
Araştırma Makalesi / Research Article

Synthesis and Characterization of Dioxouranium (VI) Complexes of Schiff Bases (Mixed-Ligands Part 1)

Erdal CANPOLAT*¹

¹Faculty of Education, Department of Science and Mathematics for Secondary Education,
University of Firat, 23119, Elazığ, Turkey

Abstract

New series of mixed-ligands complexes of dioxouranium (VI) with the Schiff bases and H₂O, py, DMF, bipy, phen and Et₃N have been synthesized. The elemental analyses, IR, ¹H-NMR and ¹³C-NMR spectra, electronic spectra, magnetic susceptibility measurements and thermogravimetric analyses were used in the synthesis of complexes. The general compositions of the complexes are [(L¹)(UO₂)(OAc)(H₂O)].2H₂O (**1**), [(L²)(UO₂)(OAc)(py)].H₂O (**2**), [(L³)(UO₂)(OAc)(DMF)].2H₂O (**3**), [(L⁴)(UO₂)(OAc)(bipy)].H₂O (**4**), [(L⁵)(UO₂)(OAc)(phen)].H₂O (**5**) and [(L⁶)(UO₂)(OAc)(Et₃N)].3H₂O (**6**).

Keywords: Schiff bases, UO₂(VI) complexes of Schiff bases.

Schiff Bazlarının Dioksouranyum (VI) Komplekslerinin Sentezi ve Karakterizasyonu (Karışık Ligandlar Bölüm 1)

Özet

Schiff Bazları ve piridin, dimetilformamid, 2,2'-bipridin, 1,10-fenantrolin yada trietilamin ile dioksouranyum (VI)'un yeni seri karışık-ligand kompleksleri sentezlenmiştir. Komplekslerin sentezinde elementel analiz, İnfrared, proton NMR, karbon-13 NMR, elektronik spektrum, manyetik süssebtilite ve termogravimetric analiz kullanılmıştır. Komplekslerin genel formülü [(L¹)(UO₂)(OAc)(H₂O)].2H₂O (**1**), [(L²)(UO₂)(OAc)(py)].H₂O (**2**), [(L³)(UO₂)(OAc)(DMF)].2H₂O (**3**), [(L⁴)(UO₂)(OAc)(bipy)].H₂O (**4**), [(L⁵)(UO₂)(OAc)(phen)].H₂O (**5**) and [(L⁶)(UO₂)(OAc)(Et₃N)].3H₂O (**6**). şeklindedir.

Anahtar Kelimeler: Schiff Bazları, Schiff Bazlarının UO₂(VI) Kompleksleri

1. Introduction

Schiff bases are important class of ligands having many applications They possess various biological activities such as fungicidal and insecticidal [1]. Chelating ligands containing O and N donor atoms show broad biological activity and are of special interest because of the variety of ways in which they are bonded to metal ions[2]. Aromatic hydroxyaldehydes form stable complexes and the presence of a phenolic hydroxyl group at their opposition imparts an additional donor site in the molecule making it bidentate. Such a molecule coordinates with the metal ion through the carbonyl oxygen and deprotonated hydroxyl group. The chelating properties of Schiff bases derived from o-hydroxyaldehydes are well established [3]. A large number of mixed-ligand complexes involving heterocyclic bases such as pyridine, 2,2'-bipyridine, o-phenanthroline, py, DMF, Et₃N etc. were reported by many workers [4–6] due to their bio-inorganic applications and thermal stability. The complexes make these compounds effective and stereospecific catalysts for oxidation, reduction and hydrolysis and they show biological activity, and other transformations of organic and inorganic chemistry [7]. It is well known that some drugs have higher activity when administered as metal complexes than as free ligands [8].

* Corresponding author: eecanpolat@gmail.com

In this study, we synthesized bidentate Schiff base ligands and their mixed-ligands dioxouranium (VI) complexes. All of the complexes were characterized by elemental analyses, IR, $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ spectra, electronic spectra, magnetic susceptibility measurements and thermogravimetric analyses.

2. Experimental

2.1. Measurements

Elemental analyses (C, H, N) were carried out using LECO-932 CHNSO by Technical and Scientific Research Council of Turkey, TUBITAK. IR spectra were recorded on a Mattson 1000 FT-IR Spectrometer as KBr pellets. $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ spectra were recorded on a Bruker GmbH Dpx-400 MHz High Performance Digital FT-NMR Spectrometers. UV-Vis measurements were done using a Shimadzu UV Probe 2.1. Magnetic susceptibilities were determined on a Sherwood Scientific Magnetic Susceptibility Balance (Model MK1) at room temperature using $\text{Hg}[\text{Co}(\text{SCN})_4]$ as a calibrate; diamagnetic corrections were calculated from Pascal's constants. TGA curves were recorded on a Shimadzu TG-50 thermo balance.

2.2. Preparation of Ligands

The synthesis and characterization of the ligands used in this study have been described previously [9-14].

2.3. Preparation of Dioxouranium (VI) Complexes

The uranyl Schiff base complex $[(\text{L}^1)(\text{UO}_2)(\text{OAc})(\text{H}_2\text{O})].2\text{H}_2\text{O}$, was prepared from the addition of uranyl acetate dihydrate (3 mmol) in ethanol of the Schiff base (3 mmol) in ethanol. The mixture was then stirred at 50°C temperature for about 7h. The product was filtered off, washed several times with H_2O , cold EtOH and dried under vacuum. The other five complexes $[(\text{L}^2)(\text{UO}_2)(\text{OAc})(\text{py})].\text{H}_2\text{O}$, $[(\text{L}^3)(\text{UO}_2)(\text{OAc})(\text{DMF})].2\text{H}_2\text{O}$, $[(\text{L}^4)(\text{UO}_2)(\text{OAc})(\text{bipy})].\text{H}_2\text{O}$, $[(\text{L}^5)(\text{UO}_2)(\text{OAc})(\text{phen})].\text{H}_2\text{O}$, and $[(\text{L}^6)(\text{UO}_2)(\text{OAc})(\text{Et}_3\text{N})].3\text{H}_2\text{O}$ were prepared in a similar way to that described above by the reaction of $\text{UO}_2(\text{CH}_3\text{COO})_2.2\text{H}_2\text{O}$ and the Schiff base together with pyridine, dimethylformamide, 2,2' bipyridine, 1,10-phenanthroline or triethylamine, respectively in a molar ratio 1:1 using ethanol as a solvent.

3. Results and Discussion

In this study, six Schiff bases ligands used salicyliden-*p*-aminoacetophenoneoxime, 5-bromosalicyliden-*p*-aminoacetophenoneoxime, 5-nitrosalicyliden-*p*-aminoacetophenoneoxime, 3-methoxysalicyliden-*p*-aminoacetophenoneoxime, 3-hydroxysalicyliden-*p*-aminoacetophenoneoxime and naphthyliden-*p*-aminoacetophenoneoxime were obtained in good yield by literature methods [9-14]. The Schiff bases (mixed ligands) were used for the complexation reaction with dioxouranium (VI). The results of the elemental analyses data show that the metal to ligand ratio in all the complexes is 1:1. The complexes were of the general formula $[(\text{L}^1)(\text{UO}_2)(\text{OAc})(\text{H}_2\text{O})].2\text{H}_2\text{O}$ (**1**), $[(\text{L}^2)(\text{UO}_2)(\text{OAc})(\text{py})].\text{H}_2\text{O}$ (**2**), $[(\text{L}^3)(\text{UO}_2)(\text{OAc})(\text{DMF})].2\text{H}_2\text{O}$ (**3**), $[(\text{L}^4)(\text{UO}_2)(\text{OAc})(\text{bipy})].\text{H}_2\text{O}$ (**4**), $[(\text{L}^5)(\text{UO}_2)(\text{OAc})(\text{phen})].\text{H}_2\text{O}$ (**5**) and $[(\text{L}^6)(\text{UO}_2)(\text{OAc})(\text{Et}_3\text{N})].3\text{H}_2\text{O}$ (**6**). The analytical data of the complexes are given in Table 1. The probable structure of the complexes is shown in Figure 1. The complexes were characterized by the following physical studies.

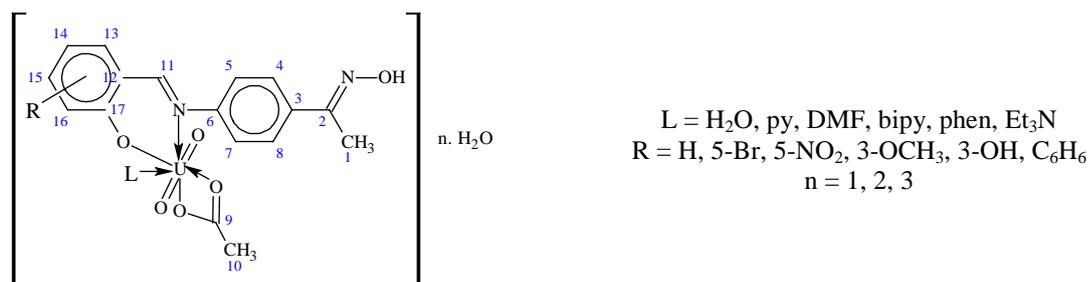


Figure 1. Suggested structure of the dioxouranium (VI) omplexes

The characteristic IR bands of the ligands and their complexes as KBr pellets are given in Table 2. The ligands contain four potential donor sites: **1**) the phenolic oxygen, **2**) the azomethine nitrogen, **3**) the oxime oxygen, **4**) the oxime nitrogen. In the IR spectrum of the dioxouranium (VI) complexes, the $\nu(\text{C}=\text{N})$ azomethine stretching band appearing at *ca.* 1617-1644 cm^{-1} in the ligands [9-14] are shifted *ca.* 1599-1619 cm^{-1} for the complexes. At the same time, the $\nu(\text{C}-\text{O})$ phenolic band at *ca.* 1210-1310 cm^{-1} in the free ligands [9-14], was moved to a lower frequency of at *ca.* 11-88 cm^{-1} after complexes formation. These suggest that the ligands are coordinated to metal ions through the nitrogen and oxygen donors. The practically unchanged $\nu(\text{O}-\text{H})$ oxime) at *ca.* 3245-3455 and $\nu(\text{C}=\text{N})$ oxime at *ca.* 1558-1617 cm^{-1} confirm that the oxime group itself does not coordinate to metal atoms by neither oxygen nor nitrogen atoms [15,16]. The coordination of an acetato group in the complexes are indicated by the appearance of two new bands in the regions 1649-1671 and 1320-1360 cm^{-1} assignable to $\nu(\text{COO})$ asymmetric and $\nu(\text{COO})$ symmetric modes, respectively [17]. In the dioxouranium (VI) complexes two additional sharp bands are observed at *ca.* 910-940 and 805-820 cm^{-1} which are assigned to $\nu(\text{UO}_2)$ asymmetric and $\nu(\text{UO}_2)$ symmetric modes, respectively. These observation suggests that the $\text{O}=\text{U}=\text{O}$ moiety are virtually linear in these complexes [18]. The IR spectrum of the **(1)** complex shows differences compared with the spectra of the other complexes. Beside the medium band at *ca.* 3540 cm^{-1} which is characteristic for the coordinated H_2O , there is a medium band at 1630 cm^{-1} as expected for bending vibrations of coordinated H_2O [19]. These two bands are absent from the spectra of the other complexes. The **(3)** complex in its IR spectrum show a very strong absorption band at 1666 cm^{-1} characteristic for DMF. The IR spectra of **(4)** and **(5)** complexes show a change in the ring (=N-) nitrogen frequencies of (bipy) and (phen). The bands in bipy and phen at *ca.* 1570-1575 and 445-460 cm^{-1} , respectively in **(4)** and **(5)** complexes. The spectra of the complexes **(2)** and **(6)** show the $\nu(\text{U}-\text{N})$ (N of py and Et_3N) at 437 and 460 cm^{-1} , respectively.

Table 1. Analytical and physical data of the dioxouranium (VI) complexes

Compounds	Formula (F.W) g/mol^{-1}	Yield (%)	Calculated (Found) %		
			C	H	N
(1) $[(\text{L}^1)(\text{UO}_2)(\text{OAc})(\text{H}_2\text{O})].2\text{H}_2\text{O}$	$\text{C}_{17}\text{H}_{22}\text{N}_2\text{O}_9\text{U}$ (636.39)	71	32.08 (31.84)	3.48 (3.63)	4.40 (4.05)
(2) $[(\text{L}^2)(\text{UO}_2)(\text{OAc})(\text{py})].\text{H}_2\text{O}$	$\text{C}_{22}\text{H}_{22}\text{N}_3\text{O}_7\text{BrU}$ (758.36)	62	34.84 (35.02)	2.92 (3.01)	5.54 (5.83)
(3) $[(\text{L}^3)(\text{UO}_2)(\text{OAc})(\text{DMF})].2\text{H}_2\text{O}$	$\text{C}_{20}\text{H}_{26}\text{N}_4\text{O}_{11}\text{U}$ (736.47)	65	32.62 (32.80)	3.56 (3.62)	7.61 (7.64)
(4) $[(\text{L}^4)(\text{UO}_2)(\text{OAc})(\text{bipy})].\text{H}_2\text{O}$	$\text{C}_{28}\text{H}_{28}\text{N}_4\text{O}_8\text{U}$ (786.57)	59	42.76 (43.12)	3.59 (3.97)	7.12 (6.99)
(5) $[(\text{L}^5)(\text{UO}_2)(\text{OAc})(\text{phen})].\text{H}_2\text{O}$	$\text{C}_{29}\text{H}_{26}\text{N}_4\text{O}_8\text{U}$ (796.57)	54	43.73 (44.09)	3.29 (2.95)	7.03 (6.87)
(6) $[(\text{L}^6)(\text{UO}_2)(\text{OAc})(\text{Et}_3\text{N})].3\text{H}_2\text{O}$	$\text{C}_{27}\text{H}_{39}\text{N}_3\text{O}_9\text{U}$ (787.64)	68	41.17 (40.97)	4.99 (4.61)	5.33 (5.04)

Table 2. Characteristic IR bands of the dioxouranium (VI) complexes as KBR pellets (in cm^{-1}).

Compounds	1	2	3	4	5	6
$\nu(\text{O}-\text{H})/\text{H}_2\text{O}$	3290-3540	3300-3440	3285-3450	3250-3410	3245-3455	3300-3435
$\nu(\text{C}=\text{N})_{\text{azomethine}}$	1611	1605	1619	1612	1614	1599
$\nu(\text{C}=\text{N})_{\text{oxime}}$	1597	1586	1589	1593	1590	1585
$\nu(\text{C}-\text{O})$	1319	1312	1321	1308	1298	1310
$\nu(\text{N}-\text{O})$	1008	1006	1009	1004	1001	1005
$\nu_{\text{as}}(\text{COO})_{\text{acetato}}$	1658	1664	1671	1649	1652	1660
$\nu_{\text{s}}(\text{COO})_{\text{acetato}}$	1334	1342	1360	1320	1331	1336
$\nu(\text{O}=\text{U}=\text{O})$	896	899	905	908	913	910
$\nu(\text{M}-\text{O})$	555	561	568	564	559	551
$\nu(\text{M}-\text{N})$	452	448	439	451	447	443
Others		437 (U-N) Py	1666 DMF	1575 (=N-), 460 Bipy	445, 1570 Phen	460 (U-N) (Et_3N)

The ^1H and ^{13}C -NMR spectra of the ligands and dioxouranium (VI) complexes were recorded in DMSO- d_6 . The ^1H and ^{13}C -NMR chemical shifts for the ligands and dioxouranium (VI) complexes are listed in Table 3 and 4. Characteristic ^1H -NMR peaks of ligands occur at *ca.* 13.09-13.99 $\delta(\text{OH})$ phenolic, 10.74-11.46 $\delta(\text{OH})$ oxime, 8.62-9.53 $\delta(\text{N}=\text{CH})$, 6.79-7.76 $\delta(\text{Arom}-\text{H})$ and 2.16-2.20 ppm $\delta(-\text{CH}_3)$ [13,15]. As can be seen in the ^1H -NMR spectra of complexes there is no OH peaks expected. The absence of the phenolic $\delta(\text{OH})$ proton signals in the complexes indicates the coordination by phenolic oxygen to the metal ion after deprotonation [20]. The coordination of the azomethine nitrogen is inferred by the upfield shifting of the $\delta(\text{CH}=\text{N})$ proton signal from 8.62-9.53 ppm in the ligands to 8.55-9.11 ppm in the complexes [21]. In the ^1H -NMR spectrums the complexes show the presence of a coordinated acetate molecule at *ca.* 1.99-2.07 ppm. ^1H -NMR spectrum of the complexes have $\delta(\text{H}_2\text{O})$ proton at *ca.* 3.1-3.4 ppm. The new signals belonging to py; DMF; bipy; phen and Et_3N groups were listed in Table 3.

Table 3. ^1H -NMR spectra of the dioxouranium (VI) complexes in DMSO- d_6 in $\delta(\text{ppm})$

Compounds	δOH	$\delta \text{N}=\text{CH}$	$\delta \text{Ar}-\text{H}$	$\delta -\text{CH}_3$	δ	
					OOCCH_3	Others
1	10.74 (s, 1H)	8.58 (s, 1H)	6.85-7.70 (m, 8H)	2.20 (s, 3H)	2.05 (s, 3H)	3.3 (H_2O)
2	10.78 (s, 1H)	8.63 (s, 1H)	6.89-7.74 (m, 8H)	2.14 (s, 3H)	2.01 (s, 3H)	3.4 (H_2O) 7.21; 7.59; 8.55 (py)
3	10.89 (s, 1H)	8.91 (s, 1H)	6.81-7.60 (m, 8H)	2.17 (s, 3H)	1.99 (s, 3H)	3.2 (H_2O) 2.78; 2.89; 7.91 (DMF)
4	11.05 (s, 1H)	9.08 (s, 1H)	6.97-7.84 (m, 8H)	2.22 (s, 3H)	2.03 (s, 3H)	3.3 (H_2O) 3.84 ($-\text{OCH}_3$) ^a 7.30; 7.79; 8.29; 8.68 (bipy)
5	11.14 (s, 1H)	9.01 (s, 1H)	6.80-7.75 (m, 8H)	2.15 (s, 3H)	2.07 (s, 3H)	3.1 (H_2O) 8.90 ($-\text{OH}$) ^b 7.61; 7.79; 8.24; 9.16 (phen)
6	10.80 (s, 1H)	8.71 (s, 1H)	6.74-7.65 (m, 8H)	2.25 (s, 3H)	2.00 (s, 3H)	3.4 (H_2O) 1.08; 2.56 (Et_3N)

Table 4. ^{13}C -NMR spectra of the dioxouranium (VI) complexes in DMSO- d_6 in $\delta(\text{ppm})$

1	11.30 (C_1), 21.37 (C_{10}), 106.07 (C_9), 116.58 (C_{16}), 118.79 (C_{14}), 119.29 (C_{12}), 120.72 (C_5 and C_7), 126.49 (C_4 and C_8), 132.19 (C_{13}), 132.97 (C_{15}), 135.73 (C_3), 148.00 (C_2), 152.81 (C_6), 160.59 (C_{11}), 162.48 (C_{17})
2	11.52 (C_1), 22.01 (C_{10}), 109.11 (C_9), 109.93 (C_{14}), 114.49 (C_{16}), 118.49 (C_{12}), 120.10 (C_5 and C_7), 123.59 (py), 126.33 (C_4 and C_8), 134.00 (C_{13}), 135.09 (C_3), 135.98 (C_{15}), 136.22 (py), 147.93 (C_2), 150.11 (py), 154.11 (C_6), 161.51 (C_{11}), 163.99 (C_{17})
3	11.21 (C_1), 21.76 (C_{10}), 31.50 (DMF), 35.71 (DMF), 108.66 (C_9), 121.37 (C_{12}), 122.49 (C_{16}), 123.95 (C_5 and C_7), 127.61 (C_{13}), 129.98 (C_4 and C_8), 132.44 (C_{15}), 135.70 (C_3), 137.13 (C_{14}), 147.58 (C_2), 153.67 (C_6), 161.62 (C_{11}), 162.59 (DMF), 163.11 (C_{17})
4	11.41 (C_1), 21.55 (C_{10}), 55.51 (C_a), 105.43 (C_9), 108.37 (C_{14}), 112.93 (C_{12}), 119.98 (C_5 and C_7), 120.53 (bpy), 121.80 (bpy), 121.99 (C_{13}), 123.64 (C_{15}), 133.26 (C_4 and C_8), 135.56 (C_3), 136.77 (bpy), 144.45 (C_2), 149.66 (bpy), 150.75 (C_{16}), 153.29 (C_6), 155.89 (C_{17}), 156.51 (bpy), 170.01 (C_{11})
5	11.88 (C_1), 21.69 (C_{10}), 101.17 (C_9), 119.19 (C_{13}), 119.58 (C_{14}), 119.91 (C_{12}), 121.35 (phen), 121.79 (C_5 and C_7), 123.33 (C_{15}), 126.50 (phen), 127.00 (C_4 and C_8), 129.00 (phen), 135.92 (phen), 136.00 (C_3), 146.09 (phen), 146.20 (C_{16}), 148.47 (C_2), 150.02 (C_6), 150.21 (phen), 152.79 (C_{17}), 164.44 (C_{11})
6	11.35 (C_1), 12.84 (Et_3N), 21.83 (C_{10}), 49.24 (Et_3N), 107.33 (C_9), 119.79 (C_{12}), 121.45 (C_{14}), 123.15 (C_{16}), 123.86 (C_5 and C_7), 123.97 (C_{20}), 127.90 (C_{15}), 128.59 (C_{18}), 128.91 (C_{17}), 132.90 (C_4 and C_8), 133.33 (C_{13}), 136.29 (C_{19}), 137.69 (C_3), 147.99 (C_2), 154.08 (C_6), 163.00 (C_{11}), 165.78 (C_{21})

More detailed information about the structure of the ligands was provided by ^{13}C -NMR spectral data $\delta(\text{C}-\text{OH})$, $\delta(\text{CH}=\text{N})$ and $\delta(\text{C}-\text{N})$ carbon atoms are observed at *ca.* 152.88-163.09, 159.74-166.45 and 149.88-153.75 ppm respectively for ligands. ^{13}C -NMR spectra of the complexes $\delta(\text{C}-\text{OH})$, $\delta(\text{CH}=\text{N})$, $\delta(\text{C}-\text{N})$ $\delta(\text{CH}_3\text{COO})$ and $\delta(\text{CH}_3\text{COO})$ carbon atoms are observed at *ca.* 152.77-162.99,

159.62-166.35, 149.81-153.69, 101.17-109.11 and 21.37-21.83 ppm respectively. The rest of carbon atoms likewise showed similar diagnostic features for the free ligands as well as their complexes as expected. The signals corresponding to the $\delta(\text{OH})$ proton and $\delta(\text{CH}=\text{N})$ carbon (both in oxime) [22] groups are unchanged in the ^1H and ^{13}C -NMR spectra of the complexes, indicating that these oxime groups do not take part in complexation. The ^{13}C -NMR chemical shifts for the py, DMF, bipy, phen and Et_3N groups are listed in Table 4.

The dioxouranium (VI) complexes were found to be diamagnetic as expected and did not give any significant values for the magnetic moment.

The diffuse reflectance spectrums of complexes two spectral band at *ca.* 260-269 nm ($\epsilon = 550\text{-}561 \text{ L mol}^{-1} \text{ cm}^{-1}$) and 330-341 nm ($\epsilon = 396\text{-}418 \text{ L mol}^{-1} \text{ cm}^{-1}$) assignable to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transition, respectively, of the azomethine linkage. The third band appearing at *ca.* 416-424 nm ($\epsilon = 380\text{-}399 \text{ L mol}^{-1} \text{ cm}^{-1}$) in these complexes are assigned to the $^1\Sigma_g^+ \rightarrow ^3\Pi_u$ transition typical of the O-U-O symmetric frequency, $\nu_s(\text{O}=\text{U}=\text{O})$, for the first excited state [23].

The Thermal behavior of the complexes has been investigated using thermogravimetric techniques in the temperature range from ambient to 800°C at a heating rate of 10°C/min. The TGA curve of complexes shows a first weight loss was observed at *ca.* 95-120°C due to elimination of lattice water [24] molecules (Found/Calcd. %; 5.66/6.09, two water molecules for (1), Found/Calcd. %; 2.38/1.87, one water molecules for (2), Found/Calcd. %; 4.89/5.65, one water molecules for (3), Found/Calcd. %; 2.29/1.94, one water molecules for (4), Found/Calcd. %; 2.26/1.95, one water molecules for (5), Found/Calcd. %; 6.86/7.37, three water molecules for (6)). The complex (1) shows a further weight loss of 2.83% (calc. 3.22%) at 174 °C corresponding to removal of one coordinated water molecule [25]. The two weight loss was observed at *ca.* 276-319 °C (Found/Calcd. %; 7.78/8.16 for (2), Found/Calcd. %; 8.01/7.66 for (3), Found/Calcd. %; 7.50/7.81) for (4), Found/Calcd. %; 7.41/7.72 for (5), Found/Calcd. %; 7.49/7.88) for (6) and may be due to loss of one acetate group from the coordination sphere. The inflation of the TGA curves of all the complexes at a temperature below 750 °C indicates the decomposition of the fully organic part of the chelate, leaving the metallic oxide at the final temperature [26]. All the complexes undergo complete decomposition to the corresponding thermodynamically stable metal oxides, UO_2 (residue: 43.05% for (1), 36.18% for (2), 37.28% for (3), 33.88% for (4), 34.41% for (5), 33.90% for (6)).

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