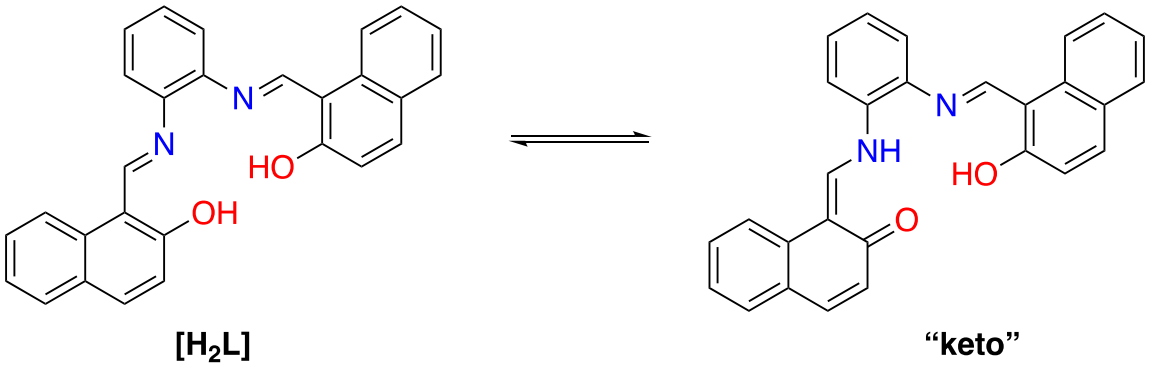
Supplemental Information

Solid-state structural elucidation and electrochemical analysis of uranyl naphthylsalophen

Julie E. Niklas,[a] Emily E. Hardy,[a] and Anne E. V. Gorden*[a]

Department of Chemistry and Biochemistry, Auburn University, 179 Chemistry Building, Auburn, Alabama 36849, USA.

Table of Contents



Scheme S1. Projection of the "enolimine" and "ketoamine" tautomers of **[H_2L]**. The solution phase data and metal complexes suggest the equilibrium lies on the "enolimine" form, but the solid-state data is the "ketoamine" form.

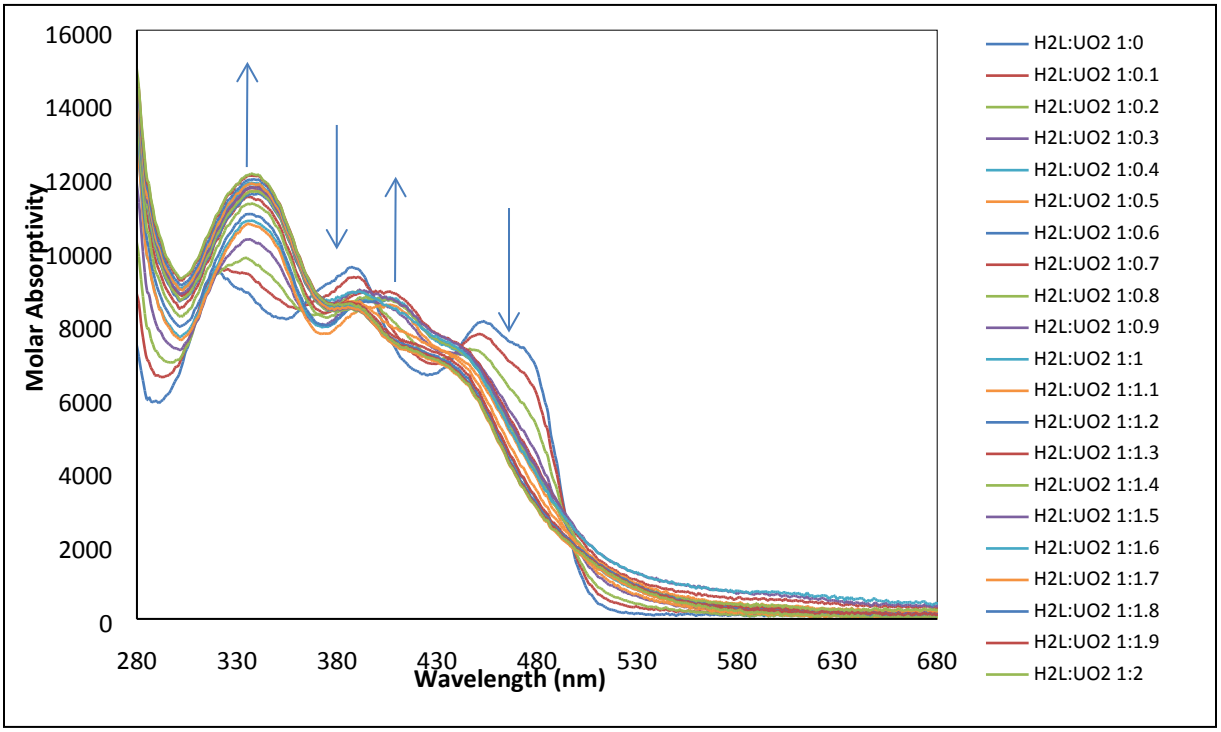


Figure S1. Serial titration of 31 µM solution of H_2L in MeOH with increasing ratios of $UO_2(NO_3)_2$• $6H_2O$ dissolved in water. Change in the UV spectrum plateaued after a 1:1 addition of metal salt. Minimal water was added through this titration (~100 µL) in 2.5 mL of MeOH.

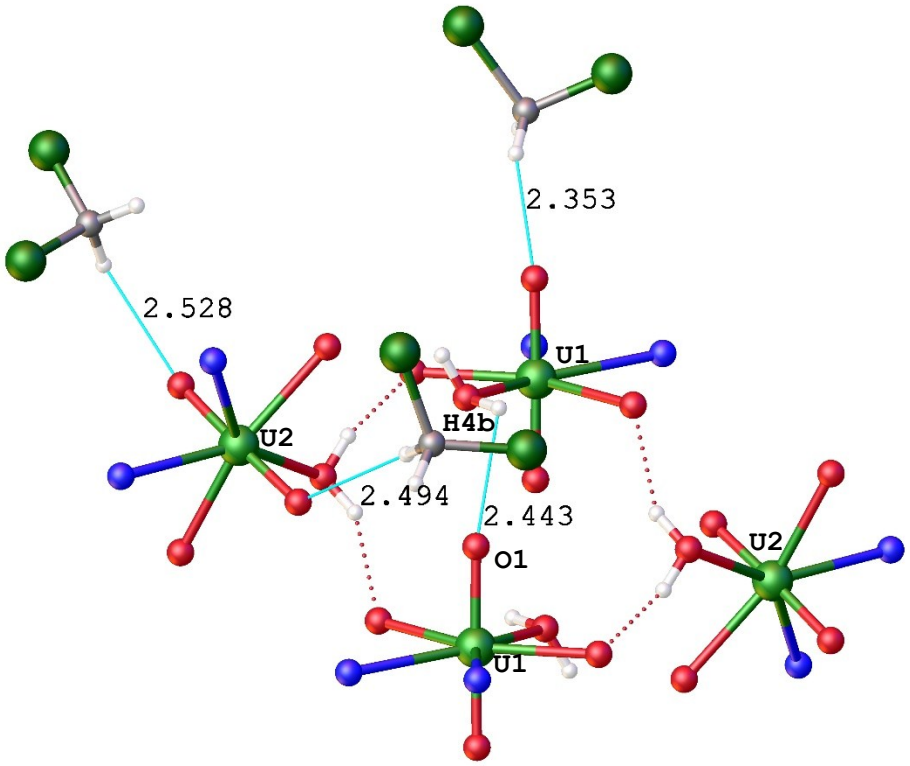


Figure S2. Coordination spheres and -yl oxo distances to hydrogen atoms on coordinated H_2O and interstitial CH_2Cl_2. All ligand carbon and hydrogen atoms omitted for clarity.

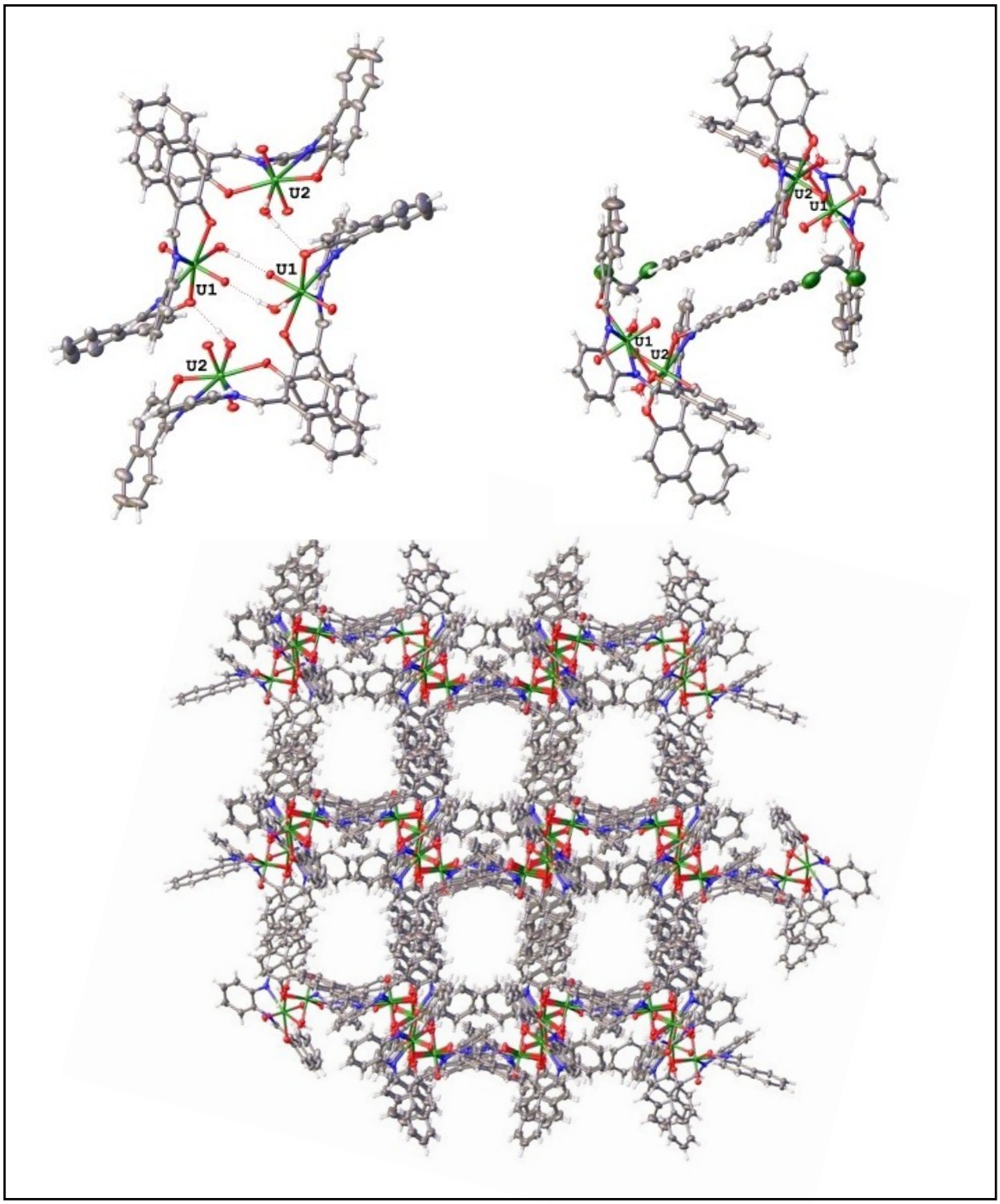


Figure S3. Projections of the UO_2[**L**] complex highlighting the hydrogen bonded tetramers and π-π stacking interactions resulting in an interesting supramolecular structure. Interstitial dichloromethane molecules have been removed for clarity.

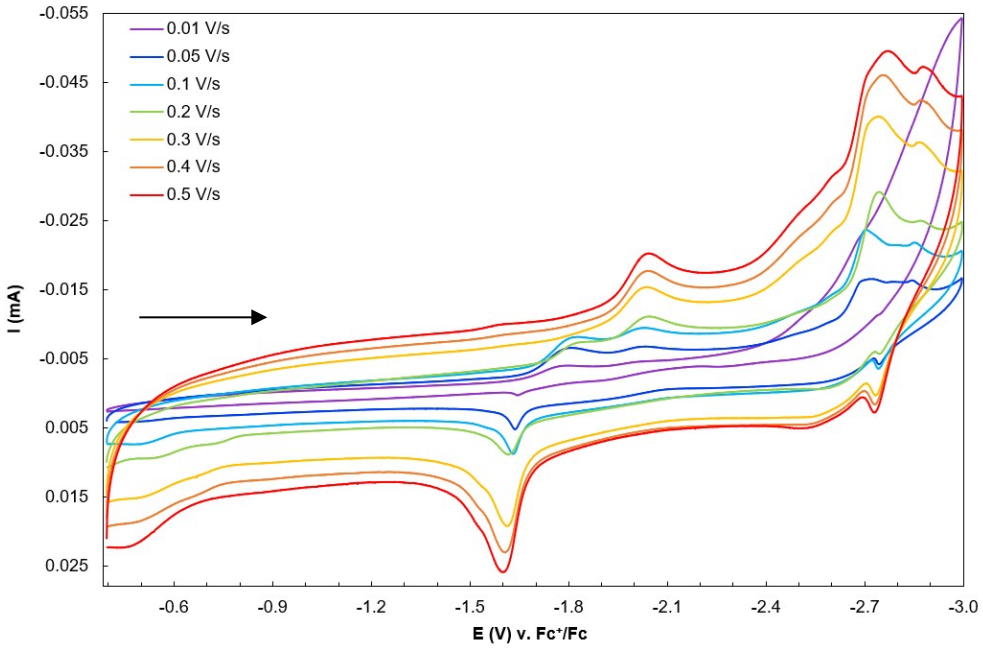


Figure S4. Cyclic voltammograms of $[H_2L]$ (0.25 mM in CH_3CN, 0.1M $TBAClO_4$) at scan rates of 0.01 V/s – 0.5 V/s.

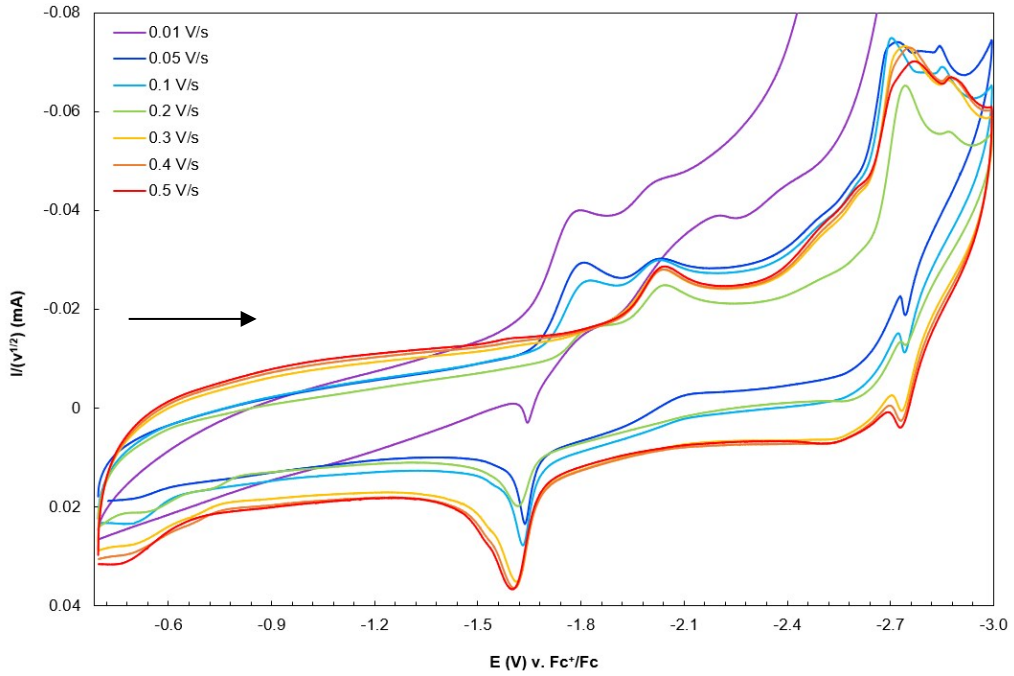


Figure S5. Scan rate normalized cyclic voltammograms of $[H_2L]$ (0.25 mM in CH_3CN, 0.1M $TBAClO_4$) at scan rates of 0.01 V/s – 0.5 V/s. *((1000)*(I/v$^{1/2}$); I= current (A), v= scan rate (V/s).*

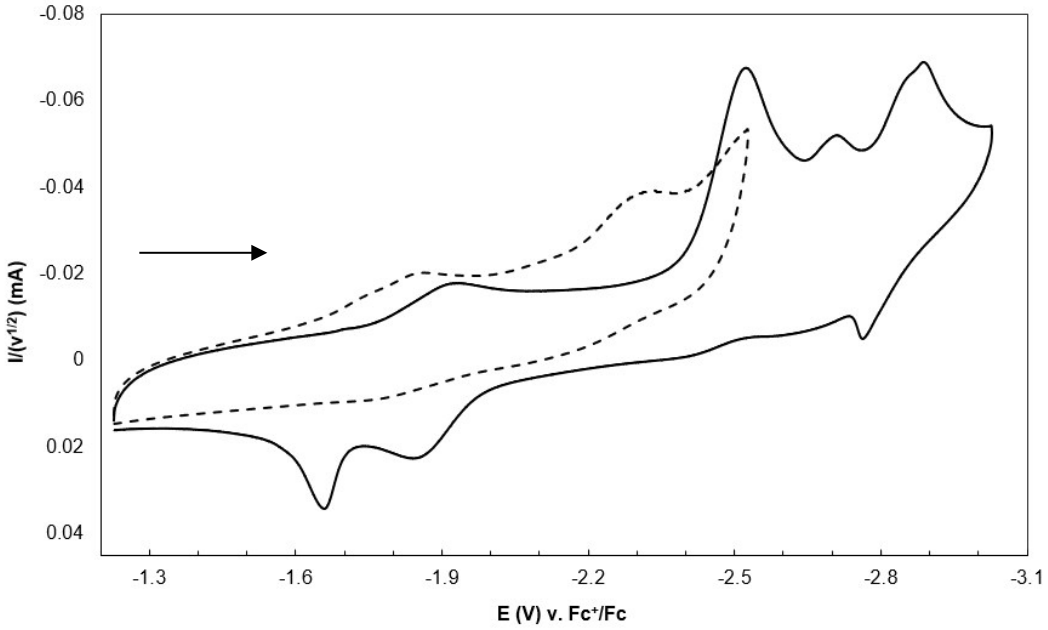


Figure S6. Scan-rate normalized cyclic voltammograms of UO_2[L] between -1.2 and -3.0 V (solid, original scan rate 0.2 V/s), and -1.2 and -2.5 V (dashed, original scan rate 0.1 V/s). *((1000)*(I/v$^{1/2}$); I= current (A), v= scan rate (V/s).*

Experimental Details

Caution! Standard precautions for handling radioactive materials or heavy metals, such as $UO_2(NO_3)_4$•$6H_2O$ as used in this study, were followed.

Any solvents not specifically identified were ACS grade, purchased from EMD, and used as received without further purification. The reagents acetonitrile (99.9%, HPLC grade, BDH), 1,2-diaminobenzene (99.5%, Aldrich), 2-hydroxynaphthaldehyde (98%, Alfa Aesar), were used as received without further purification. $UO_2(NO_3)_2$•$6H_2O$ (98%, J. T. Baker) was recrystallized from a 50% nitric acid solution and stored under hexanes until use. Anhydrous dichloromethane (BDH) was purchased, stored under argon, and dispensed from a solvent purification system. Triethylamine (99%, Alfa Aesar) was distilled and stored under argon until use. $(CD_3)_2SO$ and $CDCl_3$ were purchased from Cambridge Isotope Laboratories and stored in a desiccator.

[H_2L]: 1,2-diaminobenzene (0.433 g, 7.23 mmol) and 2-hydroxynaphthaldehyde (1.32 g, 15.0 mmol) were added to 150 mL of MeOH, and stirred for 6 hours at reflux temperature, during which time the color changed from yellow to dark orange and a precipitate formed. The solution was allowed to cool to room temperature, and the solid was filtered and washed with hexanes to yield a bright orange powder (2.54 g, 84%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.18 (d, 2H, J = 9.2 Hz), 7.34 (t, 2H, J = 7.4 Hz), 7.44-7.39 (m, 4H), 7.51 (t, 2H, J = 8.2 Hz), 7.73 (d, 2H, J = 7.9 Hz), 7.82 (d, 2H, J = 9.2 Hz), 8.14 (d, 2H, J = 8.5 Hz), 9.46 (d, 2H, J = 3.6 Hz), 15.08 (s, 2OH); ^{13}C NMR (151 MHz, $CDCl_3$) δ 109.40, 119.03, 119.15, 122.09, 123.63, 127.42, 127.51, 128.08, 129.41, 133.21, 136.67, 139.73, 156.17, 169.08; TOF MS (ESI) m/z (M^+ + 1) Calcd 417.1525, Found 417.1550. CCDC: 1523762.

UO_2[L]: The naphthylsalophen ligand [H_2L] (970. mg, 2.33 mmol) and NEt_3 (1 mL) were added to 40 mL of DCM and 20 mL of MeOH in a 100 mL round bottom flask. The mixture was stirred until all the solids were dissolved. Uranyl nitrate hexahydrate (963 mg, 2.27 mmol) was then added to the flask. The reaction was heated to 40 °C and heated with stirring for 2 hours. The solution volume was reduced by half by rotary evaporation, and subsequently put in ice for 30 minutes. A red solid precipitated, and was filtered off and rinsed with hexanes (132 mg, 83%). Crystals suitable for x-ray diffraction were grown in 3 days from layering a saturated DCM solution of UO_2[**L**] with hexanes. The ^{1}H NMR (600 MHz, d_6-DMSO) δ 7.31 (t, 2H, J = 7.4 Hz), 7.38 (d, 2H, J = 9.1 Hz), 7.53-7.55 (m, 4H), 7.85-7.88 (m, 4H), 8.20 (d, 2H, J = 8.1 Hz), 8.36 (d, 2H, J = 8.5 Hz), 10.20 (s, 2H) ppm; ^{13}C NMR (151 MHz, d_6-DMSO) δ 114.72, 120.33, 121.36, 122.92, 124.20, 127.03, 127.91, 128.49, 128.75, 134.49, 137.12, 147.38, 160.09, 171.33 ppm; TOF MS (ESI) m/z (M^+ + 1) Calcd 685.1774, Found 685.1777, ($M(H_2O)^+$ + 1) Calcd 703.1880, Found 703.1902. CCDC: 1827293.

NMR Spectroscopy

^{1}H NMR spectra were recorded with a Bruker spectrometer at 600 MHz. ^{13}C NMR spectra were recorded with a Bruker spectrometer at 151 MHz. NMR spectroscopic data were collected using deuterated chloroform ($CDCl_3$) or deuterated DMSO (D_6-DMSO). Chemical shifts are reported in parts per million (δ) and are referenced against TMS or residual internal solvent signals.

Single Crystal X-ray Diffraction

Crystals suitable for single crystal X-ray diffraction were selected and mounted on a glass fiber using Paratone-N oil and data set collection was completed on a 'Bruker APEX CCD' diffractometer using Mo Kα radiation. The crystal was kept at 180 K during unit cell and data collection. SMART (v. 5.624) was used for preliminary determination of cell constants and data collection control. Determination of integrated intensities and global cell refinement were performed with the Bruker SAINT software package, and empirical absorption correction (SADABS) was applied. The structures were solved with the ShelXS structure solution program using Direct Methods[1,2] and refined with the olex2.refine refinement package using Gauss-Newton minimisation.[3,4] Projections were created on Olex2.1.[3]

Absorbance Spectroscopy

All solution phase absorbance spectra were collected on a VARIAN Cary 50 WinUV Spectrometer with a xenon lamp with absorbance spectra from 200 nm to 900 nm with a 1 cm path length quartz cuvette. Serial titrations were completed by introducing a known amount of the metal salt ($(UO_2(NO_3)_2$ in H_2O) to a solution of the free base ligand [H_2L] in MeOH. A solution of uranyl nitrate ($UO_2(NO_3)_2$ in H_2O was prepared by dissolving 3.0 mg of recrystallized uranyl nitrate in water, and then bringing this to volume in a 25.0 mL volumetric flask. Titrations were completed in MeOH by serial additions of 20 μL of the (0.24 mM) uranyl nitrate aqueous solution, maintaining less than 1% water in MeOH. The solutions were agitated for 5 seconds, the cuvette replaced in the spectrometer, and the absorbance spectrum collected. This procedure was repeated until a large excess of metal salt was present. A control series to observe any

absorption change upon of water addition was also conducted, and no significant change was observed. The absorbance was adjusted for concentration by this serial dilution method by the reporting the data as extinction coefficient.

Electrochemical Analysis

Electrochemical measurements were carried out using a CH Instruments 660 E potentiostat in HPLC-grade MeCN or (BDH Chemicals) with tetrabutylammonium perchlorate ($TBAClO_4$) supporting electrolyte (0.1 M). Ligand and complex solutions (0.25 mM) were purged with N_2 for 30 minutes immediately prior to experiments. A three-electrode cell consisting of a glassy-carbon-disk working electrode, Pt-wire counter electrode, and Ag/AgCl/saturated KCl/H_2O reference electrode was used. Data were corrected to versus ferrocene based on averaged values for $E_{1/2}(Fc^+/Fc)$ collected using the same three-electrode cell before and after each set of experiments. All data reported was using an initial cathodic sweep and anodic return sweep.

Crystallographic Tables

$[H_2L]$

Table S1 Crystal data and structure refinement for $[H_2L]$

Empirical formula	$C_{29}H_{22}Cl_2N_2O_2$
Formula weight	501.42
Temperature/K	180.45
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	7.1378(10)
b/Å	16.682(2)
c/Å	20.056(3)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	2388.1(6)
Z	4
ρ_{calc}mg/mm^3	1.3945
m/mm^{-1}	0.303
F(000)	1041.6
Crystal size/mm^3	0.15 × 0.03 × 0.02
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection	4.06 to 57.4°
Index ranges	$-9 \le h \le 9$, $-22 \le k \le 22$, $-27 \le l \le 22$
Reflections collected	17603
Independent reflections	6177 [R_{int} = 0.0539, R_{sigma} = 0.0733]
Data/restraints/parameters	6177/0/318
Goodness-of-fit on F^2	1.036
Final R indexes [I>=2σ (I)]	R_1 = 0.0556, wR_2 = 0.1167
Final R indexes [all data]	R_1 = 0.0869, wR_2 = 0.1284
Largest diff. peak/hole / e Å^{-3}	0.65/-0.64
Flack parameter	-0.10(7)

Table S2 Bond Lengths for $[H_2L]$.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Cl1	C29	1.747(3)	C17	C16	1.392(3)
Cl2	C29	1.755(3)	C18	C19	1.438(3)
O1	C1	1.276(3)	C19	C20	1.453(3)

O2	C28	1.338(3)	C19	C28	1.395(3)
N1	C11	1.319(3)	C20	C25	1.416(3)
N1	C12	1.405(3)	C20	C21	1.423(3)
N2	C17	1.419(3)	C25	C24	1.419(4)
N2	C18	1.302(3)	C25	C26	1.412(4)
C6	C5	1.369(4)	C24	C23	1.367(4)
C6	C7	1.392(4)	C23	C22	1.396(4)
C5	C4	1.424(3)	C3	C2	1.341(4)
C4	C9	1.405(3)	C2	C1	1.446(4)
C4	C3	1.433(4)	C8	C7	1.376(3)
C9	C10	1.455(3)	C13	C14	1.380(3)
C9	C8	1.414(4)	C14	C15	1.391(4)
C10	C11	1.394(3)	C15	C16	1.381(4)
C10	C1	1.440(4)	C26	C27	1.359(4)
C12	C17	1.407(3)	C27	C28	1.407(4)
C12	C13	1.393(3)	C22	C21	1.362(3)

Table S3 Bond Angles for $[H_2L]$.

Atom	**Atom**	**Atom**	**Angle/°**	**Atom**	**Atom**	**Atom**	**Angle/°**
C12	N1	C11	127.3(2)	C21	C20	C25	117.7(2)
C18	N2	C17	118.05(19)	C24	C25	C20	119.4(2)
C7	C6	C5	119.0(2)	C26	C25	C20	119.4(2)
C4	C5	C6	120.9(2)	C26	C25	C24	121.2(2)
C9	C4	C5	120.0(2)	C23	C24	C25	121.0(3)
C3	C4	C5	120.2(2)	C22	C23	C24	119.7(3)

C3	C4	C9	119.8(2)	C2	C3	C4	121.4(2)
C10	C9	C4	119.4(2)	C1	C2	C3	122.0(3)
C8	C9	C4	117.4(2)	C10	C1	O1	122.6(2)
C8	C9	C10	123.2(2)	C2	C1	O1	119.6(2)
C11	C10	C9	121.2(2)	C2	C1	C10	117.7(2)
C1	C10	C9	119.6(2)	C7	C8	C9	121.2(2)
C1	C10	C11	119.2(2)	C8	C7	C6	121.3(3)
C10	C11	N1	123.9(2)	C14	C13	C12	120.4(2)
C17	C12	N1	118.0(2)	C15	C14	C13	120.4(2)
C13	C12	N1	122.6(2)	C16	C15	C14	119.6(2)
C13	C12	C17	119.4(2)	C15	C16	C17	120.8(2)
C12	C17	N2	117.7(2)	C27	C26	C25	121.6(2)
C16	C17	N2	123.0(2)	C28	C27	C26	120.2(2)
C16	C17	C12	119.3(2)	C19	C28	O2	122.1(2)
C19	C18	N2	122.5(2)	C27	C28	O2	116.7(2)
C20	C19	C18	121.1(2)	C27	C28	C19	121.3(2)
C28	C19	C18	120.3(2)	C21	C22	C23	121.0(3)
C28	C19	C20	118.6(2)	C22	C21	C20	121.2(3)
C25	C20	C19	119.0(2)	Cl2	C29	Cl1	111.72(16)
C21	C20	C19	123.3(2)				

Table S4 Hydrogen Bonds for [H_2L].

D	**H**	**A**	**d(D-H)/Å**	**d(H-A)/Å**	**d(D-A)/Å**	**D-H-A/°**
O2	H2	N2	0.80(3)	1.82(3)	2.574(3)	156(3)

UO_2[L]

Table S5 Crystal data and structure refinement for UO_2[L]

Empirical formula	$C_{60}H_{48}Cl_8N_4O_{10}U_2$
Formula weight	1744.75
Temperature/K	180.0
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	15.4659(12)
b/Å	19.3294(14)
c/Å	22.2346(16)

α/°	90
β/°	106.655(1)
γ/°	90
Volume/Å^3	6368.1(8)
Z	4
ρ_{calc}mg/mm^3	1.8197
m/mm^{-1}	5.476
F(000)	3269.1
Crystal size/mm^3	0.18 × 0.12 × 0.1
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection	2.84 to 51.52°
Index ranges	-18 ≤ h ≤ 18, -23 ≤ k ≤ 23, -27 ≤ l ≤ 27
Reflections collected	74217
Independent reflections	12168 [R_{int} = 0.0706, R_{sigma} = 0.0463]
Data/restraints/parameters	12168/0/759
Goodness-of-fit on F^2	1.094
Final R indexes [I>=2σ (I)]	R_1 = 0.0440, wR_2 = 0.0935
Final R indexes [all data]	R_1 = 0.0628, wR_2 = 0.1031
Largest diff. peak/hole / e Å^{-3}	3.33/-1.96

Table S6 Bond Lengths for UO_2[L].

Atom	Atom	Length/Å	Atom	Atom	Length/Å
U1	O1	1.795(5)	C22	C23	1.408(13)
U1	O2	2.285(5)	C22	C27	1.414(13)
U1	O3	1.771(5)	C23	C24	1.393(14)
U1	O4	2.424(5)	C24	C25	1.396(17)
U1	N1	2.491(6)	C25	C26	1.368(18)
U1	N2	2.516(6)	C26	C27	1.415(14)
U1	O1aa	2.288(5)	C27	C28	1.418(14)
U2	O5	2.259(5)	C28	C29	1.357(12)
U2	O6	2.466(5)	C31	C32	1.420(10)
U2	O8	1.769(5)	C31	C36	1.419(11)
U2	N3	2.538(6)	C31	O0aa	1.315(8)
U2	N4	2.515(6)	C32	C33	1.445(10)
U2	O0aa	2.311(5)	C32	C41	1.431(11)

U2	O3aa	1.771(5)	C33	C34	1.407(11)
O2	C1	1.330(9)	C33	C40	1.418(10)
O5	C46	1.315(10)	C34	C35	1.430(11)
N1	C3	1.290(10)	C34	C37	1.415(11)
N1	C4	1.428(9)	C35	C36	1.344(11)
N2	C5	1.428(10)	C37	C38	1.360(12)
N2	C6	1.307(10)	C38	C39	1.377(13)
N3	C41	1.282(9)	C39	C40	1.373(11)
N3	C42	1.425(10)	C42	C43	1.404(11)
N4	C43	1.424(10)	C42	C58	1.396(11)
N4	C44	1.294(10)	C43	C55	1.390(11)
C1	C2	1.397(11)	C44	C45	1.424(12)
C1	C29	1.422(11)	C45	C46	1.403(11)
C2	C3	1.454(11)	C45	C50	1.452(12)
C2	C22	1.433(11)	C46	C47	1.428(11)
C4	C5	1.390(11)	C47	C48	1.366(13)
C4	C21	1.409(10)	C48	C49	1.411(14)
C5	C18	1.396(11)	C49	C50	1.412(13)
C6	C7	1.437(11)	C49	C54	1.418(14)
C7	C8	1.400(11)	C50	C51	1.399(13)
C7	C12	1.455(11)	C51	C52	1.370(14)
C8	C9	1.412(10)	C52	C53	1.384(16)
C8	O1aa	1.324(9)	C53	C54	1.342(16)
C9	C10	1.360(11)	C55	C56	1.380(13)
C10	C11	1.404(12)	C56	C57	1.392(13)
C11	C12	1.411(12)	C57	C58	1.389(12)
C11	C16	1.410(12)	Cl7	C1aa	1.66(2)
C12	C13	1.404(12)	Cl8	C1aa	1.74(2)
C13	C14	1.376(14)	Cl9	C0aa	1.675(16)
C14	C15	1.379(15)	Cl10	C0aa	1.760(19)
C15	C16	1.346(14)	Cl1	C3aa	1.665(19)
C18	C19	1.393(12)	Cl2	C3aa	1.781(17)
C19	C20	1.373(13)	Cl6	C2aa	1.65(2)
C20	C21	1.392(12)	Cl0a	C2aa	1.87(3)

Table S7 Bond Angles for UO_2[L].

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O2	U1	O1	86.4(2)	C16	C11	C10	121.7(8)
O3	U1	O1	178.5(2)	C16	C11	C12	119.5(8)
O3	U1	O2	95.1(2)	C11	C12	C7	118.8(7)
O4	U1	O1	92.4(2)	C13	C12	C7	124.4(8)
O4	U1	O2	78.29(17)	C13	C12	C11	116.8(8)
O4	U1	O3	88.0(2)	C14	C13	C12	121.0(10)
N1	U1	O1	95.2(2)	C15	C14	C13	122.2(10)
N1	U1	O2	69.53(19)	C16	C15	C14	117.8(9)
N1	U1	O3	85.3(2)	C15	C16	C11	122.7(10)
N1	U1	O4	146.34(19)	C19	C18	C5	119.0(9)
N2	U1	O1	91.1(2)	C20	C19	C18	120.6(8)
N2	U1	O2	132.69(19)	C21	C20	C19	121.8(8)
N2	U1	O3	87.8(2)	C20	C21	C4	117.5(8)
N2	U1	O4	148.99(19)	C23	C22	C2	122.4(8)
N2	U1	N1	63.7(2)	C27	C22	C2	119.2(8)
O1aa	U1	O1	88.7(2)	C27	C22	C23	118.1(8)
O1aa	U1	O2	157.47(18)	C24	C23	C22	120.7(11)
O1aa	U1	O3	89.9(2)	C25	C24	C23	120.1(11)
O1aa	U1	O4	79.95(17)	C26	C25	C24	120.7(11)

O1aa	U1	N1	132.88(19)	C27	C26	C25	119.9(11)
O1aa	U1	N2	69.34(19)	C26	C27	C22	120.4(10)
O6	U2	O5	79.84(18)	C28	C27	C22	118.7(8)
O8	U2	O5	92.0(2)	C28	C27	C26	120.8(10)
O8	U2	O6	91.7(2)	C29	C28	C27	122.2(8)
N3	U2	O5	131.93(19)	C28	C29	C1	119.6(8)
N3	U2	O6	148.02(18)	C36	C31	C32	119.4(6)
N3	U2	O8	90.7(2)	O0aa	C31	C32	121.9(7)
N4	U2	O5	68.63(19)	O0aa	C31	C36	118.7(6)
N4	U2	O6	148.43(19)	C33	C32	C31	118.8(7)
N4	U2	O8	87.9(2)	C41	C32	C31	120.7(7)
N4	U2	N3	63.5(2)	C41	C32	C33	120.2(7)
O0aa	U2	O5	158.85(18)	C34	C33	C32	120.1(7)
O0aa	U2	O6	79.01(17)	C40	C33	C32	123.1(7)
O0aa	U2	O8	88.7(2)	C40	C33	C34	116.9(7)
O0aa	U2	N3	69.17(18)	C35	C34	C33	118.6(7)
O0aa	U2	N4	132.51(18)	C37	C34	C33	120.3(7)
O3aa	U2	O5	90.1(2)	C37	C34	C35	121.0(8)
O3aa	U2	O6	89.0(2)	C36	C35	C34	121.8(7)
O3aa	U2	O8	177.8(2)	C35	C36	C31	121.3(7)
O3aa	U2	N3	87.5(2)	C38	C37	C34	120.6(8)
O3aa	U2	N4	92.5(2)	C39	C38	C37	119.9(8)
O3aa	U2	O0aa	89.5(2)	C40	C39	C38	121.1(8)
C1	O2	U1	124.3(4)	C39	C40	C33	121.2(8)
C46	O5	U2	128.7(5)	C32	C41	N3	127.9(7)
C3	N1	U1	123.5(5)	C43	C42	N3	116.7(7)
C4	N1	U1	115.3(5)	C58	C42	N3	123.8(7)
C4	N1	C3	121.1(6)	C58	C42	C43	119.5(7)
C5	N2	U1	114.7(5)	C42	C43	N4	115.6(7)
C6	N2	U1	127.3(5)	C55	C43	N4	125.0(7)
C6	N2	C5	117.8(6)	C55	C43	C42	119.4(8)
C41	N3	U2	127.5(5)	C45	C44	N4	126.2(7)
C42	N3	U2	113.9(4)	C46	C45	C44	120.1(8)
C42	N3	C41	118.5(6)	C50	C45	C44	119.9(7)
C43	N4	U2	115.9(5)	C50	C45	C46	119.5(8)
C44	N4	U2	124.8(5)	C45	C46	O5	122.0(7)
C44	N4	C43	119.2(7)	C47	C46	O5	118.2(7)
C2	C1	O2	122.2(7)	C47	C46	C45	119.7(8)
C29	C1	O2	117.6(7)	C48	C47	C46	120.0(8)
C29	C1	C2	120.1(7)	C49	C48	C47	122.3(9)
C3	C2	C1	119.9(7)	C50	C49	C48	119.1(9)
C22	C2	C1	119.7(7)	C54	C49	C48	121.4(9)
C22	C2	C3	120.0(7)	C54	C49	C50	119.4(9)
C2	C3	N1	125.2(7)	C49	C50	C45	119.2(8)
C5	C4	N1	116.5(7)	C51	C50	C45	123.2(9)

C21	C4	N1	122.4(7)	C51	C50	C49	117.6(9)
C21	C4	C5	121.1(7)	C52	C51	C50	120.7(10)
C4	C5	N2	115.4(7)	C53	C52	C51	121.8(11)
C18	C5	N2	124.5(7)	C54	C53	C52	119.0(11)
C18	C5	C4	120.0(7)	C53	C54	C49	121.5(10)
C7	C6	N2	124.8(7)	C56	C55	C43	121.4(8)
C8	C7	C6	121.5(7)	C57	C56	C55	118.9(8)
C12	C7	C6	118.7(7)	C58	C57	C56	121.0(9)
C12	C7	C8	119.6(7)	C57	C58	C42	119.8(8)
C9	C8	C7	119.9(7)	C31	O0aa	U2	132.0(5)
O1aa	C8	C7	122.3(7)	C8	O1aa	U1	128.6(4)
O1aa	C8	C9	117.8(7)	Cl10	C0aa	Cl9	110.8(9)
C10	C9	C8	120.0(8)	Cl8	C1aa	Cl7	114.9(12)
C11	C10	C9	122.8(8)	Cl0a	C2aa	Cl6	101.1(9)
C12	C11	C10	118.8(7)	Cl2	C3aa	Cl1	114.3(8)

References

1. Bruker Bruker AXS Inc.: Madison, Wisconsin, USA, 2001.
2. Sheldrick, G. M., *Acta Crystallogr., Sect. A: Found. Crystallogr.* **2008,** *A64*, 112.
3. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H., *J. Appl. Cryst.* **2009,** *42*, 339-341.
4. Bourhis, L. J.; Dolomanov, O. V.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H., *Acta Crystallogr.* **2015,** *A71* (1), 59-75.