



One-dimensional Uranium(VI) Coordination Polymer Complex Containing Dimethyl and Trimethyl Phosphate Ligands: Synthesis, Spectroscopic Characterization, Thermal Analyses, and Crystal Structure

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A new one-dimensional uranium(VI) coordination polymer, $[\text{UO}_2(\mu\text{-DMP})_2(\text{TMP})]_n$ (1) (DMP is dimethyl phosphate and TMP is trimethyl phosphate), was prepared from the reaction of $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and TMP in THF (THF is tetrahydrofuran) as a solvent. Suitable crystals of this complex for crystal structure determination were obtained by slow evaporation of the produced yellow solution at room temperature. Complex 1 was characterized by elemental analysis, IR spectroscopy and single-crystal X-ray diffraction. The X-ray structural analysis indicated that the uranium(VI) is seven-coordinated in a pentagonal bipyramidal configuration by two axial uranyl oxygens, four bridged dimethyl phosphate and monodentate trimethyl phosphate ligands. Also, the thermal stability of 1 was studied by thermogravimetric and differential thermal analyses.

Keywords: Uranium(VI), Crystal structure, Trimethyl phosphate, Dimethyl phosphate

INTRODUCTION

The chemistry of uranium is dominated by hexavalent uranyl(VI) cation (UO_2^{2+}) which is a linear triatomic species capped with terminal oxygen atoms [1,2]. The uranyl(VI) cation is significant stable form of natural uranium which exists in nuclear fuel processing, and usually exhibits tetragonal, pentagonal, and hexagonal bipyramidal geometries in the equatorial plane with two oxygen atoms in axial position [3-5]. Uranium complexes are of interest owing to their importance for application in gas sorption, ion-exchange, selective oxidation catalysis, photochemical reduction, axial uranium oxygen bond activation and several others [6-13]. Trialkyl phosphate compounds, $(\text{RO})_3\text{P}=\text{O}$ (R is 2,6-diisopropylphenyl, 2,6-*t*-Bu₂-4-me-phenyl, 2,4-dichloro-6-methylphenyl, phenyl, butyl, ethyl and methyl), play a crucial role in the different areas of human activity. For example, they are widely

applied as extractants or synergists in metal extraction systems, high temperature lubricants and flame retardants [14-17]. Trialkyl phosphates are good ligands and can be coordinated to cationic of main and transition metals from one oxygen atom in neutral mode and lead to the formation of mononuclear complexes [18-21]. Also, this compounds in the anionic form, $[(\text{RO})_2\text{PO}_2]^-$, can be coordinated to cationic of main and transition metals from two oxygen atoms in the form of a bridge and lead to the formation of binuclear, trinuclear, tetranuclear, pentanuclear, hexanuclear and 1-dimensional coordination polymer complexes [21-41]. Trimethyl phosphate $[(\text{MeO})_3\text{P}=\text{O}]$ is the simplest member of trialkyl phosphate compounds. In addition to being coordinated to cationic of metals, this compound can also be present as an independent counter-ion in ion pair complexes [42,43]. Previously, we reported the synthesis and crystal structure complex of $[\text{UO}_2(\mu\text{-DMP})_2(\text{DMSO})]_n$ [32]. This complex was prepared from the reaction of $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and TMP in dimethyl sulfoxide solvent. Herein, we reported

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new one-dimensional coordination polymer of $[\text{UO}_2(\mu\text{-DMP})_2(\text{TMP})]_n$ (1). This complex was prepared according to the procedure used in complex $[\text{UO}_2(\mu\text{-DMP})_2(\text{DMSO})]_n$ except using tetrahydrofuran instead of dimethyl sulfoxide as solvent. Unlike complex $[\text{UO}_2(\mu\text{-DMP})_2(\text{DMSO})]_n$, which is easily dissolved in dimethyl sulfoxide, complex 1 did not dissolve in any solvent. Therefore, we could not identify complex 1 with spectroscopies such as NMR and UV-Vis.

EXPRIMENTAL

Materials and Physical Methods

All chemicals were purchased from Merck, and used as received. Elemental analysis was performed using an Elementar Analysensystem GmbH VarioEL. Infrared spectrum ($4000\text{-}250\text{ cm}^{-1}$) was taken on a Shimadzu-470 spectrometer in CsI pellets. Melting points were obtained on a Kofler Heizbank Rechart type 7841 melting point apparatus. Thermal behavior was measured with a STA 503 Bähr apparatus.

Preparation of Complex $[\text{UO}_2(\mu\text{-DMP})_2(\text{TMP})]_n$ (1)

Trimethyl phosphate (1.73 g, 1.35 ml, 12 mmol) was added to a solution of $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (2.04 g, 4 mmol) in THF (40 ml) and the resulting yellow solution was stirred at $40\text{ }^\circ\text{C}$ for 30 min. This solution was left to evaporate slowly at room temperature. After two weeks, yellow prismatic crystals of 1 were isolated (yield 2.12 g, 80.3%, m. p. $> 300\text{ }^\circ\text{C}$). Anal. Calcd. for $\text{C}_7\text{H}_{21}\text{O}_{14}\text{P}_3\text{U}_1$ (%): C, 12.73; H, 3.18. Found: C, 12.64; H, 3.16. IR (CsI, cm^{-1}): 3002w, 2964m, 2861m, 1452w, 1437w, 1255s, 1189s, 1116s, 1107s, 1069s, 1044s, 928s, 841s, 790m, 680m, 564w, 519m, 480m, 388m, 298m, 287m.

X-Ray Structure Analysis

The X-ray diffraction measurements for single crystal of $[\text{UO}_2(\mu\text{-DMP})_2(\text{TMP})]_n$ (1) was made on a Bruker APEX II CCD area-detector diffractometer at 298 K using graphite mono-chromated Mo $K\alpha$ radiation ($\lambda = 0.71073\text{ \AA}$). The structure of 1 was solved by SHELX-97 and absorption correction was applied using the SADABS program [44]. Data collection, cell refinement, and data reduction were performed by applying APEX II, SAINT, SHELXTL and

PLATON program packages [44-50]. The ORTEP and crystal packing diagram for title complex were drawn with the PLATON and Mercury 2.4 program [50] software's, respectively.

RESULTS AND DISCUSION

Synthesis of Complex $[\text{UO}_2(\mu\text{-DMP})_2(\text{TMP})]_n$ (1)

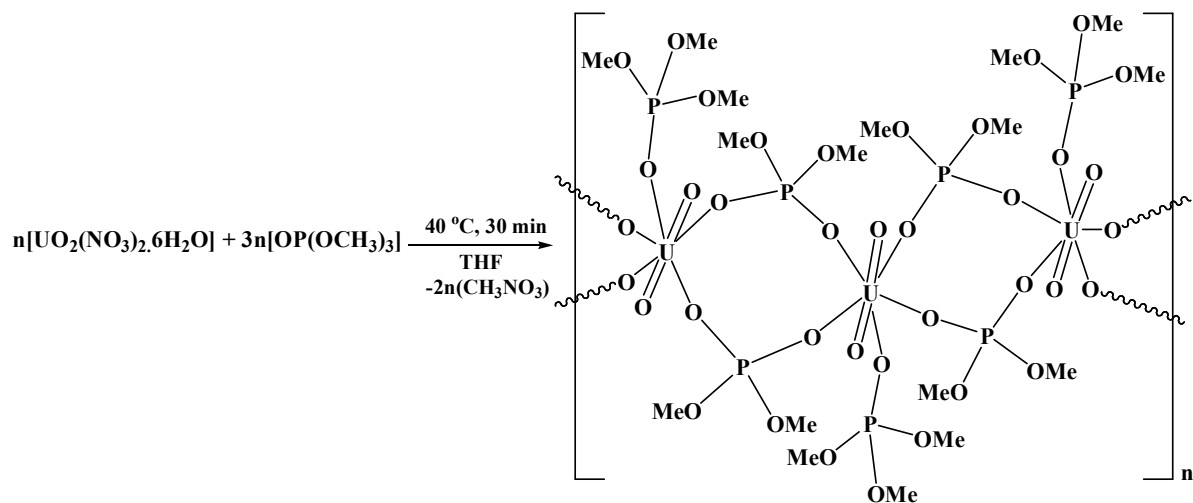
Complex 1 was prepared from the reaction of one equivalent of $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ with three equivalents of trimethyl phosphate in THF, at $40\text{ }^\circ\text{C}$ for 30 min. Suitable crystals of title complex were obtained for X-ray diffraction measurement by slow evaporation of the resulted yellow solution at room temperature. The synthetic route of title complex is shown in Scheme 1.

Spectroscopic Characterization of $[\text{UO}_2(\mu\text{-DMP})_2(\text{TMP})]_n$ (1)

This complex is air-stable and is insoluble in polar and nonpolar solvents. The IR absorption bands for title complex are listed in the experimental section. The infrared spectrum of 1 show three weak and medium absorption bands at 3002, 2964 and 2861 cm^{-1} , and two weak absorption bands at 1452 and 1437 cm^{-1} , which are assigned to the C-H stretching and bending vibrations, respectively of the methyl groups for trimethyl phosphate and dimethyl phosphate. Several strong absorption bands observed in the range $1255\text{-}790\text{ cm}^{-1}$ are assigned to P=O, C-O, P-O and P-O-C stretching vibrations. The medium and weak absorption bands observed in the range $680\text{-}480\text{ cm}^{-1}$ are assigned to the P-O bending vibrations [40,51-53]. Furthermore, in the infrared spectrum of this complex the U=O stretching vibration was observed at 388 cm^{-1} and for U-O=P and U-O-P stretching vibrations were observed at 298 and 287 cm^{-1} [54-56].

Thermal Studies of $[\text{UO}_2(\mu\text{-DMP})_2(\text{TMP})]_n$ (1)

The thermal stability of 1 between 30 and $780\text{ }^\circ\text{C}$ has been determined by thermogravimetric analysis (TGA) and differential thermal analysis (DTA) on single crystal samples in a static air atmosphere with a heating rate of $10\text{ }^\circ\text{C min}^{-1}$ (Fig. 1). The TGA curve shows that chemical decomposition starts at around $360\text{ }^\circ\text{C}$ and ends at around $750\text{ }^\circ\text{C}$ with a weight loss of 55.3% corresponding to the



Scheme 1. The preparation method for complex 1

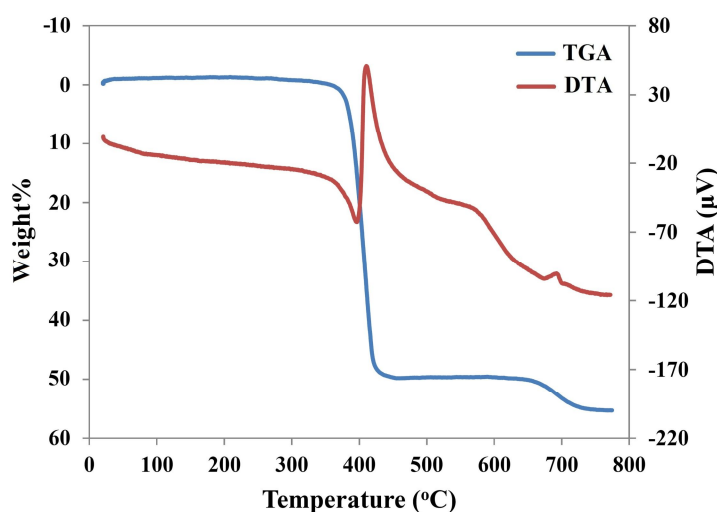


Fig. 1. Thermal behavior of 1.

removal of two dimethyl phosphate anions and one trimethyl phosphate ligand (calcd. 56.7%). The remaining weight corresponds to UO_3 [57]. The differential thermal analysis (DTA) of 1 displays one distinct exothermic peak at 396 °C and three distinct endothermic peaks at 410, 574 and 691 °C.

Description of the Molecular Structures of $[\text{UO}_2(\mu\text{-DMP})_2(\text{TMP})]_n$ (1)

Crystallographic data for this complex are given in

Table 1 and selected bond lengths and angles are presented in Table 2. This complex crystallizes in the monoclinic crystal system with $P2_1/a$ space group. The molecular structure of 1 together with the atomic labeling scheme is shown in Fig. 2. The structure of 1 consists of a $[\text{UO}_2(\mu\text{-DMP})_2(\text{TMP})]$ fragment and a repetition unit in this one-dimensional polymer. An ORTEP plot (Fig. 2a) shows that the complex consists of one uranyl(VI) cation, which is linked to other fragments *via* four O-P-O bridges of four dimethyl phosphate esters. In this polymeric complex, the

Table 1. Crystallographic and Structural Refinement Data for 1

Formula	C ₇ H ₂₁ O ₁₄ P ₃ U
Formula weight	660.18
Temperature (K)	298(2)
Wavelength λ (Å)	0.71073
Crystal system	Monoclinic
Space group	<i>P2₁/a</i>
Crystal size (mm ³)	0.30 × 0.36 × 0.60
<i>a</i> (Å)	12.1195(12)
<i>b</i> (Å)	25.913(3)
<i>c</i> (Å)	12.7698(14)
β (°)	106.530(8)
Volume (Å ³)	3844.6(7)
<i>Z</i>	8
Density (calc.) (g cm ⁻³)	2.281
θ ranges for data collection	1.57-25.69
F(000)	2496
Absorption coefficient mm ⁻¹	8.756
Index ranges	-14 ≤ <i>h</i> ≤ 14 -31 ≤ <i>k</i> ≤ 31 -15 ≤ <i>l</i> ≤ 15
Data collected	7309
Unique data (<i>R</i> _{int})	7309, (0.0780)
Parameters, restraints	446, 0
Final <i>R</i> ₁ , <i>wR</i> ₂ (Obs. data)	0.0353, 0.0774
Final <i>R</i> ₁ , <i>wR</i> ₂ (All data)	0.0493, 0.0829
Goodness of fit on <i>F</i> ² (S)	1.050
Largest diff peak and hole (e Å ⁻³)	1.141, -0.911
CCDC No.	608616

Table 2. Selected Bond Lengths (Å) and Angles (°) for 1

U1-O1	2.463(5)	O19-U2-O26	73.7(2)
U1-O5	2.352(5)	O19-U2-O27	88.1(3)
U1-O8	2.340(5)	O19-U2-O28	92.9(3)
U1-O11	2.358(6)	O22-U2-O25	72.06(19)
U1-O12	2.351(6)	O22-U2-O26	146.48(19)
U1-O13	1.750(6)	O22-U2-O27	91.6(3)
U1-O14	1.753(6)	O22-U2-O28	89.0(2)
P1-O2	1.544(10)	O15-U2-O19	144.7(2)
P1-O1	1.414(6)	O15-U2-O22	142.3(2)
P1-O3	1.516(8)	O15-U2-O25	70.4(2)
P1-O4	1.566(8)	O15-U2-O26	71.1(2)
O1-U1-O5	71.1(2)	O15-U2-O27	90.5(3)
O1-U1-O8	142.9(2)	O15-U2-O28	88.4(2)
O1-U1-O11	144.0(2)	O27-U2-O28	178.9(3)
O1-U1-O12	71.0(2)	O25-U2-O28	91.2(3)
O1-U1-O13	90.6(2)	O25-U2-O26	141.45(18)
O1-U1-O14	88.8(2)	O25-U2-O27	88.3(3)
O5-U1-O8	71.79(18)	O26-U2-O27	90.4(3)
O5-U1-O11	144.9(2)	O26-U2-O28	89.5(3)
O5-U1-O12	142.0(2)	O1-P1-O3	118.2(4)
O5-U1-O13	90.5(3)	O1-P1-O2	117.8(5)
O5-U1-O14	89.3(3)	O2-P1-O4	97.8(5)
O8-U1-O11	73.16(19)	O1-P1-O4	108.5(4)
O8-U1-O12	146.1(2)	O2-P1-O3	102.4(5)
O8-U1-O13	89.6(2)	O3-P1-O4	110.3(5)
O8-U1-O14	90.9(2)	O5-P2-O11 ^b	118.4(3)
O11-U1-O12	73.0(2)	O5-P2-O6	110.1(6)
O11-U1-O13	90.0(3)	O5-P2-O7	110.4(5)
O11-U1-O14	90.5(3)	O7-P2-O11 ^b	111.7(6)
O12-U1-O13	88.6(3)	O6-P2-O7	99.3(8)
O12-U1-O14	91.2(3)	O6-P2-O11 ^b	105.1(6)
O13-U1-O14	179.4(2)	O8-P3-O10	109.1(4)
O19-U2-O22	73.0(2)	O8-P3-O12 ^b	116.1(4)
O19-U2-O25	144.7(2)		

Symmetry code: (b) 1/2+x,1/2-y,z.

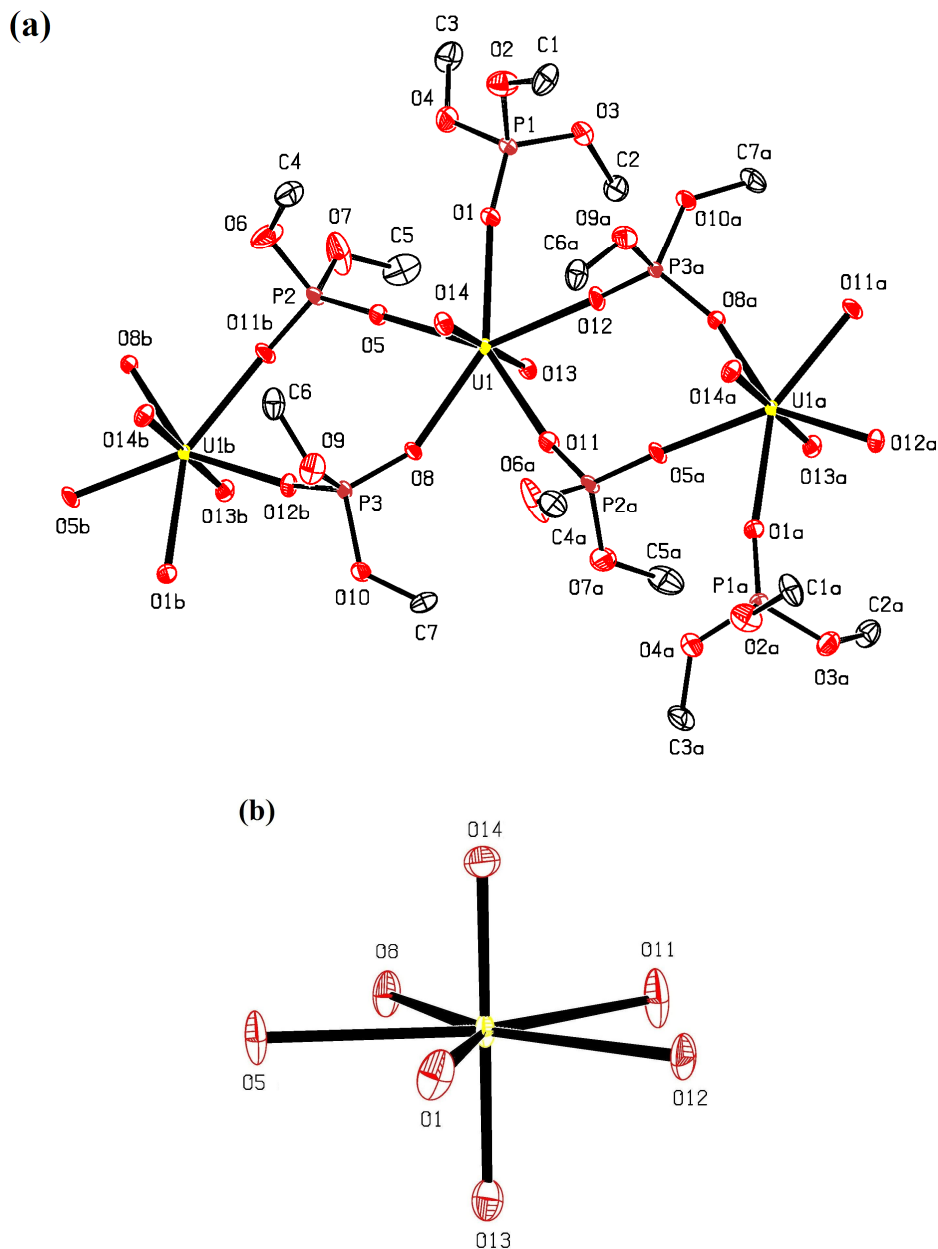


Fig. 2. (a) ORTEP drawing (30% probability displacement ellipsoids) of complex 1 with the atom numbering scheme. Hydrogen atoms are omitted for clarity. Symmetry codes: (a) $-1/2+x, 1/2-y, z$ and (b) $1/2+x, 1/2-y, z$. (b) Local coordination environment of U(VI) in 1.

uranium(VI) is seven-coordinated in a pentagonal bipyramidal configuration by two axial uranyl oxygens, four bridged dimethyl phosphate and monodentate trimethyl phosphate ligands (Fig. 2b). The U-O and P-O bond lengths

(Table 2) are in agreement with the corresponding ones in $[\text{UO}_2(\mu\text{-DEP})_4(\text{DMSO})]_n$ (DEP is diethyl phosphate) [30] and $[\text{UO}_2(\mu\text{-DMP})_4(\text{DMSO})]_n$ [32]. The phosphate groups in 1 are arranged as an abnormal distorted tetrahedron. The

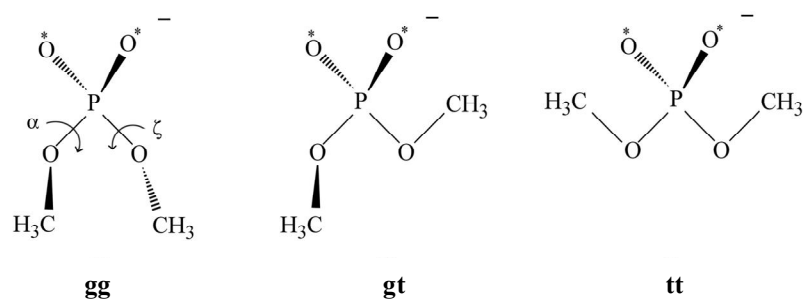


Fig. 3. Gauche-gauche (gg), gauche-trans (gt) and trans-trans (tt) conformation of dimethyl phosphate (DMP) anion.

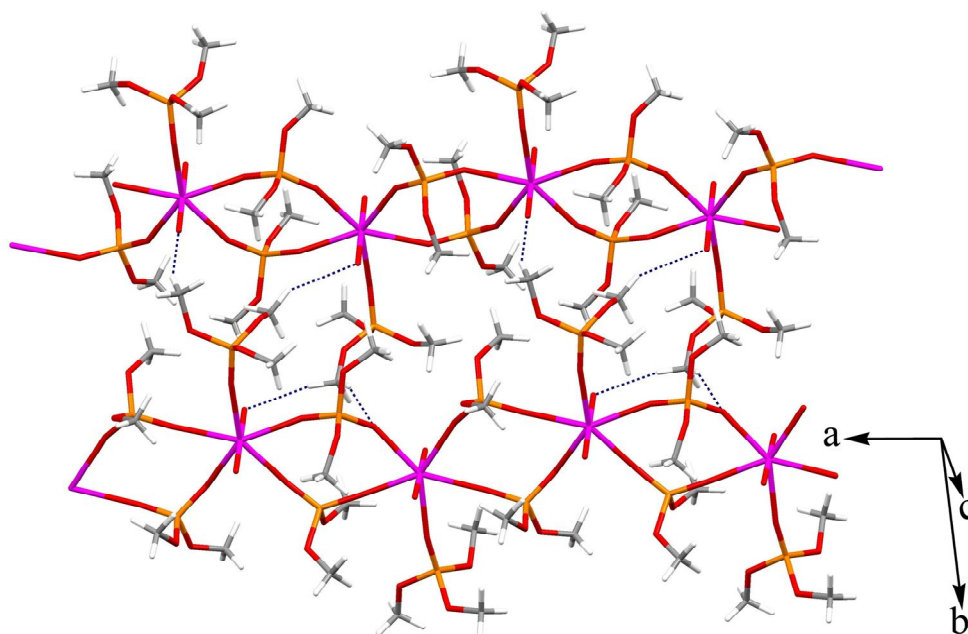


Fig. 4. Crystal packing diagram for 1. Intermolecular C-H...O hydrogen bonds are shown as dashed lines.

Table 3. Hydrogen Bond Geometry for 1 in Crystal Packing

D-H...A	D-H (Å)	H...A (Å)	D...A (Å)	D-H...A (°)	Symmetry code
C7-H7B...O8	0.96	2.470	2.890(12)	107	-
C8-H8C...O13	0.96	2.560	3.468(18)	158	1-x,-y,-z
C12-H12C...O9	0.96	2.460	3.308(17)	147	-1/2+x,1/2-y,z
C13-H13C...O24	0.96	2.480	2.81(2)	100	-
C14-H14A...O27	0.96	2.460	3.298(12)	145	2-x,-y,1-z

angles of O-P-O and MeO-P-OMe are unequal, the angle of the MeO7-P2-O6Me moiety is $99.3(8)^\circ$, whereas that of O11^b-P2-O5 (b is 1/2+x, 1/2-y, z) is $118.4(3)^\circ$, according to an inspection on similar polymeric complexes [30,37], an acceptable reason is simultaneous z-in and z-out distortions for the DMP ligands. In addition, the conformation of the DMP anion depends on the C-O-P-O angles. For description of the different phosphate conformations researchers adopt notation commonly used in spectroscopy. This convention donates conformations with COPO (α,ζ) torsional angles of $\sim \pm 60^\circ$ as gauche (g) and those of $\sim \pm 180^\circ$ as trans (t). So the conformations were showed as gauche-gauche (gg), gauche-trans (gt), and trans-trans (tt), (Fig. 3). The energies of different conformers of DMP are calculated in order to determine the relative stabilities of the (gg), (gt), and (tt) conformations with a variety of theoretical models [53,58-60]. These studies of aqueous solution of DMP have shown that the (gg) conformer to be the most stable followed by (gt) and (tt) conformations [59,60].

In complex 1, we expectedly observed the gt (gauche, trans) conformation for the DMP bridged on the dihedral angles. At this complex, for example, the dihedral angles of one of the DMP ligands are: C5-O7-P2-O6 = $156.8(14)^\circ$ and C4-O6-P2-O7 = $-99.5(14)^\circ$.

Two U=O bond lengths in complex 1 are located in axial position (Fig. 2b). The axial U=O bond lengths (U1-O13 = $1.750(6)$ Å and U1-O14 = $1.753(6)$ Å) are shorter than equatorial U-O bond lengths: U1-O1 = $2.463(5)$ Å, U1-O5 = $2.352(5)$ Å, U1-O8 = $2.340(5)$ Å, U1-O11 = $2.358(6)$ Å and U1-O12 = $2.351(5)$ Å. The U=O bond lengths in title complex as expected for seven-coordinate uranyl complexes [61,62].

In Fig. 4, the crystal packing diagram for title complex is shown. As show in this figure, in the crystal structure of the title complex intermolecular C-H...O hydrogen bonds (Table 3) between the one-dimensional coordination polymers are effective in the stabilization of the crystal structure and the formation of a three-dimensional supramolecular structure.

CONCLUSIONS

In this paper, a new one-dimensional uranium(VI) coordination polymer, $[\text{UO}_2(\mu\text{-DMP})_2(\text{TMP})]_n$ (1) has been

synthesized and characterized by infrared spectroscopic technique. The structure of this complex was defined by the single crystal diffraction method. The X-ray structural analysis specified that in this complex, the uranium(VI) is seven-coordinated in a pentagonal bipyramidal configuration by two axial uranyl oxygens (O=U=O), four bridged dimethyl phosphate and monodentate trimethyl phosphate ligands. The crystal structure of this complex has been stabilized by intermolecular C-H...O hydrogen bonds between the one-dimensional coordination polymers. Furthermore, the thermal stability of 1 was studied by thermogravimetric and differential thermal analyses.

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