

Electronic Supporting Information for:

“2,6-diiminopiperidin-1-ol: an overlooked motif relevant to uranyl and transition metal binding on poly(amidoxime) adsorbents”

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EXPERIMENTAL DETAILS

Materials and methods. All reagents were used as received from commercial sources (typically Sigma Aldrich, TCI, or Fisher Scientific). Deuterated solvents were obtained from Cambridge Isotopes. Nuclear magnetic resonance spectra were obtained using a Varian 500 MHz spectrometer. High resolution mass spectrometry was performed on an Agilent Technologies 6530 Accurate-Mass Q-ToF LC/MS with positive ionization. Standard ESI-L low concentration tuning mix with addition of 0.1 mM HP-0321 was used as calibrant. Calculations of mass spectra were obtained using the Isotope Distribution Calculator and Mass Spec Plotter program.¹ UV-visible spectra were obtained using an Agilent Technologies 8453 spectrophotometer. Elemental analysis was performed at Robertson Microlit Laboratories (Ledgewood, NJ, USA).

glutardiamidoxime ($\mathbf{H}_2\mathbf{L}^2$): Synthesized as reported by Tian et al.² ¹H and ¹³C-NMR shifts in DMSO-*d*₆ matched published values.³ ¹H-NMR (500 MHz, DMSO-*d*₆): δ ppm 8.67 (s, 2H), 5.29 (s, 4H), 1.93 (t, 4H), 1.68 (quint, 2H). ¹³C-NMR (126 MHz, DMSO-*d*₆): δ ppm 152.50, 30.31, 23.61.

$\mathbf{H}_2\mathbf{L}^2 + \text{HCl}$ experiment: A stock solution of HCl (diluted with D₂O) of appropriate concentration was added to a stirring solution of ligand $\mathbf{H}_2\mathbf{L}^2$ in DMSO-*d*₆/D₂O (90:10 v/v, total volume = 0.7 mL) in a scintillation vial to achieve a final [HCl] = 100 mM and [$\mathbf{H}_2\mathbf{L}^2$] = 10 mM. The mixture was stirred at room temperature for 7 days and analyzed by NMR. ¹H-NMR (500 MHz, DMSO-*d*₆/D₂O (90:10 v/v)): δ ppm 2.41 (t, 4H), 1.93 (quint, 2H). ¹³C-NMR (500 MHz, DMSO-*d*₆/D₂O (90:10 v/v)): δ ppm 162.19, 27.60, 24.21.

(UO₂)₂($\mathbf{H}_2\mathbf{L}^1$)₂(μ -O)₂(NO₃)₂·THF (4**):** Ligand $\mathbf{H}_2\mathbf{L}^2$ (4.0 mg, 2.5 x 10⁻⁵ mol) was dissolved in CH₃OH (0.8 mL) in a 1 dram vial. UO₂(NO₃)₂·6H₂O (18.4 mg, 3.7 x 10⁻⁵ mol) was then added resulting in a rapid (seconds) colour change from colourless to bright orange. Vapour diffusion of tetrahydrofuran into the mixture afforded **4** after 10 days as irregular yellow crystals suitable for X-ray diffraction analysis.

[Cu₃($\mathbf{H}_2\mathbf{L}^1$)₃(μ -O)₃(H₂O)₃](NO₃)₃ (5**):** Ligand $\mathbf{H}_2\mathbf{L}^2$ (26.9 mg, 1.7 x 10⁻⁴ mol) was dissolved in CH₃OH/H₂O (1:1, 2 mL total). Cu(NO₃)₂·3H₂O (84.8 mg, 3.5 x 10⁻⁴ mol) was then added to the vial resulting in a colour change from colourless to dark yellow within seconds and eventually green (after ~1 h). After 24 hrs the solution was diluted with acetone (20 mL) and cooled to 0 °C resulting in a precipitate which was isolated by filtration, washed copiously with acetone and air-dried to yield crude **5** as a blue-green powder (25.2 mg, 56 %).

Growth of single crystals: Ligand $\mathbf{H}_2\mathbf{L}^2$ (3.8 mg, 2.4 x 10⁻⁵ mol) was dissolved in CH₃OH/H₂O (1:1, 0.5 mL total) in a 1 dram vial. Cu(NO₃)₂·3H₂O (12.0 mg, 5.0 x 10⁻⁵ mol) was then added to the vial. Vapour diffusion of acetone into the mixture afforded **5** after 4 days as dark green blocks suitable for X-ray diffraction analysis. ¹H-NMR (500 MHz, DMSO-*d*₆): δ ppm 38.59 (br s, 2H), 14.75 (br s, 2H), -1.33 (br s, 4H). HRMS (ESI-TOF) *m/z*: [[Cu₃(\mathbf{HL}^1)₃(μ -O)₃]+H]⁺ Calcd for C₁₅H₂₂N₉O₃Cu₃: 566.9716; found: 566.9719, [[Cu₃($\mathbf{H}_2\mathbf{L}^1$)₂(\mathbf{HL}^1)(μ -O)₃]+NO₃]⁺ Calcd for C₁₅H₂₃N₁₀O₆Cu₃: 629.9672; found: 629.9667. [[Cu₃($\mathbf{H}_2\mathbf{L}^1$)₂(\mathbf{HL}^1)(μ -O)₃+HCO₂H+NO₃]⁺ Calcd

for $C_{16}H_{25}N_{10}O_8Cu_3$: 675.9727; found: 675.9720. Anal. Calcd. for $C_{30}H_{61}N_{23}O_{28}Cu_6$: C, 22.90%; H, 3.91%; N, 20.48%. Found: C, 22.97%; H, 3.67%; N, 20.11%.

$[Ni_3(H_2L^1)_3(\mu-O)_3(H_2O)_6]Cl_3$ (**6**): Ligand H_2L^2 (4.5 mg, 2.8×10^{-5} mol) was dissolved in CH_3OH/H_2O (1:1, 0.5 mL total) in a 1 dram vial. $Ni(NO_3)_2 \cdot 6H_2O$ (11.1 mg, 3.8×10^{-5} mol) was then added resulting in no observable colour change in solution. Vapour diffusion of acetone into the mixture afforded pale blue polycrystalline needles observed after 7 days. The mother liquor was removed and the crystals were washed with acetone and air-dried. Recrystallization was performed using a second acetone vapour diffusion process using the same conditions as during the initial crystallization to yield pale blue single crystalline rods of **6** suitable for X-ray diffraction analysis. HRMS (ESI-TOF) m/z: $[Ni_2(H_2L^1)_2(HL^1)(\mu-O)_3+H^+]$ Calcd for $C_{15}H_{24}Ni_2N_9O_3$: 494.0709; found: 494.0661. $[Ni(H_2L^1)_2(\mu-O)_2+H^+]$: Calcd for $C_{10}H_{17}NiN_6O_2$: 311.0767; found: 311.0730. Notes: Chloride counter ions are thought to arise from residual NaCl present in H_2L^2 starting material. While XRD, ESI-MS (Fig. S7), and UV analyses have been performed, conditions to isolate larger quantities of **6** have remained elusive, precluding further analysis by NMR and elemental analysis.

SINGLE CRYSTAL X-RAY DIFFRACTION DATA AND STRUCTURE REFINEMENT

Single crystals of **5** and **6** were selected and mounted on a Bruker-APEXII-CCD diffractometer. The crystals were kept at 100 K during data collection. Using Olex2,⁴ structures **5** and **6** were solved with the ShelXS⁵ structure solution program using direct methods and refined with the olex2.refine⁶ refinement package using Gauss-Newton minimisation.

A single crystal of **4** was mounted in a radiological fume hood on a MiTeGen MicroMounts[®] loop using Krytox[®] grease which was then encapsulated with a polyester tubing epoxied to the pin base and transferred to a Bruker D8 VENTURE diffractometer. Data was collected at room temperature. The initial unit cell was determined using the APEX2⁷ program package. Data integration was performed using SAINT⁸ and SADABS⁹ was used to determine the semi-empirical absorption correction. A numerical absorption correction using face-indexing was not implemented due to the difficulty in identifying the crystal faces through the polyester tubing. Subsequent data reduction, structure solution and refinement were carried out using the SHELXTL¹⁰ program. The structure was solved by direct methods and refined on F² by full-matrix least-squares techniques.

The structure of **4** is located about an inversion center, and there is a disordered tetrahydrofuran molecule of solvation in the asymmetric unit. C(3) was disordered and included using multiple components with partial site-occupancy-factors and isotropic thermal parameters. Hydrogen atoms were included using a riding model.

Table S1. Crystal data and structure refinement for complexes 4-6

| Identification code | 4 | 5 | 6 |
|---|---|---|--|
| Empirical formula | C ₁₈ H ₃₂ N ₈ O ₁₄ U ₂ | C ₁₅ H ₃₂ Cu ₃ N ₁₂ O ₁₆ | C ₁₅ H ₄₄ Cl ₃ N ₉ Ni ₃ O ₁₃ |
| Formula weight | 1060.57 | 827.13 | 841.07 |
| Temperature/K | 296 | 100 | 100 |
| Crystal system | Monoclinic | triclinic | Monoclinic |
| Space group | P 2 ₁ /n | P-1 | P 2 ₁ /c |
| a/Å | 8.3529(4) | 12.6421(9) | 17.1605(12) |
| b/Å | 21.2854(8) | 14.2612(10) | 13.4761(10) |
| c/Å | 8.9198(4) | 17.0125(11) | 14.8097(11) |
| α/° | 90.00 | 90.192(3) | 90.00 |
| β/° | 111.8841(15) | 96.380(3) | 111.774(4) |
| γ/° | 90.00 | 113.941(3) | 90.00 |
| Volume/Å ³ | 1471.61(11) | 2782.0(3) | 3180.5(4) |
| Z | 2 | 4 | 4 |
| ρ _{calc} /g/cm ³ | 2.393 | 1.9747 | 1.757 |
| μ/mm ⁻¹ | 11.068 | 2.371 | 2.081 |
| F(000) | 984.0 | 1688.9 | 1744.0 |
| Crystal size/mm ³ | 0.50 × 0.32 × 0.24 | 0.34 × 0.28 × 0.24 | 0.40 × 0.26 × 0.12 |
| Radiation | Mo Kα (λ = 0.71073) | Mo Kα (λ = 0.71073) | Mo Kα (λ = 0.71073) |
| 2θ range for data collection/° | 3.01 to 28.57 | 3.12 to 56.74 | 3.96 to 56.8 |
| Index ranges | -11 ≤ h ≤ 11, -28 ≤ k ≤ 28, -11 ≤ l ≤ 11 | -16 ≤ h ≤ 16, -19 ≤ k ≤ 19, -22 ≤ l ≤ 17 | -22 ≤ h ≤ 22, -18 ≤ k ≤ 17, -19 ≤ l ≤ 19 |
| Reflections collected | 62272 | 50230 | 32907 |
| Independent reflections | 3673 | 13844 | 7953 |
| Data/restraints/parameters | 3673/0/184 | 13844/8/903 | 7953/7/453 |
| Goodness-of-fit on F ² | 1.210 | 1.062 | 1.027 |
| Final R indexes [I >= 2σ(I)] | R ₁ = 0.0336, wR ₂ = 0.0759 | R ₁ = 0.0273, wR ₂ = 0.0651 | R ₁ = 0.0294, wR ₂ = 0.0733 |
| Final R indexes [all data] | R ₁ = 0.0375, wR ₂ = 0.0778 | R ₁ = 0.0364, wR ₂ = 0.0710 | R ₁ = 0.0351, wR ₂ = 0.0765 |
| Largest diff. peak/hole / e Å ⁻³ | 0.97/-2.66 | 1.17/-0.93 | 1.37/-0.92 |

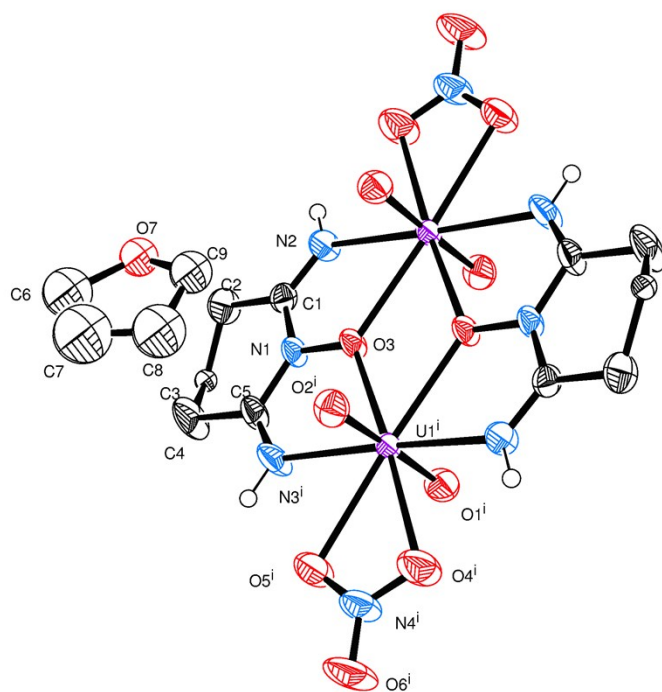


Figure S1. Crystal structure of complex **4** shown as thermal ellipsoids at 50% probability. Aliphatic hydrogen atoms omitted for clarity.

Table S2. Bond Length and Interatomic Distances for 4.

| Atom | Atom | Length/Å | Atom | Atom | Length/Å |
|------|-----------------|----------|------|-----------------|-----------|
| U1 | O2 | 1.759(4) | C1 | C2 | 1.501(8) |
| U1 | O1 | 1.766(4) | C2 | C3A | 1.479(17) |
| U1 | O3 | 2.464(4) | C2 | C3 | 1.500(13) |
| U1 | O3 ¹ | 2.468(4) | C3 | C4 | 1.554(14) |
| U1 | N3 | 2.480(5) | C3A | C4 | 1.318(18) |
| U1 | N2 | 2.493(5) | C4 | C5 | 1.507(8) |
| U1 | O4 | 2.519(5) | C5 | N3 ¹ | 1.273(8) |
| U1 | O5 | 2.546(5) | O7 | C9 | 1.36(2) |
| U1 | N4 | 2.970(5) | O7 | C6 | 1.51(2) |
| N1 | C5 | 1.358(8) | C6 | C7 | 1.18(4) |
| N1 | C1 | 1.363(7) | C7 | C8 | 1.34(4) |
| N1 | O3 | 1.393(5) | C8 | C9 | 1.37(4) |
| N2 | C1 | 1.259(8) | O7A | C6A | 1.34(2) |
| N3 | C5 ¹ | 1.273(8) | O7A | C9A | 1.45(2) |
| N4 | O6 | 1.216(7) | C6A | C7A | 1.52(3) |
| N4 | O4 | 1.256(8) | C7A | C8A | 1.50(3) |
| N4 | O5 | 1.259(8) | C8A | C9A | 1.50(3) |
| O3 | U1 ¹ | 2.468(4) | | | |

¹-X,-Y,-Z

Table S3. Bond Angles for 4.

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|-----------------|------|-----------------|------------|-----------------|------|-----------------|------------|
| O2 | U1 | O1 | 177.9(2) | C5 | N1 | O3 | 116.2(4) |
| O2 | U1 | O3 | 91.07(18) | C1 | N1 | O3 | 116.7(4) |
| O1 | U1 | O3 | 90.59(18) | C1 | N2 | U1 | 124.5(4) |
| O2 | U1 | O3 ¹ | 91.76(19) | C5 ¹ | N3 | U1 | 124.5(4) |
| O1 | U1 | O3 ¹ | 90.23(19) | O6 | N4 | O4 | 121.8(6) |
| O3 | U1 | O3 ¹ | 60.67(14) | O6 | N4 | O5 | 122.7(6) |
| O2 | U1 | N3 | 89.1(2) | O4 | N4 | O5 | 115.5(5) |
| O1 | U1 | N3 | 91.1(2) | O6 | N4 | U1 | 178.6(6) |
| O3 | U1 | N3 | 122.61(15) | O4 | N4 | U1 | 57.1(3) |
| O3 ¹ | U1 | N3 | 61.96(15) | O5 | N4 | U1 | 58.3(3) |
| O2 | U1 | N2 | 86.8(2) | N1 | O3 | U1 | 120.2(3) |
| O1 | U1 | N2 | 92.7(2) | N1 | O3 | U1 ¹ | 120.4(3) |
| O3 | U1 | N2 | 62.04(15) | U1 | O3 | U1 ¹ | 119.33(14) |
| O3 ¹ | U1 | N2 | 122.65(14) | N4 | O4 | U1 | 98.1(4) |
| N3 | U1 | N2 | 173.94(19) | N4 | O5 | U1 | 96.7(4) |
| O2 | U1 | O4 | 92.1(2) | N2 | C1 | N1 | 116.6(5) |
| O1 | U1 | O4 | 85.8(2) | N2 | C1 | C2 | 126.0(6) |
| O3 | U1 | O4 | 125.14(15) | N1 | C1 | C2 | 117.4(5) |
| O3 ¹ | U1 | O4 | 172.93(16) | C3A | C2 | C1 | 114.0(8) |
| N3 | U1 | O4 | 112.19(18) | C3 | C2 | C1 | 113.0(6) |
| N2 | U1 | O4 | 63.50(17) | C2 | C3 | C4 | 107.8(8) |
| O2 | U1 | O5 | 87.7(2) | C4 | C3A | C2 | 123.6(13) |
| O1 | U1 | O5 | 90.5(2) | C3A | C4 | C5 | 117.3(9) |
| O3 | U1 | O5 | 174.57(14) | C5 | C4 | C3 | 110.9(7) |
| O3 ¹ | U1 | O5 | 124.64(14) | N3 ¹ | C5 | N1 | 116.7(5) |
| N3 | U1 | O5 | 62.68(17) | N3 ¹ | C5 | C4 | 126.8(6) |
| N2 | U1 | O5 | 112.60(16) | N1 | C5 | C4 | 116.5(6) |
| O4 | U1 | O5 | 49.66(17) | C9 | O7 | C6 | 102.3(15) |
| O2 | U1 | N4 | 90.3(2) | C7 | C6 | O7 | 109(2) |
| O1 | U1 | N4 | 87.6(2) | C6 | C7 | C8 | 110(3) |
| O3 | U1 | N4 | 149.90(15) | C7 | C8 | C9 | 111(3) |
| O3 ¹ | U1 | N4 | 149.32(15) | O7 | C9 | C8 | 104(2) |
| N3 | U1 | N4 | 87.48(17) | C6A | O7A | C9A | 109.7(15) |
| N2 | U1 | N4 | 88.03(17) | O7A | C6A | C7A | 109.2(16) |
| O4 | U1 | N4 | 24.76(17) | C8A | C7A | C6A | 108.1(17) |
| O5 | U1 | N4 | 24.91(16) | C9A | C8A | C7A | 101.8(17) |
| C5 | N1 | C1 | 127.1(5) | O7A | C9A | C8A | 111.0(16) |

¹-X,-Y,-Z

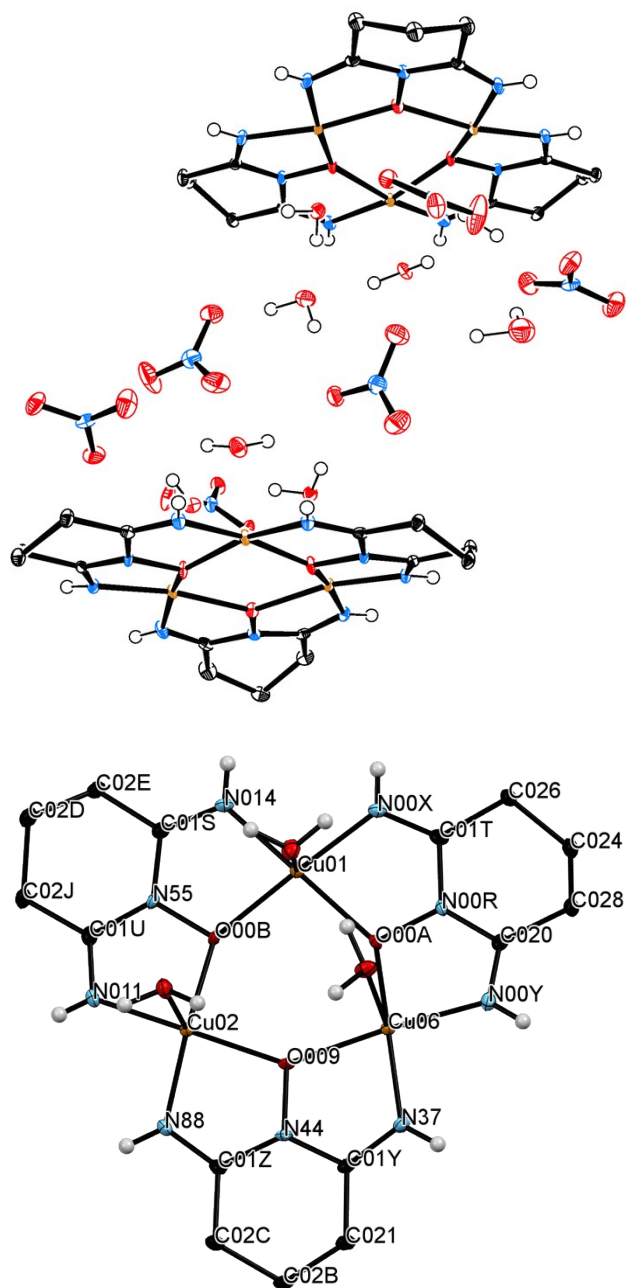


Figure S2. Full crystal structure (top) of complex **5** shown as thermal ellipsoids at 50% probability and crystal formula unit with atom labels (bottom). Copper atoms labeled tan, nitrogen atoms labeled blue, and oxygen atoms labeled red. Aliphatic hydrogen atoms omitted.

Table S4. Selected Bond Lengths for 5.

| Atom1 | Atom2 | Length/Å | Atom1 | Atom2 | Length/Å |
|-------|-------|----------|-------|-------|----------|
| Cu01 | O00A | 2.004(1) | N00Y | C020 | 1.280(2) |
| Cu01 | O00B | 1.991(1) | N011 | C01U | 1.278(2) |

| | | | | | |
|------|------|----------|------|------|----------|
| Cu01 | O00H | 2.283(2) | N014 | C01S | 1.282(2) |
| Cu01 | N00X | 1.921(2) | N55 | C01S | 1.371(2) |
| Cu01 | N014 | 1.935(2) | N55 | C01U | 1.369(2) |
| Cu02 | O009 | 2.000(1) | N44 | C01Y | 1.373(2) |
| Cu02 | O00B | 1.980(1) | N44 | C01Z | 1.370(2) |
| Cu02 | O00D | 2.366(2) | N37 | C01Y | 1.278(2) |
| Cu02 | N011 | 1.939(2) | N88 | C01Z | 1.283(3) |
| Cu02 | N88 | 1.938(2) | C01S | C02E | 1.500(3) |
| Cu06 | O009 | 2.026(1) | C01T | C026 | 1.499(3) |
| Cu06 | O00A | 1.967(1) | C01U | C02J | 1.503(3) |
| Cu06 | O00G | 2.266(2) | C01Y | C021 | 1.500(2) |
| Cu06 | N00Y | 1.955(2) | C01Z | C02C | 1.497(2) |
| Cu06 | N37 | 1.921(2) | C020 | C028 | 1.503(3) |
| O009 | N44 | 1.400(2) | C021 | C02B | 1.524(3) |
| O00A | N00R | 1.401(2) | C024 | C026 | 1.526(3) |
| O00B | N55 | 1.407(2) | C024 | C028 | 1.516(3) |
| N00R | C01T | 1.370(2) | C02B | C02C | 1.525(3) |
| N00R | C020 | 1.376(2) | C02D | C02E | 1.521(3) |
| N00X | C01T | 1.276(3) | C02D | C02J | 1.528(3) |

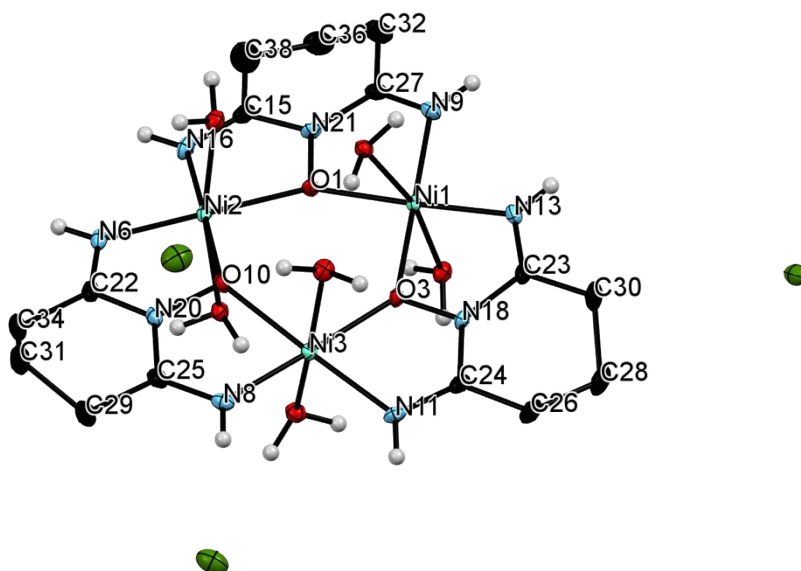


Figure S3. Crystal structure of complex **6** shown as thermal ellipsoids at 50% probability. Nickel atoms labeled aqua, nitrogen atoms labeled blue, oxygen atoms labeled red, and chloride counter ions labeled green. Aliphatic hydrogen atoms omitted.

Table S5. Selected Bond Lengths for 6.

| Atom | Atom | Length/Å | Atom | Atom | Length/Å |
|------|------|----------|------|------|----------|
| Ni1 | O1 | 2.021(2) | O10 | N20 | 1.393(3) |
| Ni1 | O3 | 2.033(2) | N11 | C24 | 1.284(3) |
| Ni1 | O5 | 2.219(1) | N13 | C23 | 1.276(3) |
| Ni1 | N9 | 2.005(2) | C15 | N16 | 1.282(4) |
| Ni1 | O12 | 2.103(1) | C15 | N21 | 1.370(3) |
| Ni1 | N13 | 1.995(2) | C15 | C38 | 1.485(4) |
| Ni2 | O1 | 2.019(2) | N18 | C23 | 1.378(3) |
| Ni2 | O2 | 2.163(1) | N18 | C24 | 1.364(3) |
| Ni2 | N6 | 1.994(2) | N20 | C22 | 1.373(3) |
| Ni2 | O10 | 2.065(1) | N20 | C25 | 1.372(3) |
| Ni2 | O14 | 2.128(1) | N21 | C27 | 1.371(3) |
| Ni2 | N16 | 2.004(2) | C22 | C34 | 1.502(4) |
| Ni3 | O3 | 2.021(2) | C23 | C30 | 1.498(3) |
| Ni3 | O4 | 2.138(1) | C24 | C26 | 1.495(3) |
| Ni3 | O7 | 2.200(1) | C25 | C29 | 1.501(4) |
| Ni3 | N8 | 1.993(2) | C26 | C28 | 1.530(4) |
| Ni3 | O10 | 2.067(1) | C27 | C32 | 1.498(3) |
| Ni3 | N11 | 2.007(2) | C28 | C30 | 1.518(3) |
| O1 | N21 | 1.385(2) | C29 | C31 | 1.518(4) |
| O3 | N18 | 1.387(2) | C31 | C34 | 1.508(3) |
| N6 | C22 | 1.278(3) | C32 | C36 | 1.503(5) |
| N8 | C25 | 1.277(3) | C36 | C38 | 1.445(7) |
| N9 | C27 | 1.284(3) | | | |

ESI-MS DATA

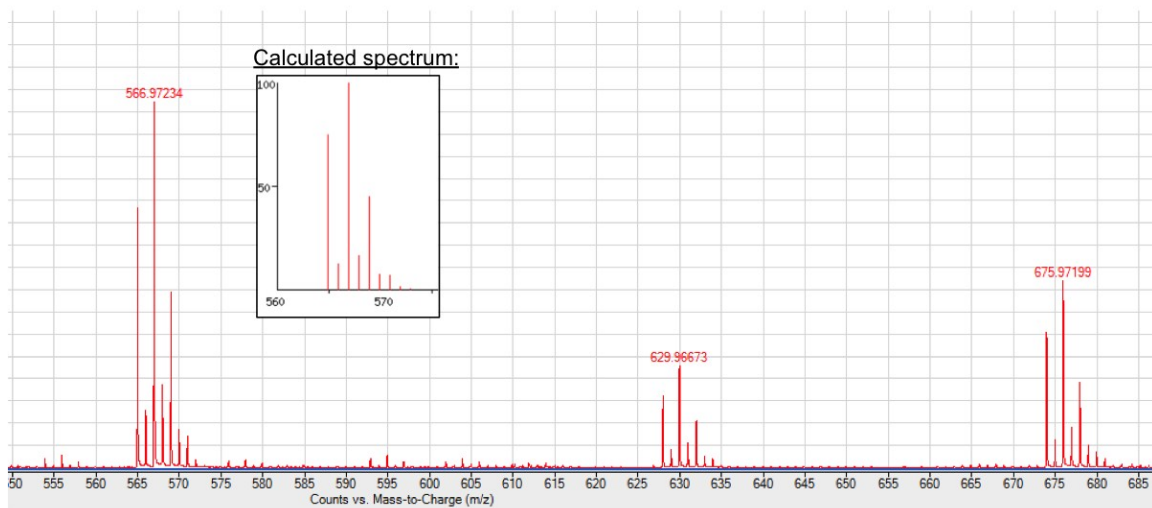


Figure S4. ESI-MS spectrum of complex **5** dissolved in H₂O with calculated mass spectrum for $[[\text{Cu}_3(\text{HL}^1)_3(\mu\text{-O})_3]\text{H}]^+$ at $m/z = 567$ (inset).

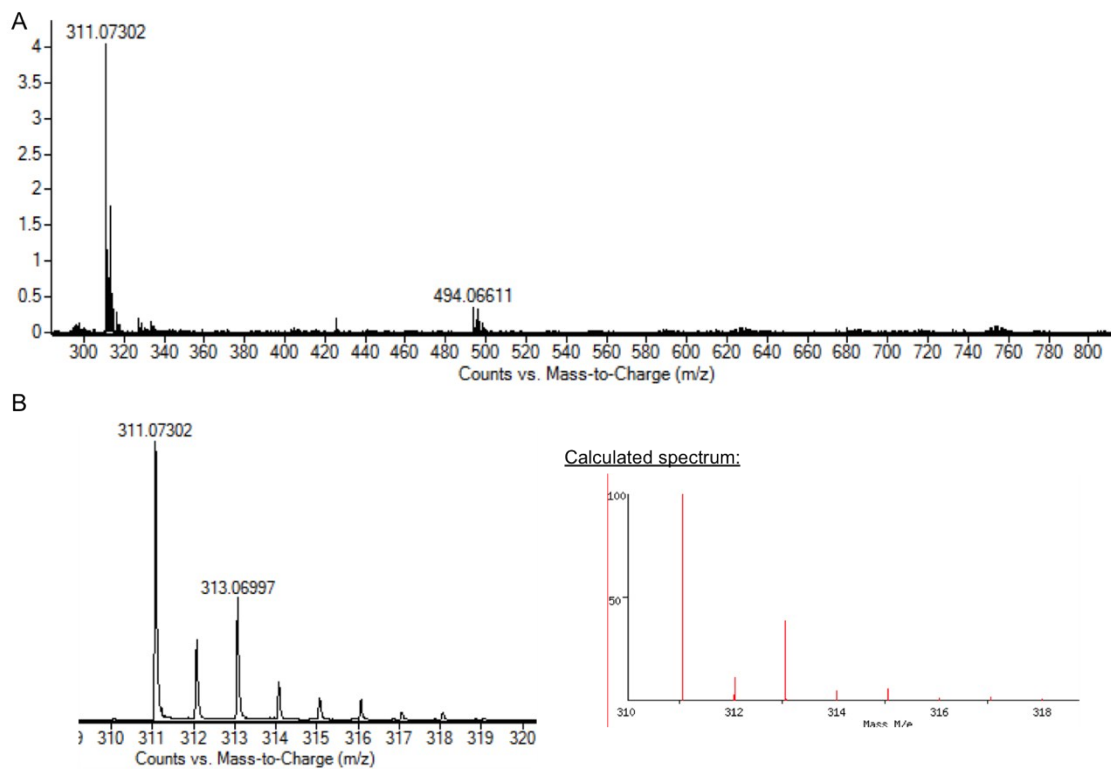


Figure S5. (A) ESI-MS spectrum of complex **6** dissolved in H₂O. (B) Zoom of isotopic pattern for [Ni(H₂L¹)₂(μ-O)₂+H]⁺ at m/z = 311 (left) and calculated pattern in this region (right)

NMR Spectra

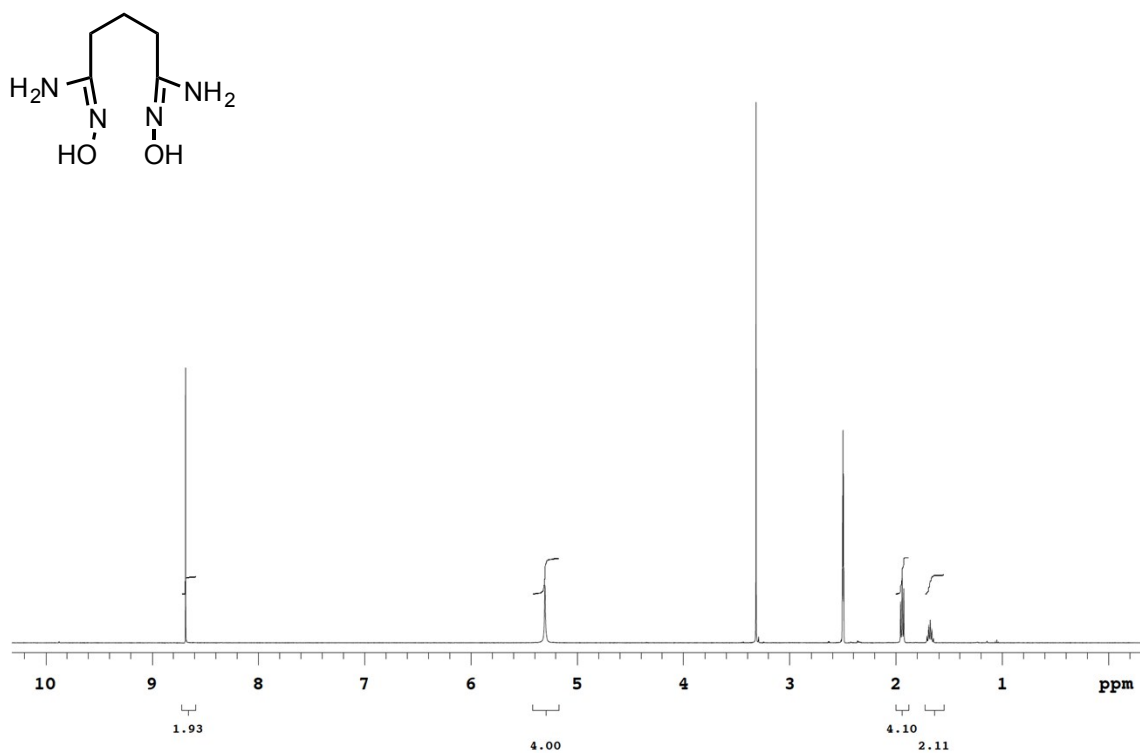


Figure S6. $^1\text{H-NMR}$ spectrum of H_2L^2 in DMSO-d_6 recorded at 500 MHz.

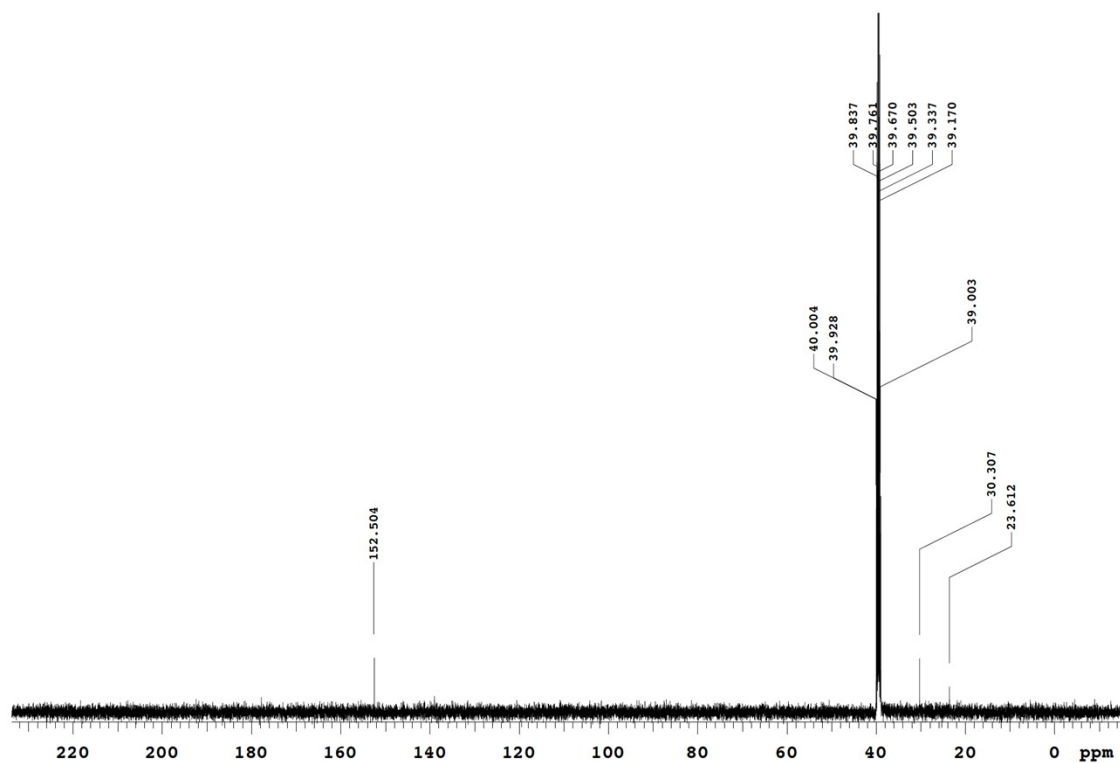


Figure S7. $^{13}\text{C-NMR}$ spectrum of H_2L^2 in DMSO-d_6 recorded at 500 MHz.

H_2L^2 (10 mM) + HCl (100 mM) experiment

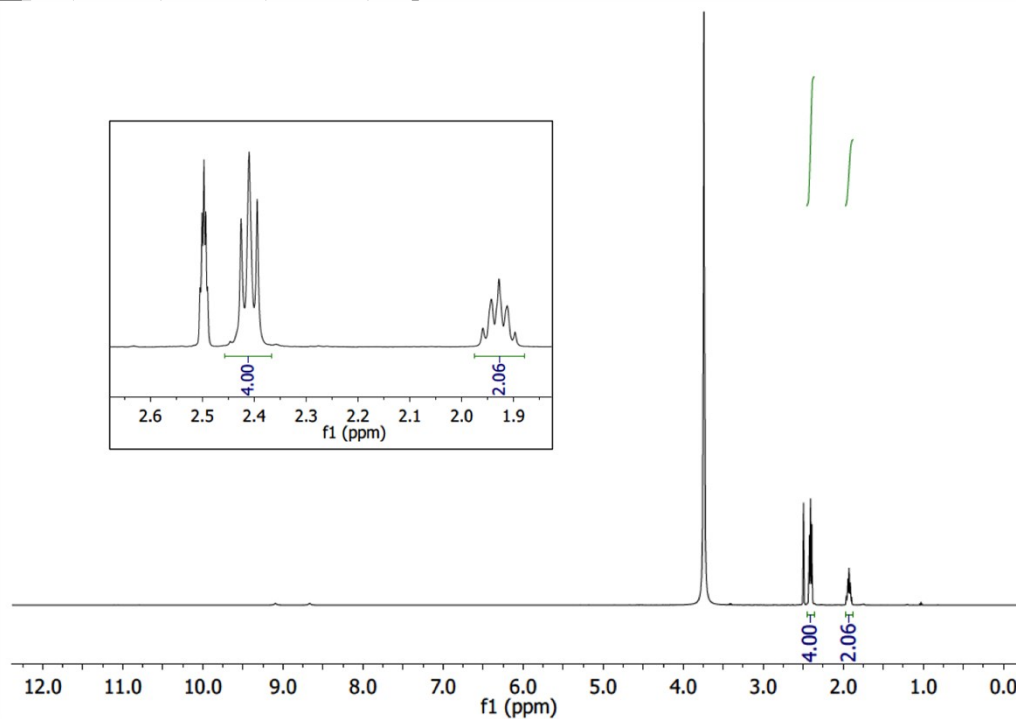


Figure S8. ^1H -NMR spectrum of H_2L^2 (10 mM) + HCl (100 mM) in 90% DMSO-d_6 / 10% H_2O recorded after 7 days at 500 MHz with zoom of upfield region (inset).

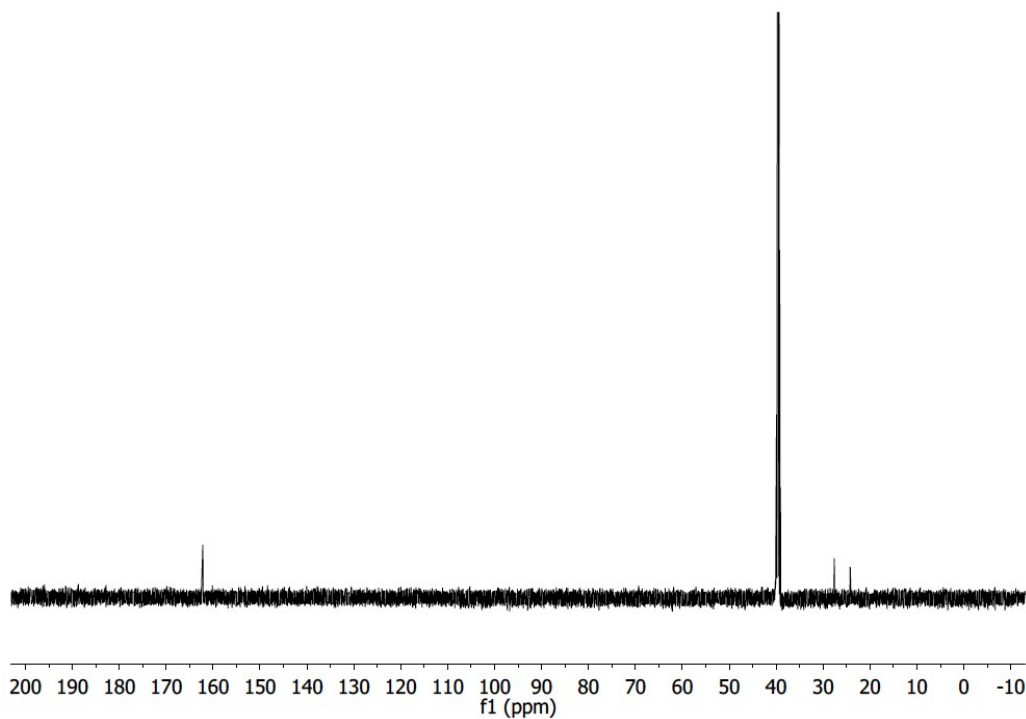


Figure S9. ^{13}C -NMR spectrum of H_2L^2 (10 mM) + HCl (100 mM) in 90% DMSO-d_6 / 10% H_2O recorded after 7 days at 500 MHz.

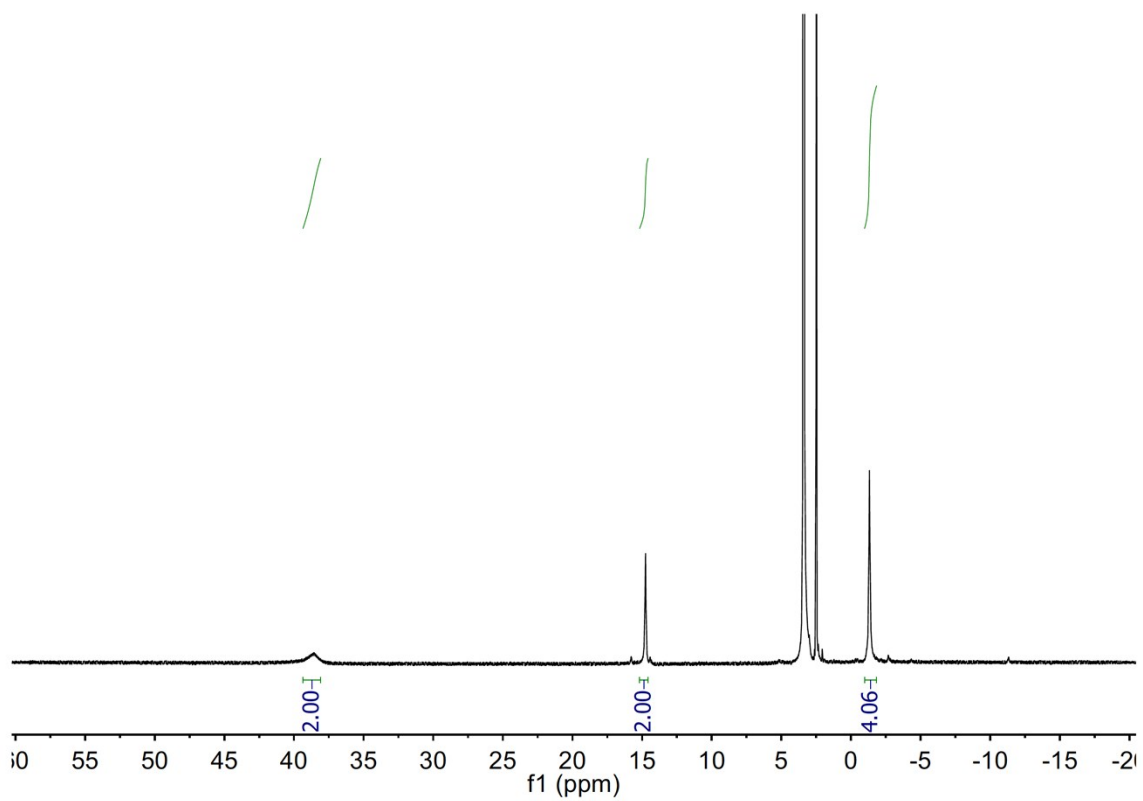


Figure S10. $^1\text{H-NMR}$ spectrum of complex **5** in DMSO-d_6 recorded at 500 MHz.

UV Spectra of Complexes 5 and 6

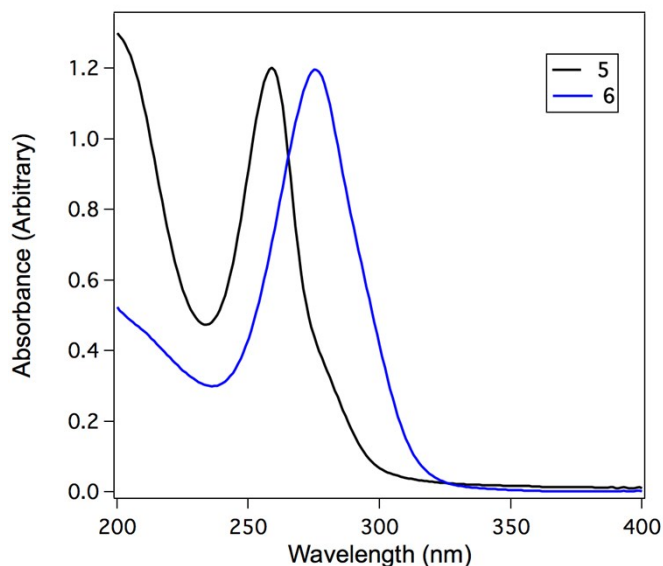


Figure S11. UV spectra for **5** ($[\text{Cu}_3(\text{H}_2\text{L}^1)_3(\mu\text{-O})_3(\text{H}_2\text{O})_3](\text{NO}_3)_3$) and **6** ($[\text{Ni}_3(\text{H}_2\text{L}^1)_3(\mu\text{-O})_3(\text{H}_2\text{O})_6]\text{Cl}_3$) in deionized H_2O .

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