Supporting Information for

Reactivity of Uranium(IV) Bridged Chalcogenido Complexes U^{IV}–E–U^{IV} (E = S, Se) with Elemental Sulfur and Selenium: Synthesis of Polychalcogenide-Bridged Uranium Complexes

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Experimental Section

General Methods. All experiments were performed under dry nitrogen atmosphere using standard Schlenk techniques or an MBraun inert-gas glovebox. All solvents were purified using a two-column solid-state purification system (Glasscontour System, Irvine, CA) and transferred to the glovebox without exposure to air, unless otherwise indicated.

Magnetism data of crystalline powdered samples (20 – 30 mg) were recorded with a SQUID magnetometer (Quantum Design) at 10 kOe (2 – 300 K for **3**, **4**, **5** and **7**). Values of the magnetic susceptibility were corrected for the underlying diamagnetic increment ($\chi_{dia} = -1275.04 \times 10^{6}$ cm^3mol^{-1} (**3**), -1258.54×10^{6} cm^3mol^{-1} (**4**), -1208.54×10^{6} cm^3mol^{-1} (**7**), -1339.18×10^{6} cm^3mol^{-1} (**5**)) by using tabulated Pascal constants and the effect of the blanksample holders (gelatin capsule/ straw).[1] Samples used for magnetization measurement were recrystallized multiple times and checked for chemical composition and purity by elemental analysis (C, H, N and S) and ^{1}H NMR spectroscopy. Data reproducibility was also carefully checked on independently synthesized samples. Results from elemental analysis were obtained from the Analytical Laboratories at the Friedrich-Alexander-University Erlangen-Nürnberg (Erlangen, Germany) on Euro EA 3000. ^{1}H NMR spectra were recorded on JEOL 270 and 400 MHz instruments, operating at the respective frequencies of 269.714 and 400.178 MHz with a probe temperature of 23 °C in benzene-d_6. Chemical shifts were referenced to *protio* solvent impurities (δ 7.09 (benzene-d_6)) and are reported in parts per million (ppm).

Starting Materials. Precursor complexes [(UI_3(dioxane)$_{1,5}$] and [$U(N(SiMe_3)_2)_3$] were prepared as described by Kiplinger *et al.*[2] The tris(2-hydroxy-3-adamantyl-5-methylbenzyl)amine ligand was synthesized from a modified literature procedure.[3] The uranium starting material [$((^{Ad}ArO)_3N)U^{III}(DME)$] and the two bridging chalcogenides [$\{((^{Ad}ArO)_3N)U(DME)\}_2(\mu\text{-S})$] and [$\{((^{Ad}ArO)_3N)U(DME)\}_2(\mu\text{-Se})$] were synthesized according to literature procedures.[4, 5] Elemental selenium powder (≥ 99.5 %) and elemental sulfur (≥ 99.5 %) were purchased from Sigma-Aldrich and used without further purification. Anhydrous 1-propanol (99.7 %) and anhydrous 1,2-dimethoxyethane (≥ 99 %) were purchased from Sigma-Aldrich. THF and 1,2-dimethoxyethane were further dried by distilling over sodium benzophenone. Triphenylphosphine sulfide (99.8 %) and triphenylphosphine selenide (≥ 98 %) were purchased from Sigma-Aldrich and used as received.

Synthesis of [$\{((^{Ad}ArO)_3N)U\}_2(\mu\text{-}Se_2)(\mu\text{-DME})$] (3). Selenium Powder (8 mg, 0.10 mmol) was added as a suspension in DME (~ 1mL) to a stirring suspension of [$((^{Ad}ArO)_3N)U^{III}(DME)$] (**1**) (100 mg, 0.09 mmol) in DME (2 mL). The solution turned to brown after 10 min. and was stirred for another 6 h. The reaction mixture was filtered and the volatiles were removed *in vacuo*. Brown crystals of **3** can be obtained from a concentrated solution of DME. Yield: 85.0 mg (0.037 mmol, 83 %).

Alternatively, complex **3** can be synthesized from the addition of a suspension of selenium powder (6.8 mg, 0.08 mmol) in 1 mL benzene to a stirring solution of [{((^{Ad}ArO)$_3$N)U(DME)}$_2$(μ-Se)] (100 mg, 0.04 mmol) in 3 mL benzene. Over 10 min., the solution turned brown and was stirred for another 6 h. The reaction mixture was filtered through a pipet and the volatiles removed *in vacuo*. Yield: 78 mg (0.034 mmol, 85 %). ^{1}H NMR (270 MHz, C_6D_6): δ [ppm] = 52.54, 51.18, 50.24, 44.22, 29.76, 25.12, 24.30, 21.31, 10.26, 8.16, 4.43, 4.02, 2.74, -1.04, -2.08, -3.90, -9.47, -10.95, -13.92, -15.47, -18.37, -29.86, -36.47, -41.94, -43.82, -45.71, -49.55, -52.36, -55.13, -89.92. Elemental analysis (%) calcd. for [{((^{Ad}ArO)$_3$N)U}$_2$(μ-Se$_2$)(μ-DME)]· DME, C 58.63, H 6.64, N 1.14; found C 58.57, H 6.51, N 1.17.

Synthesis of [{((^{Ad}ArO)$_3$N)U}$_2$(μ-η^3: η^3-Se$_4$)] (4). Selenium Powder (29 mg, 0.36 mmol) was added as a suspension in DME (~ 1mL) to a stirring suspension of **1** (100 mg, 0.09 mmol) in DME (3 mL). The solution turned to brown after 10 min. and was stirred for another 3 days after which an olive green precipitate formed. The reaction mixture was filtered, washed with cold DME (~ 1 mL) and dried *in vacuo*. Brown crystals of **4** can be obtained by diffusion of *n*-hexane into a concentrated toluene solution. Yield: 92.8 mg (0.040 mmol, 90 %).

Alternatively, complex **4** can be synthesized by reacting either **2** or **3** with stoichiometric amounts of selenium powder for 3 days after which an olive green precipitate forms. The reaction was filtered, washed with cold DME (~ 1 mL) and dried *in vacuo*. Yields: 79-81 % ^{1}H NMR (270 MHz, C_6D_6): δ [ppm] = 53.94, 52.09, 31.23, 13.23, 13.04, 10.44, 9.05, 4.92, 4.35, -0.63, -1.56, -9.04, -11.74, -14.79, -17.87, -32.05, -43.46, -45.98, -69.04. Elemental analysis (%) calcd. for [{((^{Ad}ArO)$_3$N)U}$_2$(μ-η^3: η^3-Se$_4$)]· DME, C 55.22, H 5.87, N 1.15; found C 55.57, H 6.17, N 1.02.

Synthesis of [{((^{Ad}ArO)$_3$N)U(THF)}$_2$(μ-η^2: η^2-Se$_4$)] (5). Selenium Powder (29 mg, 0.36 mmol) was added as a suspension in THF (~ 1mL) to a stirring suspension of **1** (100 mg, 0.09 mmol) in THF (3 mL). The solution turned to brown after 10 min. and was stirred for another 3 days after which an olive green precipitate formed. The reaction mixture was filtered, washed with cold THF (~ 1 mL) and dried *in vacuo*. Brown crystals of **5** can be obtained by diffusion of *n*-hexane into a concentrated toluene solution. Yield: 81.5 mg (0.033 mmol, 73 %).

Alternatively, complex **5** can be synthesized by reacting either **2** or **3** with stoichiometric amounts of selenium powder for 3 days in THF after which an olive green precipitate forms or by suspending complex **4** in THF and stirring the reaction mixture for 2 h. The reaction mixture was filtered, washed with cold DME (~ 1 mL) and dried *in vacuo*. Yields: 70-78 % ^{1}H NMR (270 MHz, C_6D_6): δ [ppm] = 53.60, 51.53, 15.10, 14.41, 13.09, 11.78, 10.27, 9.03, 7.39, 6.80, 4.80, 4.32, 3.24, 2.09, 1.17, 1.06, 0.81, 0.24, -0.69, -1.42, -8.97, -9.78, -11.55, -13.65, -14.50, -17.62, -19.72, -24.56, -31.72, -43.19, -45.80, -68.93. Elemental analysis (%) calcd. for [{((^{Ad}ArO)$_3$N)U(THF)}$_2$(μ-η^2: η^2-Se$_4$)]· 2 THF, C 55.95, H 5.99, N 1.12; found C 55.79, H 6.39, N 1.13.

Synthesis of [{((^{Ad}ArO)$_3$N)U}$_2$(μ-S$_2$)$_2$] (7). Elemental sulfur (6 mg, 0.09 mmol) was added as a suspension in DME (~ 1mL) to a stirring suspension of [((^{Ad}ArO)$_3$N)U^{III}(DME)] (100 mg, 0.09 mmol) in DME (2 mL). The solution immediately turned to black and was stirred for another 2 h. The reaction mixture was filtered, washed with cold DME (~ 1 mL) and dried *in vacuo*. Dark brown crystals of **7** can be obtained from a concentrated solution of DME or benzene. Yield: 75.2 mg (0.035 mmol, 78 %).

Alternatively, complex **7** can be synthesized from the addition of a suspension of elemental sulfur (1.4 mg, 5.6 µmol) in 1 mL DME to a stirring solution of [{((^{Ad}ArO)$_3$N)U(DME)}$_2$(μ-S)] (100 mg, 0.04 mmol) in 3 mL DME. Over 10 min., the solution turned black and was stirred for another 3 h. The reaction mixture was filtered and dried *in vacuo*. Yield: 81.8 mg (0.038 mmol, 85 %) ^{1}H NMR (270 MHz, C$_6$D$_6$): δ [ppm] = 22.89, 13.14, 12.12, 11.94, 11.25, 10.05, 8.80, 8.07, 7.64, 7.59, 7.10, 6.85, 6.74, 6.67, 6.63, 6.44, 6.41, 6.14, 5.94, 5.69, 5.65, 5.53, 4.19, 3.69, 3.61, 3.39, 3.30, 3.27, 3.04, 2.96, 2.92, 2.47, 2.43, 2.12, 1.33, 1.24, 1.12, 1.07, 1.01, 0.96, 0.88, 0.84, 0.76, 0.73, 0.61, 0.56, 0.41, 0.38, 0.30, 0.26, 0.24, 0.12, 0.07, 0.04, -0.05, -0.10, -0.39, -0.42, -1.44, -3.24, -3.28, -13.79, -13.82, -15.11. Elemental analysis (%) calcd. for [{((^{Ad}ArO)$_3$N)U}$_2$(μ-S$_2$)$_2$]· DME, C 60.09, H 6.16, N 1.30, S 5.94; found C 60.04, H 6.15, N 1.25, S 5.17.

X-Ray Crystal Structure Determinations

CCDC-965196 (for **3**), CCDC-965197 (for **4**), CCDC-965198 (for **5**), and CCDC-965199 (for **7**) contain the supplementary crystallographic data for this paper. This data can be obtained free of charge via http://www.ccdc.cam.ac.uk/products/csd/request/
(or from Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge, CB2 1EZ, UK. fax: ++44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

Crystallographic Details. Brown prisms of **3** grown from a concentrated solution of DME, brown blocks of of **4** and **5** grown from diffusion of *n*-hexane into a concentrated toluene solution, and dark brown prisms of **7** grown from further concentrating a THF solution *via* solvent diffusion into DME were coated with isobutylene oil on a microscope slide. Intensity data were collected using Mo$K\alpha$ radiation (λ = 0.71073 Å) either at 120 K on a Bruker Kappa APEX 2 *IµS* Duo diffractometer equipped with QUAZAR focusing Montel optics for compound **4**, at 100 K on a Bruker-Nonius KappaCCD (graphite monochromator) for compounds **5**, or at 100 K on a Bruker-SMART APEX2 (graphite monochromator) for compounds **3** and **7**. Data were corrected for Lorentz and polarization effects, semi-empirical absorption corrections were performed on the basis of multiple scans using *SADABS*.[6] All structures were solved by direct methods and refined by full-matrix least-squares procedures on F^2 using *SHELXTL NT* 6.12.[7] All hydrogen atoms were placed in positions of optimized geometry, their isotropic displacement parameters were tied to those of the corresponding carrier atoms by a factor of 1.2 or 1.5.

Compound **3** crystallized with two molecules of DME both of which were disordered. Two alternative orientations were refined in each case with the following refined site occupancies for the affected atoms of 65(2) and 35(2) for O10 and O10A in the case of the first DME and of 47.9(8) and 52.1(8) % for O13, C117, C119 and O14, C121, C122, respectively, in the case of the second DME.

In the investigated crystal of compound **4** disorder was observed in the central U-Se_4-U unit with a small fraction (< 7 %) of the corresponding bis μ-selenido complex $[\{((^{Ad}ArO)_3N)U\}_2(\mu\text{-Se})_2]$ being present. Compound **4** crystallized with a total of two molecules of toluene and 0.25 molecules of *n*-hexane per formula unit. One of the toluene molecules was disordered over two different sites. The refined site occupancies were 69.1(5) and 30.9(5) % for the atoms C201 – C207 and C211 – C217, respectively. The second site (atoms C211 – C217) suffered from further disorder (50 % each) as it was located on an inversion centre. The *n*-hexane molecule (atoms C401 – C403) was located on an inversion centre and this site was only partially occupied by 50 %. SIMU and ISOR restraints were applied in the treatment of the disordered solvent molecules.

Compound **5** is situated on a crystallographic inversion centre and exhibits C_i molecular symmetry.
The major part of the crystal structure is composed of the U-Se-Se-Se-Se-U complex (94.5(2) %). There seemed to be a small fraction of a U-Se-Se-Se-U compound (5.5(2) %) present with the Se_3 bridge being subjected to orientational disorder though. SIMU, ISOR, DFIX, and SADI restraints were applied in the refinement of the disordered Se_3 bridge.
The compound crystallized with a total of 2 molecules of toluene. This solvent molecule was disordered. Two alternative orientations were refined with resulting site occupancies of 53.1(9) and 46.9(9) %.

Compound **7** crystallized with two independent molecules of the U complex and five molecules of DME in the asymmetric unit. One of the DME molecules was disordered, with two alternative orientations refined for the atoms C404 – C406 (53.1(6) % occupancy) and C414 – C416 (46.9(6) % occupancy). Another DME was disordered over two different sites being occupied by 50 % each. SIMU restraints were applied in the refinement of the solvent molecules while ISOR restraints were applied for the atoms C190 and C194.

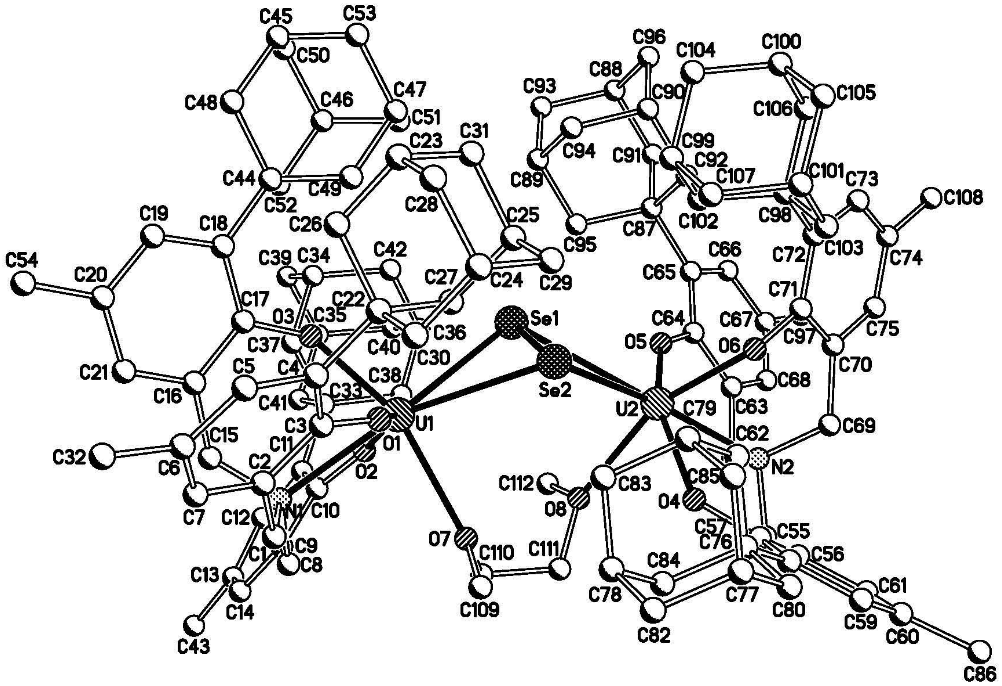


Fig. S1. Molecular structure of **3** with atom numbering scheme as balls & sticks representation (hydrogen atoms were omitted for clarity).

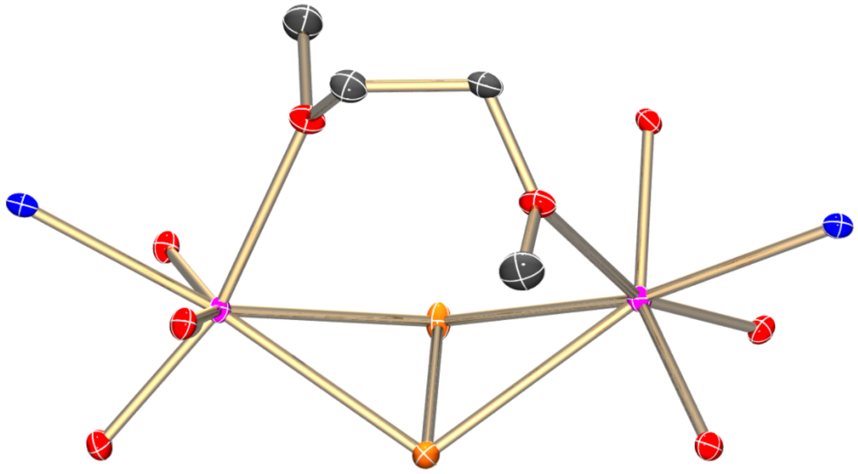


Fig. S2. Core structure of complex **3** (ellipsoids at 50 % probability, carbon and hydrogen atoms were omitted for clarity).

Crystal data and structure refinement for 3.

Identification code	sf1104	
Empirical formula	C120 H162 N2 O12 Se2 U2	
Formula weight	2458.50 g/mol	
Temperature	100(2) K	
Wavelength	0.71073 A	
Crystal system, space group	Triclinic, P−1	
Unit cell dimensions	a = 14.6186(1) Å	α = 90.0175(5) °
	b = 14.8584(1) Å	β = 93.0811(5) °
	c = 27.0913(3) Å	γ = 116.8421(4) °
Volume	5240.86(8) A^3	
Z, Calculated density	2, 1.558 Mg/m^3	
Absorption coefficient	3.844 mm^{-1}	
F(000)	2488	
Crystal size	0.42 x 0.28 x 0.16 mm	
Theta range for data collection	1.51 to 29.57°	
Limiting indices	-19<=h<=19, -20<=k<=19, -37<=l<=35	
Reflections collected / unique	109240 / 26524 [R(int) = 0.0502]	
Completeness to theta = 26.00	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.746 and 0.574	
Refinement method	Full-matrix-block least-squares on F^2	
Data / restraints / parameters	26524 / 0 / 1292	
Goodness-of-fit on F^2	1.022	
Final R indices [I>2sigma(I)]	R1 = 0.0299, wR2 = 0.0571	
R indices (all data)	R1 = 0.0457, wR2 = 0.0616	
Largest diff. peak and hole	1.284 and -0.767 e.A^{-3}	

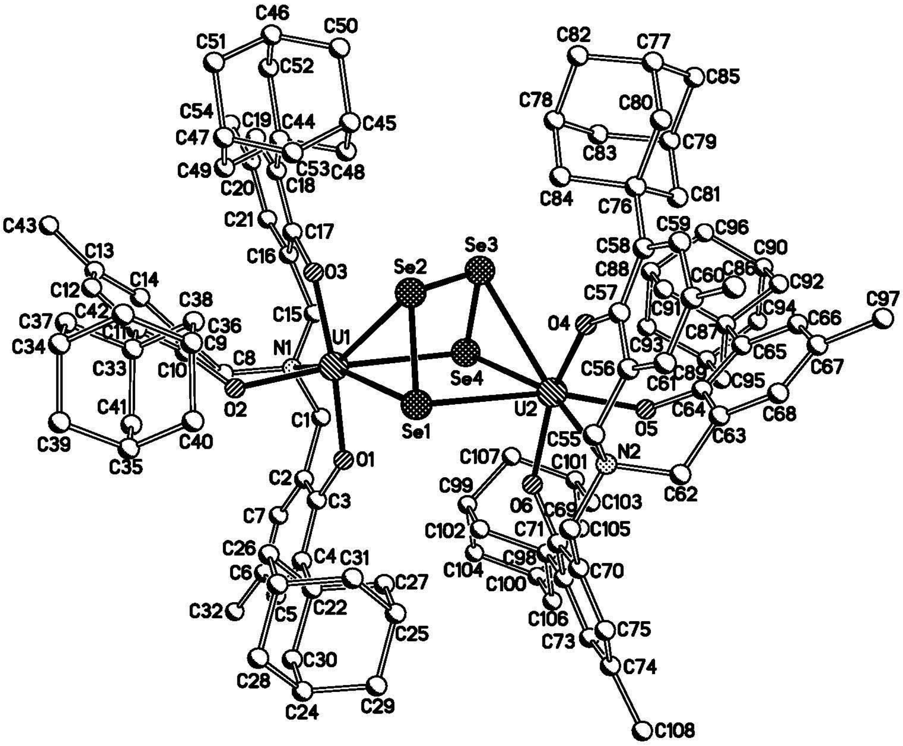


Fig. S3. Molecular structure of **4** with atom numbering scheme as balls & sticks representation (hydrogen atoms were omitted for clarity).

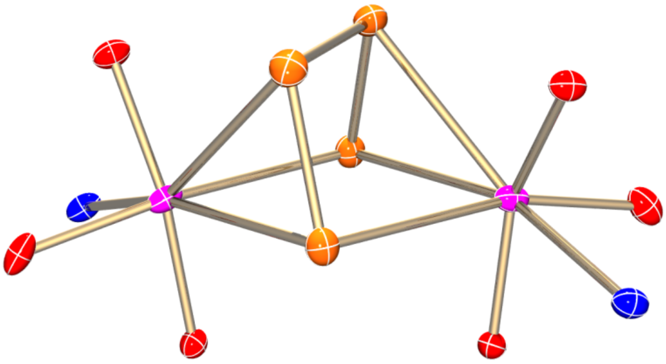


Fig. S4. Core structure of complex **4** (ellipsoids at 50 % probability, carbon and hydrogen atoms were omitted for clarity).

Crystal data and structure refinement for 4.

Identification code	sf1217a	
Empirical formula	C123.50 H151.50 N2 O6 Se3.86 U2	
Formula weight	2540.81 g/mol	
Temperature	120(2) K	
Wavelength	0.71073 A	
Crystal system, space group	Triclinic, P−1	
Unit cell dimensions	a = 11.2872(8) Å	α = 85.729(2) °
	b = 16.8161(12) Å	β = 87.925(2) °
	c = 28.943(2) Å	γ = 89.368(2) °
Volume	5474.6(7) A^3	
Z, Calculated density	2, 1.541 Mg/m^3	
Absorption coefficient	4.293 mm^{-1}	
F(000)	2539	
Crystal size	0.18 x 0.16 x 0.11 mm	
Theta range for data collection	2.92 to 27.88°	
Limiting indices	-14<=h<=14, -22<=k<=22, -38<=l<=38	
Reflections collected / unique	100676 / 26070 [R(int) = 0.0304]	
Completeness to theta = 27.88	99.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.604 and 0.489	
Refinement method	Full-matrix-block least-squares on F^2	
Data / restraints / parameters	26070 / 315 / 1339	
Goodness-of-fit on F^2	1.036	
Final R indices [I>2sigma(I)]	R1 = 0.0296, wR2 = 0.0752	
R indices (all data)	R1 = 0.0379, wR2 = 0.0789	
Largest diff. peak and hole	1.812 and -0.888 e.A^{-3}	

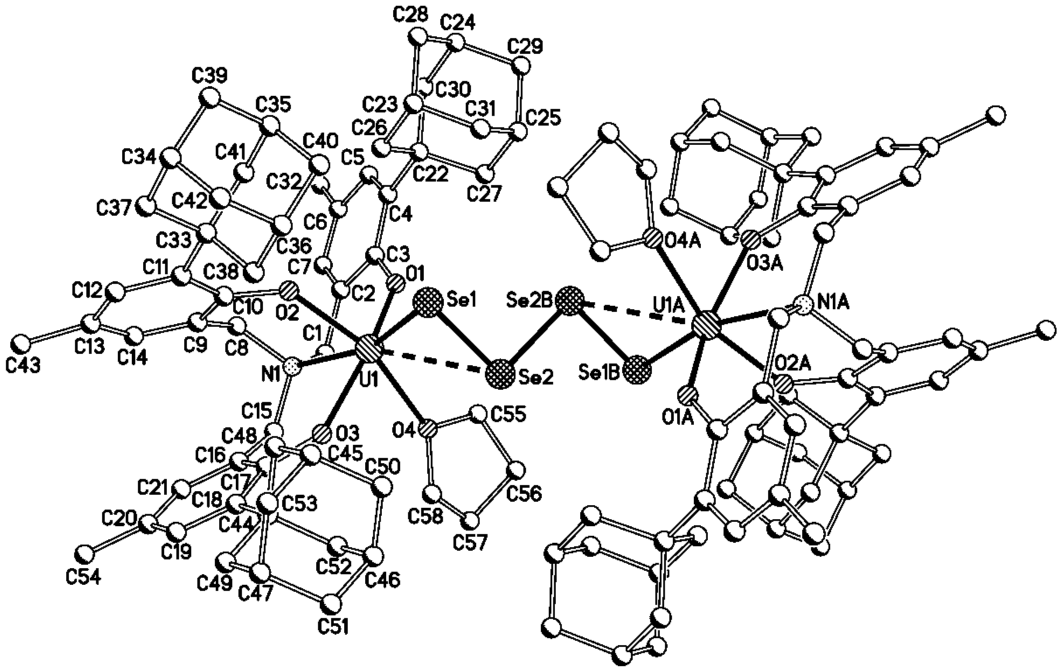


Fig. S5. Molecular structure of **5** with atom numbering scheme as balls & sticks representation (hydrogen atoms were omitted for clarity).

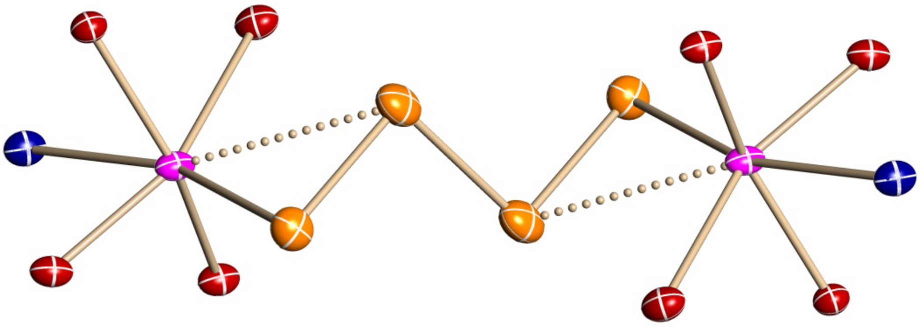


Fig. S6. Core structure of complex **5** (ellipsoids at 50 % probability, carbon and hydrogen atoms were omitted for clarity).

Crystal data and structure refinement for 5.

Identification code	sf1309
Empirical formula	C130 H164 N2 O8 Se4.11 U2
Formula weight	2683.22 g/mol
Temperature	100(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 18.092(2) Å α = 90 ° b = 12.3650(6) Å β = 118.117(12) ° c = 28.315(3) Å γ = 90 °
Volume	5586.8(9) A^{3}
Z, Calculated density	2, 1.595 Mg/m^{3}
Absorption coefficient	4.294 mm^{-1}
F(000)	2691
Crystal size	0.16 x 0.15 x 0.12 mm
Theta range for data collection	3.33 to 27.10°
Limiting indices	-23<=h<=23, -15<=k<=15, -36<=l<=36
Reflections collected / unique	85074 / 12292 [R(int) = 0.0640]
Completeness to theta = 27.10	99.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.746 and 0.601
Refinement method	Full-matrix-block least-squares on F^{2}
Data / restraints / parameters	12292 / 34 / 732
Goodness-of-fit on F^{2}	1.573
Final R indices [I>2sigma(I)]	R1 = 0.0441, wR2 = 0.0754
R indices (all data)	R1 = 0.0711, wR2 = 0.0802
Largest diff. peak and hole	1.937 and -1.597 e.A^{-3}

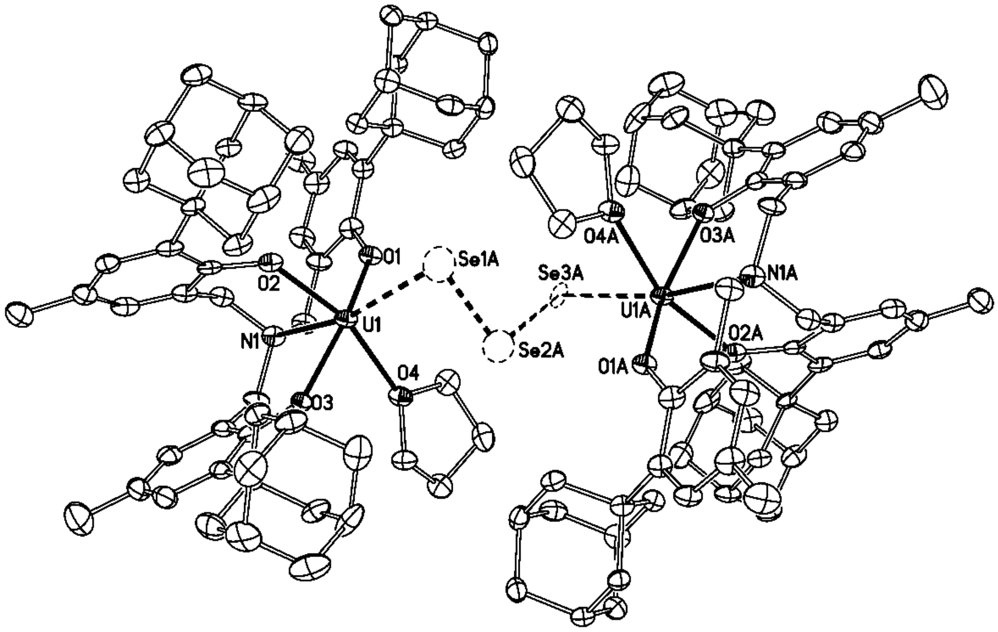


Fig. S7. Molecular structure of complex $[\{((^{Ad}ArO)_3N)U\}_2(\mu\text{-}Se_3)]$ with atom numbering scheme (ellipsoids at 50 % probability, hydrogen atoms were omitted for clarity). This compound was detected as a 5.5 % impurity in the crystallographic data of complex **5** and could not be isolated in pure form.

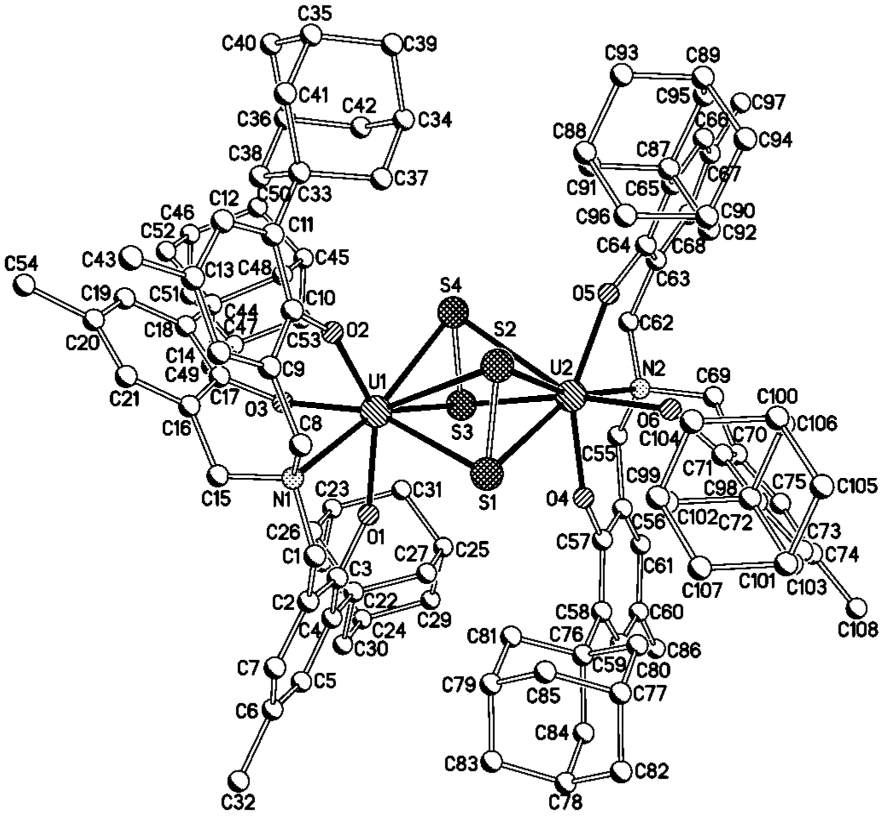


Fig. S8. Molecular structure of complex **7A** with atom numbering scheme as balls & sticks representation (hydrogen atoms were omitted for clarity).

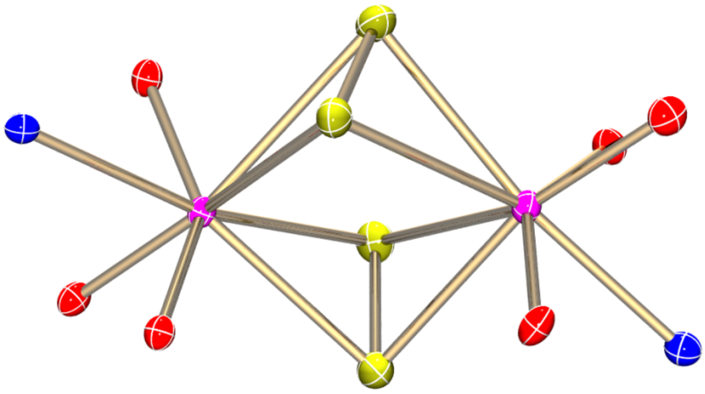


Fig. S9. Core structure of complex **7A** (ellipsoids at 50 % probability, carbon and hydrogen atoms were omitted for clarity).

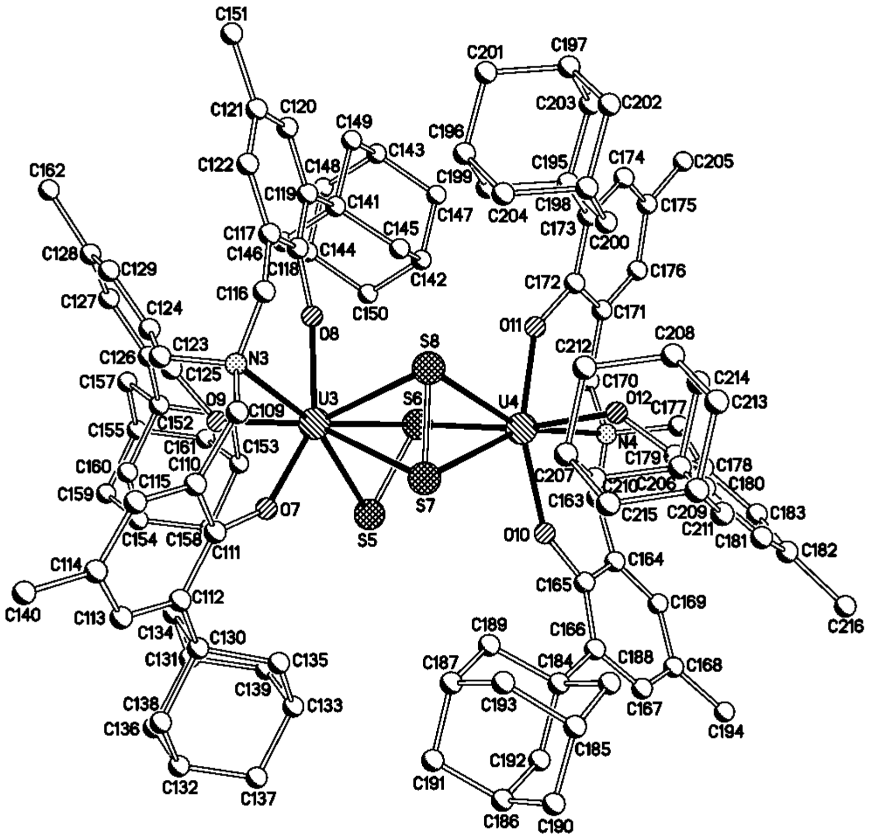


Fig. S10. Molecular structure of complex **7B** with atom numbering scheme as balls & sticks representation (hydrogen atoms were omitted for clarity).

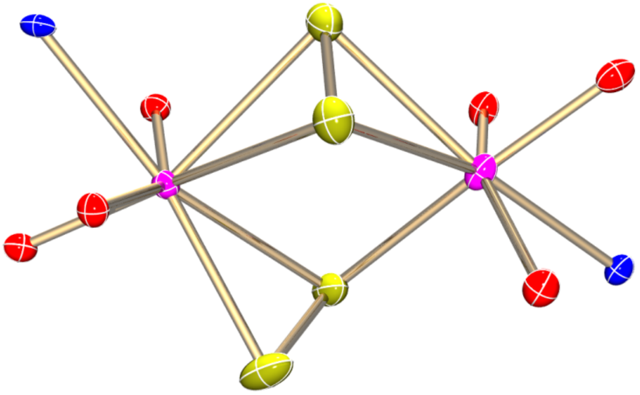


Fig. S11. Core structure of complex **7B** (ellipsoids at 50 % probability, carbon and hydrogen atoms were omitted for clarity).

Crystal data and structure refinement for 7.

Identification code	sf1117	
Empirical formula	C236 H314 N4 O22 S8 U4	
Formula weight	4767.51	
Temperature	100(2) K	
Wavelength	0.71073 A	
Crystal system, space group	Triclinic, P–1	
Unit cell dimensions	a = 20.5440(3) Å	α = 88.407(1) °
	b = 22.5155(3) Å	β = 84.055(1) °
	c = 24.1171(3) Å	γ = 75.504(1) °
Volume	10742.3(3) A^3	
Z, Calculated density	2, 1.474 Mg/m^3	
Absorption coefficient	3.149 mm^{-1}	
F(000)	4860	
Crystal size	0.48 x 0.46 x 0.0.32 mm	
Theta range for data collection	2.55 to 27.10 deg.	
Limiting indices	-25<=h<=25, -28<=k<=27, -30<=l<=30	
Reflections collected / unique	165269 / 43190 [R(int) = 0.0597]	
Completeness to theta = 25.00	98.5 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.501 and 0.314	
Refinement method	Full-matrix-block least-squares on F^2	
Data / restraints / parameters	43190 / 417 / 2570	
Goodness-of-fit on F^2	1.240	
Final R indices [I>2sigma(I)]	R1 = 0.0428, wR2 = 0.0980	
R indices (all data)	R1 = 0.0680, wR2 = 0.1063	
Largest diff. peak and hole	2.790 and -1.539 $e.A^{-3}$	

^{1}H NMR Spectroscopy

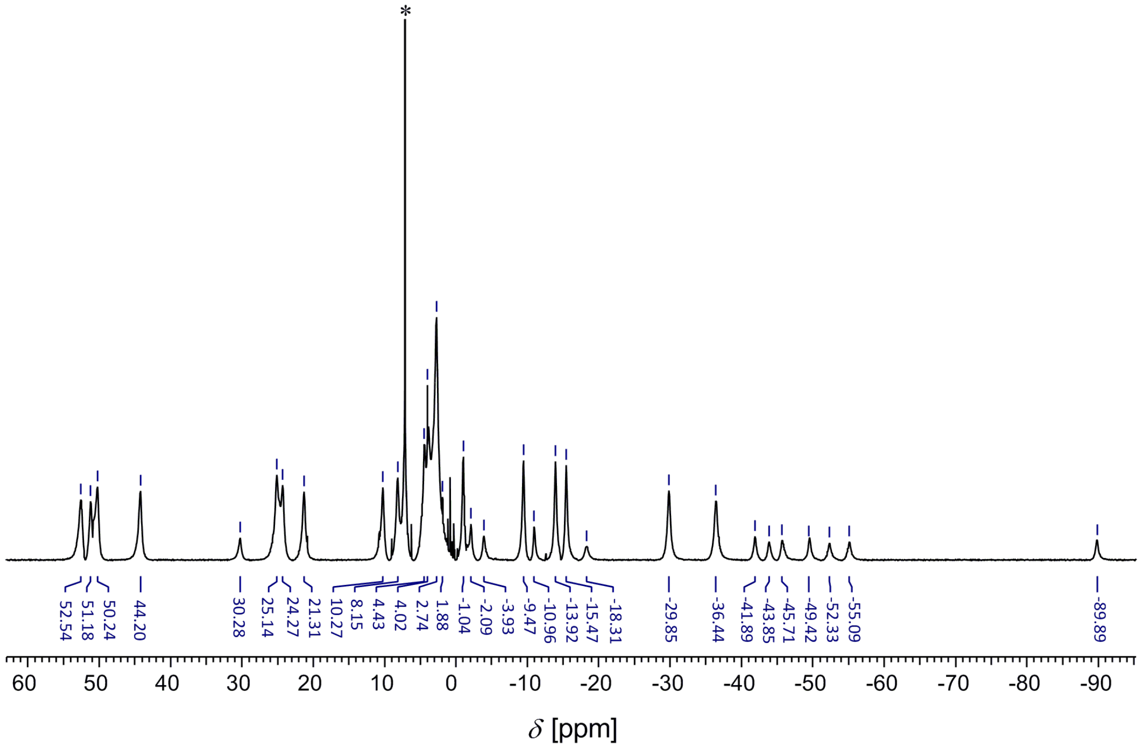


Fig. S12. ^{1}H NMR spectrum of **3** recorded in benzene-d_6.

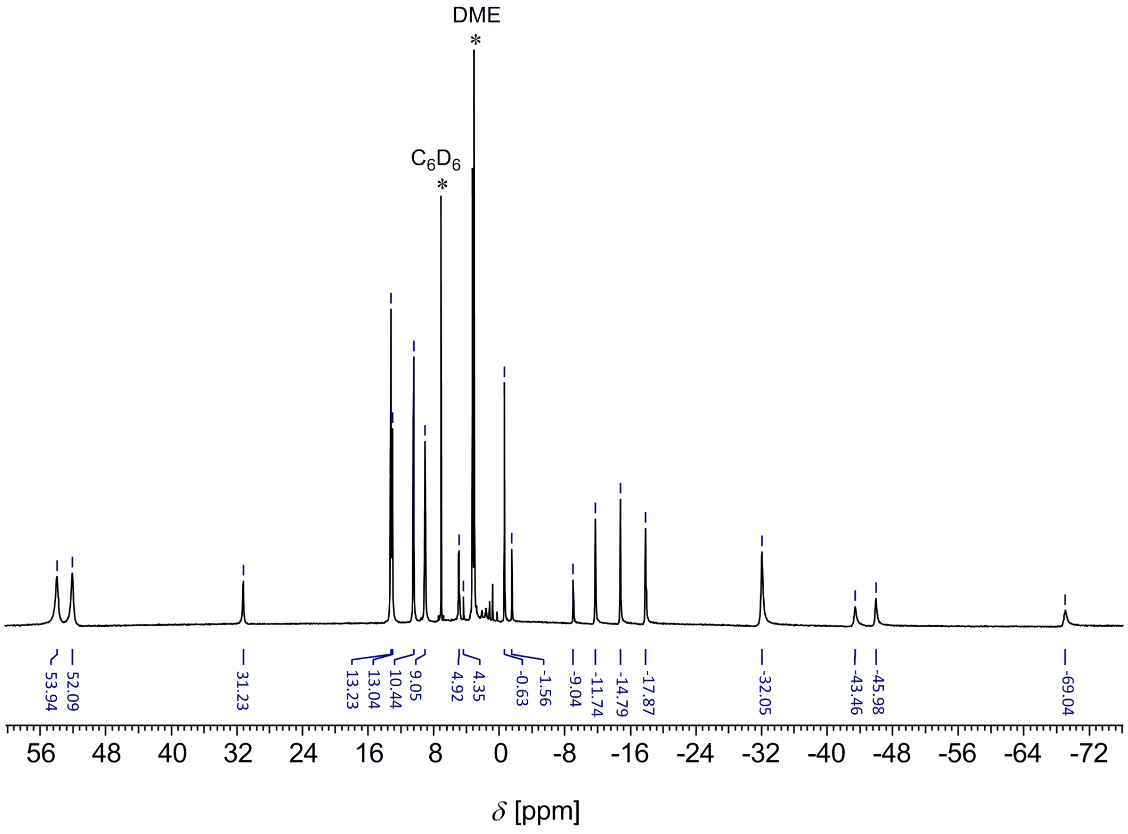


Fig. S13. ^{1}H NMR spectrum of **4** recorded in benzene-d_6.

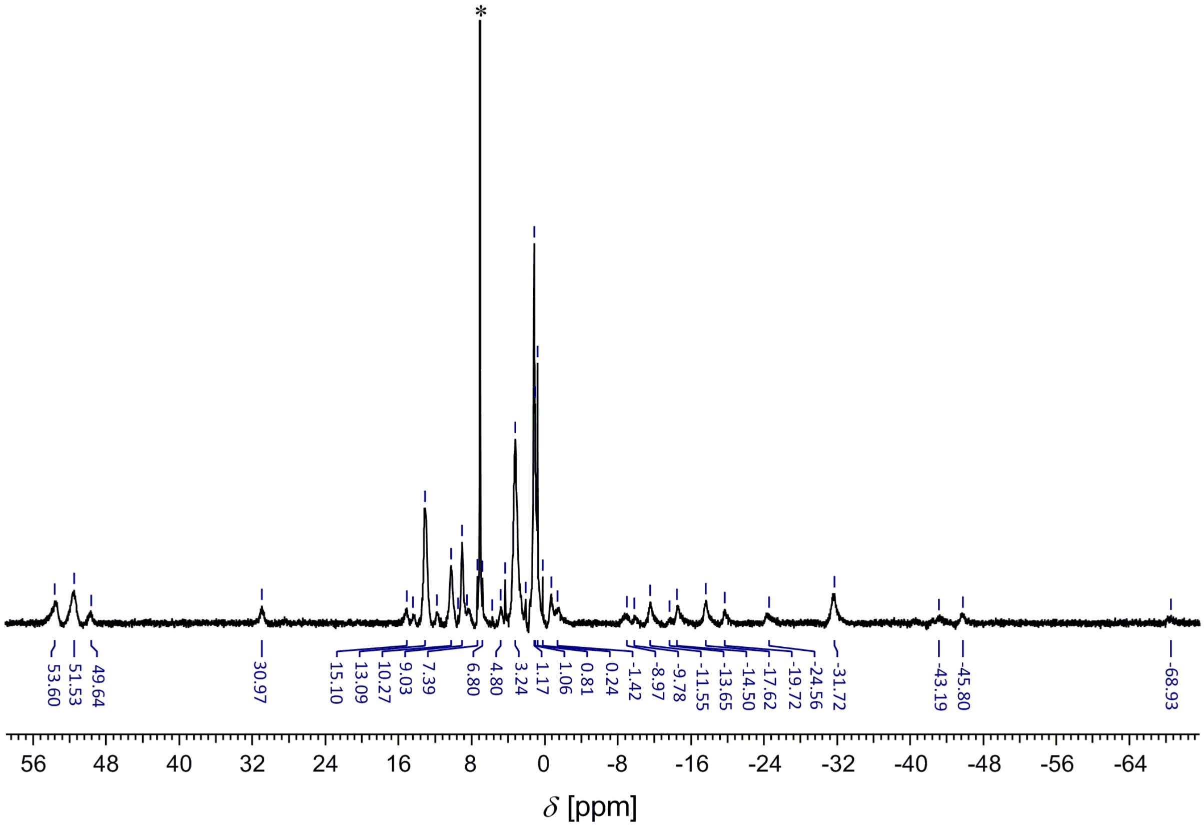


Fig. S14. ^{1}H NMR spectrum of **5** recorded in benzene-d_6.

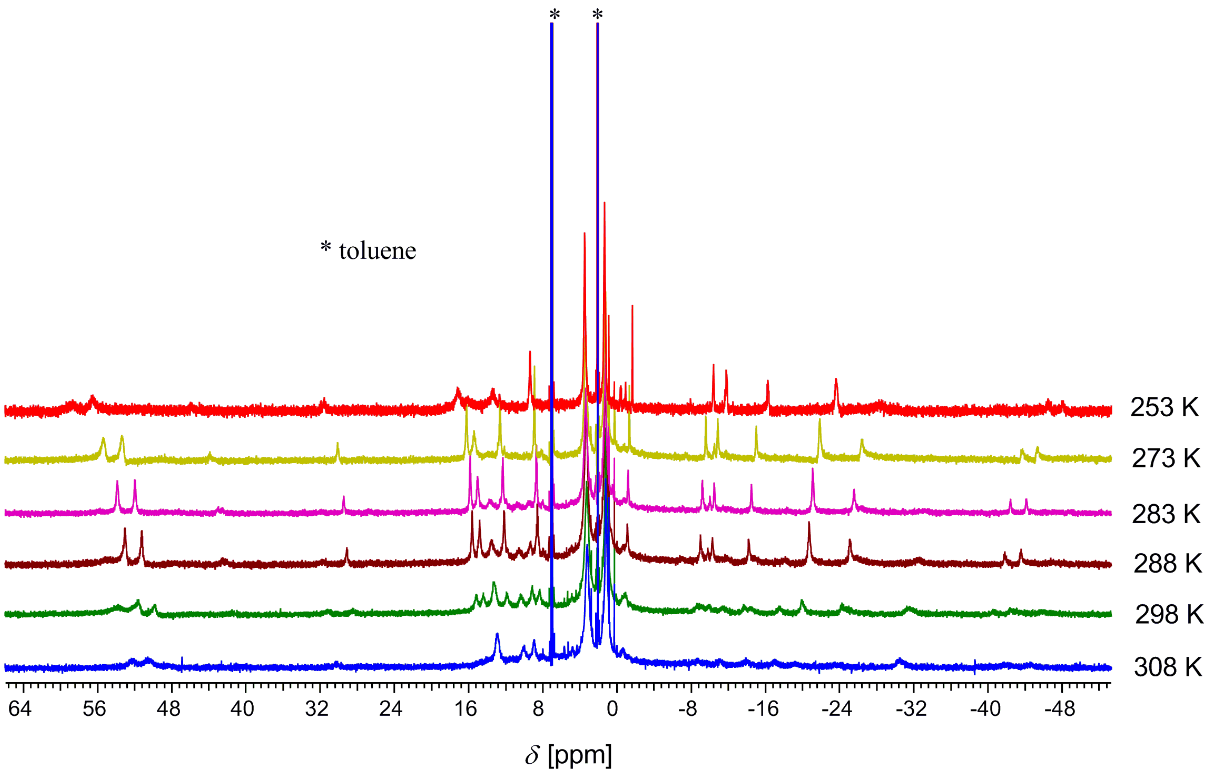


Fig. S15. VT-NMR spectra of **5** recorded in toluene-d_8.

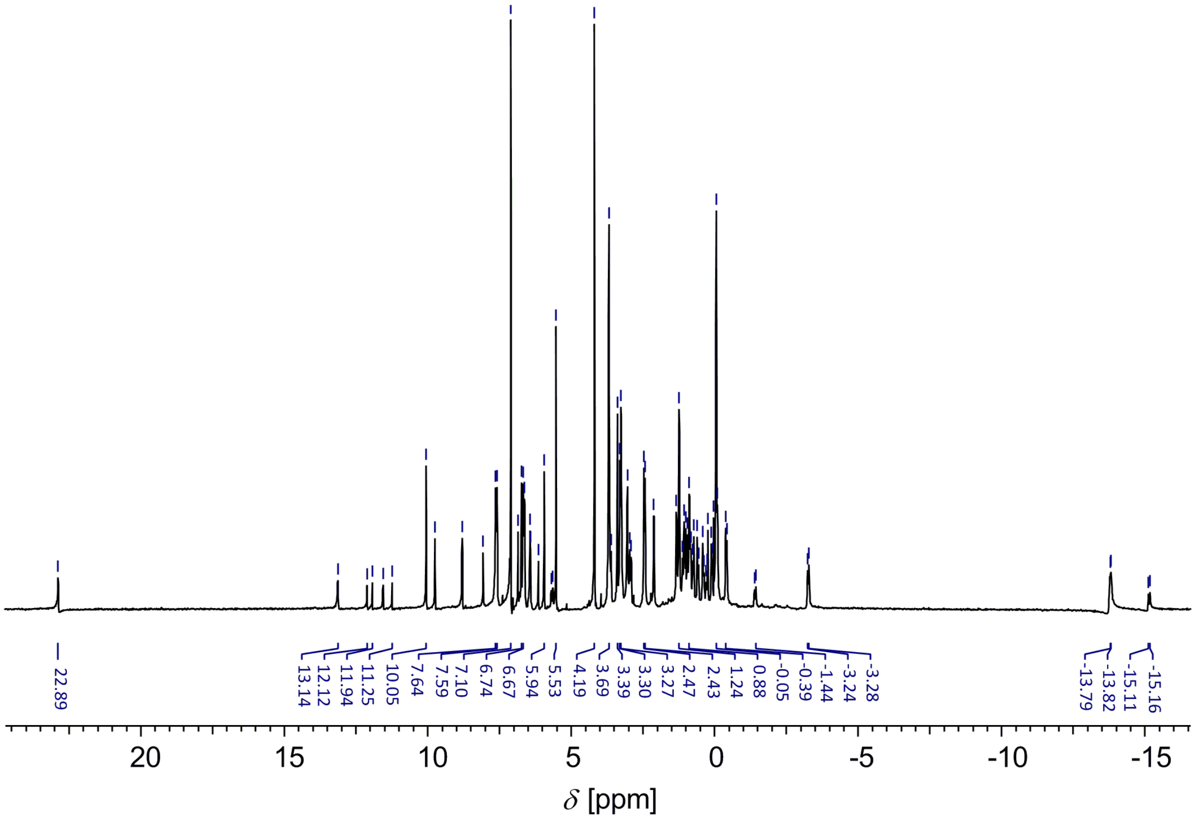


Fig. S16. ^{1}H NMR spectrum of **7** recorded in benzene-d_6.

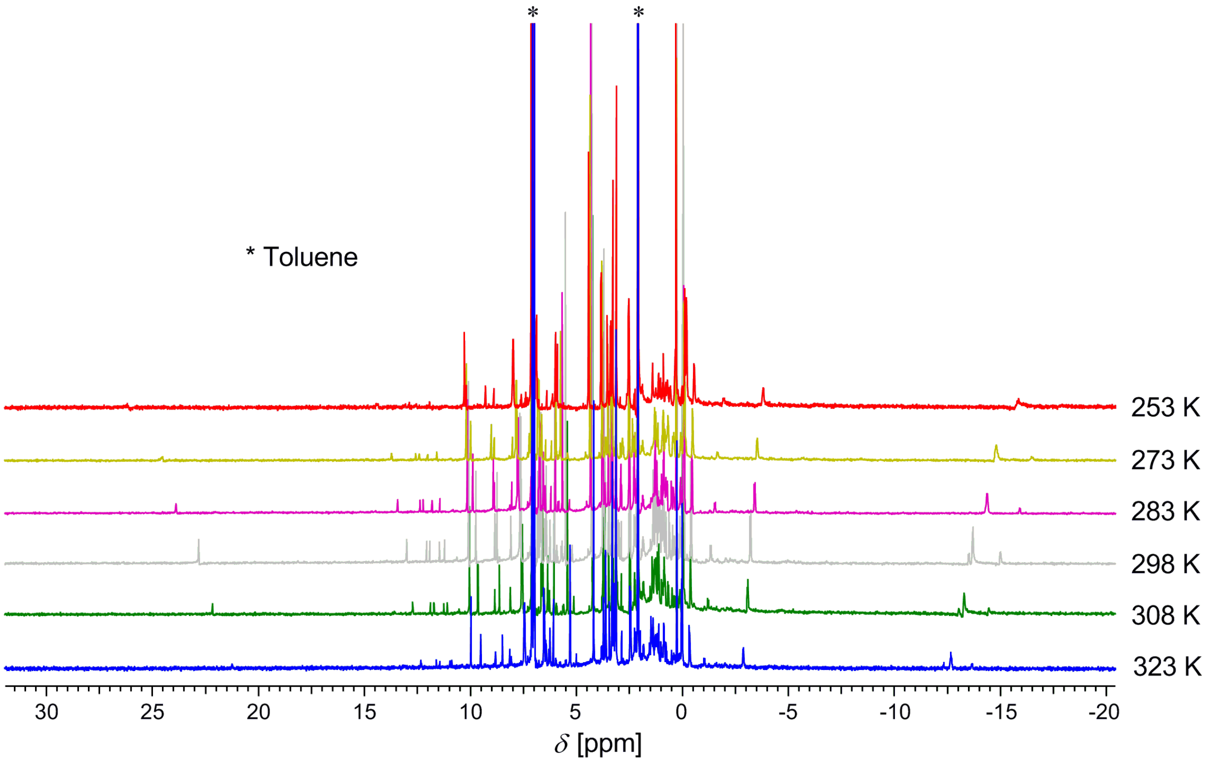


Fig. S17. VT-NMR spectra of **7** recorded in toluene-d_8.

SQUID Magnetization Data

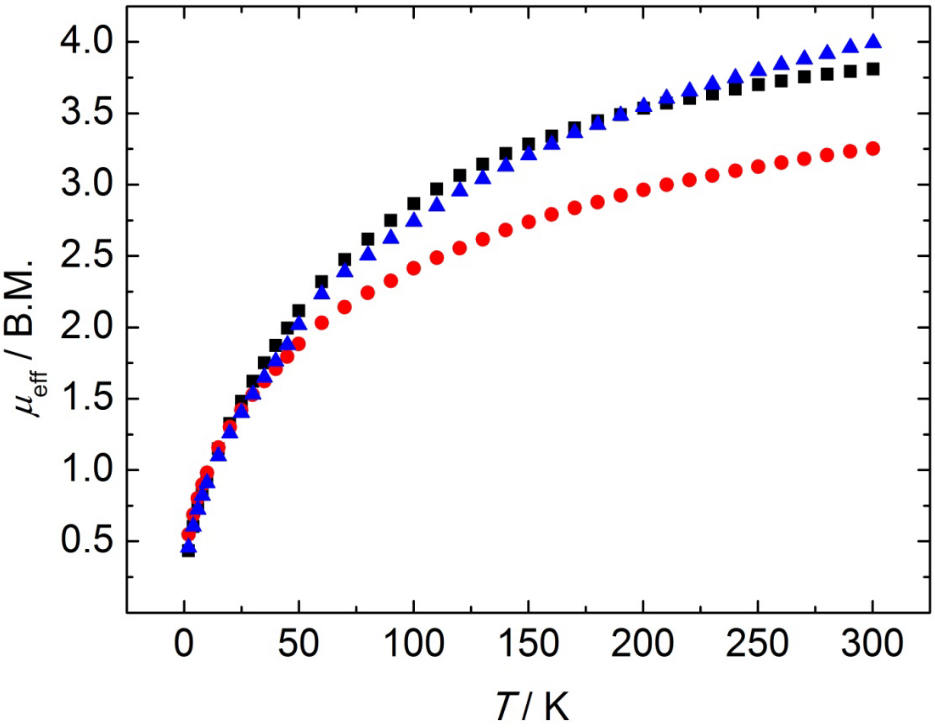
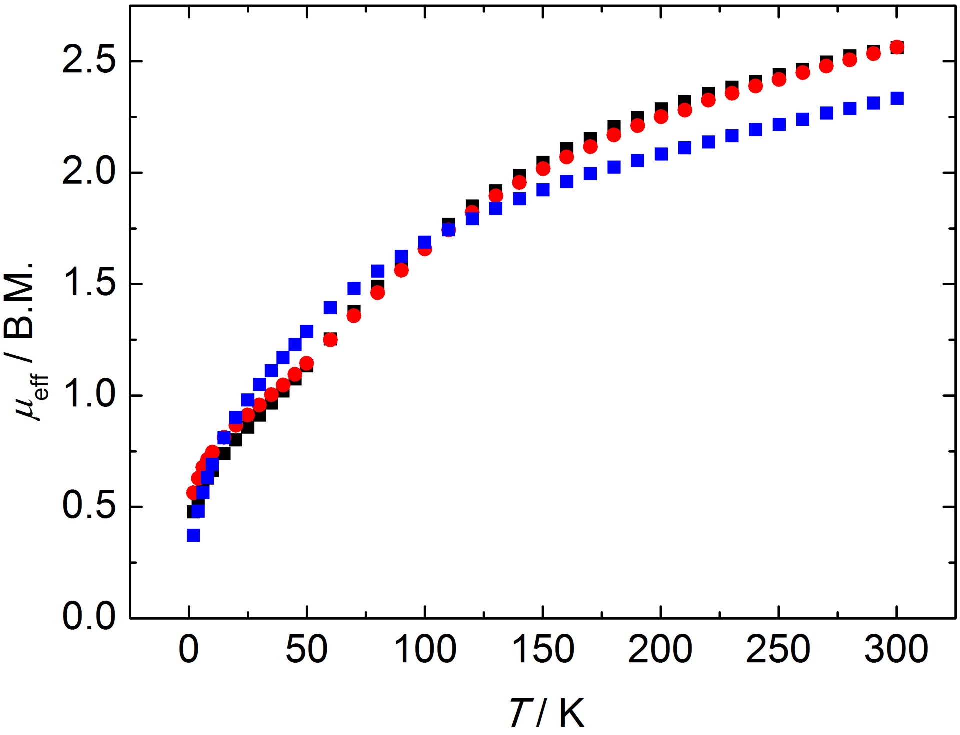


Fig. S18. Temperature-dependant SQUID magnetization data of compounds **3**, **4**, **5** (top) and three independently synthesized batches of **7** (bottom). Plot of the effective magnetic moment μ_{eff} against T. Data were corrected for underlying diamagnetism.



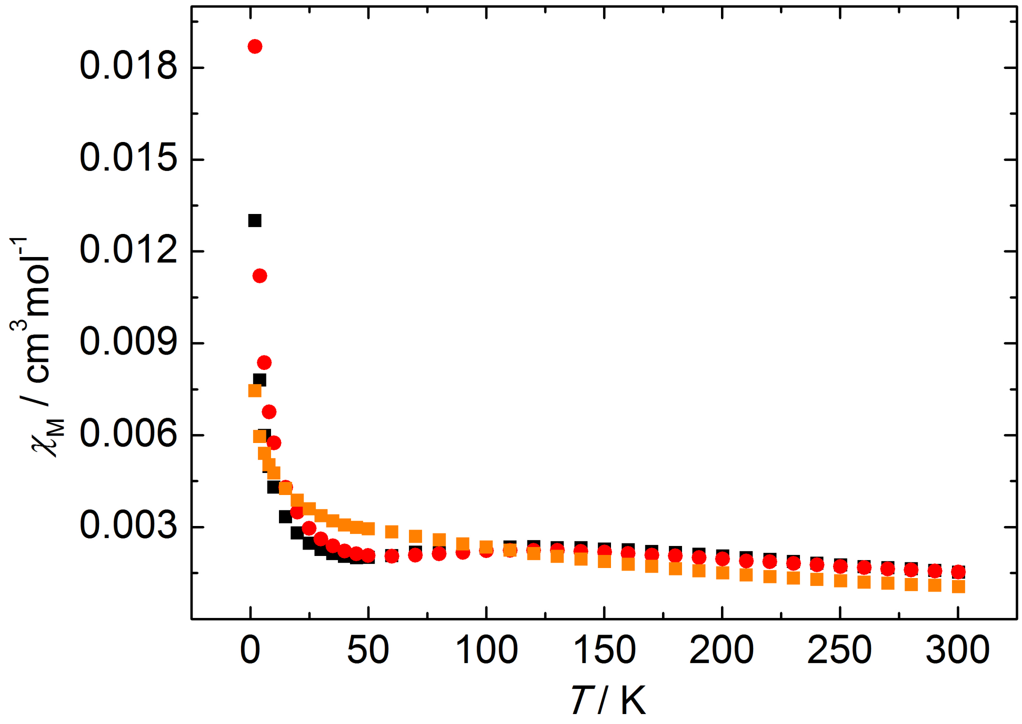


Fig. S19. Temperature-dependent SQUID magnetization data of three independently synthesized batches of compound **7**. Plot of the molar susceptibility χ_M against T. Data were corrected for underlying diamagnetism.

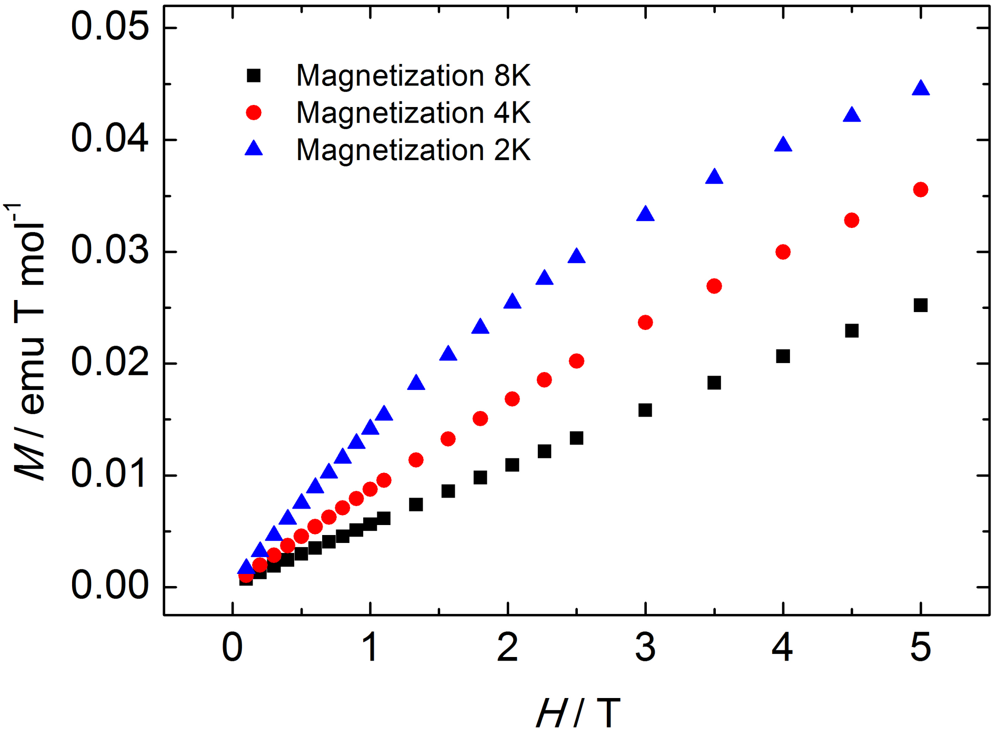


Fig. S20. Field-dependent SQUID magnetization data of complex **7** at 2 K, 4 K and 8 K. Plot of the magnetization M against the magnetic field H.

UV-vis Spectroscopy

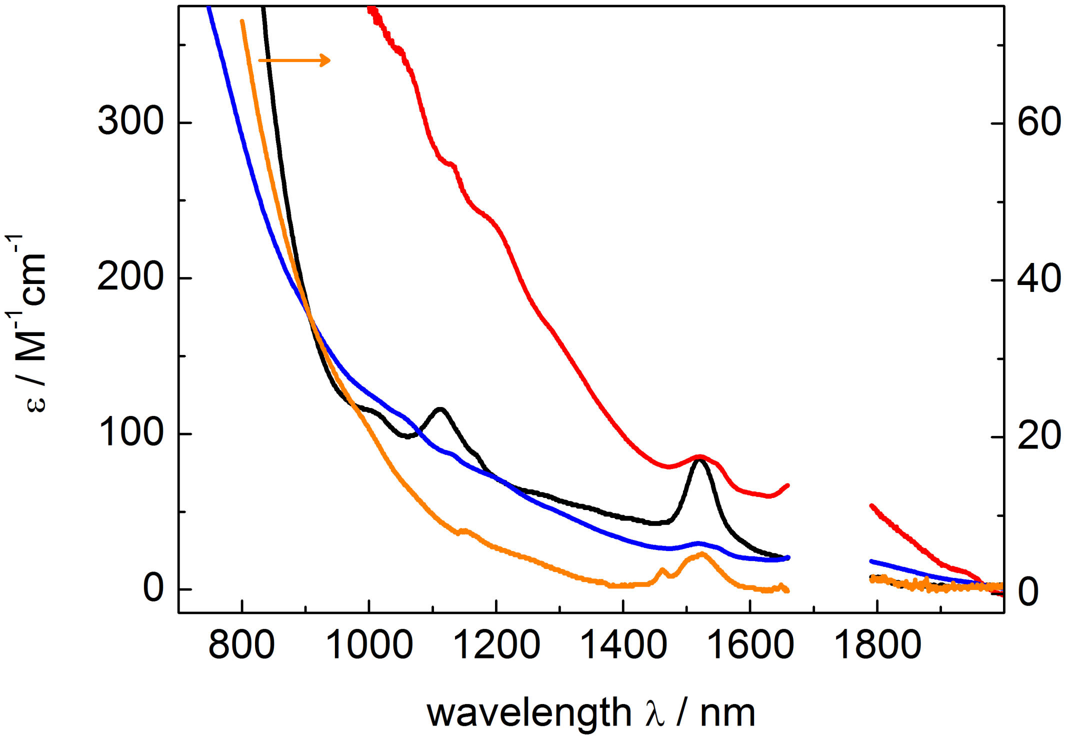


Fig. S21. UV-vis spectrum of complexes **3** (4.2 mM, orange), **4** (6.6 mM, red), **5** (6.9 mM, blue), and **7** (1.2 mM, black) recorded in toluene at 25 ˚C.

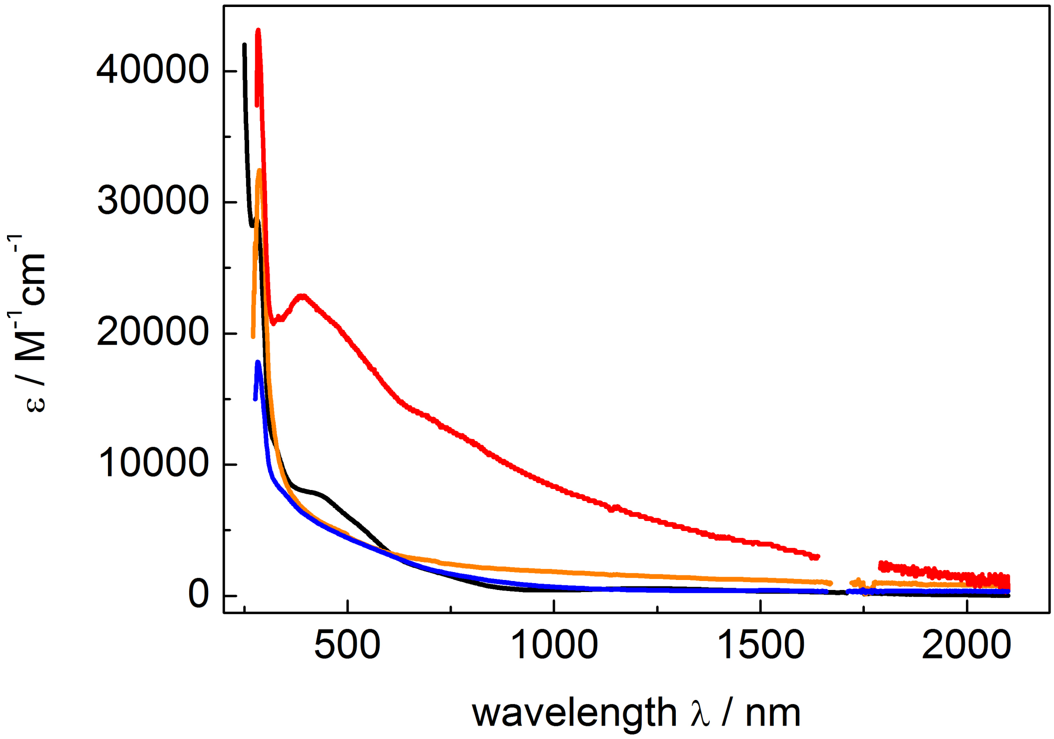


Fig. S22. UV-vis spectrum of complexes **3** (48 μM, orange), **4** (6.6 μM, red), **5** (69 μM, blue), and **7** (0.12 mM, black) recorded in toluene at 25 ˚C.

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