# Supplementary Information:

# Synthesis and characterization of a tris(hydroxyphenyl) methane-based cryptand and its triiron(III) complex

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## Electronic Supplementary Information

## Experimental

**General Considerations.** All reactions were performed under dry, air free condition using standard Schlenk techniques or in an Innovative Technologies glovebox unless otherwise noted. Solvents were either purchased anhydrous and used as received (Sigma-Aldrich) or extracted from an Innovative Technologies solvent purification system. NMR spectra were recorded on either a 500 Mhz Inova or 300 Mhz Mercury spectrophotometer with the spectra referenced to the residual protonated solvent signal, 7.27 ppm for $CDCl_3$-$d_1$ and 1.94 for $CH_3CN$-$d_3$. Deuterated solvents were purchased from Cambridge Isotope Labs and dried by standard methods as described elsewhere.[1] Infrared spectra were recorded as solids on a Bruker Vertex 80v FTIR using a Pike GladiATR stage. UV/VIS spectra were recorded on a Varian Cary 50 UV/VIS spectrophotometer using screw-top quartz cuvettes with a 1 cm path-length. X-Ray Intensity data were collected at 100 K on a Bruker DUO diffractometer using MoKα radiation (λ = 0.71073 Å) or CuKα radiation (λ = 1.54178 Å) from an ImuS power source, and an APEXII CCD area detector. Raw data frames were read by the SAINT[2] program and integrated using 3D profiling algorithms. The resulting data were reduced to produce hkl reflections and their intensities and estimated standard deviations. The data were corrected for Lorentz and polarization effects and numerical absorption corrections were applied based on indexed and measured faces. The structure was solved and refined in SHELXTL6.1, using full-matrix least-squares refinement.[3] The non-H atoms were refined with anisotropic thermal parameters and all of the H atoms were calculated in idealized positions and refined riding on their parent atoms unless otherwise stated. Cyclic voltammetry was performed under a nitrogen atmosphere using a standard three electrode setup. Electrodes were purchased from either BASi, Inc. or CH Instruments, Inc. Potential sweeps were controlled by a Princeton Applied Research Versastat II

potentiostat. Magnetic data were acquired using a Quantum Design MPMS XL superconducting quantum interference device (SQUID) magnetometer. 10.28 mg of $[K(MeCN)_2]_3[(FeCl_2)_3\mathbf{L}]$ microcrystals were introduced in a 0.273 ml PE sample vial along with about 20 mg of 1-eicosene. Once in the magnetometer, the vial was heated to 308 K for 10 minutes (1-eicoscene mp= 303 K) at 0 Gauss in order to homogenize the sample and 1-eicosene mixture before data were collected. The sample is cooled down from room temperature to 5K (10 K $min^{-1}$) in an applied field of 100 Gauss (field-cooled, FC) or zero Gauss (zero field-cooled, ZFC). Magnetization is measured while warming from 5 K to 300 K and then expressed in terms of magnetic susceptibility. Mass spectra were recorded on an Agilent 6210 TOF mass spectrometer by introducing samples into the MS by direct infusion. Samples were dissolved in anhydrous solvent in septum capped vials and transferred to the MS with a 10 μL syringe. All data were processed using MassHunter™ software. Elemental analyses were performed by Complete Analysis Laboratories, Inc (Parsippany, NJ). 2,6-Pyridinedicarboxylic acid chloride purchased from Sigma Aldrich was purified by vacuum sublimation prior to use. 4-(tert-butyl)-2-isopropylphenol (**1**) and 5-(tert-butyl)-2-hydroxy-3-isopropylbenzaldehyde (**2**) were synthesized according to literature procedures.[4,5] All other reagents were purchased from Sigma-Aldrich and used without further purification.

**6,6',6''-methanetriyltris(4-(tert-butyl)-2-isopropylphenol) (3).** Compound **3** was synthesized as described elsewhere with minor modifications.[6] Briefly, a sample of 5-(tert-butyl)-2-hydroxy-3-isopropylbenzaldehyde (**2**) (5.08g, 24.3 mmol) was combined with 2 equivalents of 4-(tert-butyl)-2-isopropylphenol (**1**) (9.17 g, 47.7 mmol) and 14 mL of methanol in a 100mL schlenk flask. With a sodium carbonate trap connected to the reaction flask, the reaction mixture was cooled to 0°C and thionyl chloride (11.4 mL, 157.7 mmol) was added

dropwise causing an immediate change from orange/yellow to dark purple for the reaction mixture. The reaction was stirred overnight. The precipitate that was formed was collected by filtration. The residue was recrystallized from hexanes to afford **3** as a pale purple solid (6.88 g) in 48% yield with respect to the starting salicylaldehyde. $^{1}$H NMR (*$CDCl_3$-d*) δ ppm 1.20 (27 H, s), 1.26 (18 H, d, *J*=6.9 Hz), 3.22 (3 H, spt, *J*=6.9 Hz), 4.91 (3 H, s), 5.83 (1 H, s), 6.81 (3 H, d, *J*=2.3 Hz), 7.18 (3 H, d, *J*=2.3 Hz). $^{13}$C-{$^{1}$H} NMR , δ 22.7, 27.5, 31.4, 34.3, 41.8, 122.2, 123.7, 125.7, 135.0, 143.5, 148.9. Anal. calcd. for $C_{40}H_{58}O_3$: C, 81.86; H, 9.96; N, 0.00. Found: C, 81.79; H, 10.32; N, 0.00.

**Tris(2-ethoxy-3-isopropyl-5-nitrophenyl)methane (4).** A dry 250 mL Schlenk flask was charged with **3** (9.17 g, 15.6 mmol) and anhydrous dimethylformamide (120 mL). The resulting solution was cooled to 0°C. NaH (1.24 g, 51.5 mmol) was added under $N_2$ flow, followed by addition of ethyl iodide (4.1 mL, 51 mmol) via syringe, and the reaction allowed to warm to room temperature overnight. Saturated aqueous $NH_4Cl$ was added to neutralize remaining NaH which generated a white precipitate. The precipitate was isolated by filtration and thoroughly rinsed with water, and subsequently recrystallized from methanol to yield tris(5-(*tert*-butyl)-2-ethoxy-3-isopropylphenyl)methane as a white crystalline solid (6.83 g) in 65% yield. $^{1}$H NMR (*$CDCl_3$-d*) δ ppm 1.17 (s, 27 H), 1.20 (d, *J*=6.8 Hz, 18 H), 1.30 (t, *J*=7.1 Hz, 9 H), 3.28 (spt, *J*=7.1 Hz, 3 H), 3.55 (q, *J*=7.1 Hz, 6 H), 6.59 (s, 1 H), 6.77 (d, *J*=2.3 Hz, 3 H), 7.07 (d, *J*=2.5 Hz, 3 H). $^{13}$C-{$^{1}$H} NMR , δ 15.7, 24.2, 26.6, 31.4, 34.3, 38.6, 69.3, 120.4, 125.7, 136.8, 140.6, 145.2, 152.1. Anal. calcd. for $C_{46}H_{70}O_3$: C, 82.33; H, 10.51; N, 0.00. Found: C, 82.27; H, 10.86; N, 0.00. A portion of the crystalline material (9.19g, 13.7 mmol) was dissolved in dichloromethane (48 mL) in a 250 mL round-bottom flask. To this solution was added

trifluoroacetic acid (9.4 mL, 123.3 mmol), causing a sudden color change to red. Concentrated nitric acid (7.9 mL, 125 mmol) was then slowly added to the reaction mixture. After stirring overnight, the solvent and trifluoroacetic acid were removed *in vacuo* from the bright orange reaction mixture. Methanol (112 mL) was added inducing precipitation of the crude product. The resulting suspension was cooled to approximately 0°C, filtered, and rinsed with cold methanol, giving **4** as a yellow powder (7.43 g, 85% yield). $^{1}$H NMR (*$CDCl_3$-d*) δ ppm 1.26 (9 H, t, *J*=6.8 Hz), 1.27 (18 H, d, *J*=6.8 Hz), 3.27 (3 H, spt, *J*=6.8 Hz), 3.51 (6 H, q, *J*=6.9 Hz), 6.71 (1 H, s), 7.64 (3 H, d, *J*=2.8 Hz), 8.14 (3 H, d, *J*=2.8 Hz). $^{13}$C-{$^{1}$H} NMR , δ 15.4, 23.6, 27.1, 38.8, 70.5, 122.3, 122.5, 137.3, 144.3, 144.6, 159.8. Anal. calcd. for $C_{34}H_{43}N_3O_9$: C, 64.03; H, 6.80; N, 6.59. Found: C, 63.67; H, 6.78; N, 6.30.

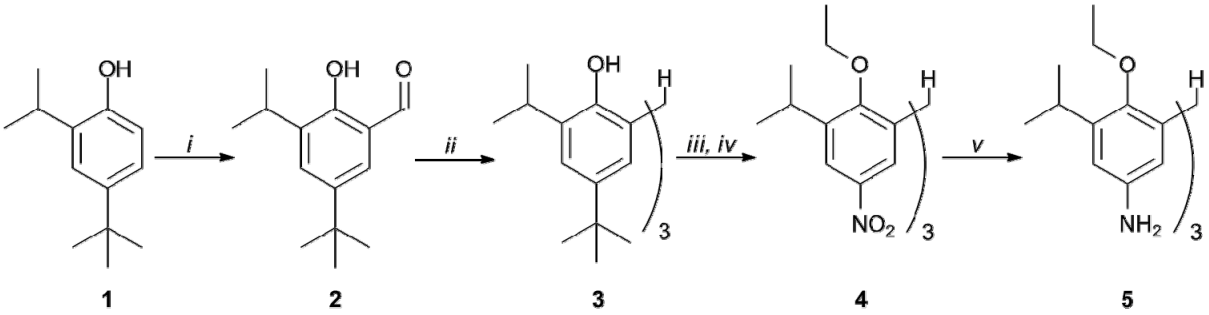
**5,5',5''-methanetriyltris(4-ethoxy-3-isopropylaniline) (5)**. In a three-neck round-bottom flask, a sample of **4** (3.63 g, 5.69 mmol) was dissolved in a solution of EtOH/THF (25/180 mL). Rainey nickel (~ 100 mg) was added to the mixture, which was then degassed by the freeze-pump-thaw method and the flask was backfilled with anhydrous $H_2$ gas. The reaction was stirred at room temperature under a constant $H_2$ stream for two days and then filtered to remove the Rainey nickel (*CAUTION: Raney Nickel is pyrophoric when dry!*). The solvent was removed under reduced pressure. The residue was dissolved in diethyl ether and extracted with a 10% HCl solution (3x, 50 mL). The aqueous phase was washed with hexanes (3x50 mL) followed by addition of 2 M NaOH to a final pH of 10. The precipitated solid was extracted into dichloromethane (3x50 mL). The combined organics were dried with sodium sulfate and the volatiles removed under reduced pressure. The residue was then recrystallized from methanol yielding 2.53 g (81%) of **5** as a colorless crystalline solid. $^{1}$H NMR (*$CDCl_3$-d*) δ ppm 1.14 (18 H,

d, *J*=6.9 Hz), 1.19 (9 H, t, *J*=7.0 Hz), 3.09 (6 H, br. s.), 3.21 (3 H, spt, *J*=6.9 Hz), 3.38 (6 H, q, *J*=6.9 Hz), 6.26 (3 H, d, *J*=2.9 Hz), 6.37 (3 H, d, *J*=2.7 Hz), 6.51 (1 H, s). $^{13}$C-{$^{1}$H} NMR , δ 15.5, 24.0, 26.3, 38.4, 69.4, 111.1, 115.2, 138.9, 141.6, 142.9, 147.3. Anal. calcd. for $C_{34}H_{49}N_3O_3$: C, 74.55; H, 9.02; N, 7.67. Found: C, 74.26; H, 9.24; N, 7.59.

**Di-(*N2*,*N2'*,*N2''*-(methanetriyltris(4-ethoxy-3-isopropylbenzene-5,1-diyl)))tris(pyridine-2,6-dicarboxamide) ($H_6$L).** In a Schlenk flask, **5** (2.67 g, 4.88 mmol) was dissolved in 100 mL of THF. The flask was fitted with an addition funnel containing 2,6-pyridinedicarboxylic acid chloride (1.49 g, 7.31 mmol) dissolved in 35 mL of THF. The reaction vessel was cooled to 0°C and the acid chloride solution was added dropwise over approximately 40 minutes. The reaction was allowed to warm to room temperature and stirred overnight. The solvent was removed from the reaction under reduced pressure and the resulting residue was purified by passage over a plug of silica gel (20:1 DCM:THF) yielding a pale yellow solid after removal of solvent. The resulting residue was further purified by crystallization in slowly evaporating THF yielding $H_6$**L** as a colorless crystalline solid (1.78 g, 48%). Single crystals suitable for x-ray diffraction studies were grown by diffusion of pentane into a THF solution of $H_6$**L**. $^{1}$H NMR (*$CDCl_3$-d*) δ ppm 1.14 - 1.20 (36 H, m), 1.31 (18 H, d, *J*=6.9 Hz), 3.26 (7 H, spt, *J*=6.8 Hz), 3.36 (6 H, m, *J*=8.8, 7.1 Hz), 3.83 (6 H, m, *J*=8.9, 7.1 Hz), 6.30 (6 H, d, *J*=2.7 Hz), 6.43 (2 H, s), 8.07 (3 H, t, *J*=7.8 Hz), 8.34 (6 H, d, *J*=2.6 Hz), 8.42 (6 H, d, *J*=7.8 Hz), 8.88 (6 H, s). $^{13}$C-{$^{1}$H} NMR , δ 15.8, 24.0, 24.4, 27.1, 40.7, 69.9, 119.0, 119.2, 126.0, 133.3, 138.4, 139.5, 143.6, 150.2, 152.0, 162.1. Sample analyzed as **5**·$(H_2O)_2$. Anal. calcd. for $C_{89}H_{105}N_9O_{14}$: C, 70.10; H, 6.94; N, 8.27. Found: C, 70.10; H, 6.95; N, 8.14.

**$[K(MeCN)_2]_3[(FeCl_2)_3L]\cdot(THF)_2$**. A portion of $H_6$**L** (203.6 mg, 0.1368 mmol) was dissolved 10 mL of THF and cooled to -78°C. A solution of KHMDS (180.2 mg, 0.9032 mmol) in 2 mL of THF was also cooled to -78°C and then added dropwise to the solution of $H_6$**L** causing an immediate change from a colorless to a yellow solution. The reaction was stirred for approximately 30 minutes during which time a solution of $FeCl_3$ (73.3mg, 0.452 mmol) was made in a minimum volume of THF and cooled to -78°C. The $FeCl_3$ solution was added dropwise to the yellow reaction mixture causing an immediate change to a dark red-brown solution. The reaction was stirred overnight, then filtered and the solvent was removed under vacuum. The residue was dissolved in a minimal volume of acetonitrile, precipitated with diethyl ether and the precipitate collected by filtration. Crystallization was afforded by a bulk diffusion of $Et_2O$ into acetonitrile to yield black microcrystalline powder. Single crystals for x-ray diffraction were grown by a slow diffusion of diethyl ether into a solution of the complex in acetonitrile. (-)ESI-MS (DMF): 951.9 m/z, $[K(FeCl_2)_3\mathbf{L}]^{2-}$. Anal. calcd. for $C_{97}H_{111}Cl_6Fe_3K_3N_9O_{14}$: C, 54.84; H, 5.27; N, 5.93. Found: C, 54.53; H, 5.16; N, 5.78.

**Scheme S1.** Synthesis of 5,5',5''-methanetriyltris(4-ethoxy-3-isopropylaniline), **5**




*i)* Ref. 5 *ii)* **1**, $SOCl_2$, MeOH *iii)* EtI, NaH, DMF i*v)* $HNO_3$, TFA *vi* Raney Ni, $H_{2(g)}$, THF/EtOH

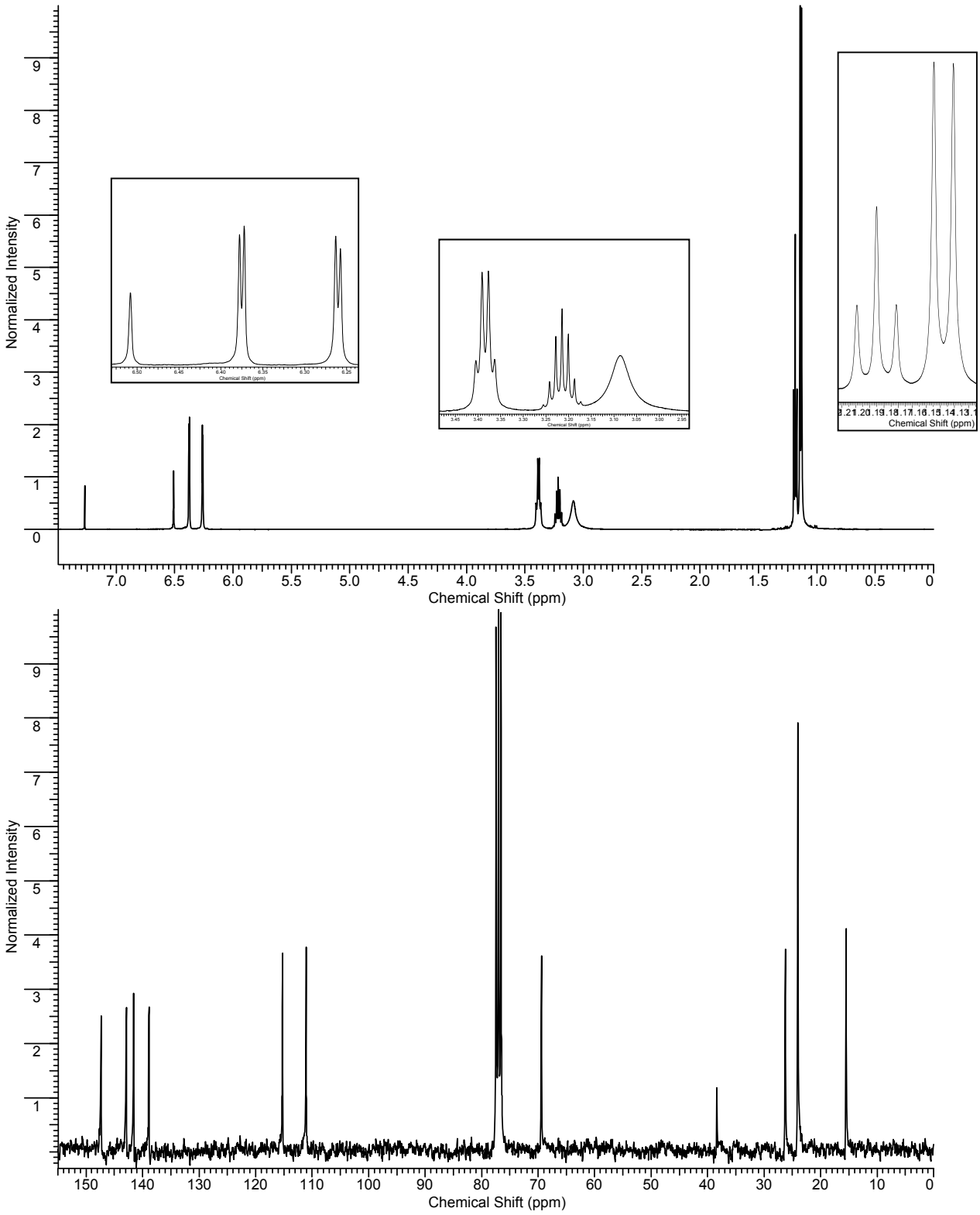



**Figure S1.** $^{1}H$ and $^{13}C$-NMR spectra for **5** in $CDCl_3$ at RT.

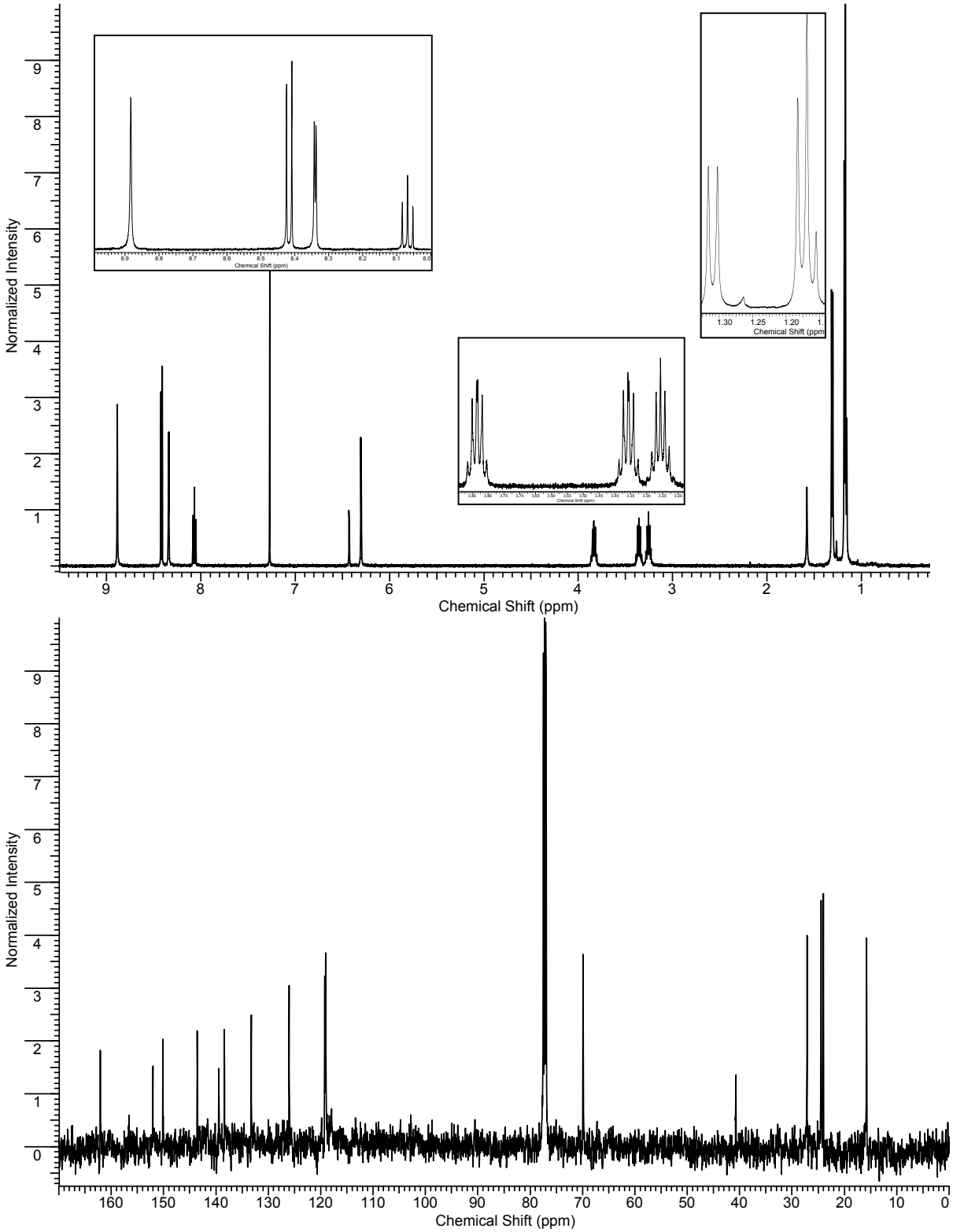



**Figure S2.** $^{1}$H and $^{13}$C-NMR spectra for **$H_6$L** in $CDCl_3$ at RT.

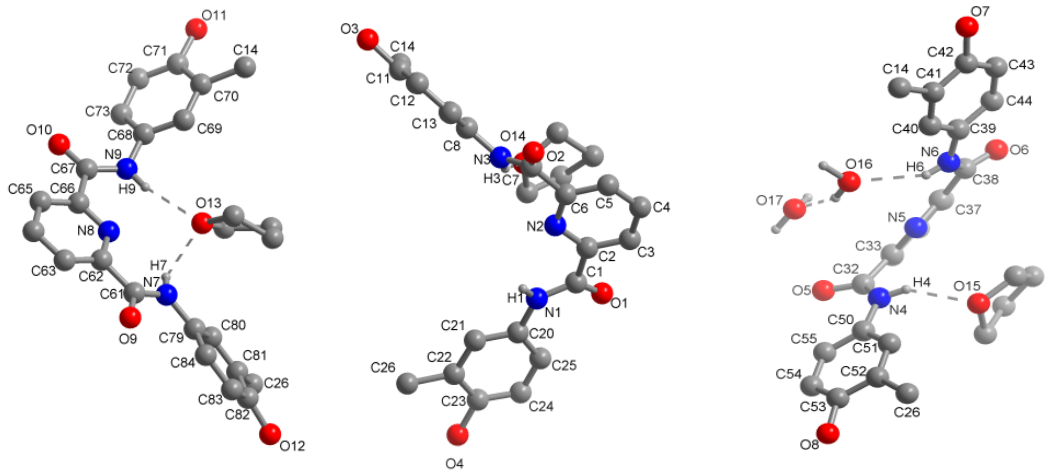



**Figure S3**. A portion of the x-ray structure of $H_6$**L**·$(THF)_3$·$(H_2O)_2$ highlighting the specific hydrogen bonds in each ligand arm. Carbon, oxygen, nitrogen, and hydrogen atoms are represented by grey, red, blue, and light gray spheres, respectively. Alkyl substitution on the triphenoxymethane caps have been removed along with all but the amide N-H hydrogens for clarity. Hydrogen atoms are placed in idealized positions. Non-interacting pentane molecules in the asymmetric unit were removed using SQUEEZE. Atoms are represented as standard radii spheres.

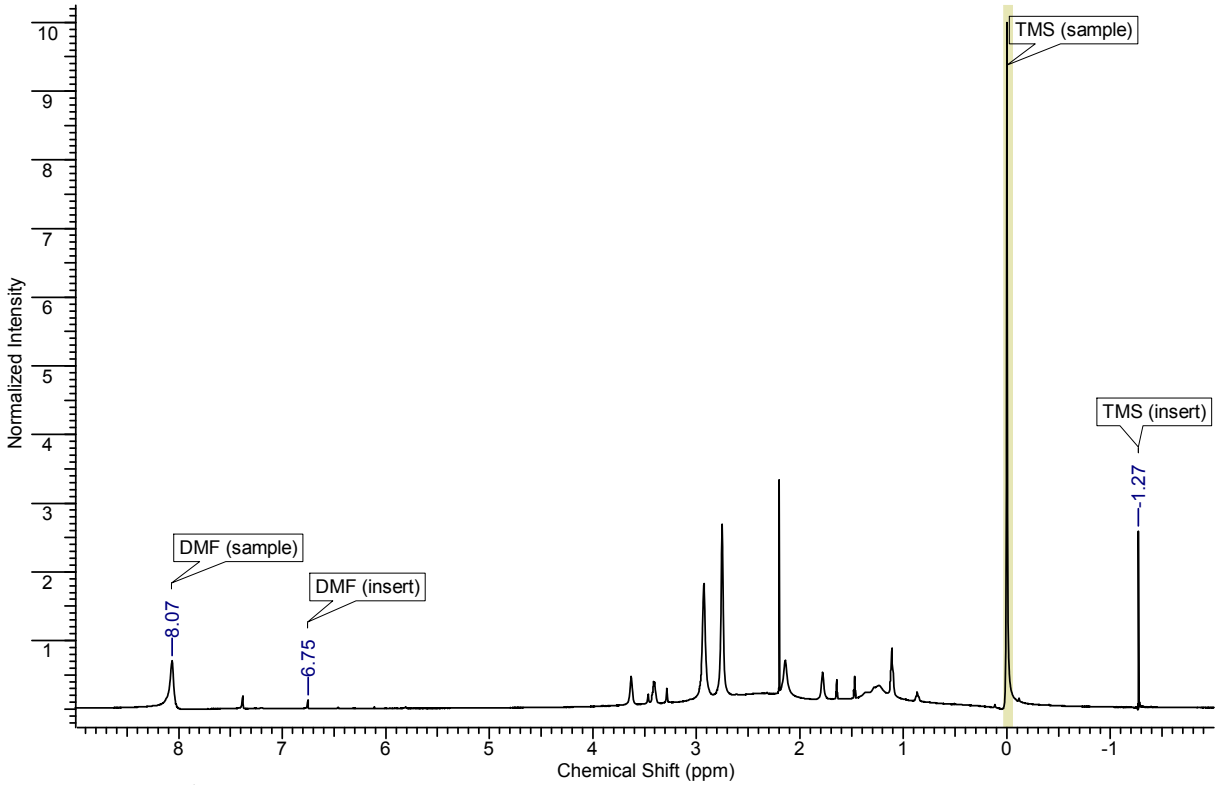



**Figure S4.** $^{1}$H-NMR spectrum of $[K(MeCN)_2]_3[(Fe^{III}Cl_2)_3\mathbf{L}]$ in DMF-$d_7$ at RT.

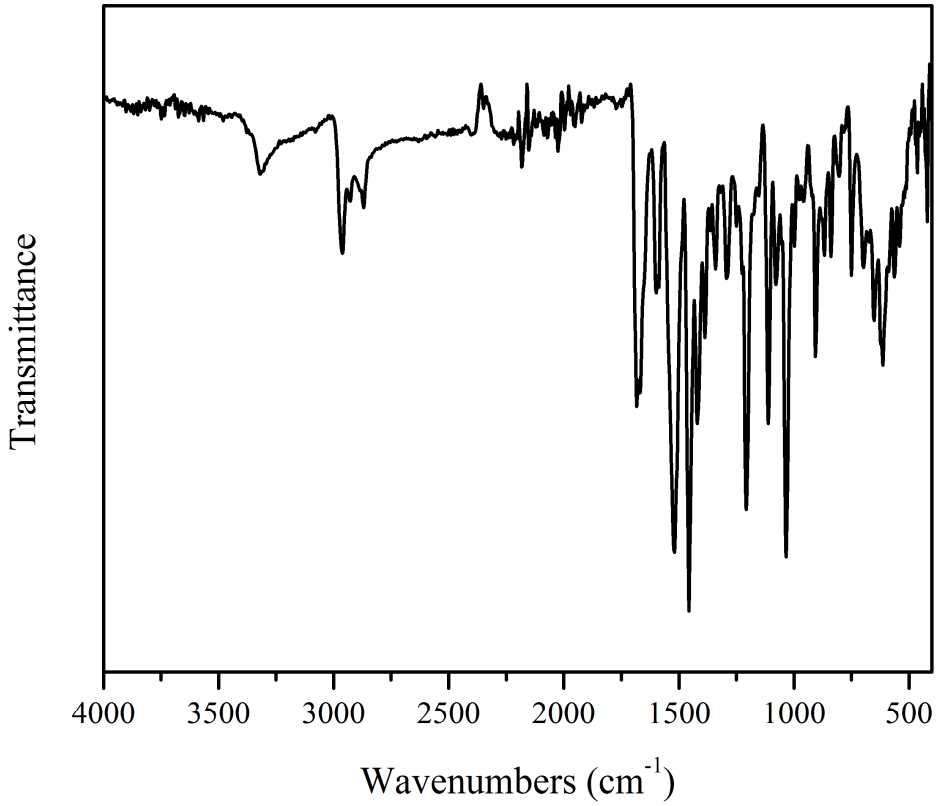



**Figure S5.** IR spectrum of $\mathbf{H_6L}$.

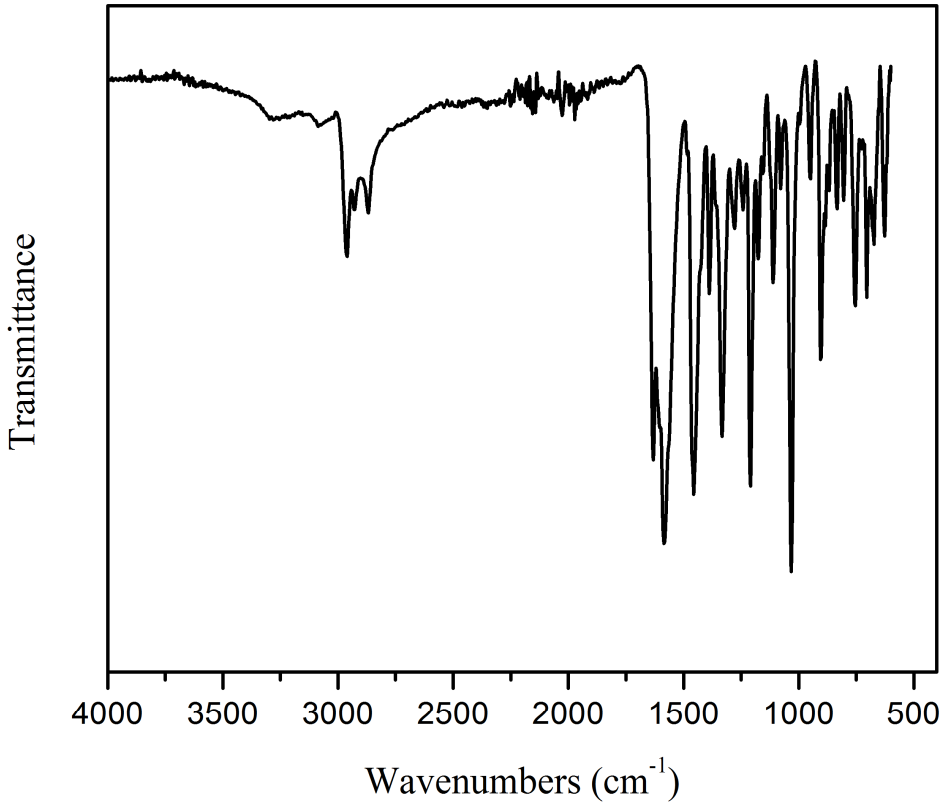



**Figure S6.** IR spectrum of $[K(MeCN)_2]_3[(FeCl_2)_3\mathbf{L}]$

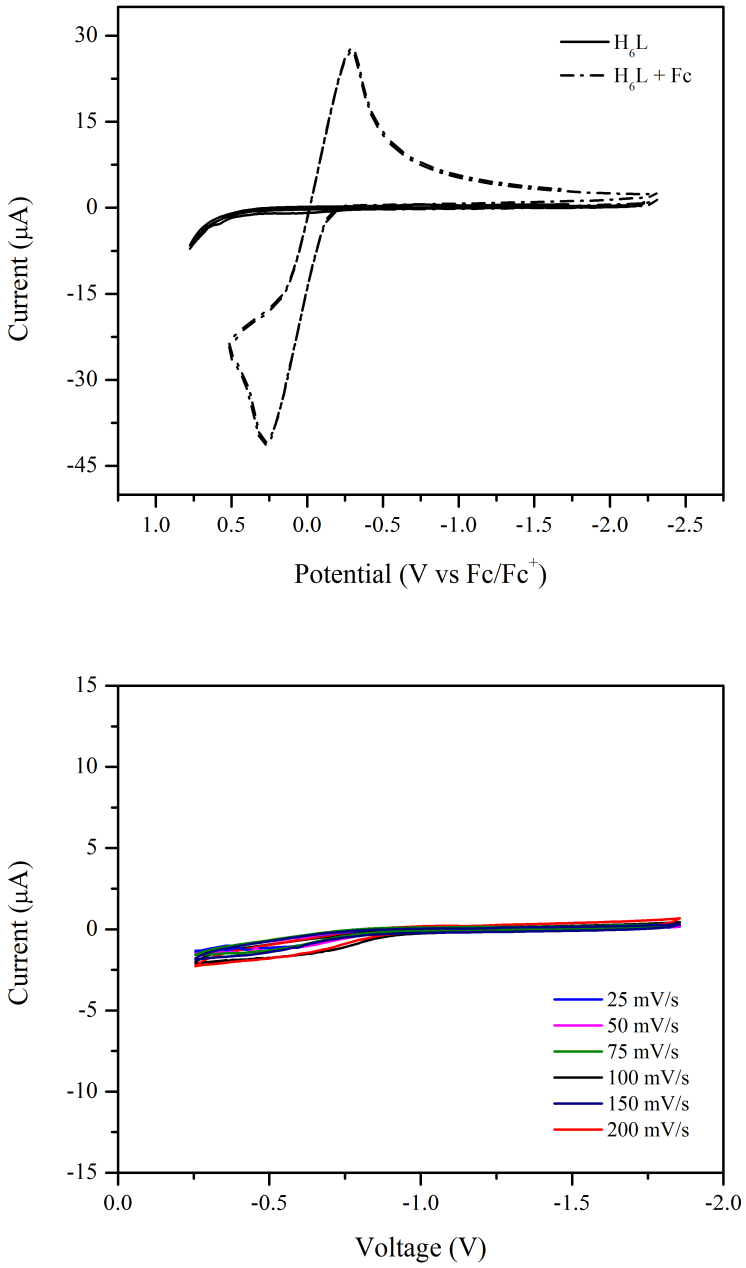



**Figure S7.** Cyclic voltammograms of (top) **$H_6$L** in tetrahydrofuran with and without ferrocinium as an internal standard (scan rate 100 mV/s for all scans) and (bottom) the reaction of **$H_6$L** and KHMDS in THF. For both, 0.1M tetrabutylammonium hexafluorophosphate was used as supporting electrolyte. Working electrode: 1mm Pt button, auxillary electrode: Pt wire, reference electrode: $Ag/AgNO_3$ in MeCN.

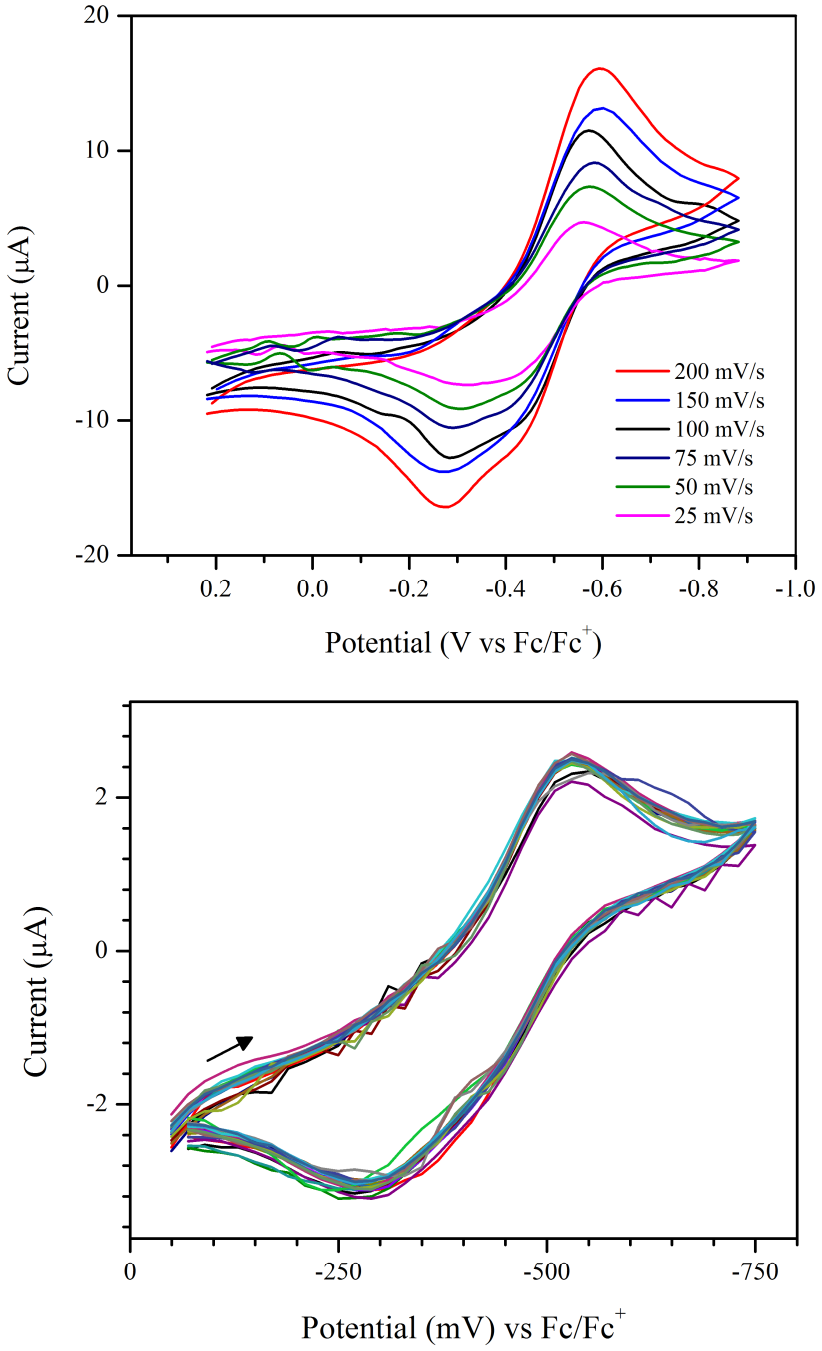



**Figure S8.** Cyclic voltammagrams of $[K(MeCN_2)]_3[(Fe^{III}Cl_2)_3\mathbf{L}]$. (top) Scanning rate dependence and (bottom) repeated scans at a rate of 100 mV/s. All scans performed in DMF with 0.1M tetrabutylammonium hexafluorophosphate used as supporting electrolyte. Working electrode: 2 mm Pt button, auxillary electrode: Pt wire, reference electrode: $Ag/AgNO_3$ in MeCN.

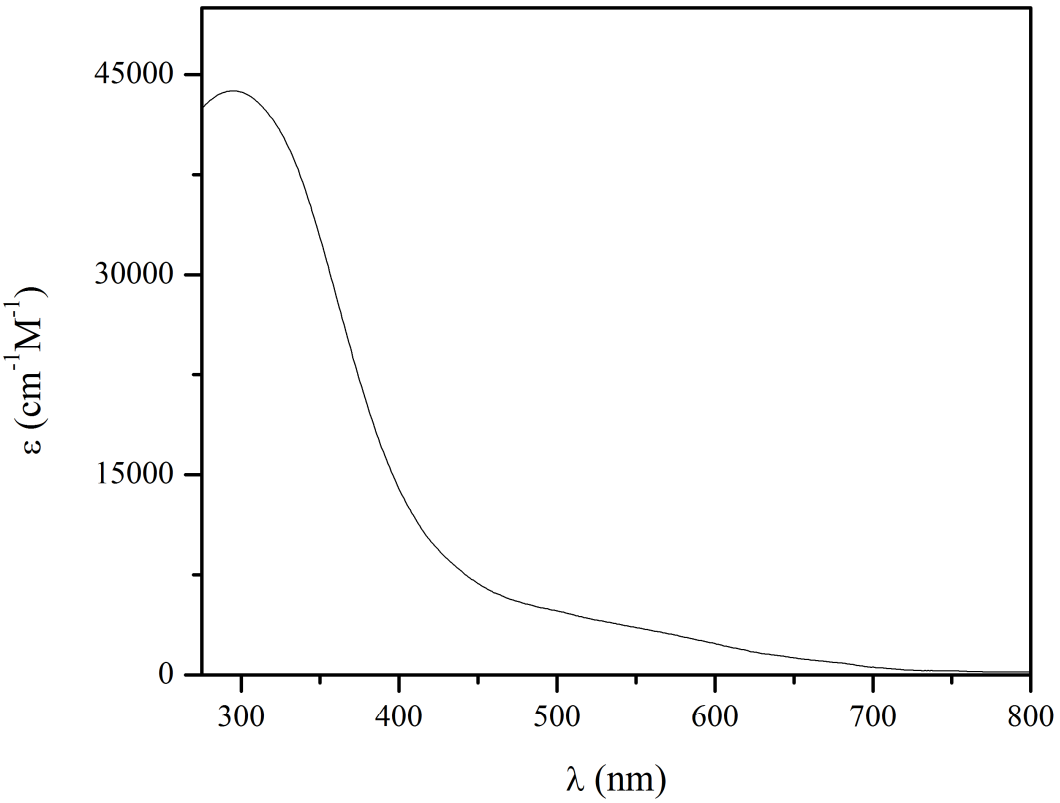



**Figure S9.** UV/VIS spectrum of $[K(MeCN_2)]_3[(Fe^{III}Cl_2)_3\mathbf{L}]$ in DMF.

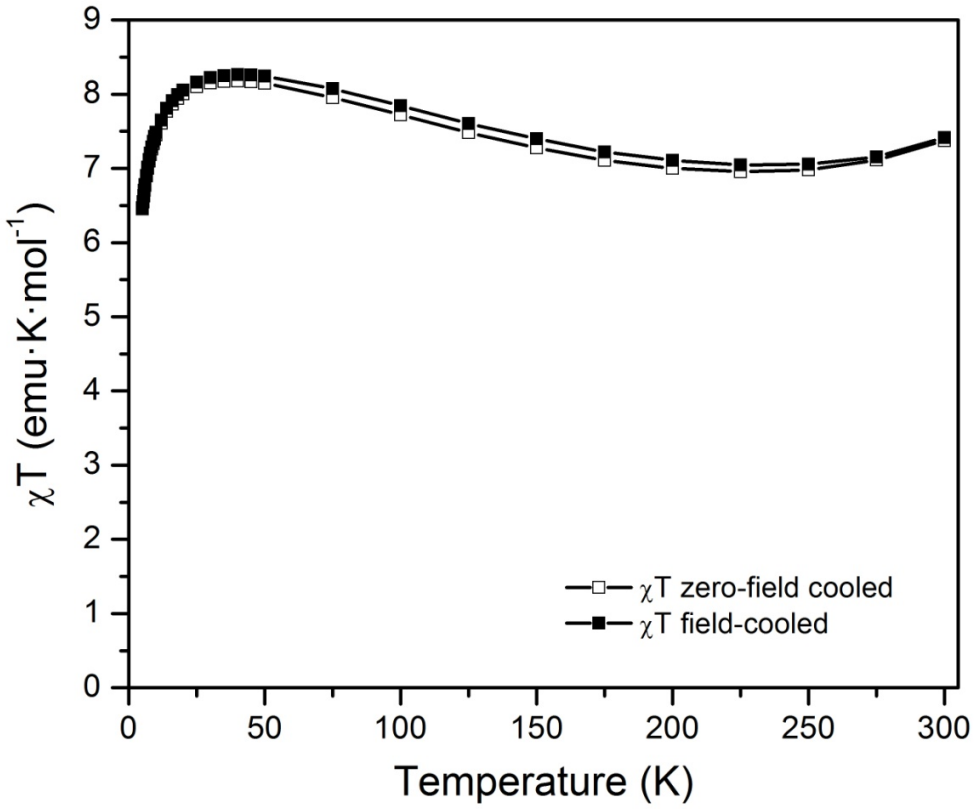



**Figure S10.** The field cooled magnetic susceptibility multiplied by temperature, χT, H=100 G as a function of temperature ( ■ ), and the zero-field cooled magnetic susceptibility multiplied by temperature, χT, H=100G, as a function of temperature ( □ ). Lines are guides for the eyes.

**Table S1.** Crystal data and structure refinement for **$H_6L$**.

| | | |
|---|---|---|
| Identification code | gui10 | |
| Empirical formula | C101 H129 N9 O17 | |
| Formula weight | 1741.13 | |
| Temperature | 100(2) K | |
| Wavelength | 1.54178 Å | |
| Crystal system | Triclinic | |
| Space group | P-1 | |
| Unit cell dimensions | a = 17.9708(2) Å | α= 73.327(1)°. |
| | b = 19.0228(3) Å | β= 67.465(1)°. |
| | c = 19.2837(3) Å | γ = 66.260(1)°. |
| Volume | 5504.66(14) $Å^3$ | |
| Z | 2 | |
| Density (calculated) | 1.050 $Mg/m^3$ | |
| Absorption coefficient | 0.577 $mm^{-1}$ | |
| F(000) | 1868 | |
| Crystal size | 0.26 x 0.05 x 0.02 $mm^3$ | |
| Theta range for data collection | 2.51 to 66.50°. | |
| Index ranges | $-19\leq h\leq 21$, $-18\leq k\leq 21$, $-21\leq l\leq 22$ | |
| Reflections collected | 67204 | |
| Independent reflections | 18677 [R(int) = 0.0957] | |
| Completeness to theta = 66.50° | 96.2 % | |
| Absorption correction | Numerical | |
| Max. and min. transmission | 0.9891 and 0.8635 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 18677 / 30 / 1173 | |
| Goodness-of-fit on $F^2$ | 0.837 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0603, wR2 = 0.1448 [9470] | |
| R indices (all data) | R1 = 0.1096, wR2 = 0.1609 | |
| Largest diff. peak and hole | 0.581 and -0.353 $e.Å^{-3}$ | |

$R1 = \Sigma(||F_o| - |F_c||) / \Sigma|F_o|$

$wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]]^{1/2}$

$S = [\Sigma[w(F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$

$w= 1/0[\sigma^2(F_o^2)+(m*p)^2+n*p]$, $p = [\max(F_o^2,0)+ 2* F_c^2]/3$, m & n are constants.

**Table S2.** Bond lengths [Å] and angles [°] for **$H_6$5**.

| | | | |
|---|---|---|---|
| O1-C1 | 1.241(4) | N7-C61 | 1.354(4) |
| O2-C7 | 1.223(4) | N7-C79 | 1.404(4) |
| O3-C11 | 1.387(4) | N7-H7 | 0.86(3) |
| O3-C15 | 1.448(3) | N8-C66 | 1.337(4) |
| O4-C23 | 1.391(4) | N8-C62 | 1.345(3) |
| O4-C27 | 1.443(4) | N9-C67 | 1.349(4) |
| O5-C32 | 1.261(4) | N9-C68 | 1.433(3) |
| O5'-C32 | 1.287(9) | N9-H9 | 0.87(3) |
| O6-C38 | 1.234(3) | C1-C2 | 1.496(5) |
| O7-C42 | 1.397(4) | C2-C3 | 1.382(4) |
| O7-C45 | 1.401(4) | C3-C4 | 1.389(5) |
| O8-C53 | 1.387(3) | C3-H3A | 0.9500 |
| O8-C56 | 1.436(4) | C4-C5 | 1.377(5) |
| O9-C61 | 1.235(3) | C4-H4A | 0.9500 |
| O10-C67 | 1.215(4) | C5-C6 | 1.398(4) |
| O11-C71 | 1.396(3) | C5-H5A | 0.9500 |
| O11-C74 | 1.438(4) | C6-C7 | 1.501(5) |
| O12-C82 | 1.376(4) | C8-C13 | 1.387(4) |
| O12-C85 | 1.436(3) | C8-C9 | 1.400(4) |
| N1-C1 | 1.339(4) | C9-C10 | 1.380(4) |
| N1-C20 | 1.416(4) | C9-H9A | 0.9500 |
| N1-H1 | 0.78(3) | C10-C11 | 1.398(4) |
| N2-C6 | 1.339(4) | C10-C14 | 1.529(4) |
| N2-C2 | 1.362(4) | C11-C12 | 1.407(4) |
| N3-C7 | 1.351(4) | C12-C13 | 1.400(5) |
| N3-C8 | 1.410(4) | C12-C17 | 1.508(5) |
| N3-H3 | 0.86(4) | C13-H13A | 0.9500 |
| N4-C32 | 1.320(4) | C14-C41 | 1.521(4) |
| N4-C50 | 1.417(3) | C14-C70 | 1.537(4) |
| N4-H4 | 0.86(4) | C14-H14A | 1.0000 |
| N5-C33 | 1.332(4) | C15-C16 | 1.504(5) |
| N5-C37 | 1.344(3) | C15-H15A | 0.9900 |
| N6-C38 | 1.342(4) | C15-H15B | 0.9900 |
| N6-C39 | 1.416(4) | C16-H16A | 0.9800 |
| N6-H6 | 0.81(4) | C16-H16B | 0.9800 |

| | | | |
|---|---|---|---|
| C16-H16C | 0.9800 | C30-H30B | 0.9800 |
| C17-C18 | 1.524(5) | C30-H30C | 0.9800 |
| C17-C19 | 1.525(6) | C31-H29B | 1.5306 |
| C17-H17A | 1.0000 | C31-H31A | 0.9800 |
| C18-H18A | 0.9800 | C31-H31B | 0.9800 |
| C18-H18B | 0.9800 | C31-H31C | 0.9800 |
| C18-H18C | 0.9800 | C30'-H30D | 0.9800 |
| C19-H19A | 0.9800 | C30'-H30E | 0.9800 |
| C19-H19B | 0.9800 | C30'-H30F | 0.9800 |
| C19-H19C | 0.9800 | C31'-H31D | 0.9800 |
| C20-C25 | 1.387(5) | C31'-H31E | 0.9800 |
| C20-C21 | 1.393(4) | C31'-H31F | 0.9800 |
| C21-C22 | 1.401(4) | C32-C33 | 1.504(4) |
| C21-H21A | 0.9500 | C33-C34 | 1.391(4) |
| C22-C23 | 1.404(5) | C34-C35 | 1.375(4) |
| C22-C26 | 1.521(4) | C34-H34A | 0.9500 |
| C23-C24 | 1.388(5) | C35-C36 | 1.384(5) |
| C24-C25 | 1.392(5) | C35-H35A | 0.9500 |
| C24-C29 | 1.517(5) | C36-C37 | 1.382(4) |
| C25-H25A | 0.9500 | C36-H36A | 0.9500 |
| C26-C81 | 1.518(4) | C37-C38 | 1.500(4) |
| C26-C52 | 1.527(4) | C39-C40 | 1.375(4) |
| C26-H26A | 1.0000 | C39-C44 | 1.400(4) |
| C27-C28 | 1.513(5) | C40-C41 | 1.383(4) |
| C27-H27A | 0.9900 | C40-H40A | 0.9500 |
| C27-H27B | 0.9900 | C41-C42 | 1.401(4) |
| C28-H28A | 0.9800 | C42-C43 | 1.394(5) |
| C28-H28B | 0.9800 | C43-C44 | 1.401(5) |
| C28-H28C | 0.9800 | C43-C47 | 1.534(4) |
| C29-C30' | 1.310(9) | C44-H44A | 0.9500 |
| C29-C31 | 1.411(7) | C45-C46 | 1.439(6) |
| C29-C31' | 1.634(11) | C45-H45A | 0.9900 |
| C29-C30 | 1.651(8) | C45-H45B | 0.9900 |
| C29-H29A | 1.0000 | C46-H46A | 0.9800 |
| C29-H29B | 1.0000 | C46-H46B | 0.9800 |
| C30-H30A | 0.9800 | C46-H46C | 0.9800 |

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|---|---|---|---|
| C47-C48 | 1.519(5) | C63-H63A | 0.9500 |
| C47-C49 | 1.531(6) | C64-C65 | 1.384(4) |
| C47-H47A | 1.0000 | C64-H64A | 0.9500 |
| C48-H48A | 0.9800 | C65-C66 | 1.388(4) |
| C48-H48B | 0.9800 | C65-H65A | 0.9500 |
| C48-H48C | 0.9800 | C66-C67 | 1.508(4) |
| C49-H49A | 0.9800 | C68-C73 | 1.379(4) |
| C49-H49B | 0.9800 | C68-C69 | 1.384(4) |
| C49-H49C | 0.9800 | C69-C70 | 1.392(4) |
| C50-C55 | 1.393(4) | C69-H69A | 0.9500 |
| C50-C51 | 1.395(4) | C70-C71 | 1.393(4) |
| C51-C52 | 1.383(4) | C71-C72 | 1.392(4) |
| C51-H51A | 0.9500 | C72-C73 | 1.404(4) |
| C52-C53 | 1.410(4) | C72-C76 | 1.521(4) |
| C53-C54 | 1.383(4) | C73-H73A | 0.9500 |
| C54-C55 | 1.382(4) | C74-C75 | 1.491(4) |
| C54-C58 | 1.531(4) | C74-H74A | 0.9900 |
| C55-H55A | 0.9500 | C74-H74B | 0.9900 |
| C56-C57 | 1.484(6) | C75-H75A | 0.9800 |
| C56-H56A | 0.9900 | C75-H75B | 0.9800 |
| C56-H56B | 0.9900 | C75-H75C | 0.9800 |
| C57-H57A | 0.9800 | C76-C78 | 1.508(5) |
| C57-H57B | 0.9800 | C76-C77 | 1.523(5) |
| C57-H57C | 0.9800 | C76-H76A | 1.0000 |
| C58-C60 | 1.515(6) | C77-H77A | 0.9800 |
| C58-C59 | 1.536(6) | C77-H77B | 0.9800 |
| C58-H58A | 1.0000 | C77-H77C | 0.9800 |
| C59-H59A | 0.9800 | C78-H78A | 0.9800 |
| C59-H59B | 0.9800 | C78-H78B | 0.9800 |
| C59-H59C | 0.9800 | C78-H78C | 0.9800 |
| C60-H60A | 0.9800 | C79-C84 | 1.400(4) |
| C60-H60B | 0.9800 | C79-C80 | 1.402(4) |
| C60-H60C | 0.9800 | C80-C81 | 1.385(4) |
| C61-C62 | 1.491(4) | C80-H80A | 0.9500 |
| C62-C63 | 1.388(4) | C81-C82 | 1.409(4) |
| C63-C64 | 1.368(5) | C82-C83 | 1.403(4) |

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|---|---|---|---|
| C83-C84 | 1.382(4) | C94-H94B | 0.9900 |
| C83-C87 | 1.530(4) | C94-H94C | 0.9900 |
| C84-H84A | 0.9500 | C94-H94D | 0.9899 |
| C85-C86 | 1.494(5) | C95-C96 | 1.692(16) |
| C85-H85A | 0.9900 | C95-H95A | 0.9900 |
| C85-H85B | 0.9900 | C95-H95B | 0.9900 |
| C86-H86A | 0.9800 | C96-C97 | 1.527(9) |
| C86-H86B | 0.9800 | C96-H96A | 0.9900 |
| C86-H86C | 0.9800 | C96-H96B | 0.9900 |
| C87-C89 | 1.535(4) | C95'-C96' | 1.373(18) |
| C87-C88 | 1.539(4) | C95'-H95C | 0.9900 |
| C87-H87A | 1.0000 | C95'-H95D | 0.9900 |
| C88-H88A | 0.9800 | C96'-C97 | 1.596(11) |
| C88-H88B | 0.9800 | C96'-H96C | 0.9900 |
| C88-H88C | 0.9800 | C96'-H96D | 0.9900 |
| C89-H89A | 0.9800 | C97-H97A | 0.9900 |
| C89-H89B | 0.9800 | C97-H97B | 0.9900 |
| C89-H89C | 0.9800 | C97-H97C | 0.9900 |
| O13-C93 | 1.436(4) | C97-H97D | 0.9898 |
| O13-C90 | 1.453(4) | O15-C98 | 1.424(8) |
| C90-C91 | 1.524(5) | O15-C101 | 1.448(10) |
| C90-H90A | 0.9900 | C98-C99 | 1.481(10) |
| C90-H90B | 0.9900 | C98-H98A | 0.9900 |
| C91-C92 | 1.485(5) | C98-H98B | 0.9900 |
| C91-H91A | 0.9900 | C99-C100 | 1.49(1) |
| C91-H91B | 0.9900 | C99-H99A | 0.9900 |
| C92-C93 | 1.494(5) | C99-H99B | 0.9900 |
| C92-H92A | 0.9900 | C100-C101 | 1.631(10) |
| C92-H92B | 0.9900 | C100-H10A | 0.9900 |
| C93-H93A | 0.9900 | C100-H10B | 0.9900 |
| C93-H93B | 0.9900 | C101-H10C | 0.9900 |
| O14-C94 | 1.417(5) | C101-H10D | 0.9900 |
| O14-C97 | 1.421(5) | O15'-C102 | 1.491(15) |
| C94-C95 | 1.504(11) | O15'-C105 | 1.556(15) |
| C94-C95' | 1.593(17) | C102-C103 | 1.443(15) |
| C94-H94A | 0.9900 | C102-H10E | 0.9900 |

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|---|---|---|---|
| C102-H10F | 0.9900 | C79-N7-H7 | 116(2) |
| C103-C104 | 1.525(15) | C66-N8-C62 | 117.0(2) |
| C103-H10G | 0.9900 | C67-N9-C68 | 123.4(3) |
| C103-H10H | 0.9900 | C67-N9-H9 | 120.1(19) |
| C104-C105 | 1.551(15) | C68-N9-H9 | 114.8(19) |
| C104-H10I | 0.9900 | O1-C1-N1 | 125.0(3) |
| C104-H10J | 0.9900 | O1-C1-C2 | 119.5(3) |
| C105-H10K | 0.9900 | N1-C1-C2 | 115.4(3) |
| C105-H10L | 0.9900 | N2-C2-C3 | 123.2(3) |
| O16-H16' | 1.0660 | N2-C2-C1 | 118.3(2) |
| O16-H16" | 0.9412 | C3-C2-C1 | 118.5(3) |
| O17-H17' | 0.9294 | C2-C3-C4 | 118.3(3) |
| O17-H17" | 0.9219 | C2-C3-H3A | 120.9 |
| | | C4-C3-H3A | 120.9 |
| C11-O3-C15 | 115.9(2) | C5-C4-C3 | 119.3(3) |
| C23-O4-C27 | 116.5(2) | C5-C4-H4A | 120.3 |
| C42-O7-C45 | 121.2(3) | C3-C4-H4A | 120.3 |
| C53-O8-C56 | 114.8(3) | C4-C5-C6 | 119.1(3) |
| C71-O11-C74 | 114.6(2) | C4-C5-H5A | 120.5 |
| C82-O12-C85 | 116.8(2) | C6-C5-H5A | 120.5 |
| C1-N1-C20 | 128.6(3) | N2-C6-C5 | 122.4(3) |
| C1-N1-H1 | 116(2) | N2-C6-C7 | 118.6(2) |
| C20-N1-H1 | 115(2) | C5-C6-C7 | 118.8(3) |
| C6-N2-C2 | 117.5(2) | O2-C7-N3 | 125.4(3) |
| C7-N3-C8 | 128.3(3) | O2-C7-C6 | 121.1(3) |
| C7-N3-H3 | 113(3) | N3-C7-C6 | 113.3(3) |
| C8-N3-H3 | 118(3) | C13-C8-C9 | 118.6(3) |
| C32-N4-C50 | 128.2(2) | C13-C8-N3 | 124.4(3) |
| C32-N4-H4 | 114(3) | C9-C8-N3 | 116.9(3) |
| C50-N4-H4 | 116(3) | C10-C9-C8 | 121.7(3) |
| C33-N5-C37 | 116.8(3) | C10-C9-H9A | 119.1 |
| C38-N6-C39 | 128.5(2) | C8-C9-H9A | 119.1 |
| C38-N6-H6 | 113(2) | C9-C10-C11 | 118.9(2) |
| C39-N6-H6 | 116(3) | C9-C10-C14 | 121.2(3) |
| C61-N7-C79 | 125.3(2) | C11-C10-C14 | 119.9(3) |
| C61-N7-H7 | 117(2) | O3-C11-C10 | 117.1(2) |

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|---|---|---|---|
| O3-C11-C12 | 121.8(3) | H18A-C18-H18C | 109.5 |
| C10-C11-C12 | 120.8(3) | H18B-C18-H18C | 109.5 |
| C13-C12-C11 | 118.4(3) | C17-C19-H19A | 109.5 |
| C13-C12-C17 | 120.2(3) | C17-C19-H19B | 109.5 |
| C11-C12-C17 | 121.4(3) | H19A-C19-H19B | 109.5 |
| C8-C13-C12 | 121.4(3) | C17-C19-H19C | 109.5 |
| C8-C13-H13A | 119.3 | H19A-C19-H19C | 109.5 |
| C12-C13-H13A | 119.3 | H19B-C19-H19C | 109.5 |
| C41-C14-C10 | 112.1(2) | C25-C20-C21 | 119.9(3) |
| C41-C14-C70 | 111.0(2) | C25-C20-N1 | 122.8(3) |
| C10-C14-C70 | 112.9(2) | C21-C20-N1 | 117.2(3) |
| C41-C14-H14A | 106.8 | C20-C21-C22 | 120.7(3) |
| C10-C14-H14A | 106.8 | C20-C21-H21A | 119.6 |
| C70-C14-H14A | 106.8 | C22-C21-H21A | 119.6 |
| O3-C15-C16 | 108.0(3) | C21-C22-C23 | 117.9(3) |
| O3-C15-H15A | 110.1 | C21-C22-C26 | 121.7(3) |
| C16-C15-H15A | 110.1 | C23-C22-C26 | 120.3(2) |
| O3-C15-H15B | 110.1 | C24-C23-O4 | 120.3(3) |
| C16-C15-H15B | 110.1 | C24-C23-C22 | 121.8(3) |
| H15A-C15-H15B | 108.4 | O4-C23-C22 | 117.7(3) |
| C15-C16-H16A | 109.5 | C23-C24-C25 | 118.9(3) |
| C15-C16-H16B | 109.5 | C23-C24-C29 | 121.2(3) |
| H16A-C16-H16B | 109.5 | C25-C24-C29 | 119.8(3) |
| C15-C16-H16C | 109.5 | C20-C25-C24 | 120.7(3) |
| H16A-C16-H16C | 109.5 | C20-C25-H25A | 119.6 |
| H16B-C16-H16C | 109.5 | C24-C25-H25A | 119.6 |
| C12-C17-C18 | 111.0(3) | C81-C26-C22 | 113.8(2) |
| C12-C17-C19 | 110.9(3) | C81-C26-C52 | 113.6(3) |
| C18-C17-C19 | 112.6(4) | C22-C26-C52 | 111.3(2) |
| C12-C17-H17A | 107.4 | C81-C26-H26A | 105.8 |
| C18-C17-H17A | 107.4 | C22-C26-H26A | 105.8 |
| C19-C17-H17A | 107.4 | C52-C26-H26A | 105.8 |
| C17-C18-H18A | 109.5 | O4-C27-C28 | 106.6(3) |
| C17-C18-H18B | 109.5 | O4-C27-H27A | 110.4 |
| H18A-C18-H18B | 109.5 | C28-C27-H27A | 110.4 |
| C17-C18-H18C | 109.5 | O4-C27-H27B | 110.4 |

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| C28-C27-H27B | 110.4 | H29B-C31-H31B | 147.5 |
| H27A-C27-H27B | 108.6 | C29-C31-H31C | 109.5 |
| C27-C28-H28A | 109.5 | H29B-C31-H31C | 94.2 |
| C27-C28-H28B | 109.5 | C29-C30'-H30D | 109.5 |
| H28A-C28-H28B | 109.5 | C29-C30'-H30E | 109.5 |
| C27-C28-H28C | 109.5 | H30D-C30'-H30E | 109.5 |
| H28A-C28-H28C | 109.5 | C29-C30'-H30F | 109.5 |
| H28B-C28-H28C | 109.5 | H30D-C30'-H30F | 109.5 |
| C30'-C29-C31 | 124.7(6) | H30E-C30'-H30F | 109.5 |
| C30'-C29-C24 | 119.1(6) | C29-C31'-H31D | 109.5 |
| C31-C29-C24 | 113.7(4) | C29-C31'-H31E | 109.5 |
| C30'-C29-C31' | 116.4(6) | H31D-C31'-H31E | 109.5 |
| C31-C29-C31' | 24.9(4) | C29-C31'-H31F | 109.5 |
| C24-C29-C31' | 108.9(5) | H31D-C31'-H31F | 109.5 |
| C30'-C29-C30 | 36.4(5) | H31E-C31'-H31F | 109.5 |
| C31-C29-C30 | 108.9(4) | O5-C32-O5' | 39.8(4) |
| C24-C29-C30 | 111.3(4) | O5-C32-N4 | 123.4(3) |
| C31'-C29-C30 | 89.6(5) | O5'-C32-N4 | 118.7(5) |
| C30'-C29-H29A | 71.4 | O5-C32-C33 | 119.6(3) |
| C31-C29-H29A | 107.6 | O5'-C32-C33 | 114.7(5) |
| C24-C29-H29A | 107.6 | N4-C32-C33 | 116.3(3) |
| C31'-C29-H29A | 130.0 | N5-C33-C34 | 123.7(3) |
| C30-C29-H29A | 107.6 | N5-C33-C32 | 117.9(3) |
| C30'-C29-H29B | 104.3 | C34-C33-C32 | 118.4(3) |
| C31-C29-H29B | 76.7 | C35-C34-C33 | 118.7(3) |
| C24-C29-H29B | 104.1 | C35-C34-H34A | 120.7 |
| C31'-C29-H29B | 101.6 | C33-C34-H34A | 120.7 |
| C30-C29-H29B | 136.9 | C34-C35-C36 | 118.6(3) |
| H29A-C29-H29B | 36.2 | C34-C35-H35A | 120.7 |
| C29-C30-H30A | 109.5 | C36-C35-H35A | 120.7 |
| C29-C30-H30B | 109.5 | C37-C36-C35 | 118.9(3) |
| C29-C30-H30C | 109.5 | C37-C36-H36A | 120.5 |
| C29-C31-H29B | 39.5 | C35-C36-H36A | 120.5 |
| C29-C31-H31A | 109.5 | N5-C37-C36 | 123.3(3) |
| H29B-C31-H31A | 81.7 | N5-C37-C38 | 117.4(3) |
| C29-C31-H31B | 109.5 | C36-C37-C38 | 119.3(2) |

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| O6-C38-N6 | 124.6(3) | C48-C47-H47A | 107.7 |
| O6-C38-C37 | 120.5(3) | C49-C47-H47A | 107.7 |
| N6-C38-C37 | 114.9(2) | C43-C47-H47A | 107.7 |
| C40-C39-C44 | 119.9(3) | C47-C48-H48A | 109.5 |
| C40-C39-N6 | 116.6(2) | C47-C48-H48B | 109.5 |
| C44-C39-N6 | 123.4(3) | H48A-C48-H48B | 109.5 |
| C39-C40-C41 | 121.9(2) | C47-C48-H48C | 109.5 |
| C39-C40-H40A | 119.0 | H48A-C48-H48C | 109.5 |
| C41-C40-H40A | 119.0 | H48B-C48-H48C | 109.5 |
| C40-C41-C42 | 118.4(3) | C47-C49-H49A | 109.5 |
| C40-C41-C14 | 120.1(2) | C47-C49-H49B | 109.5 |
| C42-C41-C14 | 121.5(3) | H49A-C49-H49B | 109.5 |
| C43-C42-O7 | 118.5(2) | C47-C49-H49C | 109.5 |
| C43-C42-C41 | 120.7(3) | H49A-C49-H49C | 109.5 |
| O7-C42-C41 | 120.4(3) | H49B-C49-H49C | 109.5 |
| C42-C43-C44 | 119.8(2) | C55-C50-C51 | 119.2(2) |
| C42-C43-C47 | 121.0(3) | C55-C50-N4 | 122.4(2) |
| C44-C43-C47 | 119.3(3) | C51-C50-N4 | 118.3(2) |
| C39-C44-C43 | 119.3(3) | C52-C51-C50 | 121.3(2) |
| C39-C44-H44A | 120.4 | C52-C51-H51A | 119.3 |
| C43-C44-H44A | 120.4 | C50-C51-H51A | 119.3 |
| O7-C45-C46 | 112.6(4) | C51-C52-C53 | 118.0(2) |
| O7-C45-H45A | 109.1 | C51-C52-C26 | 122.1(2) |
| C46-C45-H45A | 109.1 | C53-C52-C26 | 119.7(2) |
| O7-C45-H45B | 109.1 | C54-C53-O8 | 120.6(2) |
| C46-C45-H45B | 109.1 | C54-C53-C52 | 121.4(2) |
| H45A-C45-H45B | 107.8 | O8-C53-C52 | 117.8(2) |
| C45-C46-H46A | 109.5 | C55-C54-C53 | 119.3(2) |
| C45-C46-H46B | 109.5 | C55-C54-C58 | 119.0(3) |
| H46A-C46-H46B | 109.5 | C53-C54-C58 | 121.6(3) |
| C45-C46-H46C | 109.5 | C54-C55-C50 | 120.8(3) |
| H46A-C46-H46C | 109.5 | C54-C55-H55A | 119.6 |
| H46B-C46-H46C | 109.5 | C50-C55-H55A | 119.6 |
| C48-C47-C49 | 112.0(4) | O8-C56-C57 | 107.0(4) |
| C48-C47-C43 | 110.5(3) | O8-C56-H56A | 110.3 |
| C49-C47-C43 | 111.2(3) | C57-C56-H56A | 110.3 |

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| O8-C56-H56B | 110.3 | C63-C64-C65 | 118.7(3) |
| C57-C56-H56B | 110.3 | C63-C64-H64A | 120.6 |
| H56A-C56-H56B | 108.6 | C65-C64-H64A | 120.6 |
| C56-C57-H57A | 109.5 | C64-C65-C66 | 118.6(3) |
| C56-C57-H57B | 109.5 | C64-C65-H65A | 120.7 |
| H57A-C57-H57B | 109.5 | C66-C65-H65A | 120.7 |
| C56-C57-H57C | 109.5 | N8-C66-C65 | 123.5(3) |
| H57A-C57-H57C | 109.5 | N8-C66-C67 | 118.1(2) |
| H57B-C57-H57C | 109.5 | C65-C66-C67 | 118.4(3) |
| C60-C58-C54 | 111.3(3) | O10-C67-N9 | 124.6(3) |
| C60-C58-C59 | 111.9(3) | O10-C67-C66 | 120.4(3) |
| C54-C58-C59 | 110.1(3) | N9-C67-C66 | 114.9(2) |
| C60-C58-H58A | 107.8 | C73-C68-C69 | 120.6(2) |
| C54-C58-H58A | 107.8 | C73-C68-N9 | 122.1(2) |
| C59-C58-H58A | 107.8 | C69-C68-N9 | 117.3(2) |
| C58-C59-H59A | 109.5 | C68-C69-C70 | 120.6(2) |
| C58-C59-H59B | 109.5 | C68-C69-H69A | 119.7 |
| H59A-C59-H59B | 109.5 | C70-C69-H69A | 119.7 |
| C58-C59-H59C | 109.5 | C69-C70-C71 | 118.0(2) |
| H59A-C59-H59C | 109.5 | C69-C70-C14 | 122.2(2) |
| H59B-C59-H59C | 109.5 | C71-C70-C14 | 119.8(2) |
| C58-C60-H60A | 109.5 | C72-C71-C70 | 122.5(2) |
| C58-C60-H60B | 109.5 | C72-C71-O11 | 120.2(2) |
| H60A-C60-H60B | 109.5 | C70-C71-O11 | 117.2(2) |
| C58-C60-H60C | 109.5 | C71-C72-C73 | 117.6(2) |
| H60A-C60-H60C | 109.5 | C71-C72-C76 | 122.2(2) |
| H60B-C60-H60C | 109.5 | C73-C72-C76 | 120.1(2) |
| O9-C61-N7 | 123.6(3) | C68-C73-C72 | 120.5(2) |
| O9-C61-C62 | 119.9(3) | C68-C73-H73A | 119.7 |
| N7-C61-C62 | 116.4(3) | C72-C73-H73A | 119.7 |
| N8-C62-C63 | 122.9(3) | O11-C74-C75 | 107.2(3) |
| N8-C62-C61 | 118.0(3) | O11-C74-H74A | 110.3 |
| C63-C62-C61 | 119.1(3) | C75-C74-H74A | 110.3 |
| C64-C63-C62 | 119.3(3) | O11-C74-H74B | 110.3 |
| C64-C63-H63A | 120.3 | C75-C74-H74B | 110.3 |
| C62-C63-H63A | 120.3 | H74A-C74-H74B | 108.5 |

| | | | |
|---|---|---|---|
| C74-C75-H75A | 109.5 | C84-C83-C82 | 119.3(3) |
| C74-C75-H75B | 109.5 | C84-C83-C87 | 118.9(3) |
| H75A-C75-H75B | 109.5 | C82-C83-C87 | 121.8(3) |
| C74-C75-H75C | 109.5 | C83-C84-C79 | 120.8(3) |
| H75A-C75-H75C | 109.5 | C83-C84-H84A | 119.6 |
| H75B-C75-H75C | 109.5 | C79-C84-H84A | 119.6 |
| C78-C76-C72 | 110.8(3) | O12-C85-C86 | 106.1(3) |
| C78-C76-C77 | 110.2(3) | O12-C85-H85A | 110.5 |
| C72-C76-C77 | 112.2(3) | C86-C85-H85A | 110.5 |
| C78-C76-H76A | 107.8 | O12-C85-H85B | 110.5 |
| C72-C76-H76A | 107.8 | C86-C85-H85B | 110.5 |
| C77-C76-H76A | 107.8 | H85A-C85-H85B | 108.7 |
| C76-C77-H77A | 109.5 | C85-C86-H86A | 109.5 |
| C76-C77-H77B | 109.5 | C85-C86-H86B | 109.5 |
| H77A-C77-H77B | 109.5 | H86A-C86-H86B | 109.5 |
| C76-C77-H77C | 109.5 | C85-C86-H86C | 109.5 |
| H77A-C77-H77C | 109.5 | H86A-C86-H86C | 109.5 |
| H77B-C77-H77C | 109.5 | H86B-C86-H86C | 109.5 |
| C76-C78-H78A | 109.5 | C83-C87-C89 | 110.0(2) |
| C76-C78-H78B | 109.5 | C83-C87-C88 | 111.3(2) |
| H78A-C78-H78B | 109.5 | C89-C87-C88 | 110.5(3) |
| C76-C78-H78C | 109.5 | C83-C87-H87A | 108.3 |
| H78A-C78-H78C | 109.5 | C89-C87-H87A | 108.3 |
| H78B-C78-H78C | 109.5 | C88-C87-H87A | 108.3 |
| C84-C79-C80 | 119.1(3) | C87-C88-H88A | 109.5 |
| C84-C79-N7 | 122.3(3) | C87-C88-H88B | 109.5 |
| C80-C79-N7 | 118.6(2) | H88A-C88-H88B | 109.5 |
| C81-C80-C79 | 121.5(3) | C87-C88-H88C | 109.5 |
| C81-C80-H80A | 119.3 | H88A-C88-H88C | 109.5 |
| C79-C80-H80A | 119.3 | H88B-C88-H88C | 109.5 |
| C80-C81-C82 | 118.4(3) | C87-C89-H89A | 109.5 |
| C80-C81-C26 | 122.7(2) | C87-C89-H89B | 109.5 |
| C82-C81-C26 | 118.7(3) | H89A-C89-H89B | 109.5 |
| O12-C82-C83 | 120.7(2) | C87-C89-H89C | 109.5 |
| O12-C82-C81 | 118.0(3) | H89A-C89-H89C | 109.5 |
| C83-C82-C81 | 121.0(3) | H89B-C89-H89C | 109.5 |

| | | | |
|---|---|---|---|
| C93-O13-C90 | 107.9(2) | O14-C94-H94C | 110.4 |
| O13-C90-C91 | 105.2(3) | C95-C94-H94C | 127.8 |
| O13-C90-H90A | 110.7 | C95'-C94-H94C | 106.2 |
| C91-C90-H90A | 110.7 | H94A-C94-H94C | 90.6 |
| O13-C90-H90B | 110.7 | H94B-C94-H94C | 20.4 |
| C91-C90-H90B | 110.7 | O14-C94-H94D | 111.3 |
| H90A-C90-H90B | 108.8 | C95-C94-H94D | 90.8 |
| C92-C91-C90 | 104.8(3) | C95'-C94-H94D | 115.5 |
| C92-C91-H91A | 110.8 | H94A-C94-H94D | 21.1 |
| C90-C91-H91A | 110.8 | H94B-C94-H94D | 124.7 |
| C92-C91-H91B | 110.8 | H94C-C94-H94D | 109.0 |
| C90-C91-H91B | 110.8 | C94-C95-C96 | 94.6(7) |
| H91A-C91-H91B | 108.9 | C94-C95-H95A | 112.8 |
| C91-C92-C93 | 101.2(3) | C96-C95-H95A | 112.8 |
| C91-C92-H92A | 111.5 | C94-C95-H95B | 112.8 |
| C93-C92-H92A | 111.5 | C96-C95-H95B | 112.8 |
| C91-C92-H92B | 111.5 | H95A-C95-H95B | 110.3 |
| C93-C92-H92B | 111.5 | C97-C96-C95 | 94.9(7) |
| H92A-C92-H92B | 109.4 | C97-C96-H96A | 112.8 |
| O13-C93-C92 | 105.3(3) | C95-C96-H96A | 112.8 |
| O13-C93-H93A | 110.7 | C97-C96-H96B | 112.8 |
| C92-C93-H93A | 110.7 | C95-C96-H96B | 112.8 |
| O13-C93-H93B | 110.7 | H96A-C96-H96B | 110.2 |
| C92-C93-H93B | 110.7 | C96'-C95'-C94 | 106.3(12) |
| H93A-C93-H93B | 108.8 | C96'-C95'-H95C | 110.5 |
| C94-O14-C97 | 108.2(3) | C94-C95'-H95C | 110.5 |
| O14-C94-C95 | 105.7(5) | C96'-C95'-H95D | 110.5 |
| O14-C94-C95' | 104.1(6) | C94-C95'-H95D | 110.5 |
| C95-C94-C95' | 27.1(6) | H95C-C95'-H95D | 108.7 |
| O14-C94-H94A | 110.6 | C95'-C96'-C97 | 98.8(10) |
| C95-C94-H94A | 110.6 | C95'-C96'-H96C | 112.0 |
| C95'-C94-H94A | 133.0 | C97-C96'-H96C | 112.0 |
| O14-C94-H94B | 110.6 | C95'-C96'-H96D | 112.0 |
| C95-C94-H94B | 110.6 | C97-C96'-H96D | 112.0 |
| C95'-C94-H94B | 86.7 | H96C-C96'-H96D | 109.7 |
| H94A-C94-H94B | 108.7 | O14-C97-C96 | 108.2(4) |

| | | | |
|---|---|---|---|
| O14-C97-C96' | 105.7(5) | C99-C100-H10B | 111.6 |
| C96-C97-C96' | 30.0(4) | C101-C100-H10B | 111.6 |
| O14-C97-H97A | 110.1 | H10A-C100-H10B | 109.4 |
| C96-C97-H97A | 110.1 | O15-C101-C100 | 101.2(7) |
| C96'-C97-H97A | 134.6 | O15-C101-H10C | 111.5 |
| O14-C97-H97B | 110.1 | C100-C101-H10C | 111.5 |
| C96-C97-H97B | 110.1 | O15-C101-H10D | 111.5 |
| C96'-C97-H97B | 83.8 | C100-C101-H10D | 111.5 |
| H97A-C97-H97B | 108.4 | H10C-C101-H10D | 109.3 |
| O14-C97-H97C | 110.5 | C102-O15'-C105 | 98.9(11) |
| C96-C97-H97C | 132.4 | C103-C102-O15' | 116.1(13) |
| C96'-C97-H97C | 111.3 | C103-C102-H10E | 108.3 |
| H97A-C97-H97C | 81.2 | O15'-C102-H10E | 108.3 |
| H97B-C97-H97C | 29.5 | C103-C102-H10F | 108.3 |
| O14-C97-H97D | 111.1 | O15'-C102-H10F | 108.3 |
| C96-C97-H97D | 81.0 | H10E-C102-H10F | 107.4 |
| C96'-C97-H97D | 109.3 | C102-C103-C104 | 105.3(12) |
| H97A-C97-H97D | 31.0 | C102-C103-H10G | 110.7 |
| H97B-C97-H97D | 130.9 | C104-C103-H10G | 110.7 |
| H97C-C97-H97D | 108.9 | C102-C103-H10H | 110.7 |
| C98-O15-C101 | 105.3(6) | C104-C103-H10H | 110.7 |
| O15-C98-C99 | 110.1(6) | H10G-C103-H10H | 108.8 |
| O15-C98-H98A | 109.6 | C103-C104-C105 | 104.4(12) |
| C99-C98-H98A | 109.6 | C103-C104-H10I | 110.9 |
| O15-C98-H98B | 109.6 | C105-C104-H10I | 110.9 |
| C99-C98-H98B | 109.6 | C103-C104-H10J | 110.9 |
| H98A-C98-H98B | 108.2 | C105-C104-H10J | 110.9 |
| C98-C99-C100 | 106.0(7) | H10I-C104-H10J | 108.9 |
| C98-C99-H99A | 110.5 | C104-C105-O15' | 107.7(12) |
| C100-C99-H99A | 110.5 | C104-C105-H10K | 110.2 |
| C98-C99-H99B | 110.5 | O15'-C105-H10K | 110.2 |
| C100-C99-H99B | 110.5 | C104-C105-H10L | 110.2 |
| H99A-C99-H99B | 108.7 | O15'-C105-H10L | 110.2 |
| C99-C100-C101 | 100.7(6) | H10K-C105-H10L | 108.5 |
| C99-C100-H10A | 111.6 | H16'-O16-H16" | 108.3 |
| C101-C100-H10A | 111.6 | H17'-O17-H17" | 106.1 |

**Table S3.** Crystal data and structure refinement for $[K(MeCN)_2]_3[(Fe^{III}Cl_2)_3\mathbf{L}]$.

| | | |
|---|---|---|
| Identification code | gui6 | |
| Empirical formula | C101.50 H107 Cl6 Fe3 K3 N15.50 O12 | |
| Formula weight | 2233.58 | |
| Temperature | 100(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Cubic | |
| Space group | $P2_13$ | |
| Unit cell dimensions | a = 22.8525(16) Å | α= 90°. |
| | b = 22.8525(16) Å | β= 90°. |
| | c = 22.8525(16) Å | γ = 90°. |
| Volume | 11934.4(14) $Å^3$ | |
| Z | 4 | |
| Density (calculated) | 1.243 $Mg/m^3$ | |
| Absorption coefficient | 0.657 $mm^{-1}$ | |
| F(000) | 4630 | |
| Crystal size | 0.20 x 0.20 x 0.17 $mm^3$ | |
| Theta range for data collection | 1.26 to 23.38°. | |
| Index ranges | $-17\leq h\leq 17$, $0\leq k\leq 18$, $1\leq l\leq 25$ | |
| Reflections collected | 5832 | |
| Independent reflections | 5832 [R(int) = 0.0000] | |
| Completeness to theta = 23.38° | 100.0 % | |
| Absorption correction | Numerical | |
| Max. and min. transmission | 0.8964 and 0.8786 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 5832 / 9 / 408 | |
| Goodness-of-fit on $F^2$ | 1.078 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0597, wR2 = 0.1588 [4639] | |
| R indices (all data) | R1 = 0.0794, wR2 = 0.1684 | |
| Absolute structure parameter | -0.02(3) | |
| Largest diff. peak and hole | 0.522 and -0.395 e.$Å^{-3}$ | |

$R1 = \sum(||F_o| - |F_c||) / \sum|F_o|$

$wR2 = [\sum[w(F_o^2 - F_c^2)^2] / \sum[w(F_o^2)^2]]^{1/2}$

$S = [\sum[w(F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$

$w= 1/[\sigma^2(F_o^2)+(m*p)^2+n*p]$, $p = [max(F_o^2,0)+ 2* F_c^2]/3$, m & n are constants.

**Table S4.** Bond lengths [Å] and angles [°] for $[K(MeCN)_2]_3[(Fe^{III}Cl_2)_3\mathbf{L}]$.

| | | | |
|---|---|---|---|
| Fe1-N3 | 2.063(5) | C3-H3A | 0.9500 |
| Fe1-N2 | 2.098(5) | C4-C5 | 1.326(12) |
| Fe1-N1 | 2.104(5) | C4-H4A | 0.9500 |
| Fe1-Cl2 | 2.1968(17) | C5-C6 | 1.409(10) |
| Fe1-Cl1 | 2.2198(19) | C5-H5A | 0.9500 |
| K1-O1#1 | 2.595(7) | C6-C7 | 1.485(10) |
| K1-O4 | 2.722(5) | C8-C13 | 1.390(9) |
| K1-N5 | 2.775(9) | C8-C9 | 1.407(9) |
| K1-N7 | 2.778(14) | C9-C10 | 1.412(8) |
| K1-N6 | 2.79(2) | C9-H9A | 0.9500 |
| K1-N4 | 3.02(2) | C10-C11 | 1.388(9) |
| K1-C21#1 | 3.209(6) | C10-C14 | 1.506(6) |
| K1-C20#1 | 3.287(6) | C11-C12 | 1.412(10) |
| K1-C22#1 | 3.295(7) | C12-C13 | 1.368(10) |
| K1-C23#1 | 3.469(7) | C12-C17 | 1.544(10) |
| K1-C25#1 | 3.491(7) | C13-H13A | 0.9500 |
| O1-C1 | 1.248(8) | C14-C10#2 | 1.506(6) |
| O1-K1#2 | 2.595(7) | C14-C10#1 | 1.506(6) |
| O2-C7 | 1.239(8) | C14-H14A | 0.9599 |
| O3-C15' | 1.25(2) | C15-C16 | 1.53(2) |
| O3-C15 | 1.311(16) | C15-H15A | 0.9900 |
| O3-C11 | 1.381(8) | C15-H15B | 0.9900 |
| O4-C27 | 1.37(2) | C16-H16A | 0.9800 |
| O4-C23 | 1.417(9) | C16-H16B | 0.9800 |
| O4-C27' | 1.47(3) | C16-H16C | 0.9800 |
| N1-C1 | 1.325(9) | C15'-C16' | 1.51(3) |
| N1-C20 | 1.411(9) | C15'-H15C | 0.9900 |
| N2-C6 | 1.319(8) | C15'-H15D | 0.9900 |
| N2-C2 | 1.325(8) | C16'-H16D | 0.9800 |
| N3-C7 | 1.342(8) | C16'-H16E | 0.9800 |
| N3-C8 | 1.405(8) | C16'-H16F | 0.9800 |
| C1-C2 | 1.449(10) | C17-C18 | 1.400(19) |
| C2-C3 | 1.377(10) | C17-C19 | 1.46(2) |
| C3-C4 | 1.359(12) | C17-C18' | 1.54(3) |

| | | | |
|---|---|---|---|
| C17-C19' | 1.79(3) | C28-H28C | 0.9800 |
| C18-H18A | 0.9800 | C27'-C28' | 1.51(4) |
| C18-H18B | 0.9800 | C27'-H27C | 0.9900 |
| C18-H18C | 0.9800 | C27'-H27D | 0.9900 |
| C19-H19A | 0.9800 | C28'-H28D | 0.9800 |
| C19-H19B | 0.9800 | C28'-H28E | 0.9800 |
| C19-H19C | 0.9800 | C28'-H28F | 0.9800 |
| C18'-H18D | 0.9800 | C29-C31' | 1.42(3) |
| C18'-H18E | 0.9800 | C29-C30 | 1.456(18) |
| C18'-H18F | 0.9800 | C29-C31 | 1.635(18) |
| C19'-H19D | 0.9800 | C29-C30' | 1.66(2) |
| C19'-H19E | 0.9800 | C30-H30A | 0.9800 |
| C19'-H19F | 0.9800 | C30-H30B | 0.9800 |
| C20-C21 | 1.378(9) | C30-H30C | 0.9800 |
| C20-C25 | 1.391(9) | C31-H31A | 0.9800 |
| C20-K1#2 | 3.287(6) | C31-H31B | 0.9800 |
| C21-C22 | 1.376(9) | C31-H31C | 0.9800 |
| C21-K1#2 | 3.209(6) | C30'-H30D | 0.9800 |
| C21-H21A | 0.9500 | C30'-H30E | 0.9800 |
| C22-C23 | 1.421(10) | C30'-H30F | 0.9800 |
| C22-C26 | 1.523(7) | C31'-H31D | 0.9800 |
| C22-K1#2 | 3.295(7) | C31'-H31E | 0.9800 |
| C23-C24 | 1.372(11) | C31'-H31F | 0.9800 |
| C23-K1#2 | 3.469(7) | N4-C32 | 1.19(3) |
| C24-C25 | 1.404(11) | C32-C39#3 | 0.87(3) |
| C24-C29 | 1.541(11) | C32-C33 | 1.28(4) |
| C25-K1#2 | 3.491(7) | C32-C38#3 | 1.56(3) |
| C25-H25A | 0.9500 | C33-C39#3 | 0.57(3) |
| C26-C22#2 | 1.523(7) | C33-C38#3 | 1.13(3) |
| C26-C22#1 | 1.523(7) | C33-H33A | 0.9800 |
| C26-H26A | 0.9599 | C33-H33B | 0.9800 |
| C27-C28 | 1.50(2) | C33-H33C | 0.9800 |
| C27-H27A | 0.9900 | N5-C34 | 1.137(9) |
| C27-H27B | 0.9900 | C34-C35 | 1.396(11) |
| C28-H28A | 0.9800 | C35-H35A | 0.9602 |
| C28-H28B | 0.9800 | C35-H35B | 0.9600 |

| | | | |
|---|---|---|---|
| C35-H35C | 0.9600 | O4-K1-N6 | 92.5(6) |
| N6-N7 | 1.10(3) | N5-K1-N6 | 97.7(6) |
| N6-C36 | 1.160(17) | N7-K1-N6 | 22.8(6) |
| C36-C37 | 1.394(17) | O1#1-K1-N4 | 55.3(4) |
| C37-C36#3 | 1.394(17) | O4-K1-N4 | 78.1(4) |
| C37-C36#4 | 1.394(17) | N5-K1-N4 | 163.1(4) |
| C37-H37A | 0.9838 | N7-K1-N4 | 88.0(5) |
| C37-H37B | 0.9581 | N6-K1-N4 | 65.5(7) |
| C37-H37C | 0.9149 | O1#1-K1-C21#1 | 71.02(17) |
| N7-C38 | 1.112(13) | O4-K1-C21#1 | 76.63(15) |
| C38-C33#4 | 1.13(3) | N5-K1-C21#1 | 132.2(2) |
| C38-C39 | 1.427(15) | N7-K1-C21#1 | 152.0(3) |
| C38-C32#4 | 1.56(3) | N6-K1-C21#1 | 130.0(6) |
| C39-C33#4 | 0.57(3) | N4-K1-C21#1 | 64.5(4) |
| C39-C32#4 | 0.87(3) | O1#1-K1-C20#1 | 52.63(17) |
| C39-H39A | 0.9601 | O4-K1-C20#1 | 101.08(16) |
| C39-H39B | 0.9598 | N5-K1-C20#1 | 126.2(2) |
| C39-H39C | 0.9600 | N7-K1-C20#1 | 149.8(4) |
| | | N6-K1-C20#1 | 129.1(6) |
| N3-Fe1-N2 | 74.9(2) | N4-K1-C20#1 | 69.8(4) |
| N3-Fe1-N1 | 145.1(2) | C21#1-K1-C20#1 | 24.45(16) |
| N2-Fe1-N1 | 73.6(2) | O1#1-K1-C22#1 | 94.29(17) |
| N3-Fe1-Cl2 | 98.91(15) | O4-K1-C22#1 | 63.91(16) |
| N2-Fe1-Cl2 | 142.00(15) | N5-K1-C22#1 | 111.9(2) |
| N1-Fe1-Cl2 | 96.80(15) | N7-K1-C22#1 | 157.2(3) |
| N3-Fe1-Cl1 | 103.41(15) | N6-K1-C22#1 | 144.8(6) |
| N2-Fe1-Cl1 | 110.42(15) | N4-K1-C22#1 | 83.4(4) |
| N1-Fe1-Cl1 | 101.22(16) | C21#1-K1-C22#1 | 24.39(15) |
| Cl2-Fe1-Cl1 | 107.49(7) | C20#1-K1-C22#1 | 42.91(17) |
| O1#1-K1-O4 | 131.2(2) | O1#1-K1-C23#1 | 99.29(19) |
| O1#1-K1-N5 | 127.2(3) | O4-K1-C23#1 | 78.18(18) |
| O4-K1-N5 | 101.6(2) | N5-K1-C23#1 | 90.7(2) |
| O1#1-K1-N7 | 98.1(3) | N7-K1-C23#1 | 162.0(3) |
| O4-K1-N7 | 93.7(3) | N6-K1-C23#1 | 168.6(6) |
| N5-K1-N7 | 75.1(4) | N4-K1-C23#1 | 105.7(4) |
| O1#1-K1-N6 | 81.8(5) | C21#1-K1-C23#1 | 41.73(16) |

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|---|---|---|---|
| C20#1-K1-C23#1 | 48.15(17) | C3-C2-C1 | 127.3(6) |
| C22#1-K1-C23#1 | 24.08(17) | C4-C3-C2 | 118.8(7) |
| O1#1-K1-C25#1 | 61.60(19) | C4-C3-H3A | 120.6 |
| O4-K1-C25#1 | 112.21(17) | C2-C3-H3A | 120.6 |
| N5-K1-C25#1 | 102.9(2) | C5-C4-C3 | 121.2(7) |
| N7-K1-C25#1 | 153.7(3) | C5-C4-H4A | 119.4 |
| N6-K1-C25#1 | 143.4(5) | C3-C4-H4A | 119.4 |
| N4-K1-C25#1 | 92.6(4) | C4-C5-C6 | 118.8(8) |
| C21#1-K1-C25#1 | 41.47(16) | C4-C5-H5A | 120.6 |
| C20#1-K1-C25#1 | 23.44(16) | C6-C5-H5A | 120.6 |
| C22#1-K1-C25#1 | 48.30(17) | N2-C6-C5 | 119.5(7) |
| C23#1-K1-C25#1 | 39.80(19) | N2-C6-C7 | 113.5(5) |
| C1-O1-K1#2 | 133.4(5) | C5-C6-C7 | 127.0(7) |
| C15'-O3-C15 | 114.1(14) | O2-C7-N3 | 126.4(6) |
| C15'-O3-C11 | 126.5(13) | O2-C7-C6 | 120.9(6) |
| C15-O3-C11 | 117.7(9) | N3-C7-C6 | 112.7(6) |
| C27-O4-C23 | 110.9(10) | C13-C8-N3 | 120.7(6) |
| C27-O4-C27' | 18.2(14) | C13-C8-C9 | 118.2(6) |
| C23-O4-C27' | 114.8(12) | N3-C8-C9 | 121.0(5) |
| C27-O4-K1 | 129.1(9) | C8-C9-C10 | 120.8(6) |
| C23-O4-K1 | 118.7(4) | C8-C9-H9A | 119.6 |
| C27'-O4-K1 | 126.0(12) | C10-C9-H9A | 119.6 |
| C1-N1-C20 | 116.1(5) | C11-C10-C9 | 118.2(6) |
| C1-N1-Fe1 | 118.2(5) | C11-C10-C14 | 120.9(6) |
| C20-N1-Fe1 | 125.6(4) | C9-C10-C14 | 120.8(6) |
| C6-N2-C2 | 121.6(6) | O3-C11-C10 | 118.6(6) |
| C6-N2-Fe1 | 118.5(4) | O3-C11-C12 | 119.6(6) |
| C2-N2-Fe1 | 119.8(5) | C10-C11-C12 | 121.7(6) |
| C7-N3-C8 | 115.4(5) | C13-C12-C11 | 118.1(6) |
| C7-N3-Fe1 | 119.3(4) | C13-C12-C17 | 121.5(7) |
| C8-N3-Fe1 | 124.7(4) | C11-C12-C17 | 120.4(6) |
| O1-C1-N1 | 125.4(7) | C12-C13-C8 | 122.9(6) |
| O1-C1-C2 | 119.5(6) | C12-C13-H13A | 118.6 |
| N1-C1-C2 | 115.0(6) | C8-C13-H13A | 118.6 |
| N2-C2-C3 | 120.1(7) | C10#2-C14-C10 | 113.6(3) |
| N2-C2-C1 | 112.5(6) | C10#2-C14-C10#1 | 113.6(3) |

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|---|---|---|---|
| C10-C14-C10#1 | 113.6(3) | C18'-C17-C19' | 113.5(15) |
| C10#2-C14-H14A | 104.9 | C12-C17-C19' | 103.9(11) |
| C10-C14-H14A | 104.9 | C17-C18-H18A | 109.5 |
| C10#1-C14-H14A | 104.9 | C17-C18-H18B | 109.5 |
| O3-C15-C16 | 107.4(13) | H18A-C18-H18B | 109.5 |
| O3-C15-H15A | 110.2 | C17-C18-H18C | 109.5 |
| C16-C15-H15A | 110.2 | H18A-C18-H18C | 109.5 |
| O3-C15-H15B | 110.2 | H18B-C18-H18C | 109.5 |
| C16-C15-H15B | 110.2 | C17-C19-H19A | 109.5 |
| H15A-C15-H15B | 108.5 | C17-C19-H19B | 109.5 |
| C15-C16-H16A | 109.5 | H19A-C19-H19B | 109.5 |
| C15-C16-H16B | 109.5 | C17-C19-H19C | 109.5 |
| H16A-C16-H16B | 109.5 | H19A-C19-H19C | 109.5 |
| C15-C16-H16C | 109.5 | H19B-C19-H19C | 109.5 |
| H16A-C16-H16C | 109.5 | C17-C18'-H18D | 109.5 |
| H16B-C16-H16C | 109.5 | C17-C18'-H18E | 109.5 |
| O3-C15'-C16' | 111.7(19) | H18D-C18'-H18E | 109.5 |
| O3-C15'-H15C | 109.3 | C17-C18'-H18F | 109.5 |
| C16'-C15'-H15C | 109.3 | H18D-C18'-H18F | 109.5 |
| O3-C15'-H15D | 109.3 | H18E-C18'-H18F | 109.5 |
| C16'-C15'-H15D | 109.3 | C17-C19'-H19D | 109.5 |
| H15C-C15'-H15D | 107.9 | C17-C19'-H19E | 109.5 |
| C15'-C16'-H16D | 109.5 | H19D-C19'-H19E | 109.5 |
| C15'-C16'-H16E | 109.5 | C17-C19'-H19F | 109.5 |
| H16D-C16'-H16E | 109.5 | H19D-C19'-H19F | 109.5 |
| C15'-C16'-H16F | 109.5 | H19E-C19'-H19F | 109.5 |
| H16D-C16'-H16F | 109.5 | C21-C20-C25 | 119.0(7) |
| H16E-C16'-H16F | 109.5 | C21-C20-N1 | 120.3(6) |
| C18-C17-C19 | 97.8(12) | C25-C20-N1 | 120.6(6) |
| C18-C17-C18' | 30.8(11) | C21-C20-K1#2 | 74.6(4) |
| C19-C17-C18' | 127.1(14) | C25-C20-K1#2 | 86.5(4) |
| C18-C17-C12 | 116.6(10) | N1-C20-K1#2 | 106.9(4) |
| C19-C17-C12 | 113.1(10) | C22-C21-C20 | 121.9(6) |
| C18'-C17-C12 | 105.9(12) | C22-C21-K1#2 | 81.3(4) |
| C18-C17-C19' | 82.7(13) | C20-C21-K1#2 | 81.0(4) |
| C19-C17-C19' | 24.2(11) | C22-C21-H21A | 119.1 |

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|---|---|---|---|
| C20-C21-H21A | 119.1 | C27-C28-H28B | 109.5 |
| K1#2-C21-H21A | 108.5 | H28A-C28-H28B | 109.5 |
| C21-C22-C23 | 117.4(7) | C27-C28-H28C | 109.5 |
| C21-C22-C26 | 122.1(6) | H28A-C28-H28C | 109.5 |
| C23-C22-C26 | 120.5(6) | H28B-C28-H28C | 109.5 |
| C21-C22-K1#2 | 74.3(4) | O4-C27'-C28' | 114(2) |
| C23-C22-K1#2 | 84.8(4) | O4-C27'-H27C | 108.7 |
| C26-C22-K1#2 | 110.6(4) | C28'-C27'-H27C | 108.7 |
| C24-C23-O4 | 120.8(7) | O4-C27'-H27D | 108.7 |
| C24-C23-C22 | 122.7(7) | C28'-C27'-H27D | 108.7 |
| O4-C23-C22 | 116.4(7) | H27C-C27'-H27D | 107.6 |
| C24-C23-K1#2 | 84.3(5) | C27'-C28'-H28D | 109.5 |
| O4-C23-K1#2 | 118.9(4) | C27'-C28'-H28E | 109.5 |
| C22-C23-K1#2 | 71.1(4) | H28D-C28'-H28E | 109.5 |
| C23-C24-C25 | 117.2(7) | C27'-C28'-H28F | 109.5 |
| C23-C24-C29 | 122.4(7) | H28D-C28'-H28F | 109.5 |
| C25-C24-C29 | 120.4(7) | H28E-C28'-H28F | 109.5 |
| C20-C25-C24 | 121.7(7) | C31'-C29-C30 | 122.9(14) |
| C20-C25-K1#2 | 70.0(4) | C31'-C29-C24 | 114.0(12) |
| C24-C25-K1#2 | 83.1(4) | C30-C29-C24 | 108.8(9) |
| C20-C25-H25A | 119.2 | C31'-C29-C31 | 20.6(11) |
| C24-C25-H25A | 119.2 | C30-C29-C31 | 108.9(10) |
| K1#2-C25-H25A | 118.3 | C24-C29-C31 | 110.3(8) |
| C22#2-C26-C22 | 112.7(4) | C31'-C29-C30' | 107.0(14) |
| C22#2-C26-C22#1 | 112.7(4) | C30-C29-C30' | 20.7(10) |
| C22-C26-C22#1 | 112.7(4) | C24-C29-C30' | 109(1) |
| C22#2-C26-H26A | 106.1 | C31-C29-C30' | 90.2(11) |
| C22-C26-H26A | 106.1 | C29-C30-H30A | 109.5 |
| C22#1-C26-H26A | 106.1 | C29-C30-H30B | 109.5 |
| O4-C27-C28 | 110.5(14) | H30A-C30-H30B | 109.5 |
| O4-C27-H27A | 109.5 | C29-C30-H30C | 109.5 |
| C28-C27-H27A | 109.5 | H30A-C30-H30C | 109.5 |
| O4-C27-H27B | 109.5 | H30B-C30-H30C | 109.5 |
| C28-C27-H27B | 109.5 | C29-C31-H31A | 109.5 |
| H27A-C27-H27B | 108.1 | C29-C31-H31B | 109.5 |
| C27-C28-H28A | 109.5 | H31A-C31-H31B | 109.5 |

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|---|---|---|---|
| C29-C31-H31C | 109.5 | H33B-C33-H33C | 109.5 |
| H31A-C31-H31C | 109.5 | C34-N5-K1 | 161.4(8) |
| H31B-C31-H31C | 109.5 | N5-C34-C35 | 173.9(10) |
| C29-C30'-H30D | 109.5 | C34-C35-H35A | 108.2 |
| C29-C30'-H30E | 109.5 | C34-C35-H35B | 109.8 |
| H30D-C30'-H30E | 109.5 | H35A-C35-H35B | 109.5 |
| C29-C30'-H30F | 109.5 | C34-C35-H35C | 110.4 |
| H30D-C30'-H30F | 109.5 | H35A-C35-H35C | 109.5 |
| H30E-C30'-H30F | 109.5 | H35B-C35-H35C | 109.5 |
| C29-C31'-H31D | 109.5 | N7-N6-C36 | 135(4) |
| C29-C31'-H31E | 109.5 | N7-N6-K1 | 78.2(14) |
| H31D-C31'-H31E | 109.5 | C36-N6-K1 | 135(3) |
| C29-C31'-H31F | 109.5 | N6-C36-C37 | 172(4) |
| H31D-C31'-H31F | 109.5 | C36#3-C37-C36 | 119.9(3) |
| H31E-C31'-H31F | 109.5 | C36#3-C37-C36#4 | 119.9(3) |
| C32-N4-K1 | 161.3(18) | C36-C37-C36#4 | 119.9(3) |
| C39#3-C32-N4 | 168(3) | C36#3-C37-H37A | 106.4 |
| C39#3-C32-C33 | 21(2) | C36-C37-H37A | 107.5 |
| N4-C32-C33 | 170(3) | C36#4-C37-H37A | 60.6 |
| C39#3-C32-C38#3 | 64.8(18) | C36#3-C37-H37B | 15.1 |
| N4-C32-C38#3 | 125(2) | C36-C37-H37B | 106.1 |
| C33-C32-C38#3 | 45.7(17) | C36#4-C37-H37B | 134.1 |
| C39#3-C33-C38#3 | 110(5) | H37A-C37-H37B | 107.7 |
| C39#3-C33-C32 | 33(3) | C36#3-C37-H37C | 101.2 |
| C38#3-C33-C32 | 80(2) | C36-C37-H37C | 110.4 |
| C39#3-C33-H33A | 109.5 | C36#4-C37-H37C | 51.0 |
| C38#3-C33-H33A | 140.8 | H37A-C37-H37C | 111.3 |
| C32-C33-H33A | 138.1 | H37B-C37-H37C | 113.6 |
| C39#3-C33-H33B | 109.5 | N6-N7-C38 | 135(2) |
| C38#3-C33-H33B | 60.0 | N6-N7-K1 | 78.9(16) |
| C32-C33-H33B | 81.5 | C38-N7-K1 | 143.6(13) |
| H33A-C33-H33B | 109.5 | N7-C38-C33#4 | 152(2) |
| C39#3-C33-H33C | 109.5 | N7-C38-C39 | 174.4(19) |
| C38#3-C33-H33C | 52.5 | C33#4-C38-C39 | 22.0(18) |
| C32-C33-H33C | 103.9 | N7-C38-C32#4 | 151.6(18) |
| H33A-C33-H33C | 109.5 | C33#4-C38-C32#4 | 54(2) |

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|---|---|---|---|
| C39-C38-C32#4 | 33.3(10) | C32#4-C39-H39B | 142.0 |
| C33#4-C39-C32#4 | 126(5) | C38-C39-H39B | 109.4 |
| C33#4-C39-C38 | 48(4) | H39A-C39-H39B | 109.5 |
| C32#4-C39-C38 | 82(2) | C33#4-C39-H39C | 80.6 |
| C33#4-C39-H39A | 158.6 | C32#4-C39-H39C | 100.0 |
| C32#4-C39-H39A | 35.7 | C38-C39-H39C | 108.7 |
| C38-C39-H39A | 110.2 | H39A-C39-H39C | 109.5 |
| C33#4-C39-H39B | 83.4 | H39B-C39-H39C | 109. |

Symmetry transformations used to generate equivalent atoms:

#1 z,x,y  #2 y,z,x  #3 y+1/2,-z+1/2,-x+1  #4 -z+1,x-1/2,-y+1/2

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