, $\nu(C-C)$; 699 m, $\gamma(COO)$; 597 m, $\rho(CH_2)$; 553 w, $\delta(CCC)$. The most intense absorption bands in the spectra of **I** and **II** correspond to uranyl and succinate vibrations.

X-ray diffraction study was carried out on a Bruker KAPPA APEX II automated four-circle diffractometer with area detector ($MoK\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$, φ - and ω -scan mode). The unit cell parameters were refined over the whole data array [17]. The absorption corrections to the experimental reflection intensities were applied using SADABS software [18]. The structures were solved by direct methods with SHELXS97 software [19] and refined by the full-matrix least-squares method on F^2 with SHELXL-2018/3 software [20] over all data in the anisotropic approximation for all non-hydrogen atoms. The H atoms of the CH_2 groups were placed in the geometrically calculated positions with $U_{iso} = 1.2U_{equiv}(C)$. The H atoms of the ammonium cation, water molecules, and carboxyl group (in **I**) were located using the Fourier difference electron density maps. The H atoms of the ammonium cation in **I** were refined with $U_{iso} = 1.2U_{equiv}(N)$ and with imposing the equality condition on the N–H distances and the HNH angles. The H atoms of the water molecule in **I** were refined with $U_{iso} = 1.5U_{equiv}(O)$ and using restraints on the O–H distances and the HOH angle. The H atom of the carboxyl group in **I** was refined with $U_{iso} = 1.2U_{equiv}(O)$. The water H atoms in **II** were refined with the individual isotropic thermal factors and with imposing the equality condition on the O–H distances.

The X-ray diffraction experiment details and the final R -factors for the crystals of **I** and **II** are summarized in Table 1; selected bond lengths and bond angles of the UO_8 polyhedra and hydrogen bond parameters are in Tables 2 and 3. The coordination numbers of atoms in the structures were calculated using the method of overlapping spheres [21].

The atom coordinates and thermal parameters were deposited with the Cambridge Crystallographic Data Centre (CCDC nos. 2202634 (**I**) and 2202635 (**II**)).

The differential thermal and thermogravimetric analyses were performed on a Shimadzu DTG-60 derivatograph at a heating rate of $10^\circ\text{C}/\text{min}$. The sample weights were 7–8 mg. The calcination was carried out in air at 900°C in platinum crucibles using a Pt–Pt/Rh thermocouple and calcined alumina as the reference.

The IR spectra were recorded on an FT-801 FTIR spectrometer at room temperature in the 500 – 4000 cm^{-1} range. The samples were prepared by pressing with KBr. The bands were assigned using published data [22, 23].

RESULTS AND DISCUSSION

Each of the structures contains one crystallographic sort of U(VI) atoms, which occupy $4e$ general positions in **I** and $4c$ centrosymmetric positions in **II**. In both cases, the uranium coordination polyhedron (CP) is a UO_8 hexagonal bipyramid, with the oxygen atoms of UO_2^{2+} ions located on its principal axis. The uranyl ions in **I** are nearly symmetrical and linear, the $U=O$ distances are 1.769 and 1.772 \AA , and the $O=U=O$ angle is 178.8° ; in **II**, the $U=O$ distance is 1.751 \AA and the $O=U=O$ angle is 180° (Tables 2, 3). The volumes of the uranium Voronoi–Dirichlet polyhedron (VDP), shaped as the hexagonal prism, are 9.40 and 9.39 \AA^3 in **I** and **II**, respectively. This coincides within σ with the average value of $9.2(2) \text{ \AA}^3$, established for U(VI) atoms in UO_n CP for n ranging from 6 to 9 [24].

In structure **I**, four of the six equatorial oxygen atoms belong to succinate ions, which correspond to the Q^{02} coordination type while the other two belong to hydrogen succinate ions coordinated according to the B^{01} type. According to [25], the Q and B characters (the first letters of the words “quadridentate” and “bidentate”) characterize the total ligand denticity. Type Q^{02} succinate ions are bound to each coordinated uranium atom in the bidentate fashion; therefore, the superscript 2, indicating the total number of such metal atoms, is in the second position. Each hydrogen succinate ion is bound in the bidentate fashion to only one uranium atom, thus, the type of coordination of this ligand is designated as B^{01} . In structure **I**, the succinate and hydrogen succinate ions form gauche (φ^1) and transoid (φ^3) conformations, respectively, with $C-C-C-C$ torsion angles being 72.4° and 176.9° . The ligand coordination types and crystal chemical formulas (CCFs) of the complexes are given in accordance with [25]. In structure **I**, uranium atoms are combined into infinite zigzag chains $[UO_2(\text{Suc})-(\text{HSuc})]^-$, described by $AQ^{02}B^{01}$ CCF, where $A = UO_2^{2+}$, $Q^{02} = C_4H_4O_4^{2-}$, and $B^{01} = C_4H_5O_4^-$ (Fig. 1).

In structure **II**, out of the six equatorial oxygen atoms of the UO_8 bipyramid, four atoms belong to

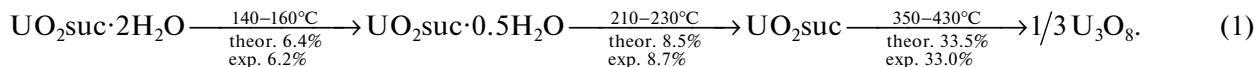
Table 1. Crystallographic data and X-ray diffraction experiment and structure refinement details for **I** and **II**

Parameter	Value	
	I	II
System	Monoclinic	Monoclinic
Space group, <i>Z</i>	<i>P</i> 2 ₁ / <i>n</i> , 4	<i>C</i> 2/ <i>c</i> , 4
<i>a</i> , Å	11.8569(4)	12.7711(11)
<i>b</i> , Å	9.5179(4)	7.5930(6)
<i>c</i> , Å	12.7079(5)	10.6556(9)
β, deg	103.437(1)	122.375(1)
<i>V</i> , Å ³	1394.86(9)	872.67(13)
ρ, g/cm ³	2.568	3.213
μ, mm ⁻¹	11.694	18.615
<i>T</i> , K	100(2)	296(2)
Crystal size, mm	0.18 × 0.16 × 0.16	0.10 × 0.08 × 0.06
θ _{max} , deg	29.997	29.976
Range of <i>h</i> , <i>k</i> , <i>l</i>	-16 ≤ <i>h</i> ≤ 16, -13 ≤ <i>k</i> ≤ 13, -17 ≤ <i>l</i> ≤ 17	-17 ≤ <i>h</i> ≤ 17, -10 ≤ <i>k</i> ≤ 10, -14 ≤ <i>l</i> ≤ 14
Number of reflections measured/unique (<i>N</i> ₁)	24143/4064	4741/1277
<i>R</i> _{int}	0.0256	0.0215
Number of reflections with <i>I</i> > 1.96σ(<i>I</i>) (<i>N</i> ₂)	3757	871
Number of refined parameters	212	70
<i>R</i> ₁ for <i>N</i> ₂	0.0294	0.0271
<i>wR</i> ₂ for <i>N</i> ₁	0.0144	0.0133
<i>S</i>	1.071	0.991
Residual electron density (min/max), e/Å ³	-0.655/0.751	-0.922/0.730

succinate anions, which exhibit the same Q⁰² type of coordination as in **I**. The other two oxygen atoms belong to water molecules, which are in the *trans*-position to each other. The succinate ions in **II** form the φ¹ conformation with the C—C—C—C torsion angle amounting to 59.4°. In structure **II**, the uranium atoms are connected by succinate ions into infinite

electrically neutral zigzag chains [UO₂(Suc)(H₂O)₂], described by AQ⁰²M₂¹, CCF, where A = UO₂²⁺, Q⁰² = C₄H₄O₄²⁻, and M¹ = H₂O (Fig. 2).

According to the data of simultaneous differential thermal and thermogravimetric analysis (Fig. 3), decomposition of **II** can be described by Scheme 1.



The numbers above the arrows indicate the temperature ranges of the observed effects, and the calculated and experimental values of the mass loss, respectively, are indicated under the arrows.

In structures **I** and **II**, it was possible to determine the coordinates of all independent atoms; therefore, the intermolecular interactions in these crystals can be analyzed using the molecular Voronoi–Dirichlet polyhedron method [26], which takes into account all possible A/Z interatomic contacts, but not only those

that are considered to be important. Since **I** and **II** contain atoms of five and four elements, respectively, 15 and 10 types of contacts, differing in the nature of the A and Z atoms, are theoretically possible in the crystal structures. However, according to the results, only six types of intermolecular contacts actually exist in both structures, with identical combinations of atoms: H/H, H/C, C/C, H/O, C/O, and O/O (Table 4). The greatest contribution to the total surface area of molecular VDP faces (⁰S in Table 4) in **I**

Table 2. Selected geometric parameters of the uranium(VI) polyhedron and parameters of hydrogen bonds in $\text{NH}_4[\text{(UO}_2\text{)(C}_4\text{H}_4\text{O}_4\text{)(C}_4\text{H}_5\text{O}_4)]\cdot\text{H}_2\text{O}$ (**I**)

Bond	$d, \text{\AA}$	$\Omega, \%$ *	Angle	ω, deg	
Hexagonal bipyramidal UO_8					
U–O(1)	1.7688(15)	21.65	O(1)UO(2)	178.79(7)	
U–O(2)	1.7722(15)	21.96	O(3)UO(4)	52.76(5)	
U–O(3)	2.4342(15)	9.78	O(7)UO(8)	52.52(5)	
U–O(4)	2.4818(15)	9.30	O(8)UO(9)	66.25(5)	
U–O(7)	2.4605(14)	9.60	O(9)UO(10)	52.48(5)	
U–O(8)	2.4839(14)	9.10	O(3)UO(10)	66.82(5)	
U–O(9)	2.4882(15)	9.13	O(4)UO(7)	69.22(5)	
U–O(10)	2.4595(14)	9.48			
Hydrogen bond parameters**					
D–H···A	Distance, \AA			$\Omega(D\text{--H}), \%$	
	D–H	H···A	D···A		
O(6)–H(1)···O(8)	0.74(3)	2.02(3)	2.723(2)	160(3)	36.48
O(11)–H(3)···O(4)	0.852(16)	1.999(18)	2.825(2)	163(3)	32.01
N(1)–H(4)···O(11)	0.790(14)	2.008(14)	2.797(2)	177(2)	24.68
N(1)–H(5)···O(9)	0.780(14)	2.398(16)	3.117(2)	154(2)	25.29
N(1)–H(6)···O(5)	0.799(14)	2.088(16)	2.816(2)	152(2)	24.14
N(1)–H(7)···O(10)	0.772(14)	2.165(15)	2.886(2)	156(2)	25.89
C(2)–H(8)···O(2)	0.99	2.55	3.423(3)	147	28.24
C(6)–H(12)···O(5)	0.99	2.54	3.467(3)	156	28.06
C(7)–H(15)···O(1)	0.99	2.50	3.257(2)	133	27.96
$\Omega(H\text{--A}), \%$					

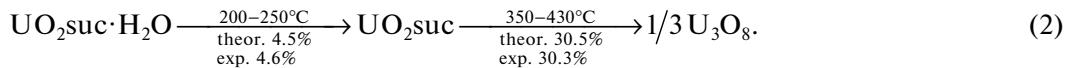
* Here and below, Ω is the solid angle (expressed in % of 4π steradian) under which the VDP face common to neighboring atoms is seen from the nucleus of any of these atoms.

** Here and below, the data are given for hydrogen bonds with $H\text{--A} < 3 \text{\AA}$, $D\text{--H}\cdots A > 130^\circ$, and $\Omega > 10\%$.

and **II** is made by hydrogen bonds (H/O contacts), the partial contributions of which are ~64 and 68%. The second most important are the H/H dispersion interactions (~25%). Together with the H/O contacts, they account for ~90–93% of the 0S value. The sum of partial contributions of the other four types of intermolecular contacts (Table 4) does not exceed 10%. Detailed characteristics of the most important hydrogen bonds in **I** and **II**, which are medium strength bonds according to the existing classification [27], are summarized in Tables 2 and 3.

It is noteworthy that, apart from uranyl succinate dihydrate **II** studied here for the first time, uranyl succinate monohydrate (**III**) have long been known as characterized α - [6], β - [7], and γ - [28] polymorphs. It

is of interest that, despite the specific features of the 3D frameworks in the crystals of α -, β -, and γ -[$\text{UO}_2(\text{Suc})(\text{H}_2\text{O})$] (the differences were discussed in detail previously [28]), these three monohydrates correspond to the same AQ^4M^1 CCF, where $\text{A} = \text{UO}_2^{2+}$, $\text{Q}^4 = \text{C}_4\text{H}_4\text{O}_4^{2-}$, and $\text{M}^1 = \text{H}_2\text{O}$. In conformity with this CCF, all U atoms in the monohydrates have C.N. 7 rather than 8, which is inherent in dihydrate **II** characterized by $\text{AQ}^{02}\text{M}_2^1$ CCF. According to the data of differential thermal and thermogravimetric analyses (Fig. 4), the decomposition of γ -[$\text{UO}_2(\text{Suc})(\text{H}_2\text{O})$] can be described by Scheme 2.



It is important that the monohydrate cannot be obtained by the thermal decomposition of the dihy-

drate, since the first stage of decomposition of **II** gives $\text{UO}_2(\text{Suc})\cdot 0.5\text{H}_2\text{O}$, as can be seen in Scheme 1. This

Table 3. Geometrical parameters of the structure $[\text{UO}_2(\text{Suc})(\text{H}_2\text{O})_2]$ (II)

Hexagonal bipyramidal UO_8				
Bond	$d, \text{\AA}$	$\Omega, \%$	Angle	ω, deg
$\text{U}(1)-\text{O}(1)$	$1.751(3) \times 2$	22.11	$\text{O}(1)\text{U}(1)\text{O}(1)$	180.0
$\text{U}(1)-\text{O}(2)$	$2.537(2) \times 2$	8.37	$\text{O}(3)\text{U}(1)\text{O}(2)$	$51.50(7) \times 2$
$\text{U}(1)-\text{O}(3)$	$2.462(2) \times 2$	9.29	$\text{O}(4)\text{U}(1)\text{O}(2)$	$64.35(9) \times 2$
$\text{U}(1)-\text{O}(4)$	$2.456(3) \times 2$	10.20	$\text{O}(4)\text{U}(1)\text{O}(3)$	$64.27(9) \times 2$

Hydrogen bond parameters					
D-H \cdots A	Distance, \AA			D-H \cdots A, angle, deg	$\Omega(\text{H}\cdots\text{A}), \%$
	D \cdots A	D-H	H \cdots A		
O(4)-H(1) \cdots O(2)	2.895(4)	0.72(4)	2.21(4)	157	17.3
O(4)-H(2) \cdots O(3)	2.813(4)	0.72(4)	2.11(4)	167	17.4
C(2)-H(3) \cdots O(1)	3.750(5)	0.97	2.84	157	12.5
C(2)-H(4) \cdots O(1)	3.334(5)	0.97	2.47	148	17.1

result may be attributed to the fact that the hypothetical monohydrate with $\text{AQ}^{02}\text{M}^1\text{CCF}$ is unstable, and the simultaneous change in the type of coordination of all succinate ions in the crystal structure from Q^{02} to Q^4 is energetically unfavorable (this would require synchronous cleavage and change in the spatial orientation for at least half of the equatorial $\text{U}-\text{O}$ bonds).

The results of IR spectroscopy for **I** and **II** are in agreement with the existing X-ray diffraction data.

FUNDING

X-ray diffraction experiments were carried out at the Center for Collective Use of Physical Investigation Methods, Frumkin Institute of Physical Chemistry and Electro-

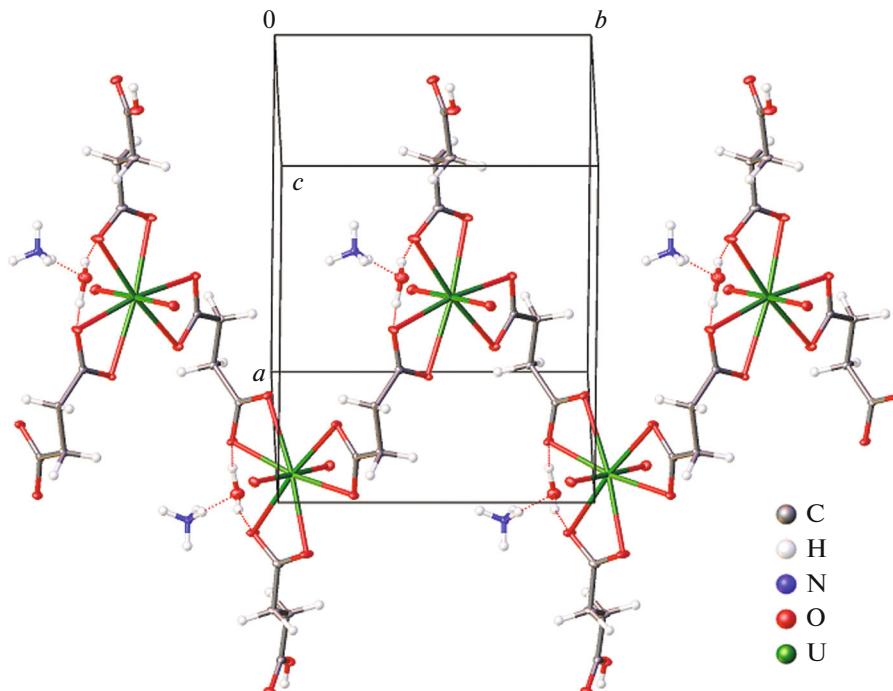


Fig. 1. Fragment of the structure of $\text{NH}_4[\text{UO}_2(\text{Suc})(\text{HSuc})]\cdot\text{H}_2\text{O}$.

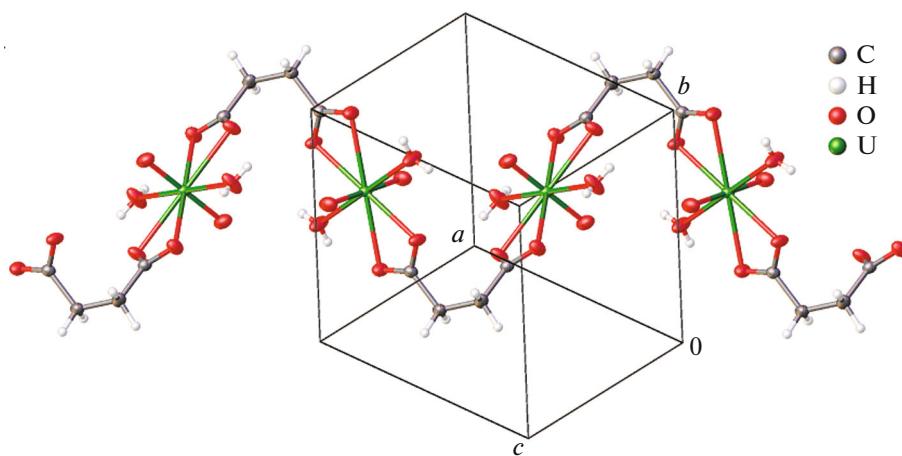


Fig. 2. Fragment of the zigzag chain $[\text{UO}_2(\text{Suc})(\text{H}_2\text{O})_2]$ with $\text{AQ}^{02}\text{M}_2^1$ CCF in structure **II**.

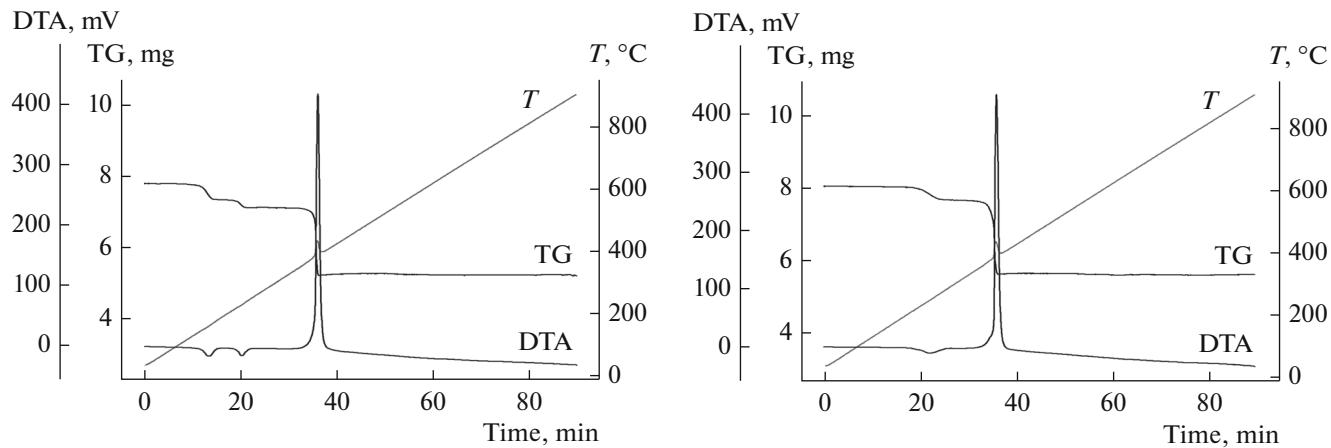


Fig. 3. Differential thermogram of $[\text{UO}_2(\text{Suc})(\text{H}_2\text{O})_2]$.

Fig. 4. Differential thermogram of γ - $[\text{UO}_2(\text{Suc})(\text{H}_2\text{O})]$.

Table 4. Selected parameters of intermolecular contacts in the crystal structures of **I** and **II***

A/Z contacts	NH ₄ [UO ₂ (Suc)(HSuc)]·H ₂ O (I)				[UO ₂ (Suc)(H ₂ O) ₂] (II)			
	k_{AZ}	$d, \text{\AA}$	$S_{\text{AZ}}, \text{\AA}^2$	$\Delta_{\text{AZ}}, \%$	k_{AZ}	$d, \text{\AA}$	$S_{\text{AZ}}, \text{\AA}^2$	$\Delta_{\text{AZ}}, \%$
H/H	83	2.35–4.29	105.88	26.03	44	2.62–4.28	47.39	24.95
H/C	20	3.04–4.24	9.22	2.27	12	2.83–3.41	6.75	3.55
C/C	4	3.60–3.77	0.42	0.10	2	3.80–3.80	0.04	0.02
H/O	140	2.00–4.31	261.70	64.33	64	2.12–4.23	129.93	68.41
C/O	16	3.00–3.95	12.47	3.07	8	3.75–4.21	0.24	0.13
O/O	31	3.04–4.31	17.14	4.21	12	3.22–4.19	5.60	2.95
Sum	294	2.00–4.31	406.84	100	142	2.12–4.28	189.94	100

* k_{AZ} is the total number of all VDP faces with the rank = 0; d is the range of the corresponding A–Z interatomic distances; S_{AZ} is the total area of all faces of this type of VDP of the atoms contained in one formula unit of the compound; Δ_{AZ} is the partial contribution of the corresponding non-covalent A/Z contacts to the integral parameter ${}^0S = \sum S_{\text{AZ}}$ of the molecular VDP.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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