

addressed by collecting high quality data at low temperature using a microfocus Cu X-ray source and employing dual-space recycling (SHELXD) for structure solution. The very large numbers of independent molecules present good exercises for the RESI and BLOC commands during refinement. For example, one structure in space group *I2* has a unit cell volume of nearly 43 000 Å³ and contains fourteen independent molecules. Despite a fascinating topological interweaving, more than one-third of the unit cell volume is occupied by solvent molecules that are disordered to the point of being impractical to model. Following application of the SQUEEZE routine in PLATON, the residual *R*_i decreased from 8.4% to 4.5%.

Keywords: Z-prime, pseudosymmetry, porous

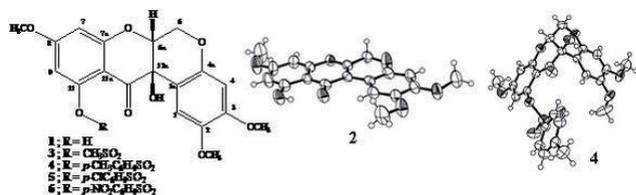
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X-ray structure-cytotoxicity relationship of the 6-deoxyclitriacetals derivatives

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6-Deoxyclitriacetals (**1**) has been identified to have a good cytotoxic activity against various types of human carcinoma, possibly due to its ability to intercalate with DNA as evidenced *in vitro* assay [1]. The sulfonate derivatives of 6-deoxyclitriacetals were synthesized to enhance its cytotoxic activities as novel anticancers [2,3]. Screening of these compounds for cytotoxic activity has shown that tosylate derivative (**4**) was more potent and selective than commercial doxorubicin hydrochloride. X-ray structures and their cytotoxic activities have considerably revealed that not only a bent-shaped structure but also the suitable functional groups at C11 play an important role in increasing their cytotoxicities. Preliminarily, molecular docking of **4** with d(CGATCG)₂ revealed that this derivative with a bent shape structure can intercalate between CG base pair, like doxorubicin behavior. Additionally, the sulfonate derivatives were evaluated their ability to inhibit topoisomerase II activity. They had potentially inhibited the topoisomerase II more 70% inhibition. Finally, we studied the DNA-binding affinity, thermal denaturation of 6-deoxyclitriacetals and its sulfonate derivatives based on UV-Visible spectroscopic techniques [4].



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Keywords: 6-deoxyclitriacetal, X-ray structure-cytotoxicity, topoisomerase II

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Synthesis, crystal structure and properties of [Be(H₂O)₄][UO₂(C₂H₃COO)₃]₂

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A novel complex compound [Be(H₂O)₄][UO₂(CH₃COO)₃]₂ (**1**) was synthesized and studied by X-Ray diffraction, IR spectroscopy and thermal analysis. Yellow prismatic crystals of **1** were obtained by slow evaporation of aqueous solution containing equimolar amounts of beryllium and uranyl acetates. **1** crystallizes in tetragonal crystal system, unit cell parameters: *a* = 10.3647, *c* = 23.4127 Å, *V* = 2515.2 Å³, *Z* = 4, space group *I4₁/a*, *R_i* = 0.0194 (for 1436 reflections). Each beryllium atom in structure of **1** is tetrahedrally surrounded by four oxygen atoms of water molecules and forms [Be(H₂O)₄]²⁺ complex. The crystal-chemical formula (CCF) of this complex is AM₁₄ (A = Be²⁺, M¹ = H₂O). CCF is given in accordance with [1]. Uranyl cation forms mononuclear [UO₂(CH₃COO)₃]⁻ complex with AB⁰₃ (A = UO₂²⁺, B⁰ = CH₃COO⁻) CCF. Coordination polyhedron of uranium atom is in shape of hexagonal bipyramid. Six oxygen atoms that are located in equatorial plane belong to three acetate groups, which act as bidentate chelate ligands (the B⁰ coordination type).

The [Be(H₂O)₄]²⁺ and [UO₂(CH₃COO)₃]⁻ in **1** are bound with each other by electrostatic interactions and hydrogen bonds. The hydrogen bonds are formed by atoms of water molecules and oxygen atoms of acetate groups. There are two crystallographically different H-bonds in **1**. According to Steiner classification [2], both of them are of moderate strength (the OH...O angles are equal to 175 and 170°, and the O...O distances are equal to 2.60 and 2.67 Å). Meanwhile Voronoi-Dirichlet polyhedron solid angles of the O...O face (expressed as a percentage of 4π steradian) calculated without hydrogen atoms equal 16.6 and 14.9% respectively. This result agrees with previously reported method [3] of H-bonds finding in structures which contain hydrogen atoms, but its coordinates have not been determined.

Thermal decomposition of **1** includes three stages. At the first stage (120-140°C) a removal of water molecules takes place. Further heating up to 220°C leads to sequential acetate anions decomposition. The product of decomposition is a black-colored mixture of BeO and U₃O₈.

IR spectrum of **1** contains uranyl, acetate and water absorption bands. It is in agreement with structure data obtained using single-crystal X-Ray diffraction experiment.

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Synthesis and structure of uranyl complexes with *n*-butyrate and *n*-valerate ions

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To date, more than 70 acetate uranyl complexes have been researched, while uranyl complexes with anions of other monocarboxylic acids are considerably less studied. Thus, structure of only one uranyl complex with *i*-butyrate ligands is determined [1], and there exists no data about U(VI) compounds containing *n*-butyrate and *n*-valerate ions. In present research we defined crystal structures of $\text{NaUO}_2(\text{n-C}_3\text{H}_7\text{COO})_3 \cdot 0.25\text{H}_2\text{O}$ (I), $\text{KUO}_2(\text{n-C}_3\text{H}_7\text{COO})_3$ (II) and $\text{UO}_2(\text{n-C}_4\text{H}_9\text{COO})_2 \cdot 2\text{H}_2\text{O}$ (III).

I and II were derived as prismatic yellow crystals while studying the interaction between uranyl nitrate and butyric acid in presence of sodium hydroxide (or potassium hydroxide). Prismatic yellow crystals III were derived as a result of interaction between uranium trioxide and aqueous solution of valeric acid. Fundamental absorption bands of UO_2^{2+} , $\text{C}_3\text{H}_7\text{COO}^-$ or $\text{C}_4\text{H}_9\text{COO}^-$ (only for III) ions and water molecules (for I and III) are present in IR spectra of I-III.

Structures of I-III were determined using X-ray diffraction. I crystallizes in the monoclinic system with the unit cell parameters $a = 13.567$, $b = 20.07$, $c = 13.614$ Å, $\beta = 106.839^\circ$, space group $P2_1$, $Z = 8$ and $R = 0.049$. II crystallizes in the orthorhombic system with the unit cell parameters $a = 17.133$, $b = 19.697$, $c = 21.969$ Å, space group $P2_12_12_1$, $Z = 16$ and $R = 0.056$. III crystallizes in the monoclinic system with the unit cell parameters $a = 7.782$, $b = 10.802$, $c = 9.512$ Å, $\beta = 104.885^\circ$, space group $C2/m$, $Z = 2$ and $R = 0.025$. Some butyrate and valerate ions in I-III are disordered over two positions.

Coordination polyhedron of every uranium atom in I-III is hexagonal bipyramid UO_8 with oxygen atoms of uranyl ion located on the main axis. In I and II there are four crystallographic sorts of atoms U and R ($R = \text{Na}$ or K with coordination numbers 6 or 5 (5 – only for Na(3), which is bonded with water molecule in I)). The structural units of I and II are mononuclear groups of the composition $[\text{UO}_2(\text{C}_3\text{H}_7\text{COO})_3]^-$ with crystal chemical formula AB^{01}_3 ($A = \text{UO}_2^{2+}$, $B^{01} = \text{C}_3\text{H}_7\text{COO}^-$) (coordination types of ligands are assigned according to [2]). Tris(butyrate)dioxidouranium(VI) complexes are joined through electrostatic interactions with sodium and potassium cations, and, in I, also through a system of hydrogen bonds with participation of water molecules. The structural units of III are uncharged complexes $[\text{UO}_2(\text{C}_4\text{H}_9\text{COO})_2(\text{H}_2\text{O})_2]$ with crystal chemical formula $AB^{01}_2M^1_2$ ($A = \text{UO}_2^{2+}$, $B^{01} = \text{C}_4\text{H}_9\text{COO}^-$, $M^1 = \text{H}_2\text{O}$), which are joined through a system of hydrogen bonds.

Using TOPOS program package [3], crystal chemical analysis of sodium and potassium uranyl complexes with acetate and butyrate ligands was carried out.

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Keywords: uranyl, butyrate, valerate

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Crystal structure of complexes of uranyl chromate with isomers of pyridinecarboxylic acid

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The structures of uranyl sulfate with some isomers of pyridinecarboxylic acid are described in [1, 2]. To investigate the influence of nature of oxoanions on the structure of the complexes, complexation of uranyl chromate with nicotinic and isonicotinic acid in aqueous solutions was studied and new compound with

compositions $[\text{UO}_2\text{CrO}_4(\text{C}_5\text{H}_4\text{NHCOO})] \cdot \text{H}_2\text{O}$ (I) ($\text{C}_5\text{H}_4\text{NHCOO}$ – piridin-4-carboxylic acid) and $[\text{UO}_2\text{CrO}_4(\text{C}_5\text{H}_4\text{NHCOO})] \cdot 0.25\text{H}_2\text{O}$ (II) ($\text{C}_5\text{H}_4\text{NHCOO}$ – piridin-3-carboxylic acid) were obtained. The structures of I and II were studied by X-ray diffraction. Both compounds crystallize in the monoclinic crystal system. The unit cell parameters for I: $a = 7.5025$, $b = 11.5188$, $c = 13.0518$ Å, $\beta = 97.877^\circ$, $V = 1117.3$ Å³, space group $P2_1/n$, $Z = 4$, $R_f = 0.0263$ (3840 reflections); and for II: $a = 7.2362$, $b = 13.8847$, $c = 10.7204$ Å, $\beta = 90.037^\circ$, $V = 1077.1$ Å³, space group $P2_1/n$, $Z = 4$, $R_f = 0.0236$ (5224 reflections).

The coordination polyhedron of the uranium atom in both structures I and II has the shape of a UO_2O_5 pentagonal bipyramid. In structure I three equatorial oxygen atoms belong to three tridentate chromate ions (T^3 type) and another two oxygen atoms belong to two bidentate bridging molecules of isonicotinic acid (B^2 type), which occupy trans positions in relation to each other in equatorial plane of uranyl ion. The main structural unit of I is a layer $[\text{UO}_2\text{CrO}_4(\text{C}_5\text{H}_4\text{NHCOO})]$ belonging to the AT^3B^2 crystal chemical group, ($A = \text{UO}_2^{2+}$, $T^3 = \text{CrO}_4^{2-}$, $B^2 = \text{C}_5\text{H}_4\text{NHCOO}$ molecules). The designations of coordination types and crystal chemical formulas are given in accordance with [3]. Layers are linked in framework by system of hydrogen bonds, which include outer sphere water molecules. In structure II chromate ions also have tridentate coordination and nicotinic acid molecules are bidentate chelate (B^{01} type). The main structural unit of II is a chain $[\text{UO}_2\text{CrO}_4(\text{C}_5\text{H}_4\text{NHCOO})]$, belonging to the AT^3B^{01} crystal chemical group, ($A = \text{UO}_2^{2+}$, $T^3 = \text{CrO}_4^{2-}$, $B^{01} = \text{C}_5\text{H}_4\text{NHCOO}$ molecules).

Influence of nature of oxoanion and type of isomer on the structure of complexes of uranyl sulfate and chromate with some isomers of pyridinecarboxylic acid was discussed.

This study was supported by the Ministry of Education and Science of the Russian Federation (project no. 02.740.11.0275).

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Keywords: uranyl, structure

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Complexes containing coordination centre OSiC_3X ($\text{X} = \text{Cl}$, Br) based on N-organosulfonyl-2-aminoacids

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Complexes with a coordination centre OSiC_3Cl obtained from N-organosulfonyl-2-aminoacid containing three biologically active moieties (sulphonamide group, the residue of amino acid, and the five-membered O-Si-chelate ring) were studied by x-ray diffraction.

The structural characteristics of the coordination polyhedron of the silicon atom in these complexes and the influence of crystal packing on their molecular structure were analyzed.

In the case of $\text{X} = \text{Cl}$ the coordination polyhedron of the silicon atom correspond to a distorted trigonal bipyramid. The deviation of the silicon atom from the plane of equatorial substituents varies in the range from $-0.0408(8)$ to $0.0809(3)$ Å (sign “-” means that the Si atom deviates towards the Cl atom). The Si-O bond length depends on the electron-acceptor properties of N-organosulfonylprolin substituent; the smallest value corresponds to the compound **1** ($R = \text{Me}$), and the largest to the compound **2** ($R = \text{Ph}$). The changes in the interatomic Si ... O distances are those for Si ... Cl are antibate ($1.924(2)$ - $2.007(16)$ and $2.256(15)$ - $2.346(11)$ Å, respectively). The strongest intermolecular