

Electronic Supplementary Information for

A Tris(2-quinolylmethyl)amine Scaffold that Promotes Hydrogen Bonding within the Secondary Coordination Sphere

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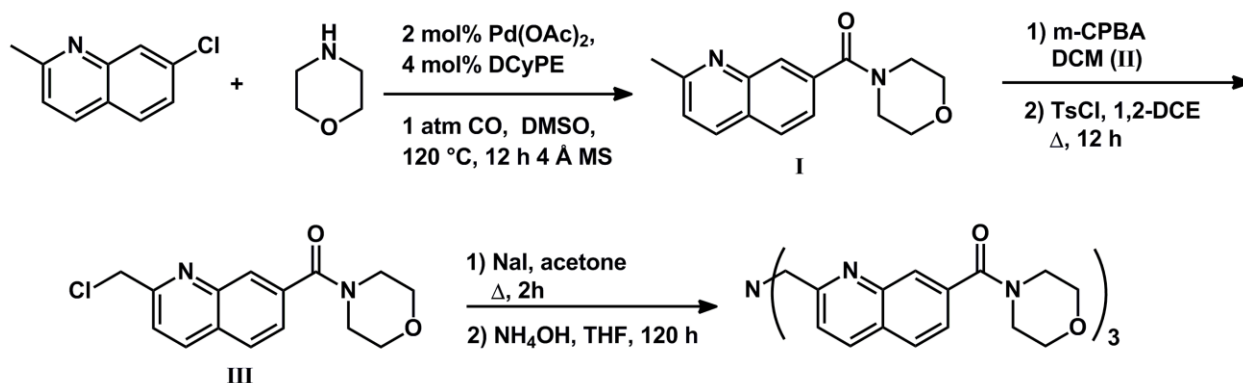
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Synthetic Preparations

Warning: Although we experienced no difficulties during the course of the present study, perchlorate complexes are potentially explosive and should be handled with extreme care.¹

General Considerations

All commercially-available reagents were used as received without further purification. TQA,² TPA,³ DCyPE,⁴ Cu(CH₃CN)₄X,⁵ Cu(TQA)PF₆² and [Cu(CH₃CN)TPA][(ClO₄)₂]⁶ were prepared as previously described. All manipulations were carried out in the air unless otherwise stated. Air sensitive reactions were performed either using standard Schlenk techniques or in an MBraun Lab Master 130 or Innovative Technologies Pure Lab^{HE} GP-1 glovebox. NMR spectra were recorded on either a Varian MR400 or a Varian vnmrs 500 spectrometer. IR spectra were collected using a Nicolet iS10 spectrometer equipped with a diamond attenuated total reflectance (ATR) accessory. UV-vis measurements were carried out using a Varian Cary50 spectrophotometer equipped with an all-quartz dip probe using fiber optic cables. Electrochemical measurements were performed using a Pine WaveNow potentiostat. The electrochemical cell consisted of a modified three electrode set-up with a glassy carbon working electrode, a platinum counter electrode and a silver wire pseudo-reference electrode. Ferrocene was used as an internal reference and introduced at the end of the experiment, then voltammograms were referenced to SCE (ferrocene/ferrocenium⁺ = 0.45 V / SCE). Voltammograms were measured in DMF with 0.1 M tetrabutylammonium hexafluorophosphate as the supporting electrolyte. X-band EPR spectra were recorded on a Bruker EMX spectrometer equipped with a Bruker 4102-ST general purpose cavity at 85 K. The spectra were recorded at a frequency of 9.258 GHz, modulation frequency of 100 kHz, modulation amplitude of 10.0 G, and 205.1 mW of power.



Scheme S1. Synthetic route for TQA'.

(2-methylquinolinyl)(morpholino)methanone (I)

To a 300 mL Schlenk flask charged with a stirbar, 4 Å molecular sieves (11.6 g) were added and activated under vacuum and heat. The flask was moved into the glovebox and charged with sodium phenoxide (15.10 g, 130 mmol) and 1,2-bis(dicyclohexylphosphino)ethane (1.10 g, 2.60 mmol). The flask was removed from the glovebox and palladium(II)acetate (0.29 g, 1.29 mmol) and 7-chloroquinoline (11.60 g, 65.3 mmol) were added quickly against an outflow of nitrogen. The flask was sealed with a septum and tape, and DMSO (125 mL) was then introduced,

followed by morpholine (17.0 mL, 194 mmol) under vigorous stirring. The headspace of the flask was evacuated and exchanged for carbon monoxide and a bubbler was attached to the flask. The flask was heated to 120 °C and allowed to stir while carbon monoxide continued to bubble through the solution. After 12 h, the brown mixture was allowed to cool to room temperature and was diluted with EtOAc (100 mL) and filtered through a pad of silica (4 cm tall x 5 cm diam.), eluting with EtOAc (400 mL). The eluent was extracted with 1 M aqueous NaOH (6 x 100 mL), dried over anhydrous Na₂SO₄ and concentrated to afford a beige solid. The combined aqueous layers were extracted with Et₂O (4 x 100 mL) and the organic extracts were then washed with brine and concentrated to a yellow oil. The yellow oil was lyophilized using benzene until a yellow gummy solid was obtained, which was triturated with cold Et₂O to provide a beige solid. This solid was combined with the original portion to give the title compound (10.8 g, 65 %) in ~95 % purity (as assessed by ¹H NMR spectroscopy; the major impurity is phenol), and was used without further purification. A crystalline sample was obtained by slow evaporation from a saturated Et₂O solution. ¹H NMR 500 MHz (CDCl₃), δ (ppm): 8.06 (d, *J* = 8.5 Hz, 1H), 7.99 (s, 1H), 7.84 (*J* = 8 Hz, 1H), 7.55 (dd, *J* = 8.5, 1.5 Hz, 1H), 7.34 (d, *J* = 8.5 Hz, 1H), 3.4 – 3.8 (br m, 8H), 2.76 (s, 3H). ¹³C NMR 125 MHz (CDCl₃), δ (ppm): 169.9, 160.4, 147.2, 136.13, 136.10, 128.6, 127.2, 127.1, 124.7, 123.3, 67.1, 25.6. IR, neat (cm⁻¹): 2976, 2921, 2844, 1628, 1615, 1431, 1270, 1110, 856.

(2-methylquinolinyl)(morpholino)methanone-N-oxide (II)

In a 500 mL round bottom flask charged with a stirbar and CH₂Cl₂ (250 mL) was added **I** (8.94 g, 34.9 mmol). With stirring, *m*-CPBA (~77 wt.%, 11.7 g, 68.0 mmol) was added and the solution was allowed to stir for 12 h. Saturated aqueous K₂CO₃ (200 mL) was then added and the solution was vigorously stirred for 2 h. The layers were then separated and the aqueous layer was extracted with CH₂Cl₂ (5 x 20 mL). The combined organic layers were washed with brine (50 mL) and dried over anhydrous Na₂SO₄, then concentrated to give the title compound as a beige powder (9.47 g, 99.7%). ¹H NMR 500 MHz (CDCl₃), δ (ppm): 8.78 (s, 1H), 7.91 (d, *J* = 8.5 Hz, 1H), 7.68 (overlapping d's, *J* = 8.5 Hz, 2H), 7.39 (d, *J* = 8.5 Hz, 1H), 3.4 – 3.9 (br m, 8H), 2.73 (s, 3H). ¹³C NMR 125 MHz (CDCl₃), δ (ppm): 169.1, 146.9, 141.0, 136.9, 129.8, 129.2, 127.0, 124.8, 124.4, 118.4, 67.0, 18.9. IR, neat (cm⁻¹): 2964, 2913, 2863, 1627, 1615, 1471, 1434, 1334, 1251, 1110, 852.

(2-(chloromethyl)quinolinyl)(morpholino)methanone (III)

In a 100 mL Schlenk flask charged with a stir bar, tosyl chloride (1.24 g, 6.50 mmol) was dissolved in deoxygenated 1,2-DCE (60 mL) under a dry dinitrogen atmosphere. To the stirring solution was added **II** (1.61 g, 5.91 mmol). The mixture was allowed to reflux under nitrogen overnight. After this time, the reaction was allowed to cool to room temperature and was quenched with saturated aqueous K₂CO₃. The phases were separated and the aqueous phase was extracted with DCM (3 x 25 mL). The combined organic layers were dried over anhydrous Na₂SO₄, filtered and concentrated to afford a yellow oil. The oil was dissolved in CH₃CN and filtered through a pad of silica (3 cm tall x 4 cm diam.) eluting with CH₃CN. The eluent was concentrated to provide a yellow oil and triturated with Et₂O to give the title compound (1.12 g, 65 %) as a beige powder in ~95% purity (as assessed by ¹H NMR spectroscopy; the major impurity is tosyl chloride). ¹H NMR 500 MHz (CDCl₃), δ (ppm): 8.23 (d, *J* = 8.5 Hz, 1H), 8.06 (s, 1H), 7.91 (d, *J* = 8 Hz, 1H), 7.68 (d, *J* = 8.5 Hz, 1H), 7.63 (dd, *J* = 8.5, 1.5 Hz, 1H), 4.84 (s,

2H), 3.4 – 3.9 (br m, 8H). ^{13}C NMR 125 MHz (CDCl_3), δ (ppm): 169.6, 158.1, 146.8, 137.3, 136.8, 128.6, 128.0, 127.8, 126.0, 121.8, 67.1, 47.3. IR, neat (cm^{-1}): 2983, 2868, 1623, 1436, 857.

TQA'

In a 50 mL round bottom flask, **III** (0.52 g, 1.8 mmol) and NaI (0.54 g, 3.6 mmol) were added followed by acetone (20 mL) with stirring. The solution was heated to reflux and stirred for 2 h resulting in a slightly yellow, cloudy solution. The mixture was allowed to cool to room temperature and was concentrated to afford a yellow oil. The oil was taken up in DCM (20 mL) and water was added (20 mL). The layers were separated and the aqueous layer was extracted with DCM (3 x 20 mL) and the organic extracts were combined and dried over anhydrous Na_2SO_4 , filtered and concentrated to an orange oil corresponding to the iodo-substituted product. ^1H NMR spectra recorded of this compound show clean and complete conversion from the chloride to iodide compound: ^1H NMR 400 MHz (CDCl_3), δ (ppm): 8.14 (d, $J = 8.4$ Hz, 1H), 8.03 (s, 1H), 7.86 (d, $J = 8.8$ Hz, 1H), 7.59 (app. t, $J = 8.5$ Hz, 2H), , 4.67 (s, 2H), 3.4 – 3.9 (br m, 8H). The iodo-compound thus prepared was used directly in the next step of the synthesis due to the instability of this compound. Storage on the bench top or in a freezer in the dark results in the slow decomposition of this product leading to a purple oil within less than a week.

The freshly prepared iodo-compound from above was dissolved in THF (20 mL) and NH_4OH was then added (0.24 mL, 14.8 M, 3.6 mmol). The flask was sealed and allowed to stir for 5 days. After that period, a white precipitate had appeared and was collected on a medium-porosity glass frit and washed with MeOH and then Et_2O and dried to a white powder (0.15 g, 33%). ^1H NMR 500 MHz (CDCl_3), δ (ppm): 8.14 (d, $J = 8$ Hz, 3H), 8.06 (s, 3H), 7.85 (d, $J = 8.5$ Hz, 3H), 7.77 (d, $J = 8.5$ Hz, 3H), 7.56 (d, $J = 8$ Hz, 3H), 4.15 (s, 6H), 3.4 – 3.9 (br m, 24H). ^{13}C NMR 125 MHz (CDCl_3), δ (ppm): 169.8, 161.1, 147.0, 136.4, 128.5, 128.0, 127.8, 125.2, 122.4, 67.1, 61.0. IR, neat (cm^{-1}): 2847, 1628, 1457, 1238, 854.

[CuTQA'] $[\text{PF}_6]$ (1)

In a nitrogen-filled glovebox, TQA' (82 mg, 0.11 mmol) and $\text{Cu}(\text{CH}_3\text{CN})_4\text{PF}_6$ (39 mg, 0.11 mmol) were dissolved in 5 mL of CH_3CN in a 20 mL scintillation vial and stirred for 30 min. Et_2O (~ 2 mL) was added to the yellow solution and then was filtered through 545 Celite. Et_2O (15 mL) was added to the filtrate and the cloudy yellow mixture was placed in a -35 °C freezer overnight to produce the title compound as a light orange powder (83 mg, 74%). Crystals suitable for a single crystal X-ray diffraction experiment were grown by vapor diffusion of Et_2O into a saturated solution of the title compound in CH_3CN . ^1H NMR 400 MHz (CD_2Cl_2), δ (ppm): 8.72 (s, 3H), 8.35 (d, $J = 8$ Hz, 3H), 7.96 (d, $J = 8.4$ Hz, 3H), 7.65 (d, $J = 8.4$ Hz, 3H), 7.56 (d, $J = 8$ Hz, 3H), 4.62 (s, 6H), 3.3 – 4.1 (br m, 24H). IR, neat (cm^{-1}): 2859, 1637, 1435, 1301, 1277, 1244, 1113, 835. ESI-MS: 841.9 $[\text{M-PF}_6]^+$ (calc. 842.3).

Replacement of $\text{Cu}(\text{CH}_3\text{CN})_4\text{PF}_6$ for $\text{Cu}(\text{CH}_3\text{CN})_4\text{ClO}_4$ yields the corresponding perchlorate complex in high yield:

In a nitrogen-filled glovebox, TQA' (50 mg, 0.064 mmol) and $\text{Cu}(\text{CH}_3\text{CN})_4\text{ClO}_4$ (19 mg, 0.058 mmol) were dissolved in 10 mL of CH_3CN in a 20 mL scintillation vial and stirred for 30 min.

Et₂O (100 mL) was added to the solution and the cloudy yellow mixture was filtered on a medium porosity frit. The orange solid was washed with Et₂O and dried to an orange powder (53 mg, 96%). ¹H NMR 400 MHz (CD₂Cl₂), δ (ppm): 8.72 (s, 3H), 8.35 (d, *J* = 8 Hz, 3H), 7.96 (d, *J* = 8.4 Hz, 3H), 7.65 (d, *J* = 8.4 Hz, 3H), 7.56 (d, *J* = 8 Hz, 3H), 4.67 (s, 6H), 3.3 – 4.1 (br m, 24H). IR, neat (cm⁻¹): 2854, 1630, 1433, 1301, 1276, 1242, 1084, 858.

[Cu(CH₃CN)TQA'][(ClO₄)₂] (2)

Method A:

In a 20 mL scintillation vial with a stir bar, Cu(ClO₄)₂·6H₂O (12.2 mg, 0.0329 mmol) was dissolved in 5 mL of CH₃CN. To the stirring pale blue solution was added solid TQA' (25.1 mg, 0.0322 mmol). The green mixture was stirred until all the solids had dissolved, then the solution was concentrated to minimal volume and filtered. Vapor diffusion of Et₂O directly into the filtrate provided bright green crystals which were washed with Et₂O and dried (32.1 mg, 92%). Crystals suitable for a single crystal X-ray diffraction experiment were grown by slow vapor diffusion of Et₂O into a saturated solution of the title compound in CH₃CN.

Method B:

In a nitrogen filled glovebox, CH₃CN (2 mL) was added to the solids Cu(TQA')ClO₄ (20 mg, 0.021 mmol) and ferrocenium perchlorate (7 mg, 0.03 mmol) with stirring. The initial blue color immediately became green and, once all of the solids dissolved, Et₂O (20 mL) was added to give a green precipitate. The green solid was collected on a medium porosity frit and washed with THF, then Et₂O, and dried to give a mint-green powder (17 mg, 74%).

UV-vis, λ_{max} (ε M⁻¹ cm⁻¹): 690 sh (110), 900 (290). X-band EPR 85 K (1:1 THF:CH₃CN): g_{||} = 2.00, g_⊥ = 2.18, A_{||} = 72 G, A_⊥ = 86 G. IR, neat (cm⁻¹): 2860, 2322, 2293, 2250, 1622, 1604, 1441, 1301, 1280, 1251, 1081. ESI-MS (performed for **2-d**): 887.0 [M – 2 ClO₄]⁺ (calc. 886.3).

Note: The use of Cu(TQA')PF₆ and ferrocenium hexafluorophosphate yields identical results; a green powder exhibiting identical EPR and UV-vis characterization. In our hands, we found that the synthesis of **2** by Method A was less reproducible than Method B. However, **2** can be prepared reproducibly using Method B and therefore we advise the use of Method B for the consistent synthesis of **2**.

[Cu(CH₃CN)TQA][(ClO₄)₂] (3)

In a 20 mL scintillation vial with a stir bar, Cu(ClO₄)₂·6H₂O (17.7 mg, 0.0478 mmol) was dissolved in 5 mL of CH₃CN. To the stirring pale blue solution was added solid TQA (21.3 mg, 0.0483 mmol). The teal mixture was stirred until all the solids had dissolved, then the solution was concentrated to minimal volume and filtered. Et₂O (20 mL) was added to produce a light blue precipitate which was collected and washed with Et₂O. The solid was dried to give the title compound as a pale turquoise powder (29.4 mg, 83%). Crystals suitable for a single crystal X-ray diffraction experiment were grown by slow vapor diffusion of Et₂O into a saturated solution of the title compound in CH₃CN. UV-vis, λ_{max} (ε M⁻¹ cm⁻¹): 660 (170), 845 sh (100). X-Band EPR 85 K (1:1 THF:CH₃CN) g_⊥ = 2.05, g_{||} = 2.27, A_{||} = 153 G. IR, neat (cm⁻¹): 2317, 2288,

1603, 1515, 1433, 1084, 822. Anal calcd. for $C_{32}H_{27}Cl_2CuN_5O_8$: C, 51.66; H, 3.66; N, 9.41.
Found: C, 51.71; H, 3.72; N, 9.42.

The preparation for the CD_3CN isotopologues was analogous to the above syntheses except that CH_3CN was replaced with CD_3CN .

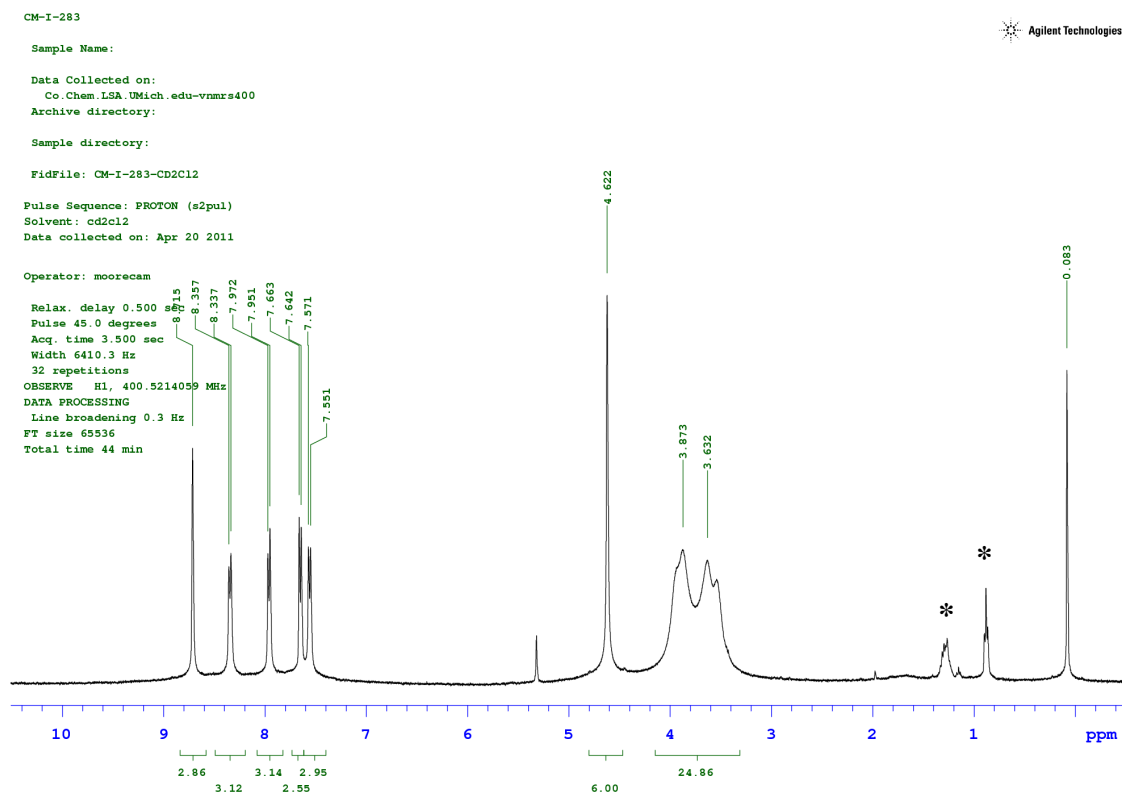


Figure S2. 1H NMR spectrum of **1** in CD_2Cl_2 showing retention of C_3 symmetry in solution (* denotes trace impurities of grease and diethyl ether).

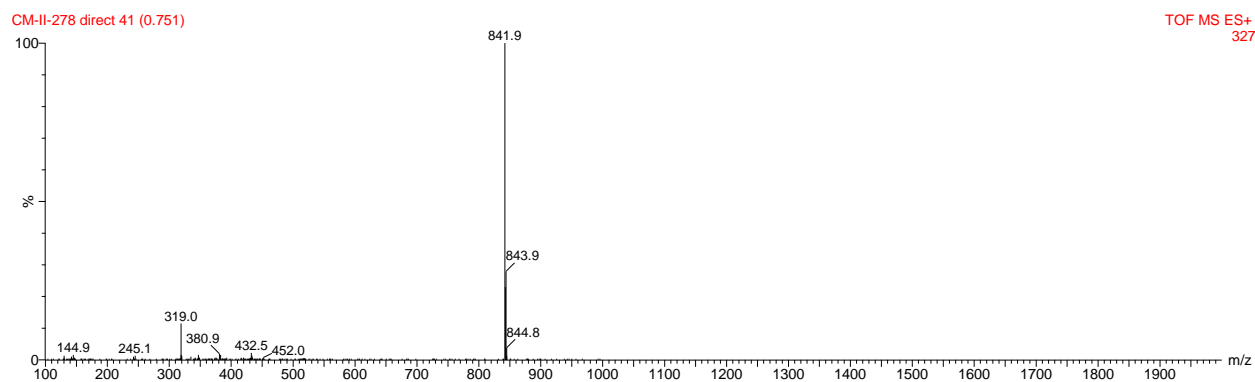


Figure S3. ESI-MS of **1** showing parent $[M-X]^+$ peak at $m/z = 841.9$ (calc. 842.3).

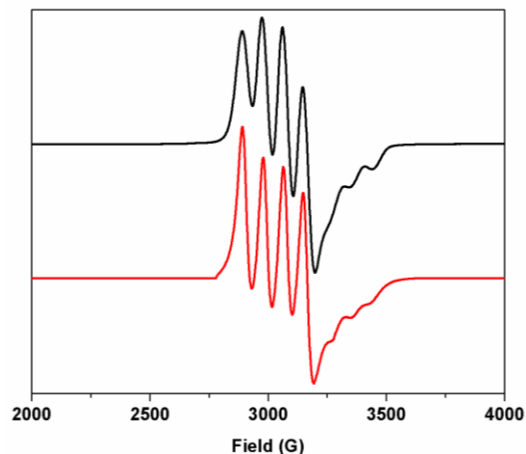


Figure S4. X-Band EPR spectrum of **2** in frozen THF/CH₃CN at 85 K (**experimental, simulated**). Simulated parameters: $g_{\perp} = 2.18$, $g_{\parallel} = 2.00$, $A_{\perp} = 86$ G, $A_{\parallel} = 72$ G

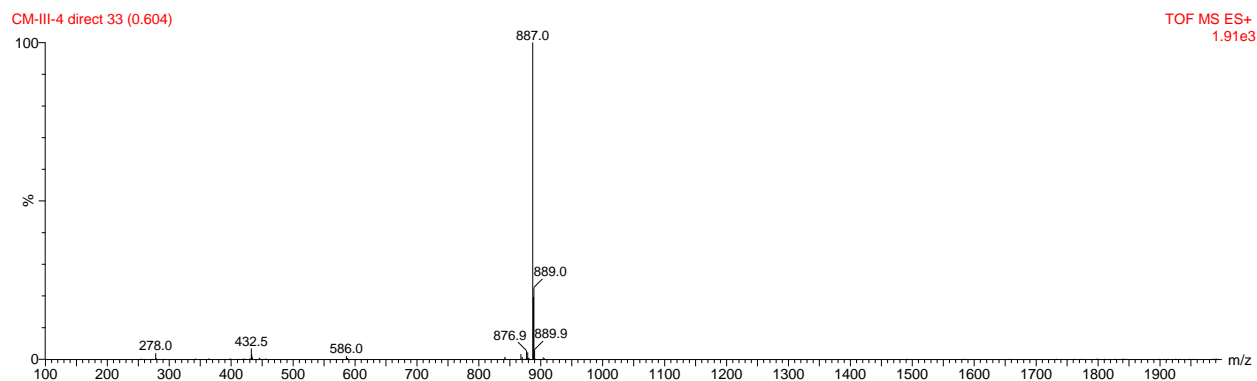


Figure S5. ESI-MS of **2-d** showing parent $[M-2X]^+$ peak at $m/z = 887.0$ (calc. 886.3).

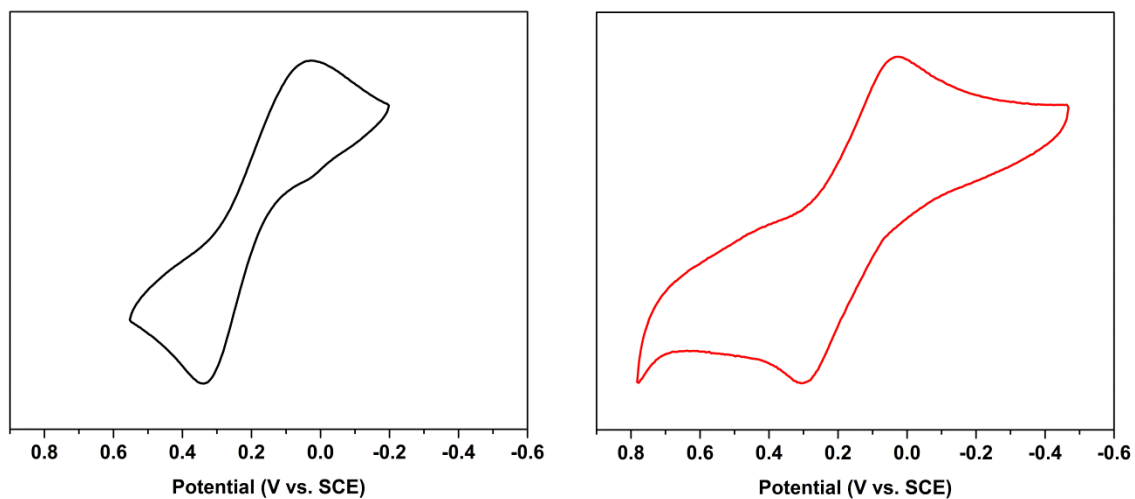


Figure S6. Normalized cyclic voltammograms of **1** (**black**) and Cu(TQA)PF₆ (**red**) collected at a scan rate of 50 mV/s. Conditions: glassy carbon working electrode, a platinum counter electrode and a silver wire pseudo-reference electrode in DMF with 0.1 M tetrabutylammonium hexafluorophosphate as the supporting electrolyte.

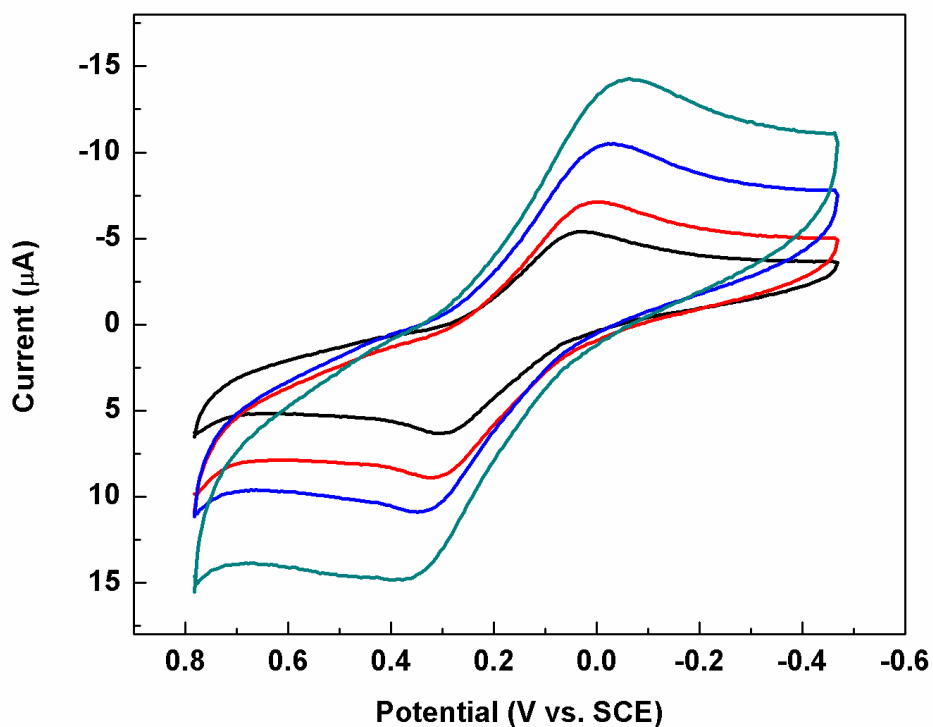


Figure S7. Cyclic voltammograms of **1** at various scan rates (50 mV/s, 100 mV/s, 200 mV/s and 400 mV/s). Conditions: glassy carbon working electrode, a platinum counter electrode and a silver wire pseudo-reference electrode in DMF with 0.1 M tetrabutylammonium hexafluorophosphate as the supporting electrolyte.

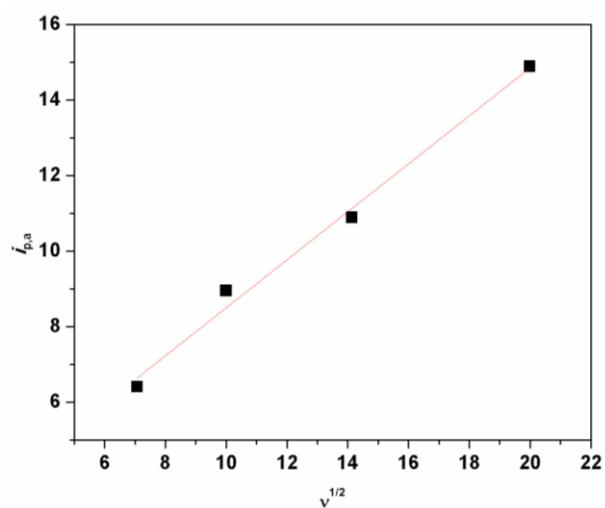


Figure S8. Linear dependence of peak current on the square-root of the scan rate of **1** during cyclic voltammetry, consistent with a reversible redox couple.

^2H NMR Studies

For the analysis of the CD_3CN isotopologues, a solution containing the complex (0.02 mmol) and 0.50 mL of MeNO_2 with an internal C_6D_6 standard (0.002 mmol) was prepared. The spectra were recorded at 77 MHz using a Varian vnmrs 500 MHz spectrometer. Samples were allowed to fully equilibrate at the desired temperature for 5 minutes before spectra were recorded.

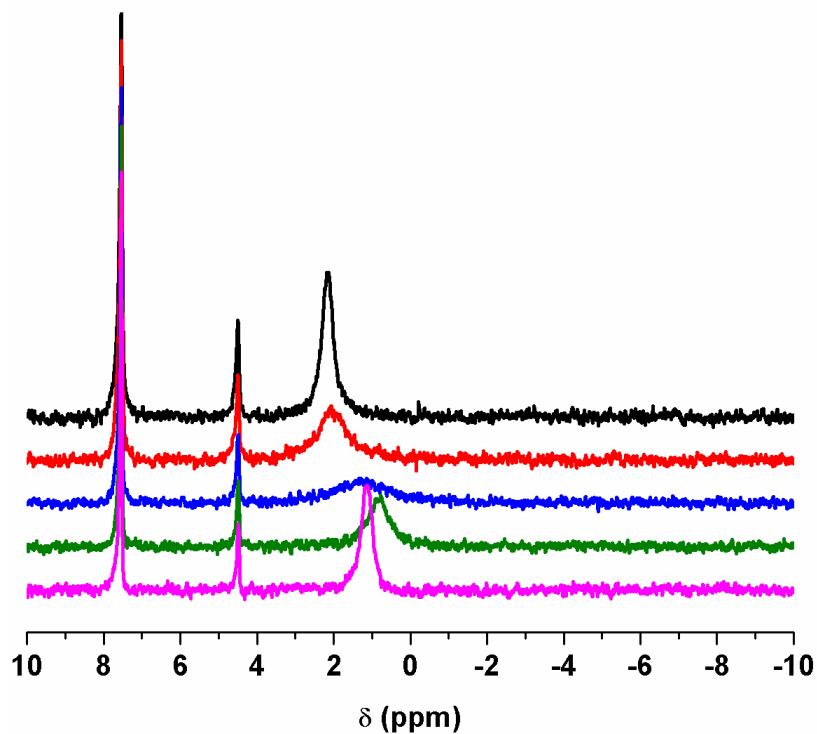


Figure S9. VT ^2H NMR spectra of **2** in MeNO_2 ($T = -20\text{ }^\circ\text{C}$, $0\text{ }^\circ\text{C}$, $20\text{ }^\circ\text{C}$, $40\text{ }^\circ\text{C}$ and $60\text{ }^\circ\text{C}$).

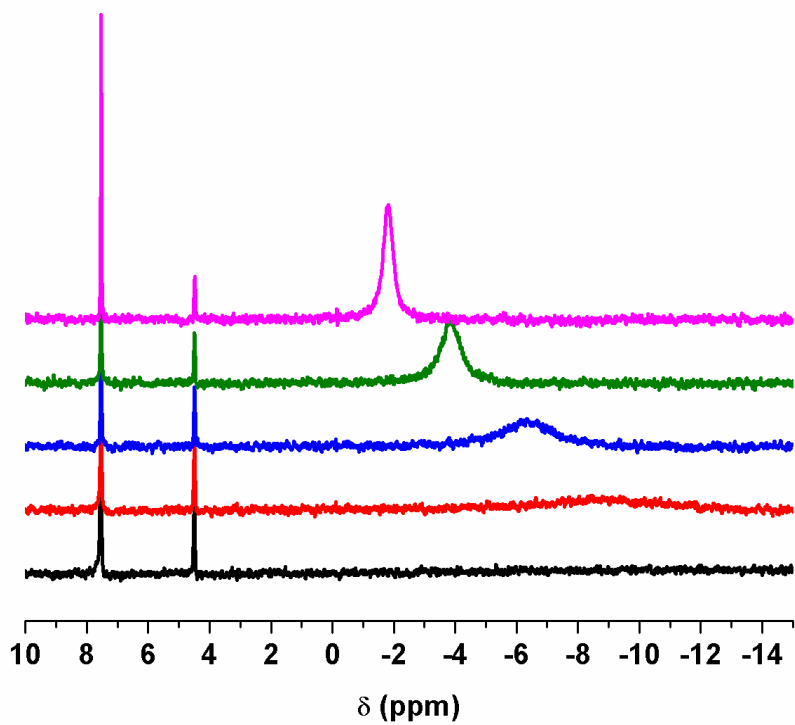


Figure S10. VT ²H NMR spectra of **3-d** in MeNO₂ (T = -20 °C, 0 °C, 20 °C, 40 °C and 60 °C).

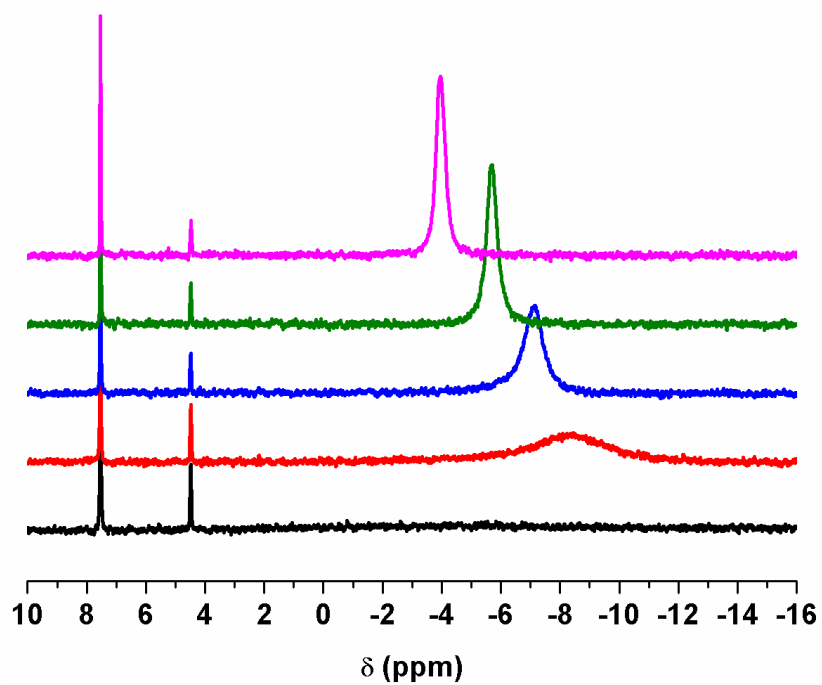


Figure S11. VT ²H NMR spectra of **4-d** in MeNO₂ (T = -20 °C, 0 °C, 20 °C, 40 °C and 60 °C).

CM-III-8inDCM-MeNO2-neg80



Sample Name:

Data Collected on:

Te-vnmrs500

Archive directory:

Sample directory:

FidFile: CM-III-8inDCM-MeNO2-neg80

Pulse Sequence: s2pul

Solvent: cd3no2

Data collected on: Feb 16 2012

Temp. -80.0 C / 193.2 K

Operator: moorecam

Relax. delay 0.100 sec

Pulse 45.0 degrees

Acq. time 1.416 sec

Width 11574.1 Hz

512 repetitions

OBSERVE 1k, 76.7674962 MHz

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 17 min

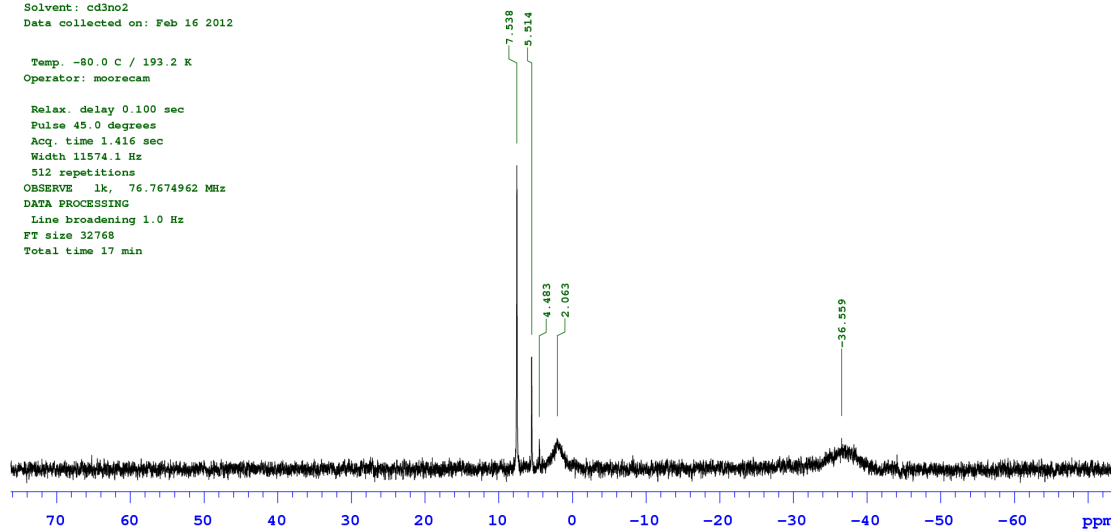


Figure S12. ^2H NMR spectrum of **4-d** in 5:1 DCM:MeNO₂ at -80 °C showing the decoalescence of **4-d** (-36.56 ppm) and free CD₃CN (2.06 ppm).

Crystallographic Details

Compound	1 · 1.5 CH₃CN	2 · 3.5 CH₃CN	3
Empirical Formula	C ₄₈ H _{49.50} CuF ₆ N _{8.50} O ₆ P	C ₅₂ H _{55.50} Cl ₂ CuN _{10.50} O ₁₄	C ₃₂ H ₂₇ Cl ₂ CuN ₅ O ₈
Formula Weight	1049.97	1186.01	744.03
Temperature	85(2)K	85(2) K	85(2) K
Wavelength	1.54178 Å	1.54178 Å	1.54178 Å
Crystal system	Monoclinic	Monoclinic	Triclinic
Space group	P 21/c	C 1 2/c 1	P-1
Unit cell dimensions	a = 14.3615(12) Å α = 90° b = 28.410(3) Å β = 115.017(8)° c = 13.7532(13) Å γ = 90°	a = 23.7111(4) Å α = 90° b = 17.7456(3) Å β = 107.050(7)° c = 26.3160(19) Å γ = 90°	a = 9.0679(2) Å α = 78.358(6)° b = 11.0145(2) Å β = 82.766(6)° c = 17.5571(12) Å γ = 67.261(5)°
Volume	5085.0(8) Å ³	10586.3(8) Å ³	1581.78(12) Å ³
Z	4	8	2
Density (calc.)	1.371 mg/m ³	1.488 mg/m ³	1.562 mg/m ³
Absorption coefficient	1.572 mm ⁻¹	2.106 mm ⁻¹	3.052 mm ⁻¹
F(000)	2172	4488	762
Crystal size	0.12 x 0.06 x 0.06 mm ³	0.18 x 0.15 x 0.10 mm ³	0.17 x 0.15 x 0.09 mm ³
Theta range for data collection	6.64 to 63.77°	3.20 to 68.25°	2.57 to 68.24°
Index ranges	-16 ≤ h ≤ 16 -30 ≤ k ≤ 32 -15 ≤ l ≤ 15	-28 ≤ h ≤ 28 -21 ≤ k ≤ 21 -28 ≤ l ≤ 31	-10 ≤ h ≤ 13 -13 ≤ k ≤ 13 -21 ≤ l ≤ 21
Reflections collected	83097	73479	40295
Independent reflections	8170 [R(int) = 0.1397]	9671 [R(int) = 0.0472]	5675 [R(int) = 0.0774]
Completeness	97.5 %	99.7 %	98.1 %
Absorption correction	Semi-empirical from equivalents	Semi-empirical from equivalents	Semi-empirical from equivalents
Max. and min. transmission	0.9116 and 0.8338	0.810 and 0.705	0.760 and 0.558
Refinement method	Full-matrix least-squares on F ²	Full-matrix least-squares on F ²	Full-matrix least-squares on F ²
Data / restraints / parameters	8170 / 66 / 706	9671 / 0 / 745	5673 / 0 / 435
Goodness-of-fit on F ²	1.052	1.053	1.056
Final R indices [I > 2σ(I)]	R1 = 0.0884 wR2 = 0.2558	R1 = 0.0348 wR2 = 0.0882	R1 = 0.0794 wR2 = 0.2305
R indices (all data)	R1 = 0.1320 wR2 = 0.2893	R1 = 0.0377 wR2 = 0.0901	R1 = 0.0875 wR2 = 0.2385
Largest diff. peak and hole	0.854 and -0.403 e.Å ⁻³	0.388 and -0.456 e.Å ⁻³	1.560 and -0.876 e.Å ⁻³

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