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Structural and electrochemical studies on uranyl(VI) complex with pentadentate Schiff base ligand: A guide to stable uranyl(V)

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Abstract. A U(VI) complex with N,N’-disalicylidenediethylenetriamine (saldien2−) was characterized by single crystal X-ray analysis and X-ray absorption fine structure (XAFS) spectroscopy, and its electrochemical behavior in a DMSO solution was studied. The obtained U(VI)-saldien2− complex recrystallized from DMSO was identified as orthorhombic UVIO2(saldien)·DMSO. All coordination sites in the equatorial plane of UO22+ are occupied by coordinating O and N atoms of saldien2−. Comparing a k3-weighted U LIII-edge EXAFS spectra of UVIO2(saldien) in DMSO and DMF solutions with that in solid state, the molecular structure of UVIO2(saldien) remains even in the solutions. Quasi-reversible redox waves of UVIO2(saldien) were observed at E°′ = −1.582 ± 0.005 V vs. Fc/Fc+ (ΔEp = 0.080-0.170 V at v = 0.010-0.500 V·s−1) in DMSO. UV-Vis-NIR absorption spectral change with the electrochemical reduction of UVIO2(saldien) using the spectroelectrochemical technique shows isosbestic points clearly, indicating that the electrochemical reaction of UVIO2(saldien) is only present. Using the absorbance change and the Nernstian equation, the electron stoichiometry in the reduction of UVIO2(saldien) remains even in the solutions. Quasi-reversible redox waves of UVIO2(saldien) were observed at E°′ = −1.582 ± 0.005 V vs. Fc/Fc+ (ΔEp = 0.080-0.170 V at v = 0.010-0.500 V·s−1) in DMSO. UV-Vis-NIR absorption spectral change with the electrochemical reduction of UVIO2(saldien) using the spectroelectrochemical technique shows isosbestic points clearly, indicating that the electrochemical reaction of UVIO2(saldien) is only present. Using the absorbance change and the Nernstian equation, the electron stoichiometry in the reduction of UVIO2(saldien) in DMSO was determined as 0.929. This quantity close to unity reveals a redox reaction, UVIO2(saldien) + e− = [UVO2(saldien)]−, i.e., UVIO2(saldien) without unidentate ligands results in the stable U(V) complex, [UVO2(saldien)]−, in DMSO. This U(V) species also shows the characteristic absorption bands of U(V) at 630, 700, 830, 1390, and 1890 nm as well as other U(V) complexes.

1. Introduction
Uranium(V) has only 1 unpaired electron in its 5f-orbital. This is one of the species with the simplest electronic configuration in actinides. Actinides (An) in oxidation states +5 and +6 form in many cases a typical “actinyl” ion, (AnO2n+). Few observations of pure U(V) are reported so far because of the unusual instability of U(V) towards disproportionation.[1,2]
To find a stable U(V) species in solution, we studied the electrochemistry of various U(VI) complexes with aqua, carbonate, β-diketonates and Schiff bases in aqueous or non-aqueous solvents in the last decades.[3-11] From these studies, we can conclude that (i) the U(V/VI) redox couple is reversible or quasireversible, (ii) the electroreduction forms relatively stable U(V) species, (iii) the resulting U(V) species are also presumably $UO_2^{2+}$, and that (iv) the coordination of multidentate ligand(s) tends to more stabilize its U(V) complex than unidentate ligands like a solvent molecule. Furthermore, a non-aqueous aprotic solvent provides an additional merit of exclusion of $H^+$ which initiates the U(V) disproportionation. Up to now, 2 stable U(V) complexes in non-aqueous solutions have been found, $[UO_2(salophen)DMSO]^-\text{in DMSO}$ (salophen = $N,N'$-disalicylidene-o-phenylenediaminate, DMSO = dimethyl sulfoxide) and $[UO_2(dbm)DMSO]^-\text{in DMSO}$ (dbm = dibenzoylmethanate).[7-11]

In the systems of $[UO_2(salophen)L]^-\text{(L = unidentate ligand as a coordinating solvent)}$, L tends to dissociate from U(V) with decreasing the concentration of L.[7] This results in the mixture of $[UO_2(salophen)L]$ and $[UO_2(salophen)]^-$, which disturbs observation of a “pure” U(V) complex. This reaction suggests an additional hint for stabilization of U(V), i.e., L should be excluded from the equatorial plane of U(V). The most probable coordination number in the equatorial plane of U(VI) is 5 or 6. On this basis, a penta- or hexadentate planar ligand should be appropriate to expel any L from the equatorial plane. As candidates of pentadentate ligand, $N,N'$-disalicylidenediethylene-triaminate (saldien$^2-$)[12-15] and superphthalocyanine[16-18] are available. In this study, we selected saldien$^2-$ because of simplicity of preparation, and used its U(VI) complex (Figure 1) as a starting material. The molecular structure of $UO_2(saliden)$ in solid and solution states and its electrochemical behavior were investigated here.

![Figure 1. Schematic structure of $UO_2(saliden)$.](image)

2. Experimental

2.1. Preparation of $UO_2(saliden)$ Complex

In ethanol (EtOH), $UO_2(NO_3)_2\cdot6H_2O$, diethylenetriamine (Aldrich) and salicylaldehyde (Fluka) in 1:1:2 molar ratio were mixed with vigorous stirring. The mixture has been refluxed at 78°C for 30 min, and then cooled to room temperature. Precipitate was filtered off, and washed with EtOH. The obtained material was dissolved in DMSO at ca. 130°C, and slowly cooled to room temperature overnight. Deposited crystals were recovered by filtration, washed with EtOH, and dried under reduced pressure. The resulting compound was identified as $UO_2(saliden)$-DMSO by single crystal X-ray analysis described below.

2.2. Analytical Methods

A single crystal of $UO_2(saliden)$-DMSO was mounted on a glass fiber and placed under N$_2$ gas stream at 173 K. Intensity data were collected by using imaging plate area detector in Rigaku RAXIS RAPID diffractometer with graphite monochromated Mo Kα radiation ($\lambda = 0.71075 \text{ Å}$). The structure of the U(VI) complex was solved by SIR92 direct method,[19] and expanded using Fourier techniques.[20] A numerical absorption correction was applied, which resulted in transmission factors described in the crystallographic information file (CIF).[21] All non-hydrogen atoms were
anisotropically refined by \textit{SHELXL-97}.\textsuperscript{[22]} Hydrogen atoms were refined as riding on their parent atoms with $U_{iso}(\text{H}) = 1.2U_{eq}(\text{C, N})$. The final cycle of full-matrix least-squares refinement on \textit{F}$_2$ was based on observed reflections and parameters, and converged with unweighted and weighted agreement factors, \textit{R} and \textit{wR}. All calculations were performed by the CrystalStructure crystallographic software package.\textsuperscript{[23]} Crystal data and other data collection parameters are summarized in Table 1. CCDC 739692 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

X-ray absorption fine structure (XAFS) spectroscopy experiments were performed at the Rossendorf Beamline (ROBL) BM20 at the European Synchrotron Radiation Facility (6 GeV; 70–90 mA).\textsuperscript{[24]} A Si(111) double-crystal monochromator was employed in channel-cut mode to monochromatize a white X-ray from the synchrotron. Uranium L$_{III}$-edge X-ray absorption spectra of the solid and solution samples were recorded in transmission mode by using Ar-filled ionization chambers at ambient temperature (295 K) and pressure. The X-ray energy in each experimental run was calibrated by Y foil (first inflection point at 17038 eV). The threshold energy, $E_{k=0}$, of U L$_{III}$-edge was defined at 17185 eV. The X-ray absorption spectrum of each sample was accumulated twice and merged. The obtained spectra were processed using Athena for background removal and extraction of EXAFS spectra, and Artemis for EXAFS curve fit.\textsuperscript{[25]} The curve fit was performed in the \textit{R} space, using phases and amplitude calculated by \textit{FEFF} 8.20.\textsuperscript{[26]} Single-scattering paths from oxygen, nitrogen and carbon, and multiple-scattering paths from the linear uranyl moiety were included in the EXAFS curve fit. The molecular structure of U$^{VI}$O$_2$(saldien) from single crystal X-ray analysis was used as initial structure model. The amplitude decay factor, $S_0^2$, was fixed at 0.9, and the shifts in the threshold energy, $\Delta E_0$, were constrained to be the same value for all shells.

Cyclic voltammetry was utilized to study the redox behavior of U$^{VI}$O$_2$(saldien) in DMSO. The CV measurements were carried out at 298 ± 1 K under dry argon atmosphere using BAS ALS660B. A three-electrode system consisted of a Pt disc working electrode (electrode surface area: 0.020 mm$^2$), a Pt wire counter electrode, and an Ag/AgCl reference electrode (3 M NaCl) with liquid junction filled with 0.1 M tetrabutylammonium perchlorate (TBAP, Fluka)/DMSO solution. A ferrocene/ferrocenium ion redox couple (Fc/Fc$^+$) was taken as the internal standard redox system.\textsuperscript{[27]} Dissolved O$_2$ in the sample solutions were removed by passing Ar gas for at least 10 min prior to starting experiments.

UV-Vis-NIR spectroelectrochemical measurements for [U$^{VI}$O$_2$(saldien)]$^-$/U$^{V}$O$_2$(saldien) in DMSO were performed with SHIMADZU UV-3150 spectrophotometer equipped with optical transparent thin layer electrode (OTTLE) cell.\textsuperscript{[28]} The optical path length of the cell was determined spectrophotometrically as 1.89 × 10$^{-2}$ cm. The three-electrode system was same as that in the CV experiment with replacement of the working electrode by a Pt gauze (80 mesh). The applied potential on OTTLE was controlled by BAS ALS660B. The electronic spectrum for each potential was recorded after equilibrium of the electrochemical reaction, which was completed within 2 min. The sample solution in the OTTLE cell was deoxygenated by passing dry Ar gas at least 1 h prior to the experiment.

\begin{table}[h]
\centering
\caption{Crystallographic Data of U$^{VI}$O$_2$(saldien)-DMSO}
\begin{tabular}{lll}
\hline
formula & C$_{20}$H$_{25}$N$_3$O$_5$SU & $T$ (K) 173 \\
formula weight & 657.52 & $D_{calcd}$ (g·cm$^{-3}$) 1.947 \\
crystal size (mm) & 0.12 × 0.18 × 0.55 & $\mu$ (mm$^{-1}$) 7.365 \\
crystal system & orthorhombic & $F_{000}$ 1256 \\
space group & $Pca2_1$ & obsd data (all) 5030 \\
a (Å) & 20.082(5) & \textit{R} (I > 2\sigma) 0.0230 \\
B (Å) & 10.396(8) & \textit{wR} (all) 0.0477 \\
c (Å) & 10.744(5) & GOF 1.066 \\
V (Å$^3$) & 2243(2) & $\Delta\rho_{max}$ (e·Å$^{-3}$) 0.738 \\
Z & 4 & $\Delta\rho_{min}$ (e·Å$^{-3}$) −0.539 \\
\hline
\end{tabular}
\end{table}

3
3. Results and Discussion

The ORTEP view of U\textsuperscript{VI}O\textsubscript{2}(saldien)-DMSO is shown in Figure 2. In this figure, coordination of 2 O and 3 N of saldien\textsuperscript{2−} is formed in the equatorial plane of U\textsuperscript{VI}O\textsubscript{2\textsuperscript{2+}}. As a result, geometry around U in U\textsuperscript{VI}O\textsubscript{2}(saldien) is pentagonal-bipyramidal. The bond distances between U and axial O atoms [U(1)−O(1), U(1)−O(2)] are 1.788(3) and 1.790(3) Å, which are typical values for U(VI) compounds. The O(1)−U(1)−O(2) bond angle, 176.2(2)°, indicates that the uranyl moiety is slightly bent in the direction to the coordination of the center N(2). In the equatorial plane, the U(1)−O(3), U(1)−O(4) bond distances [2.231(3), 2.220(4) Å] are shorter than U−N [U(1)−N(1): 2.586(4), U(1)−N(2): 2.581(4), U(1)−N(3): 2.602(3) Å], implying that the coordination of O is stronger than N. No significant differences were found among three U−N bond distances. Dihedral angles of ethylene groups, N(1)−C(8)−C(9)−N(2) and N(2)−C(10)−C(11)−N(3) are close to 60° (59.6° and 58.4°, respectively), indicating that these moieties show gauche conformation.

Figure 2. ORTEP view of U\textsuperscript{VI}O\textsubscript{2}(saldien)-DMSO at the 50% probability level. Hydrogen atoms are omitted for clarity.

The most important point in this analysis is to figure out if the unidentate ligand L is coordinated or not. Here, DMSO, which is one of the strong Lewis bases, was used as L. As shown in Figure 2, the DMSO molecule is actually included in the crystal lattice of the obtained compound. However, the interatomic distance between U(1) and O(5) is 6.00 Å, which is too long to regard it as bonding. Therefore, exclusion of L from the equatorial plane of U(VI) using the pentadentate ligand was successfully done as we expected. The DMSO molecule is included just as a solvent molecule. The similar structure was also found as U\textsuperscript{VI}O\textsubscript{2}(saldien)-EtOH in the previous publication.[15]

To clarify the molecular structure of U\textsuperscript{VI}O\textsubscript{2}(saldien) in solution, EXAFS spectra of this compound dissolved in DMSO and DMF were recorded. The \(k^3\)-weighted spectra are shown in the left part of Figure 3 together with that of solid state. Although there are minor differences among these spectra, the main feature of EXAFS oscillation is the same. This may imply the molecular structure of U\textsuperscript{VI}O\textsubscript{2}(saldien) remains unchanged in the solutions. The right part of Figure 3 shows a Fourier transform of the DMSO solution and the best result of the EXAFS curve fit. The evaluated interatomic distances (\(R\)) described in the figure caption are in agreement with those of the U\textsuperscript{VI}O\textsubscript{2}(saldien) crystalline within 0.03 Å which is the lower limit of EXAFS structure determination. Furthermore, in accordance with the derived coordination numbers (\(N\)), the equatorial plane of U(VI) seems to be fully occupied by the coordinating O and N atoms of saldien\textsuperscript{2−}, i.e., no space for additional solvent coordination is remaining. In addition, no significant enhancement of the peak intensity due to the presence of S atom of DMSO was detected around 3.6-3.7 Å. These EXAFS results demonstrate that

Figure 3. EXAFS spectra of U\textsuperscript{VI}O\textsubscript{2}(saldien) dissolved in DMSO and DMF.
no solvent coordination occurs even in the DMSO solution, i.e., the molecular structure of \( \text{UVIO}_2(\text{saldien}) \) remains unchanged.

\[
\begin{align*}
\kappa^3 &\rightarrow \\
\chi(k) &\rightarrow \\
\text{FT} &\rightarrow \\
\end{align*}
\]

\textbf{Figure 3.} Left: \( \kappa^3 \)-weighted EXAFS spectra of \( \text{UVIO}_2(\text{saldien}) \) in solid state, DMF and DMSO solutions. Right: Fourier transform of \( \kappa^3 \)-weighted EXAFS spectrum of the DMSO solution. U–O(1,2): \( N = 2 \) (fixed), \( R = 1.81 \text{ Å} \); U–O(3,4): \( N = 2.1, R = 2.26 \text{ Å} \); U–N(1-3): \( N = 3.0, R = 2.59 \text{ Å} \); U–C(1,18): \( N = 2.0, R = 3.12 \text{ Å} \); U–C(7-12): \( N = 6.0, R = 3.54 \text{ Å} \); O(1)–U–O(2) MS: \( N = 2 \) (fixed), \( R = 3.62 \text{ Å} \). \( \Delta E_0 = -2.04 \text{ eV} \). Atomic notations follow Figure 2.

Cyclic voltammograms of \( \text{UVIO}_2(\text{saldien}) \) (1.52 \( \times \) 10\(^{-3} \) M) in DMSO containing 0.106 M TBAP are shown in Figure 4. Cathodic and anodic peaks were observed at –1.65 V and –1.52 V vs. Fe/Fe\(^+\). The peak potential separation tends to increase from 0.080 to 0.170 V with increasing potential sweep rate (\( \nu \), 0.010-0.500 V·s\(^{-1} \)), indicating quasireversible system. The formal potential \( E^{°'} \) is almost constant at –1.582 ± 0.005 V vs. Fe/Fe\(^+\) regardless of \( \nu \). Using the current value at the cathodic peak (\( i_{pc} \), Figure 5) and the \( i_{pc} \sim \nu^{1/2} \) relationship for the irreversible system,[29] the diffusion coefficient of \( \text{UVIO}_2(\text{saldien}) \) was estimated as 2.3 \( \times \) 10\(^{-6} \) cm\(^2\)·s\(^{-1} \).[7]

\textbf{Figure 4.} Cyclic voltammograms of \( \text{UVIO}_2(\text{saldien}) \) (1.52 \( \times \) 10\(^{-3} \) M) in DMSO containing 0.106 M TBAP at different potential sweep rates (\( \nu \) = 0.010-0.500 V·s\(^{-1} \)). Initial scan direction: cathodic.
Figure 5. Cathodic peak current ($i_{pc}$) in Figure 4 as a function of $v^{1/2}$.

Figure 6. Electronic spectra recorded at the applied potentials in the range from –1.392 to –1.792 V vs. Fc/Fc+ for UVIO$_2$(saldien) ($4.20 \times 10^{-3}$ M) in DMSO containing 0.305 M TBAP. Wavelength regions: 260–570 nm (a), 480–2200 nm (b). Noises are due to detectors (photomultiplier and PbS detector) in the spectrophotometer and/or absorption of DMSO in the OTTLE cell. Optical path length: 1.89 $\times$ 10$^{-2}$ cm.

It is possible only to obtain qualitative information about reaction mechanism from the cyclic voltammogram. To determine the electron stoichiometry of the redox reaction in Figure 4, UV-Vis-NIR spectroelectrochemical experiment was performed for UVIO$_2$(saldien) ($4.20 \times 10^{-3}$ M) in DMSO containing 0.305 M TBAP by using the OTTLE cell. The absorption spectra were recorded at the potentials varied stepwise in the range from –1.392 to –1.792 V vs. Fc/Fc+. The resulting spectra are shown in Figure 6 together with that of initial UVIO$_2$(saldien) without the potential application. The spectral changes were observed with a decrease in the potential and converged at –1.792 V vs. Fc/Fc+. Isosbestic points were clearly observed at 349, 401 and 544 nm, indicating that the redox equilibrium of UVIO$_2$(saldien) only takes place. Using the absorbance at 373 nm which is the absorption maximum of the reductant, the concentrations of the oxidant ($C_O$, here UVIO$_2$(saldien)) and reductant ($C_R$) at each potential was calculated. The potential value $E$ was plotted as a function of natural logarithm of $C_O/C_R$. 

The slope and intercept of the best fit line of the Nernstian equation \(E = E^\circ + \left(\frac{RT}{nF}\right) \ln \left(\frac{C_O}{C_R}\right)\) to the \(E–\ln\left(\frac{C_O}{C_R}\right)\) curve were 0.0276 and –1.584, respectively. From the slope, the electron stoichiometry was calculated as 0.929, which is regarded as unity. Therefore, the reductant of \(\text{UVIO}_2(\text{saldien})\) is clarified to be the corresponding U(V) complex, \([\text{UVO}_2(\text{saldien})]^-\). From the intercept, the standard redox potential of \([\text{UVO}_2(\text{saldien})]/\text{UVIO}_2(\text{saldien})\) is –1.584 V vs. Fe/Fe^+\), which is in agreement with \(E^\circ\) from CV. It was confirmed that \([\text{UVO}_2(\text{saldien})]^-\) is stable in DMSO. The UV-Vis-NIR absorption spectrum recorded at –1.792 V vs. Fe/Fe^+ is assigned to pure \([\text{UVO}_2(\text{saldien})]^-\) in DMSO.

The electronic spectrum of \([\text{UVO}_2(\text{saldien})]^-\) in DMSO is shown in Figure 7. The absorption maxima of \([\text{UVO}_2(\text{saldien})]^-\) were observed at 373, 630, 700, 830, 1380 and 1900 nm. Since the molar absorptivity \(\varepsilon\) at 373 nm is ca. 13000 M\(^{-1}\)·cm\(^{-1}\), this absorption band can be assigned to an electric-dipole allowed transition originating from LM/MLCT and/or the coordinating saldien\(^2^-\). In contrast, the \(\varepsilon\) values of the absorption bands at > 600 nm are relatively small, 100–400 M\(^{-1}\)·cm\(^{-1}\). The peak positions and the \(\varepsilon\) values are similar to other U(V) complexes with the pentagonal-bipyramidal coordination.[11] In accordance with the previous discussion, the absorption bands of \([\text{UVO}_2(\text{saldien})]^-\) in Vis-NIR regions are attributable to f–f transition in U\(^{5+}\).

![Figure 7.](image)

Figure 7. Electronic spectrum of \([\text{UVO}_2(\text{saldien})]^-\) in DMSO containing 0.305 M TBAP at –1.792 V vs. Fe/Fe^+ in Figure 6. Wavelength regions: 260–570 nm (a), 480–2200 nm (b). Noises are due to detectors (photomultiplier and PbS detector) in the spectrophotometer and/or absorption of DMSO in the OTTLE cell.

4. Conclusion

In the present study, we investigated the molecular structure of \(\text{UVIO}_2(\text{saldien})\) in solid and solution states. Our strategy to obtain a stable U(V) species is the exclusion of unidentate ligand L from the equatorial plane of the uranyl ion. As expected, the pentadentate ligand saldien\(^2^-\) does not allow the solvent molecule L to participate in the U(VI) coordination sphere even in the solutions. The electrochemical behavior of \(\text{UVIO}_2(\text{saldien})\) in DMSO was also studied by the cyclic voltammetry and spectroelectrochemical technique. As a result, we found another stable U(V) complex, \([\text{UVO}_2(\text{saldien})]^-\) in DMSO. This species shows the characteristic absorption bands of U(V) due to f–f transition in the Vis-NIR regions as well as other U(V) complexes.

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