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A Decacationic Ferrocene-Based Metallostar

Susanne M. Rupf, Amina L. Moshtaha, Moritz Malischewski*

SUPPORTING INFORMATION

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Experimental Procedures

General Procedures

CAUTION: Mercury compounds are highly toxic. Therefore, the chemicals should be handled under a well-ventilated fume hood. Great care must be taken that organomercury compounds are not spilled on the skin. It is advisable to use multiple layers of impenetrable gloves and to remove the outer layer immediately if material is spilled on it. The face might be protected against splashes by using a face shield. Due to the hazardous effects of mercury on the environment, great care must be taken to prevent contamination of the wastewater with mercury. Moreover, SbF_5 and *a*HF are highly dangerous compounds with devastating effects on human tissue. They should only be handled in appropriate equipment by trained personnel. SO_2 and *a*HF are (toxic) gases at room temperature.

Commercially available chemicals were used as received, unless otherwise noted. SbF_5 was stored inside a glove box. Oxygen- and moisture-sensitive compounds were handled using Schlenk techniques and/or handled and stored in an argon-filled glovebox ($O_{2(g)}$ and $H_2O_{(g)}$ < 0.1 ppm). SO_2 was distilled from CaH_2 and stored in a stainless-steel cylinder. Reactions involving SO_2, *a*HF and SbF_5 were performed in PFA (tetrafluoroethene-perfluoroalkoxyvinyl-copolymer) tubes connected to stainless-steel valves. Instead of stirring, the mixtures were agitated with the help of the Mini-Vortex Mixer PV-1. $[FeC_{10}(HgO_2CCF_3)_{10}]$[1] and $[C_5F_5NH][SbF_6]$[2] were synthesized according to the literature procedure. Anhydrous MeCN and DCM were obtained from the solvent system FMBRAUN MB SPS-800. THT, MeCN and C_5F_5N as well as deuterated solvents were stored in Young flasks under argon atmosphere over molar sieve (3 Å) which was dried beforehand at 250 °C under high vacuum. Solvents were degassed by three freeze-pump-thaw cycles.

^1H, ^{13}C and ^{19}F NMR spectra were recorded on a Bruker AVANCE III 700 spectrometer by using 6mm NMR glass tubes. All reported chemical shifts (δ) are referenced to the Ξ values given in IUPAC recommendations of 2008 using the ^2H signal of the deuterated solvent as internal reference.[3] All chemical shifts (δ) are given in parts per million (ppm) and the signals are specified according to the multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad) and the coupling constants *J* in Hz. The program MestRe Nova Version 14.0.1 was used to evaluate and plot the data.[4]

Infrared spectra were measured using a Thermo-Scientific Nicolet iS10 FTIR spectrometer with DuraSamplIR accessory in attenuated total reflection at room temperature or on a Bruker ALPHA FT-IR spectrometer inside a glovebox equipped with a diamond ATR attachment. Raman spectra were recorded on a Bruker MultiRAM II equipped with a low-temperature Ge detector (1064 nm, 30-80 mW, resolution 2 cm^{-1}). The software OriginPro 2017G was used to plot the data.[5]

Cyclic voltammetry was performed on an Interface 1010 B Potentiostat/Galvanostat/ZRA from Gamry Instruments. The investigations were carried out starting from 0 V going to the oxidation first and then to the reduction. The measurements were performed in anhydrous and oxygen free solvents under argon atmosphere without supporting electrolyte and platinum wires as working-, counter-, and quasi-reference electrodes. The voltammograms were internally referenced against $FeCp_2^{0/+}$. The software OriginPro 2017G was used to plot the data.[5]

X-Ray data were collected on a BRUKER D8 Venture system. Data were collected at 100(2) K using graphite-monochromated Mo K_α radiation (λ_α = 0.71073 Å) or a Cu K_α radiation (λ=1.54178). The strategy for the data collection was evaluated by using the Smart software. The data were collected by the standard "*ψ-ω* scan techniques" and were scaled and reduced using Saint+software. The structures were solved by using Olex2,[6] the structure was solved with the XT[7] structure solution program using Intrinsic Phasing and refined with the XL refinement package[8,9] using Least Squares minimization. If it is noted, bond length and angles were measured with Diamond Crystal and Molecular Structure Visualization Version 4.6.2.[10] Drawings were generated with POV-Ray.[11] Deposition numbers CCDC 2214089 ($[FeC_{10}(HgDMAP)_{10}][TFA]_{10}$), 2213962 ($[FeC_{10}(HgTHT)_{10}][SbF_6]_{10} \cdot 24$ MeCN) and 2213956 (**[**$FeCp_2(HgMeCN)_{10}][SbF_6]_{10}[MoF_6] \cdot 2\ SO_2 \cdot 10$ HF**)** contain the supplementary crystallographic data for this paper. These data are provided free of charge by the joint Cambridge Crystallographic Data Centre and Fachinformationszentrum Karlsruhe Access Structures service www.ccdc.cam.ac.uk/structures.

Elemental analysis was performed with Vario EL elemental analyser (Elementar, Germany). UV/vis spectra were recorded on a Varian Cary 50 Bio photo spectrometer (xenon lamp) by using Suprasil quartz cuvettes (d = 1 cm) equipped with a Schlenk valve cock. Observed spectra are consistent with those of ferrocene and ferrocenium salts.[12,13]

Syntheses

*$[FeC_{10}Hg_{10}dmap_{10}][TFA]_{10}$ (**1**)*

$$[FeC_{10}(HgTFA)_{10}] \xrightarrow[\text{THF, 25 °C}]{\text{exc. dmap}} [FeC_{10}Hg_{10}dmap_{10}][TFA]_{10} \quad (\mathbf{1})$$

In a 100 mL round bottom flask $[FeC_{10}(HgTFA)_{10}]$ (580 mg, 0.175 mmol, 1.0 eq.) was dissolved in THF (25 mL). Then, dmap (370 mg, 3.03 mmol, 17 eq.) was added to the solution at room temperature, yielding an orange precipitate. The precipitate was filtered off, washed with THF (50 mL) and *n*-pentane (50 mL) and dried at high vacuum. The compound $[FeC_{10}(HgDMAP)_{10}][TFA]_{10}$ (425 mg, 0.093 mmol, 57%) was obtained as orange powder. Single crystals were obtained by diffusion of $[FeC_{10}(HgTFA)_{10}]$ and DMAP in tetrahydrofuran in a H-tube. **^{1}H NMR** (600 MHz, CD_2Cl_2, r.t.): δ [ppm] = 8.27 (m, 2H), 6.50 (m, 2H), 3.05 (s, 6H). **^{13}C{^{1}H} NMR** (101 MHz, CD_2Cl_2, r.t.): δ [ppm] = 161.4 (q, J = 34.3 Hz), 155.7, 148.8, 118.4 (q, J = 296.5 Hz), 106.9, 100.8, 39.6. **^{19}F NMR** (565 MHz, CD_2Cl_2, r.t.) δ [ppm] = −74.7 (s). **FT-IR** (ATR): $\tilde{\nu}$ [cm^{-1}] = 3094, 2931, 1790, 1621, 1549, 1446, 1396, 1235, 1196, 1174, 1122, 1072, 1023, 947, 813, 798, 718. **Raman** (1064 nm, r.t.): $\tilde{\nu}$ [cm^{-1}] = 3094, 2937, 2905, 2869, 2823, 1636, 1624, 1549, 1448, 1418, 1301, 1237, 1077, 951, 827, 765, 662, 595, 193, 100. **Elemental analysis:** m[%] = calculated: N: 6.41, C: 24.72, H: 1.38, found: N: 6.43, C: 24.76, H: 1.28.

*$[(FeC_{10}Hg_{10}(solv.)_n][SbF_6]_{10}$ (**2**)*

$$[FeC_{10}(HgTFA)_{10}] \xrightarrow[\substack{\text{- HTFA} \\ SO_2,\ -25\ °C}]{10\ [HNC_5F_5][SbF_6]} [FeC_{10}Hg_{10}(NC_5F_5)_n][SbF_6]_{10} \xrightarrow[\substack{-\ n\ NC_5F_5 \\ SO_2,\ -25\ °C}]{\text{solv.}} [FeC_{10}Hg_{10}(solv.)_{10}][SbF_6]_{10} \quad (\mathbf{2})$$

2a – solv. = C_5F_5N: Pentafluoropyridinium hexafluoroantimonate (2.99 g, 7.38 mmol, 10 eq.) was filled in a 12 mm PFA tube under an atmosphere of argon. Sulfur dioxide was condensed at −196 °C into the reaction tube. Subsequently, the reaction mixture was brought to −10 °C and mechanically shaken until a colourless solution was obtained. The mixture was brought to −196 °C again, followed by addition of $FeC_{10}(HgTFA)_{10}$ (2.45 g, 0.738 mmol, 1.0 eq.). The mixture was warmed up to 0 °C until a deep red solution was obtained. Sulfur dioxide was evaporated under reduced pressure and the product (4.60 g, 0.738 mmol, quant.) was obtained as orange, amorphous solid. The compound was stored in a glovebox under argon atmosphere. **FT-IR** (ATR): $\tilde{\nu}$ [cm^{-1}] = 3345, 1778, 1668,1615, 1545, 1516, 1489, 1324, 1186, 1112, 1077, 998, 978, 660, 633, 555. **Raman** (1064 nm, r.t.): $\tilde{\nu}$ [cm^{-1}] = 939, 650, 639, 602, 522, 497, 458, 359, 279, 112.

$[FeC_{10}Hg_{10}(NC_5F_5)_n][SbF_6]_{10}$ was placed in a flame dried 20 mL Schlenk tube. Anhydrous donor solvent (solv., ca. 0.1 mL) was added yielding a red solution. The solvent was evaporated in an oil pump vacuum of 10^{-3} mbar to afford the product as pink, amorphous solid.

2b – solv. = THT: $[FeC_{10}Hg_{10}(NC_5F_5)_n][SbF_6]_{10}$ (100.0 mg, 0.016 mmol, 1.0 eq.), THT (ca. 1 mL,), $[FeC_{10}Hg_{10}(THT)_{10}][SbF_6]_{10}$ (87.3 mg, 0.016 mmol, quant.). Single crystals were obtained, by slow cooling of **2b** dissolved in MeCN from r.t. to −35 °C. **^{1}H NMR** (600 MHz, MeCN-d_3, r.t.) δ [ppm] = 3.64 (m, 4H), 2.24 (m, 4H). **^{13}C NMR** (151 MHz, MeCN-d_3, r.t.) δ [ppm] = 111.8, 31.6. **^{19}F NMR** (565 MHz, MeCN-d_3, r.t.) δ [ppm] = −120.3 (m). **FT-IR** (ATR): $\tilde{\nu}$ [cm^{-1}] = 3607, 3535, 2955, 2869, 1629, 1491, 1435, 1275, 1260, 1194, 1143, 1077, 975, 957, 883, 811, 656, 635, 559, 518, 472. **Raman** (1064 nm, r.t.): $\tilde{\nu}$ [cm^{-1}] = strong fluorescence. **Elemental analysis:** m[%] = calculated : N: 0.00, C: 11.08, H: 1.49, S: 5.91, found: N: 0.00, C: 11.88, H: 1.66, S: 5.35.

2c – solv. = MeCN: $[FeC_{10}Hg_{10}(NC_5F_5)_n][SbF_6]_{10}$ (25.0 mg, 4.01 µmol, 1.0 eq.), MeCN (0.1 mL,), $[FeC_{10}Hg_{10}(MeCN)_{10}][SbF_6]_{10}$ (19.9 mg, 4.01 µmol, quant.). **^{1}H NMR** (700 MHz, MeCN-d_3, r.t.) δ [ppm] = 1.96 (s). **^{13}C NMR** (176 MHz, MeCN-d_3, r.t.) δ [ppm] = 108.3 (s), 97.7 (s), 1.2 (q, J = 20.7 Hz). **^{19}F NMR** (565 MHz, MeCN-d_3, r.t.) δ [ppm] = −120.3 (m). **FT-IR** (ATR): $\tilde{\nu}$ [cm^{-1}] = 3015, 2947, 2331, 2305, 2247, 1416, 1368, 1215, 1162, 1032, 957, 795, 651, 638, 560. **Raman** (1064 nm, r.t.): $\tilde{\nu}$ [cm^{-1}] = strong fluorescence. **Elemental analysis:** m[%] = calculated : N: 2.83, C: 7.28, H: 0.61, found: N: 2.75, C: 7.45, H: 1.24.

*$[(FeC_{10}Hg_{10}(solv.)_n]^{11+}$ (**3**)*

$$[FeC_{10}Hg_{10}(solv.)_n][SbF_6]_{10} \xrightarrow[-\,Ox]{Ox[An]} [FeC_{10}Hg_{10}(solv.)_n][SbF_6]_{10}[An] \qquad (\mathbf{3})$$

Table S1. Summary of oxidation attempts. Orange-red solution – Fe(II), green/blue solution – Fe(III).

starting material	oxidant	solvent	observation
$[(FeC_{10}Hg_{10}(NC_5F_5)_n]^{11+}$ (**3a**)	$NOBF_4$ (8.6 eq.)	SO_2	brown suspension
	NO_2SbF_6 (2.1 eq.)	SO_2	green solution
$[(FeC_{10}Hg_{10}(THT)_{10}]^{11+}$ (**3b**)	$AgSbF_6$	dcm	no reaction
	$NOBF_4$ (5.0 eq.)	SO_2	green solution
	NO_2SbF_6 (1.0 eq.)	SO_2	green solution
	MoF_6 (11.0 eq.)	SO_2	green solution
$[(FeC_{10}Hg_{10}(MeCN)_n]^{11+}$ (**3c**)	$NOBF_4$ (2.7 eq.)	SO_2	blue solution
	NO_2SbF_6 (1.8 eq.)	SO_2	blue solution
	MoF_6 (12.0 eq.)	SO_2	blue solution

3c – solv. = MeCN: $[FeC_{10}Hg_{10}(MeCN)_{10}][SbF_6]_{10}$ (39.6 mg, 0.008 mmol, 1.0 eq.) was filled in an 8 mm PFA tube under an atmosphere of argon and dissolved in liquid SO_2 (1 mL). Then, MoF_6 (20.00 mg, 0.095 mmol, 11.9 eq.) was added at −196 °C. The reaction mixture was brought to −10 °C and mechanically shaken until a dark blue solution was obtained. All liquid components were evaporated under reduced pressure and the product (41.5 mg, 0.008 mmol, quant.) was obtained as blue solid and stored under argon. Single crystals were obtained by recrystallisation from HF/SO_2 at −74 °C. **FT-IR** (ATR): $\tilde{\nu}$ [cm^{-1}] = 2942, 2332, 2306, 2247, 1552, 1408, 1374, 1221, 1167, 1019, 957, 865, 653, 634, 557, 520. **Raman** (1064 nm, r.t.): $\tilde{\nu}$ [cm^{-1}] = 2949, 2339, 2991, 2248, 1369, 945, 644, 393, 364, 125.

Crystallographic Data

Table S2. Crystallographic data.

Compound	**$[FeC_{10}(HgDMAP)_{10}][TFA]_{10}$**	**$[FeC_{10}(HgTHT)_{10}][SbF_6]_{10}$ · 24 MeCN**	**$[FeCp_2(HgMeCN)_{10}][SbF_6]_{10}[MoF_6]$ · 2 SO_2 ·10 HF**
Empirical formula	$FeC_{98}Hg_{10}N_{20}H_{100}O_{18}F_{27}$	$C_{98}F_{60}FeH_{150}Hg_{10}N_{24}S_{10}Sb_{10}$	$C_{30}F_{88}FeH_{42}Hg_{10}MoN_{10}O_{12}S_6Sb_{10}$
Formula weight	4420.72	6404.26	5974.28
Temperature/K	100	100.0	100
Crystal system	monoclinic	monoclinic	triclinic
Space group	$P2_1/n$	*C*2/*c*	*P*-1
a/Å	16.8907(4)	18.7149(8)	11.5091(7)
b/Å	40.5278(10)	36.9102(16)	16.0047(12)
c/Å	23.0130(5)	32.7166(14)	16.9690(11)
α/°	90	90	78.893(3)
β/°	109.0010(10)	94.377(2)	83.202(2)
γ/°	90	90	80.190(3)
Volume/Å³	14895.0(6)	22533.8(17)	3010.4(4)
Z	4	4	1
ρ_{calc}/g·cm³	1.971	1.888	3.295
μ/mm^{-1}	19.530	8.197	15.392
F(000)	8164	11768.0	2654.0
Crystal size/mm³	0.5 × 0.44 × 0.39	0.5 × 0.38 × 0.33	0.26 × 0.18 × 0.15
Crystal shape	block	block	block
Radiation	CuKα (λ=1.54178)	MoKα (λ = 0.71073)	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.61 to 134.364	3.966 to 50.328	4.18 to 56.594
Index ranges	$-20 \le h \le 20$, $-48 \le k \le 48$, $-27 \le l \le 27$	$-22 \le h \le 22$, $-44 \le k \le 44$, $-39 \le l \le 39$	$-15 \le h \le 15$, $-21 \le k \le 21$, $-22 \le l \le 22$
Reflections collected	416111	325039	66569
Independent reflections	26601 [R_{int} = 0.0781, R_{sigma} = 0.0277]	20131 [R_{int} = 0.0376, R_{sigma} = 0.0141]	14855 [R_{int} = 0.0291, R_{sigma} = 0.0243]
Data/restrains/parameters	26601/1/1710	20131/0/911	14855/0/660
Goodness-of-fit on F²	1.052	1.040	1.083
Final R indexes [I>=2σ(I)]	R_1 = 0.0519, wR_2 = 0.1389	R_1 = 0.0384, wR_2 = 0.1055	R_1 = 0.0309, wR_2 = 0.0702
Final R indexes [all data]	R_1 = 0.0569, wR_2 = 0.1439	R_1 = 0.0426, wR_2 = 0.1095	R_1 = 0.0344, wR_2 = 0.0717
Largest diff. peak/hole / e·Å³	3.38/-1.87	2.14/-1.87	2.75/-2.47

In the crystal structure of $[FeC_{10}(HgTHT)_{10}][SbF_6]_{10} \cdot 24\ MeCN$ the iron atoms produce a hexagonal closed arrangement along the ac-plane with an AB-stacking pattern (Fig. S20). However, the iron atoms of layer B are not found within the trigonal voids similar to the Mg-type but displaced along the c-axis causing a number of surrounding iron atoms of ten instead twelve as expected for a Mg-type packing. Considering only the centers of gravity for all molecular entities, no simple packing arrangement is observed for the anionic units as they are not located neither within the tetrahedral nor in the octahedral voids. For the THT-structure the decacation is surrounded by 20 anions but due to disordering of the anions no ordered arrangement of Sb atoms is visible.

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Appendix

Spectra

*[FeC$_{10}$(HgDMAP)$_{10}$][TFA]$_{10}$ (**1a**):*

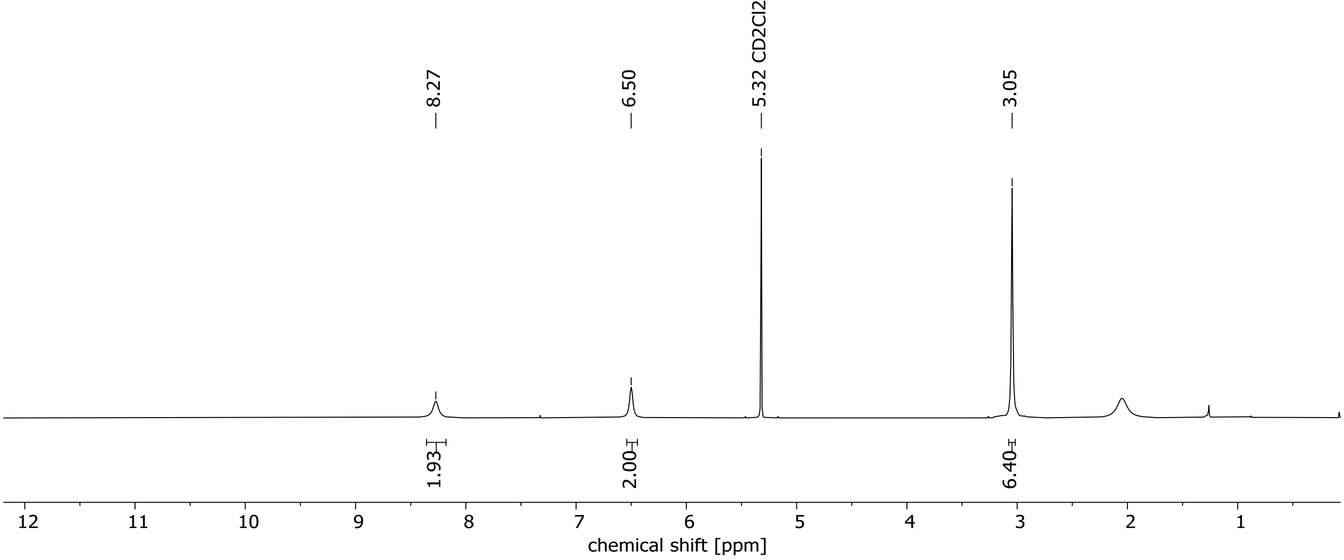

Figure S1. ^{1}H NMR spectrum of [FeC$_{10}$(HgDMAP)$_{10}$][TFA]$_{10}$ (600 MHz, CD$_2$Cl$_2$, r.t.).

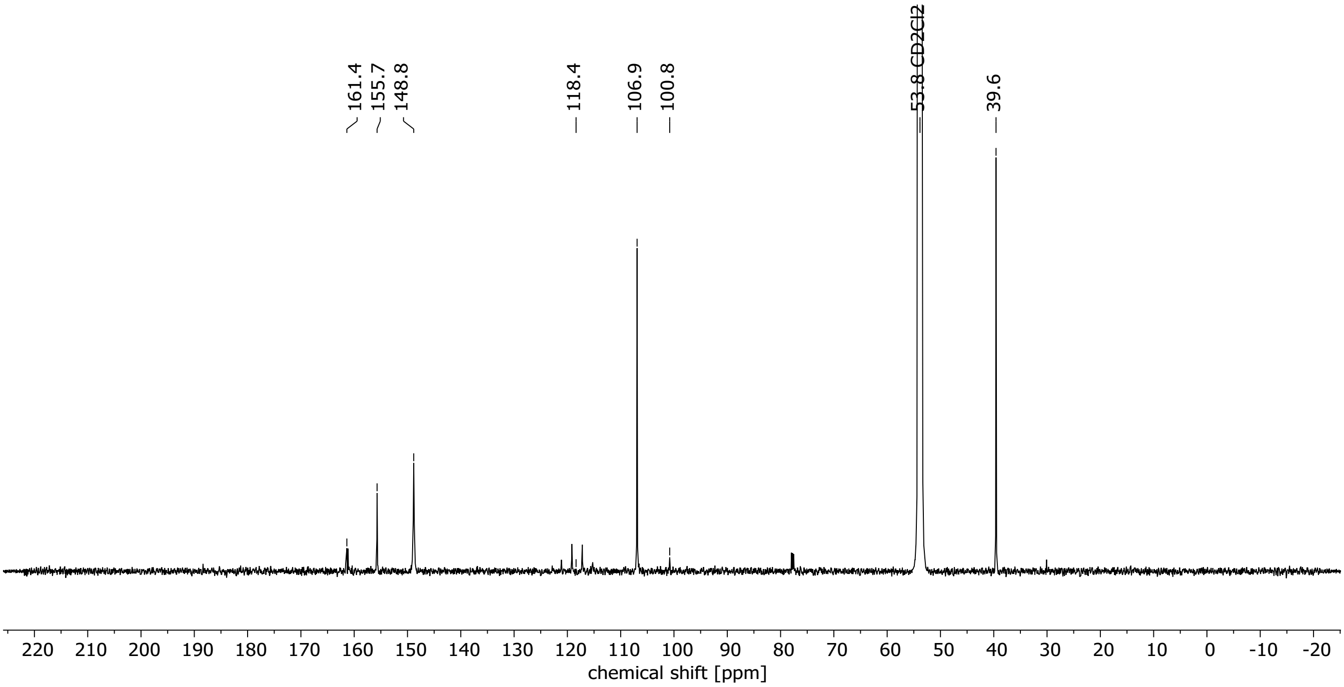

Figure S2. ^{13}C NMR spectrum of [FeC$_{10}$(HgDMAP)$_{10}$][TFA]$_{10}$ (151 MHz, CD$_2$Cl$_2$, r.t.).

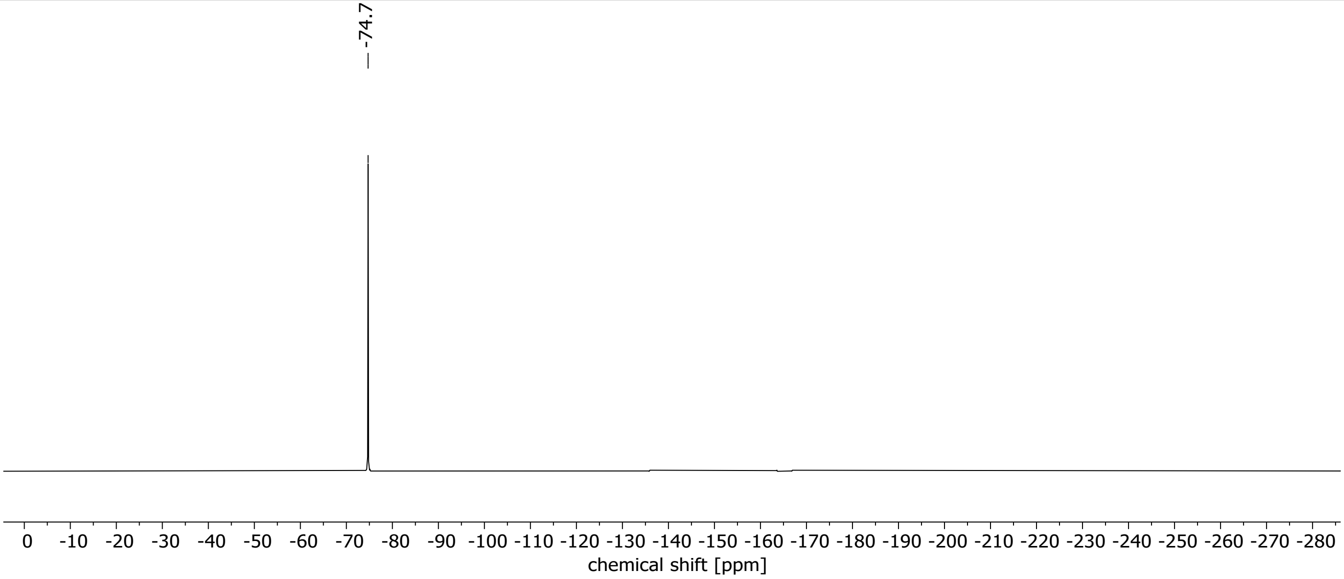

Figure S3. ^{19}F NMR spectrum of $[FeC_{10}(HgDMAP)_{10}][TFA]_{10}$ (565 MHz, CD_2Cl_2, r.t.).

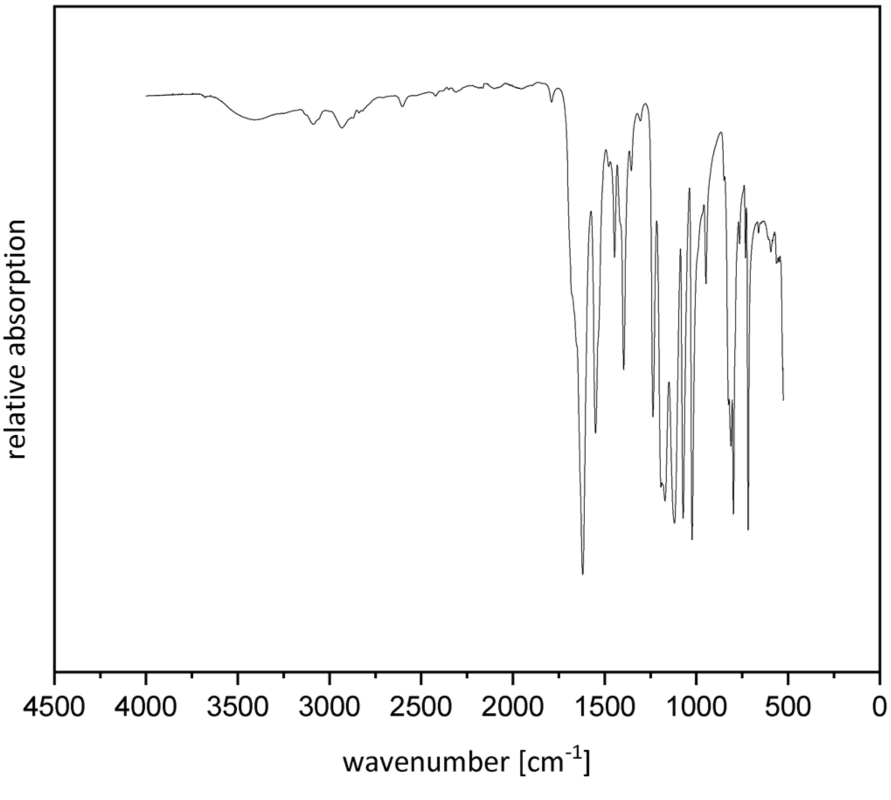

Figure S4. Infrared spectrum (ATR) of $[FeC_{10}(HgDMAP)_{10}][TFA]_{10}$.

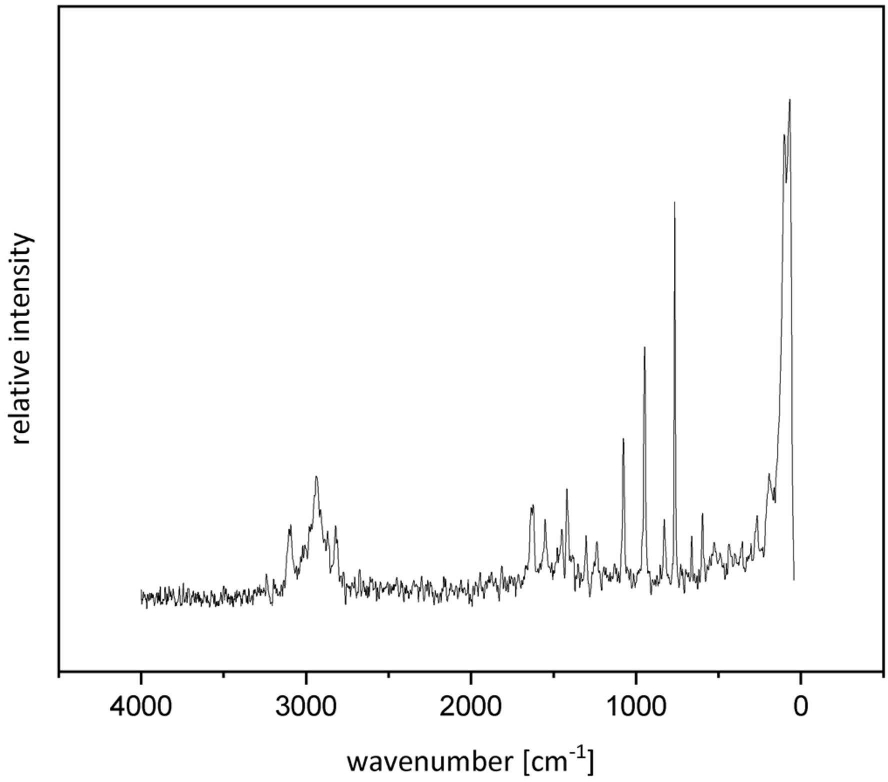

Figure S5. Raman spectrum (1064 nm, r.t.) of $[FeC_{10}(HgDMAP)_{10}][TFA]_{10}$.

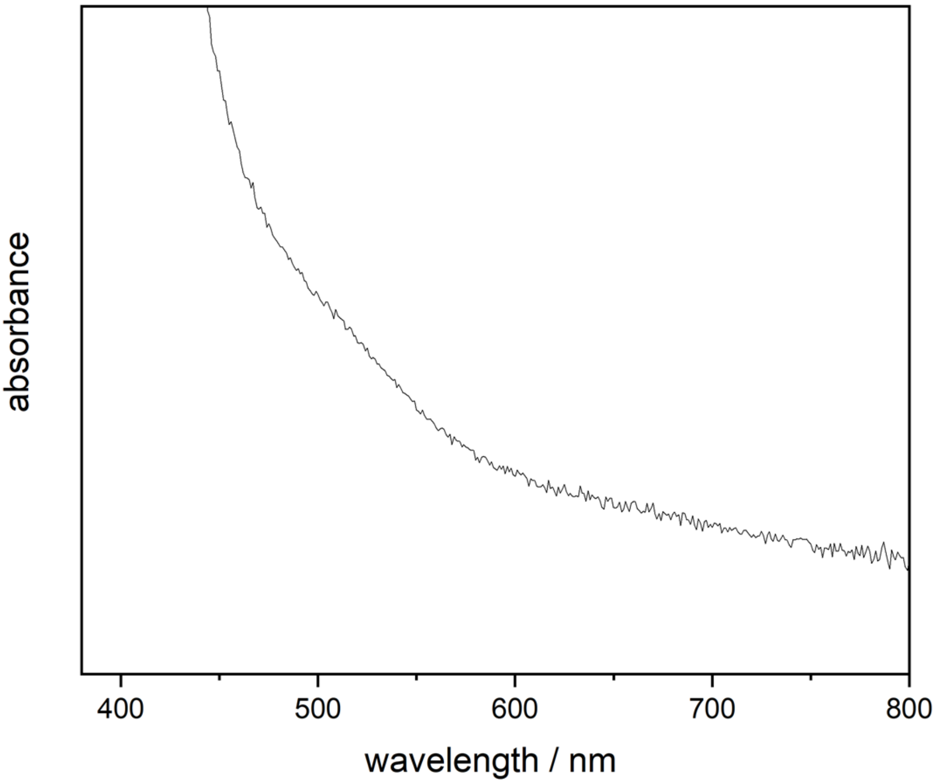

Figure S6. UV/Vis spectrum of $[FeC_{10}(HgDMAP)_{10}][TFA]_{10}$ in DCM.

*$[FeC_{10}Hg_{10}(NC_5F_5)_n][SbF_6]_{10}$ (**2a**):*

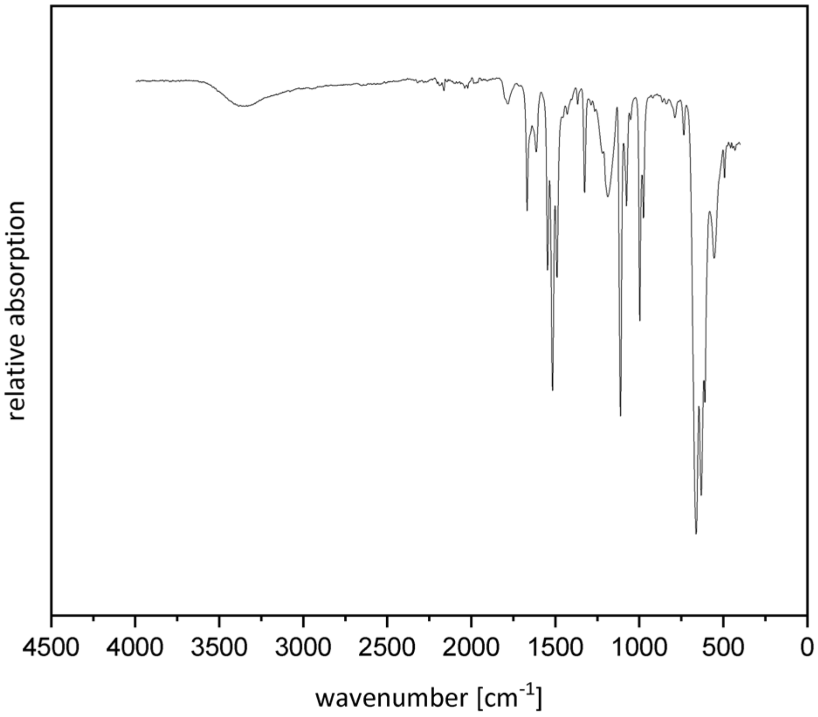

Figure S7. Infrared spectrum (ATR) of $[FeC_{10}Hg_{10}(NC_5F_5)_n][SbF_6]_{10}$.

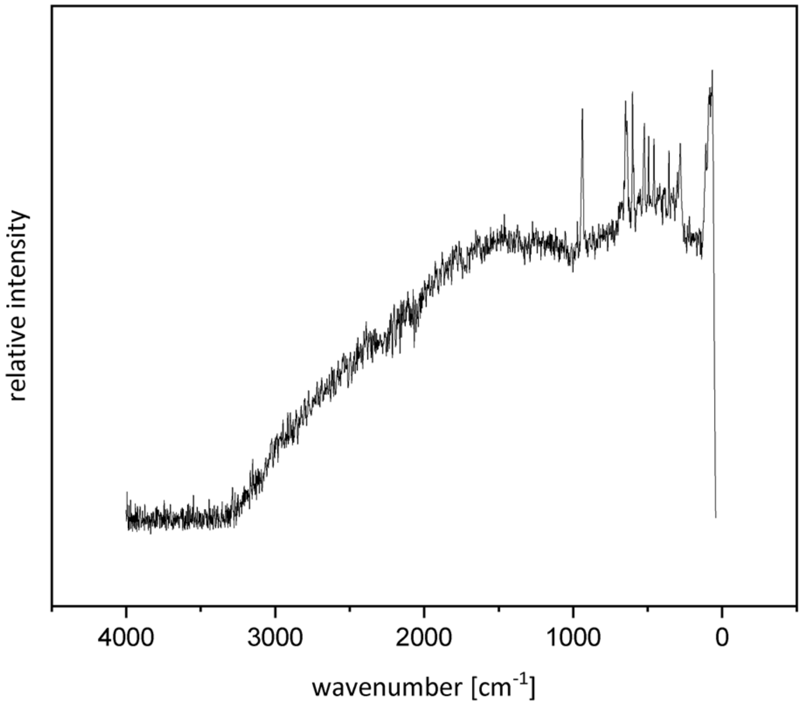

Figure S8 Raman spectrum (1064 nm, r.t.) of $[FeC_{10}Hg_{10}(NC_5F_5)_n][SbF_6]_{10}$.

*[$FeC_{10}Hg_{10}(THT)_{10}$][$SbF_6$]$_{10}$ (**2b**):*

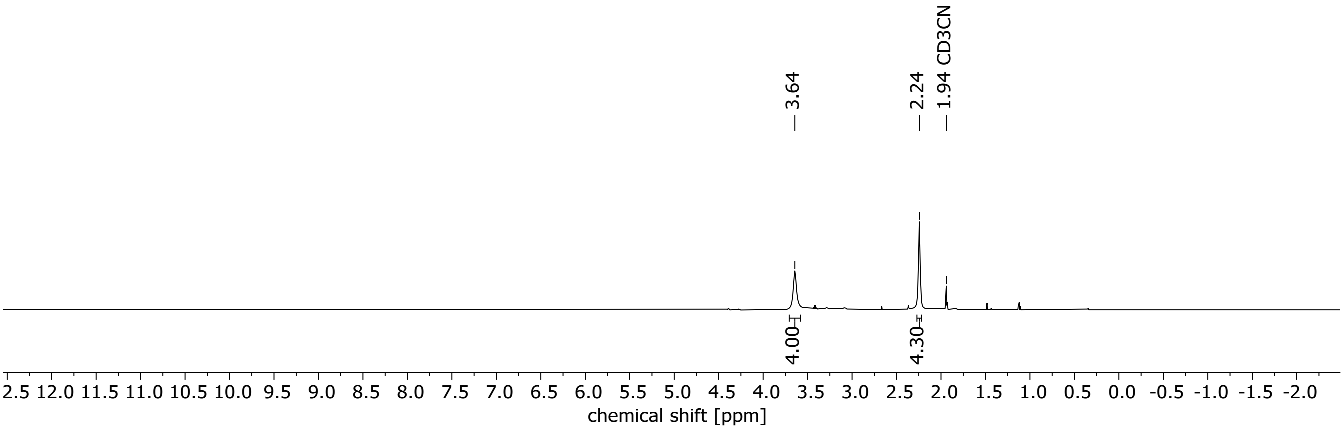

Figure S9. ^{1}H NMR spectrum of [$FeC_{10}Hg_{10}(THT)_{10}$][$SbF_6$]$_{10}$ (600 MHz, MeCN-d_3, r.t.).

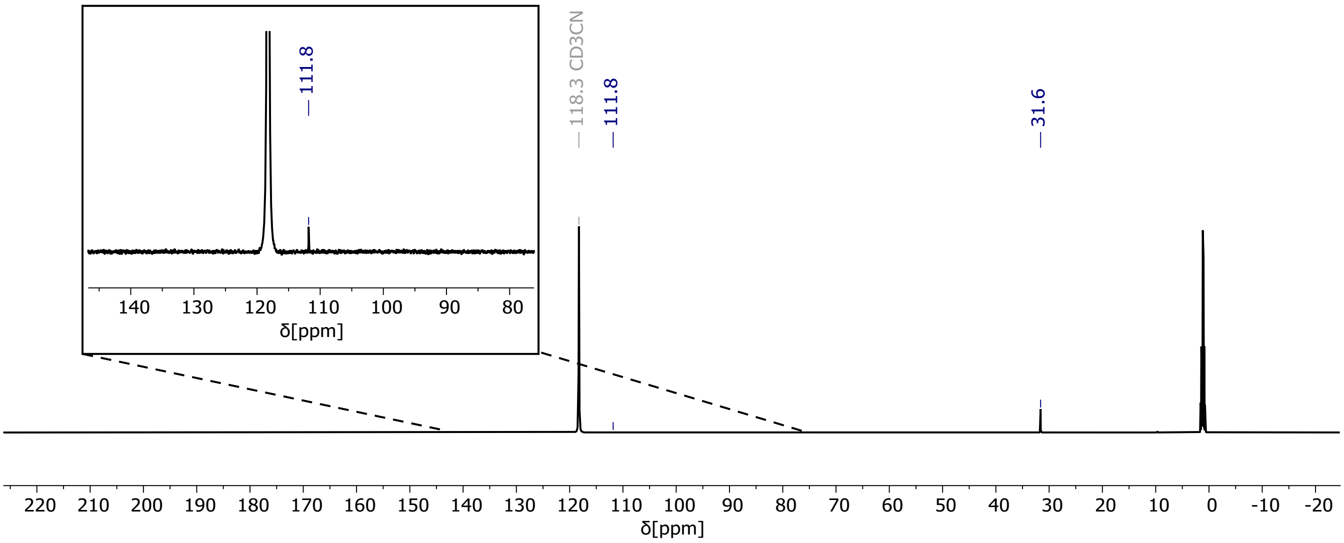

Figure S10. ^{13}C NMR spectrum of [$FeC_{10}Hg_{10}(THT)_{10}$][$SbF_6$]$_{10}$ (151 MHz, MeCN-d_3, r.t.).

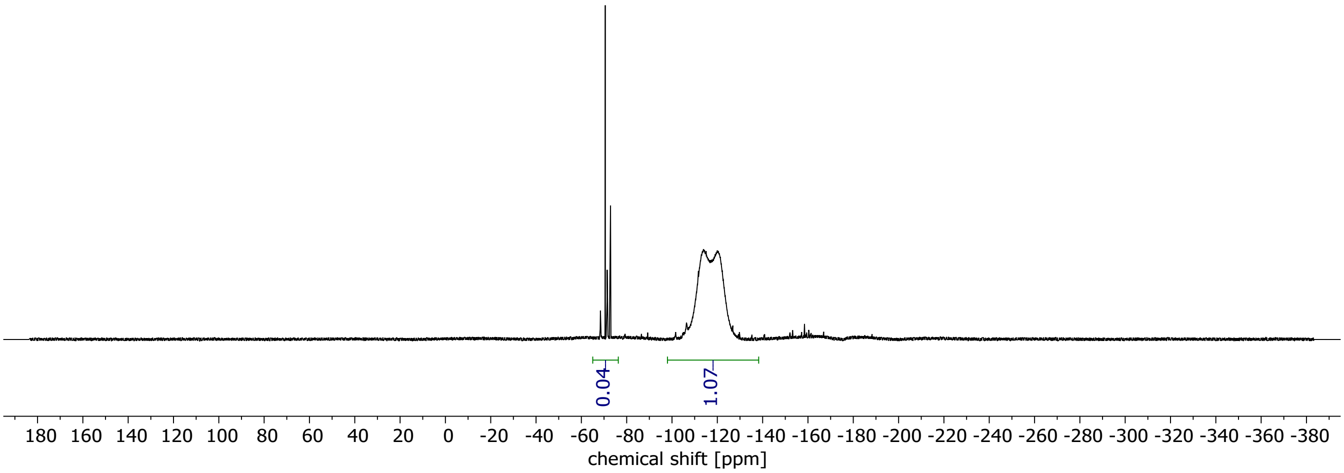

Figure S11. ^{19}F NMR spectrum of [$FeC_{10}Hg_{10}(THT)_{10}$][$SbF_6$]$_{10}$ (565 MHz, MeCN-d_3, r.t.).

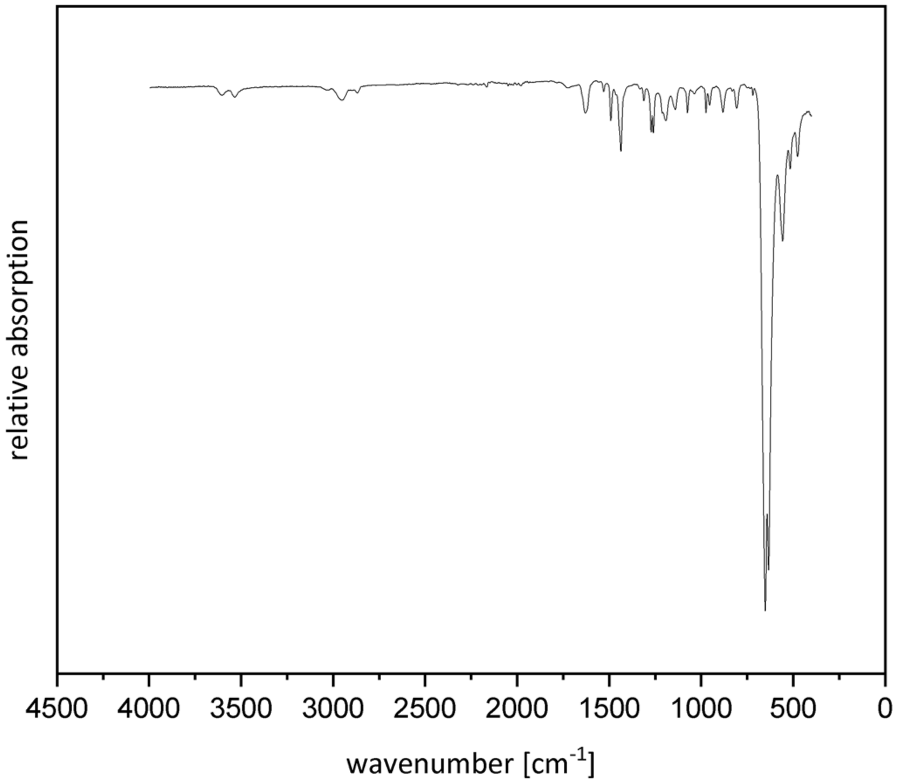

Figure S12. Infrared spectrum of [$FeC_{10}Hg_{10}(THT)_{10}$][$SbF_6$]$_{10}$ (ATR).

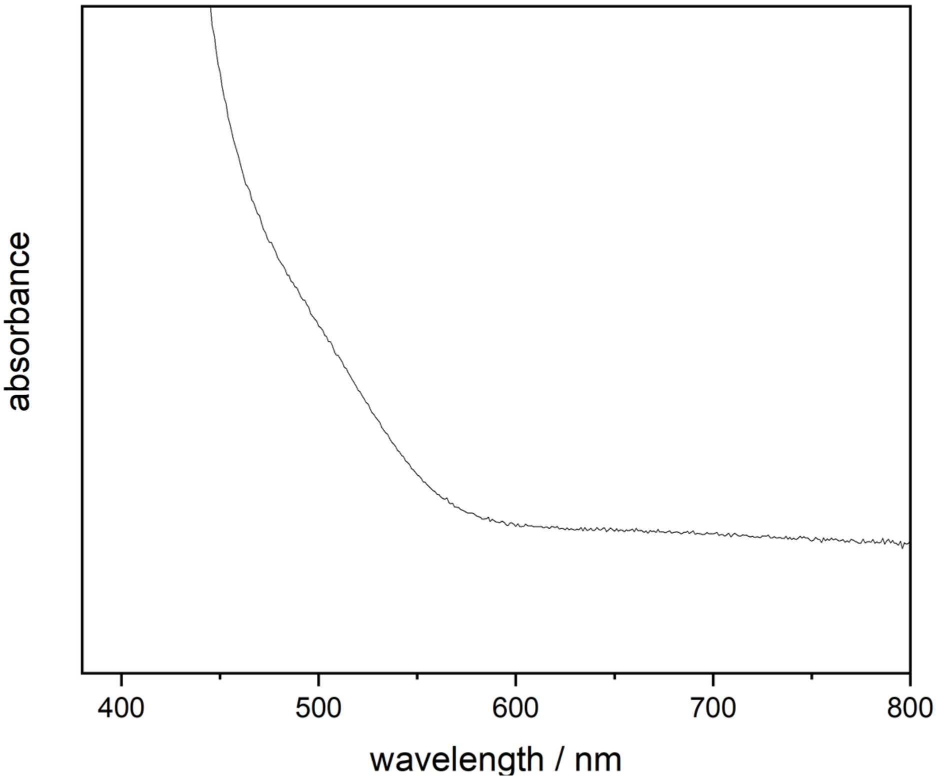

Figure S13. UV/Vis spectrum of of [$FeC_{10}Hg_{10}(THT)_{10}$][$SbF_6$]$_{10}$ in acetonitrile.

*$[FeC_{10}Hg_{10}(MeCN)_{10}][SbF_6]_{10}$ (**2c**):*

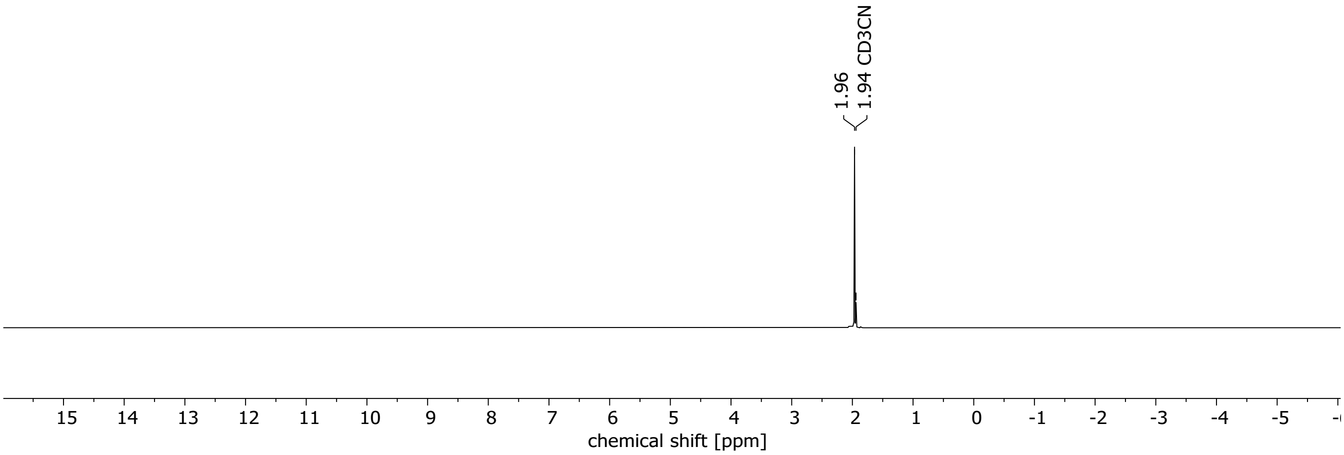

Figure S14. ^{1}H NMR spectrum of $[FeC_{10}Hg_{10}(MeCN)_{10}][SbF_6]_{10}$ (700 MHz, MeCN-d_3, r.t.).

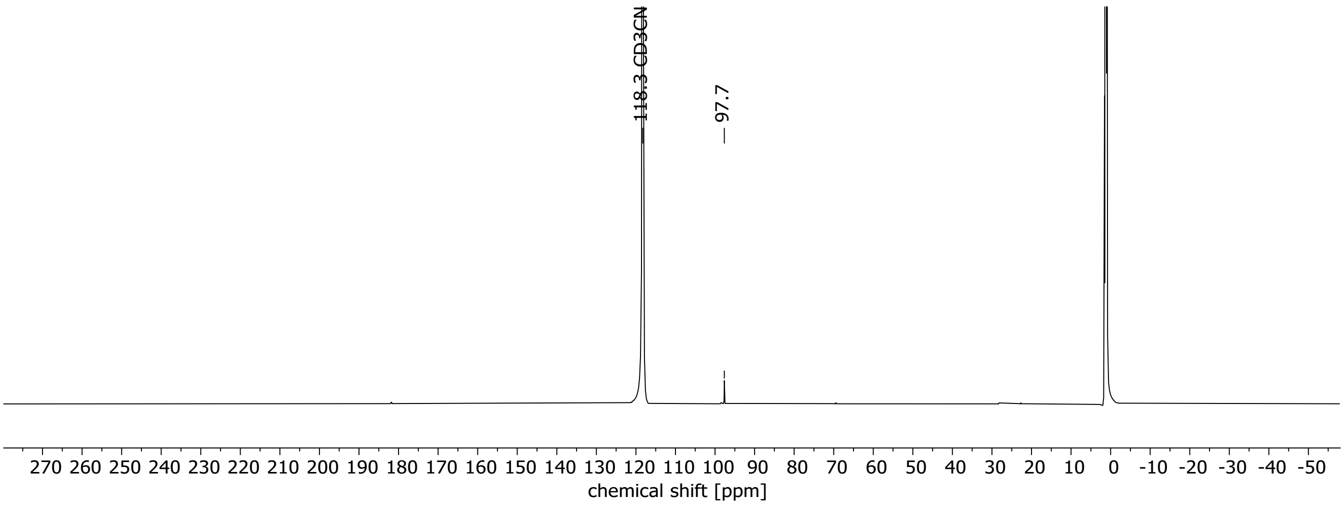

Figure S15. ^{13}C NMR spectrum of $[FeC_{10}Hg_{10}(MeCN)_{10}][SbF_6]_{10}$ (176 MHz, MeCN-d_3, r.t.).

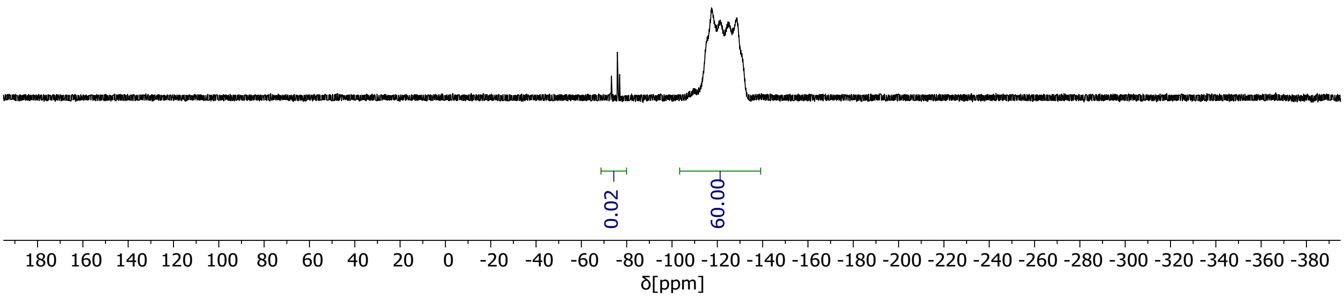

Figure S16. ^{19}F NMR spectrum of $[FeC_{10}Hg_{10}(MeCN)_{10}][SbF_6]_{10}$ (565 MHz, MeCN-d_3, r.t.).

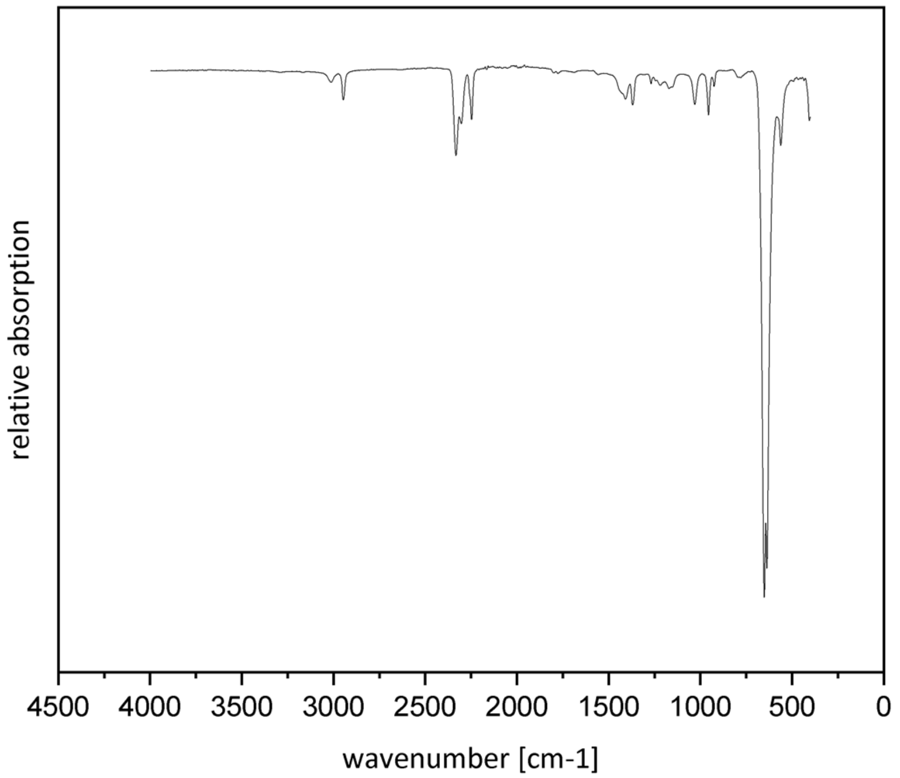

Figure S17. Infrared spectrum of $[FeC_{10}Hg_{10}(MeCN)_{10}][SbF_6]_{10}$ (ATR).

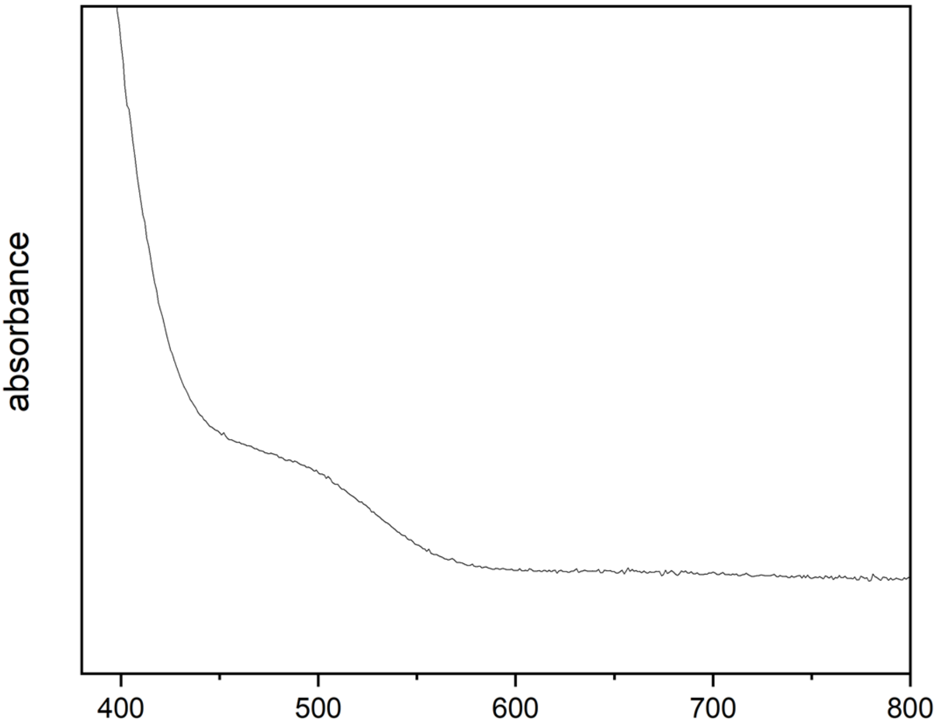

Figure S18. UV/vis spectrum of $[FeC_{10}Hg_{10}(MeCN)_{10}][SbF_6]_{10}$ in acetonitrile.

*$[FeC_{10}Hg_{10}(MeCN)_{10}][SbF_6]_{10}[MoF_6]$ (**3**):*

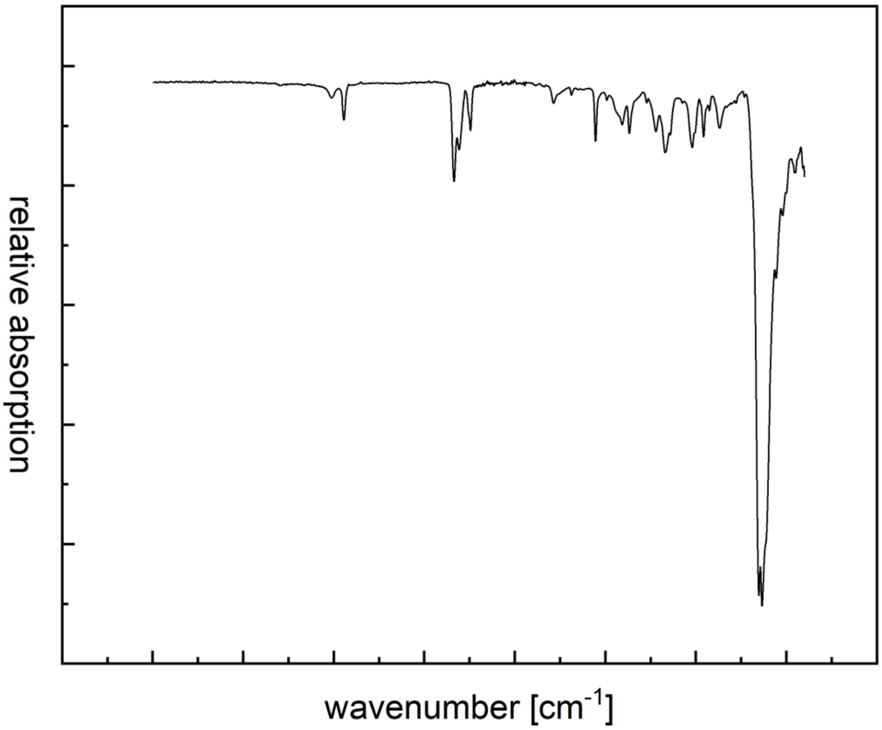

Figure S19. Infrared spectrum of $[FeC_{10}Hg_{10}(MeCN)_{10}][SbF_6]_{10}[MoF_6]$ (ATR).

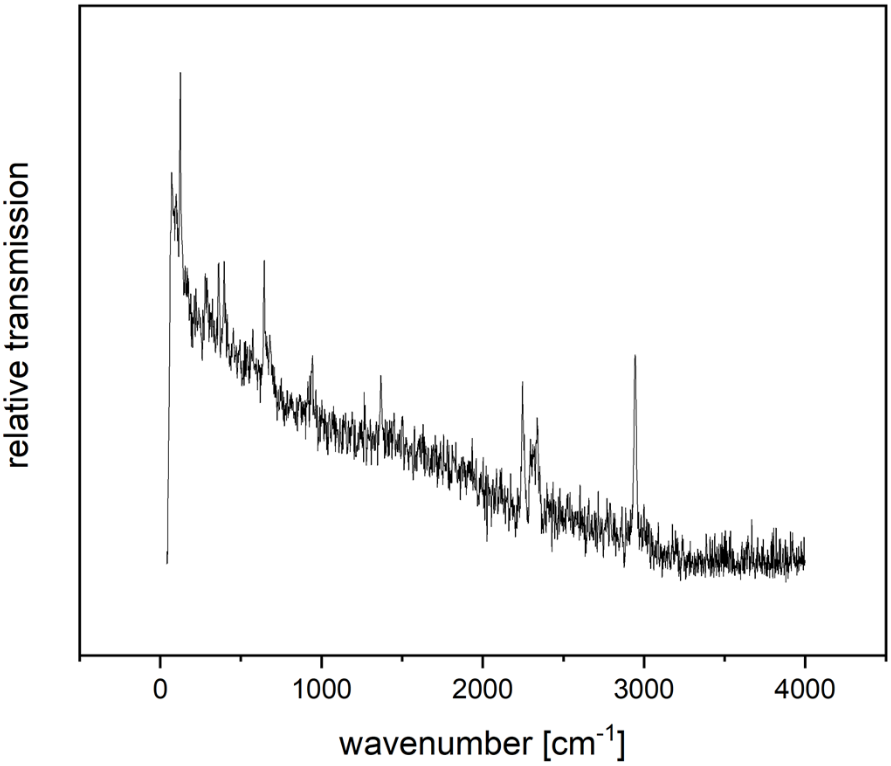

Figure S20. Raman spectrum (1064 nm, r.t.) of $[FeC_{10}Hg_{10}(MeCN)_{10}][SbF_6]_{10}[MoF_6]$.

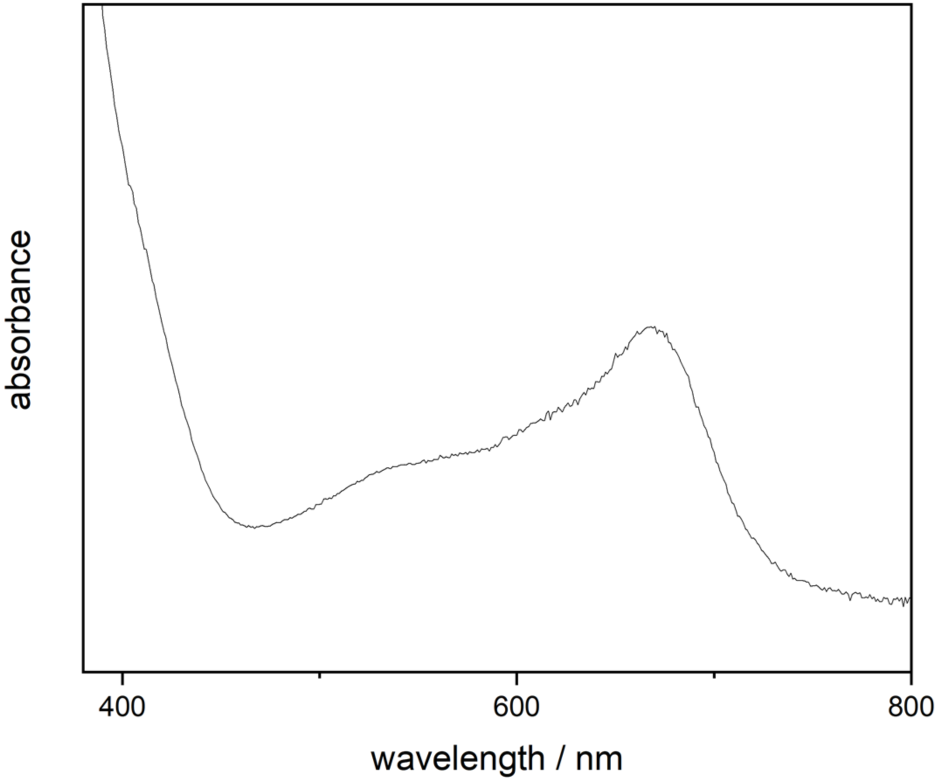

Figure S21. UV/vis spectrum of [$FeC_{10}Hg_{10}(MeCN)_{10}$][$SbF_6$]$_{10}$[$MoF_6$] in acetonitrile. λ_{max} = 668 nm.

[$FeC_{10}Hg_{10}(C_5F_5N)_n$][$SbF_6$]$_{11}$:

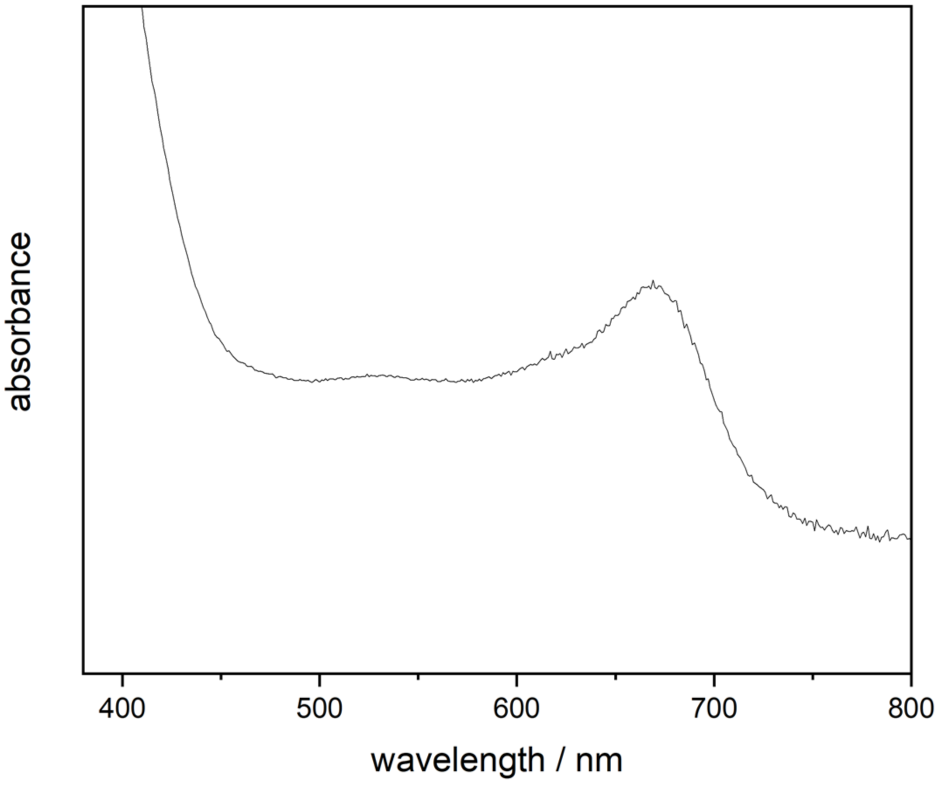

Figure S22. UV/vis spectrum of [$FeC_{10}Hg_{10}(C_5F_5N)_n$][$SbF_6$]$_{11}$ in acetonitrile synthesized by oxidation of [$FeC_{10}Hg_{10}(C_5F_5N)_n$][$SbF_6$]$_{10}$ with NO_2SbF_6. λ_{max} = 669 nm.

$[FeC_{10}Hg_{10}(THT)_{10}][SbF_6]_{11}$:

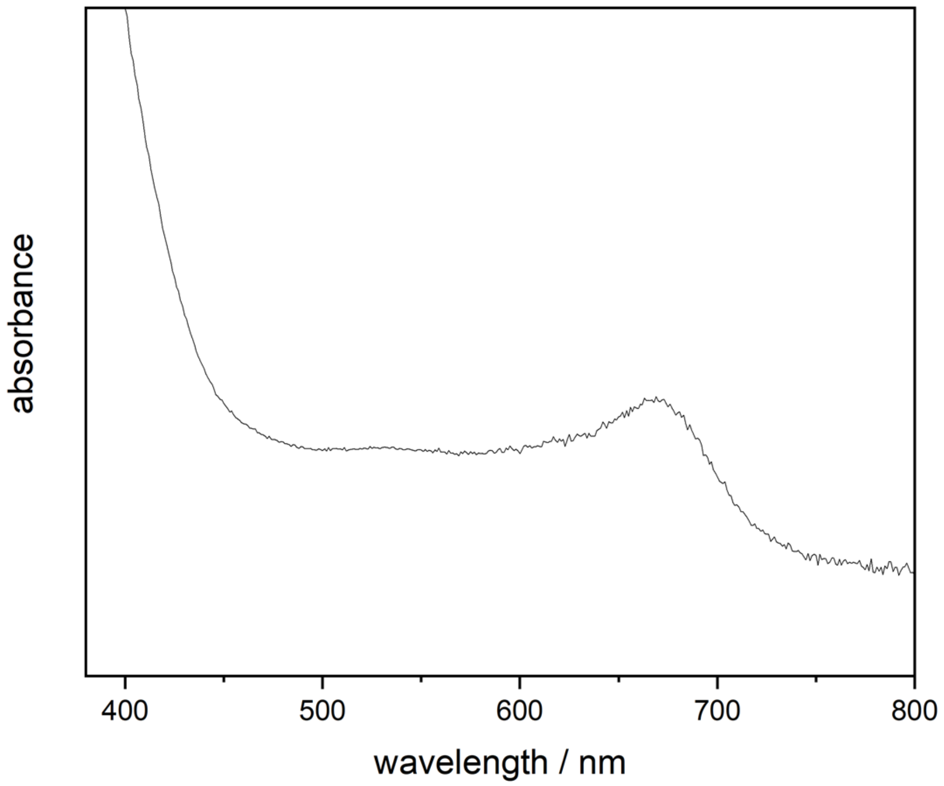

Figure S23. UV/vis spectrum of $[FeC_{10}Hg_{10}(THT)_{10}][SbF_6]_{11}$ in acetonitrile synthesized by oxidation of $[FeC_{10}Hg_{10}(THT)_{10}][SbF_6]_{10}$ with NO_2SbF_6. λ_{max} = 673 nm.

Cyclic voltammograms

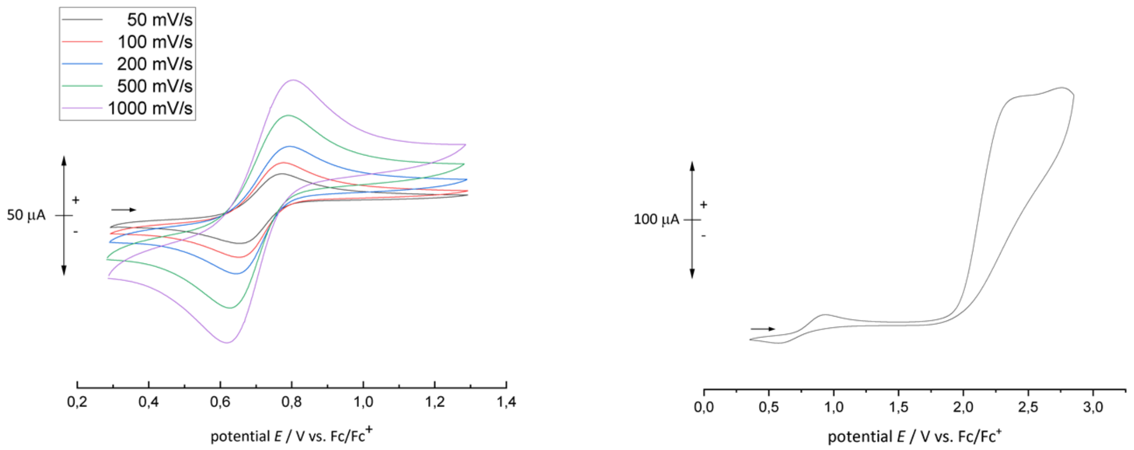

Figure S24. Cyclic voltammograms of $[FeC_{10}(HgMeCN)_{10}][SbF_6]_{10}$ in MeCN without supporting electrolyte at room temperature. First oxidation at different scan rates (left) and full cyclic voltammogram within the oxidation window of MeCN at 100 mV/s (right).

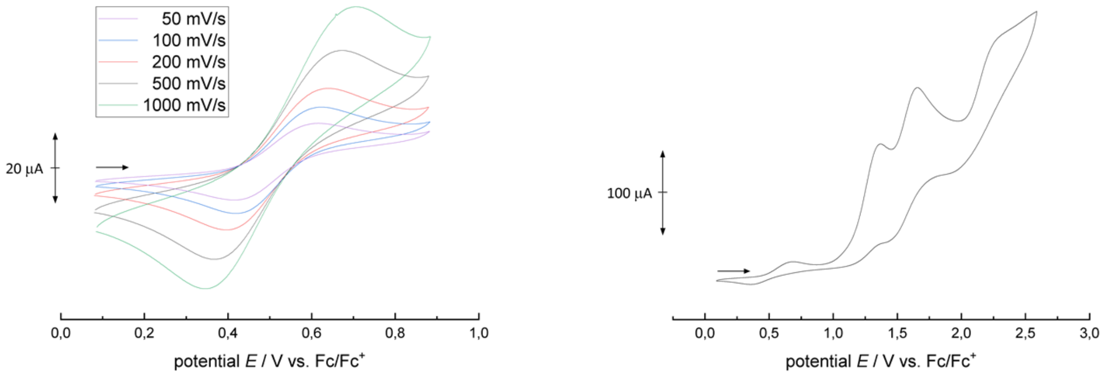

Figure S25. Cyclic voltammograms of $[FeC_{10}(HgTHT)_{10}][SbF_6]_{10}$ in MeCN without supporting electrolyte at room temperature. First oxidation at different scan rates (left) and full cyclic voltammogram within the oxidation window of MeCN at 100 mV/s (right).

Crystal Packing

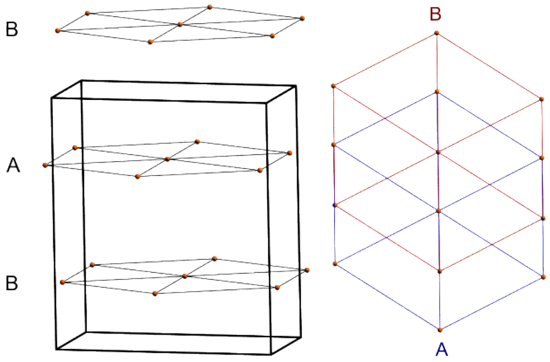

Figure S26. Packing of iron atoms in crystal structure of $[FeC_{10}(HgTHT)_{10}][SbF_6]_{10} \cdot 24\ MeCN$.