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Supporting Information

Dicopper(I) Complexes of a Binucleating, Dianionic, Naphthyridine Bis(amide) Ligand

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Experimental

General experimental details

All experiments were performed using standard Schlenk techniques or in an N_2-filled glovebox unless otherwise noted. Pentane, THF, toluene and diethyl ether for experiments inside the nitrogen-filled (6N) glovebox were dried and deaerated using a JC Meyers Phoenix SDS solvent purification system. Dichloromethane-d_2 and benzene-d_6 were dried and stored over activated 3Å molecular sieves for at least 48 hours and degassed with three freeze-pump-thaw cycles. The compound 7-dichloro-1,8-naphtyhridine was prepared according to the reported method.[1] Mesitylcopper(I) was prepared according to the procedure by Tsuda, et al.[2] All other reagents were obtained from commercial sources.

NMR spectra were acquired at 299 K using Bruker AV-400, AV-500 and AV-600 spectrometers. ^{1}H NMR spectra were referenced via residual monoprotio solvent peaks (δ 5.32 for dichloromethane-d_2, δ 7.16 for benzene-d_6, 1.72 for THF-d_8), while ^{13}C{^{1}H} NMR spectra were referenced to solvent resonances (δ 128.06 for benzene-d_6, δ 53.84 for dichloromethane-d_2, δ 67.21 for THF-d_8). Elemental analyses were performed by Elena Kramer at the Microanalytical Facility at the University of California, Berkeley. IR Spectra were recorded on a Bruker Vertex 80 FTIR spectrometer in ATR mode with a resolution of 4 cm^{-1}.

Electrochemistry

Electrochemical experiments were performed inside a nitrogen glovebox using a BASi EC Epsilon galvanostat. Glassy carbon working electrodes (3 mm diameter of active material) were polished with alumina (1.0 µm, 0.3 µm and 0.05 µm), rinsed with milliQ-grade water and high purity acetone before being transferred into the glovebox. The counter electrode consisted of a platinum wire that was cleaned in a torch flame before being rinsed with milliQ-grade water and high purity acetone and dried under nitrogen before transfer into the glovebox. The reference electrode consisted of a silver wire etched in 1M HCl in a reservoir of electrolyte solution and 0.5 mM silver tetrafluoroborate (99.999%), separated by a porous glass frit and assembled the glovebox. The electrolyte solution (Bu_4NPF_6, electrochemical grade, 0.1 M in THF) was prepared immediately before measurement. The reference electrode was calibrated by measurement of the ferrocene redox couple in electrolyte solution at regular intervals to ensure no drifting of the potential. Cyclic voltammograms were measured by scanning in the positive direction (unless otherwise noted) after an equilibration period of two seconds and blank measurements were performed before addition of the substrates.

Single crystal X-ray diffraction

Single crystal X-ray diffraction for complex **4** was performed at UC Berkeley CheXRay crystallographic facility. Data collection was performed using a Rigaku XtaLAB P200 equipped with a MicroMax 007HF rotating anode and a Pilatus 200k hybrid pixel array detector using Mo Kα radiation (λ = 0.71073 Å) monochromated by a graphite monochromator. All other diffraction studies were collected at the Advanced Light Source beamline 12.2.1 using a Bruker D85 three-circle diffractometer equipped with a PHOTON II CCD area detector using synchrotron radiation (λ = 0.7288 Å) monochromated using a double crystal Si(111) monochromator. Structure solution, modeling, and refining was performed using both the OLEX2[3] and Shelxle GUI[4] and SHELX[5] softwares while data integration was done using APEXIII[6] softwares.

Computational Details

All Calculations were performed with the Gaussian16 (Revision B.01) package and visualized with GaussView 6.0 or Chemcraft Version 1.8. The geometries of all complexes were optimized in vacuum using the corresponding solid-state molecular structures as starting points, omitting counterions and any solvent molecules. Optimization of the geometries was performed using the def2-SVP basis set with the ωB97X-D functional,[7] then further optimized in the def2-TZVP basis set.[8,9] A frequency calculation was then used to ensure that that the structures were fully optimized to a minimum.

Syntheses

Synthesis of 1,8-naphthyridine bis(2,6-diisopropylphenyl)amide (H_2**NBDA**):

A Fisher-Porter bottle (80 mL volume) was filled with 2,7-dichloro-1,8-naphthyridine (0.40 g, 2.0 mmol) and 2,6-di(isopropyl)aniline (0.83 mL, 4.4 mmol, 2.2 equiv.) and degassed by repeated evacuation and refilling under nitrogen. Under nitrogen flow, a solution of $Pd(OAc)_2$ and XantPhos in toluene (10 mM, 3.0 mL, 1.5 mol%) previously prepared inside a glovebox was added, followed by addition of triethylamine (0.7 mL, 5 mmol, 2.45 equiv.) and toluene (7 mL). The reactor was then purged with CO gas (5 atm) by repeating a pressurization and depressurization cycle 6 times. After the final purging cycle, the reactor was filled with 5 atm CO gas and heated to 100 °C for 20 h (until the internal pressure stabilized). After cooling, the reactor was depressurized and the reaction solution was diluted with 50 mL of dichloromethane (DCM). The product was transferred to a separatory funnel and washed with 1 M HCl (30 mL), K_2CO_3 solution (1.0 g, 30 mL) and finally saturated NaCl solution (15 mL). After removal of the solvent, the crude product was purified by column chromatography on silica gel using DCM/MeOH 150:1 as eluent. Finally, the product was recrystallized from hot ^{i}PrOH to give a white, crystalline material. Yield: 595 mg, 56%.

^{1}H-NMR (400 MHz, CD_2Cl_2) δ 9.67 (s, 2H), 8.64 – 8.54 (m, 4H), 7.38 (dd, J = 8.4, 7.1 Hz, 2H), 7.27 (d, J = 7.7 Hz, 4H), 3.20 (h, J = 6.9 Hz, 4H), 1.22 (d, J = 6.9 Hz, 24H).

^{13}C-NMR (151 MHz, CD_2Cl_2) δ 163.53, 154.18, 153.33, 146.96, 139.74, 131.56, 128.87, 126.28, 124.00, 121.70, 29.40, 23.85.

EA Calc. for $C_{34}H_{40}N_4O_2$: C, 76.09; H, 7.51; N, 10.44; Found: C, 75.52; H, 7.65; N, 10.30. (corresponds to 0.25 equiv. H_2O per product)

Synthesis of [NBu_4][Cu_2(**NBDA**)(μ-Cl)] (**1**):

Inside a glovebox, a vial was charged with H_2**NBDA** (27.1 mg, 0.051 mmol, 1 equiv.) which was dissolved in THF (0.5 mL). To this, a solution of mesitylcopper(I) (19.4 mg, 0.106 mmol, 2.05 equiv.) in THF (0.5 mL) was added and the solution became dark yellow. This solution was then quickly added to another vial charged with [NBu_4]Cl (14.1 mg, 0.051 mmol, 1.0 equiv.) and THF (0.8 mL), and a rapid color change to dark brown was observed. The reaction solution was stirred at 22 °C for 20 h, then filtered through celite and the solvent was removed under vacuum. The resulting brown solid was triturated with diethyl ether (5 mL) three times, then dried under vacuum for 4 h. The analytically pure product was obtained as a flaky brown solid. Yield: 41 mg, 86%. Crystals suitable for diffraction were grown by vapor diffusion of diethyl ether into a THF solution of the product. Note: addition of a CuMes solution to a mixture of H_2**NBDA** and [NBu_4]Cl in THF produced the desired product in equal yield and purity as the with the procedure described here.

^{1}H-NMR (600 MHz, CD_2Cl_2) δ 8.62 (d, J = 8.4, 2.4 Hz, 2H), 8.52 (d, J = 8.3 Hz, 2H), 7.12 – 7.08 (m, 4H), 7.07 – 7.01 (m, 2H), 3.44 – 3.35 (m, 4H), 3.06 – 2.97 (m, 8H), 1.60 – 1.56 (m, 8H), 1.43 – 1.33 (m, 8H), 1.22 (d, J = 6.9 Hz, 12H), 1.16 (d, J = 6.9 Hz, 12H), 1.02 – 0.96 (m, 12H).

^{13}C-NMR (151 MHz, CD_2Cl_2) δ 166.07, 159.36, 149.39, 147.52, 142.65, 138.95, 125.32, 123.16, 122.84, 122.28, 59.44, 28.69, 24.25, 24.22, 24.10, 23.76, 20.09, 13.72.

EA: Calc. for $C_{50}H_{74}ClCu_2N_5O_2$: C, 63.46; H, 7.88; N, 11.84; Found: C, 63.55 ; H, 7.88; N, 11.92.

Synthesis of [Cu_2(DPFN)(μ-1,1-N_3)](NTf_2) (**2**):

Inside a glovebox, a vial was charged with [Cu_2(DPFN)(MeCN)](NTf_2)$_2$ (30.0 mg, 0.024 mmol, 1 equiv.) and THF (1 mL). To this, a solution of [NBu_4]N_3 (7.4 mg, 0.026 mmol, 1.1 equiv.) in THF (2 mL) was added and the reaction mixture cleared and turned purple. The reaction was stirred for 1 h at 22 °C, then the volatiles were removed under vacuum. The crude product was triturated twice with toluene (10 mL) and twice with diethyl ether (5 mL) to remove [NBu_4]$^+$ salts. The product was dried under vacuum and obtained as a purple powder. Yield: 18.2 mg, 81%. Crystals suitable for diffraction were grown by vapor diffusion of diethyl ether into a THF solution of the product.

^{1}H-NMR (600 MHz, THF-d_8) δ 9.00 (d, J = 4.8 Hz, 4H), 8.88 (d, J = 8.6 Hz, 2H), 8.45 (dd, J = 8.6, 3.3 Hz, 2H), 8.18 (dd, J = 8.3, 3.5 Hz, 4H), 8.08 (dd, J = 7.9, 1.7 Hz, 4H), 7.58 (ddd, J = 7.6, 5.0, 1.2 Hz, 4H).

^{13}C-NMR (151 MHz, THF-d_8) δ 160.71, 160.51, 154.51, 154.31, 150.98, 150.96, 142.28, 142.26, 140.24, 140.22, 125.86, 124.44, 121.73, 121.64, 120.81, 120.70.

^{19}F-NMR (565 MHz, THF-d_8) δ –80.86, –80.87, –174.03.

EA: Calc for $C_{32}H_{20}Cu_2F_8N_{10}O_4S_2$: C, 40.38; H, 2.12; N, 14.72; Found: C, 40.17; H, 1.90; N, 14.39.

Synthesis of [NBu_4][Cu_2(**NBDA**)(μ-1,1-N_3)] (**3**):

Inside a glovebox, a vial was charged with H_2**NBDA** (55.0 mg, 0.102 mmol, 1 equiv.) and THF (2 mL). To this, a solution of mesitylcopper(I) (39.0 mg, 0.212 mmol, 2.06 equiv.) in THF (2 mL) was added and the solution became dark yellow. A solution of [NBu_4]N_3 (29.1 mg, 0.102 mmol, 1.0 equiv.) in THF (1.5 mL) was added to the reaction mixture, followed by a rapid color change to dark brown. The reaction mixture was stirred for 18 h at 22 °C, then filtered through celite and the volatile materials were then removed under vacuum. The crude product was dissolved in THF (ca 1.5 mL) and then crystallized by vapor diffusion of diethyl ether first at –30 °C (36 h), then at 22 °C (additional 36 h) to give gold-brown crystals. The supernatant was decanted and the product was washed with diethyl ether (3 x 2 mL), and finally dried under vacuum. Yield: 70 mg, 72% (Additional product were isolated from the supernatant, giving an additional 5 mg of product). Crystals suitable for diffraction were grown by vapor diffusion of diethyl ether into a THF solution of the product.

^{1}H-NMR (600 MHz, CD_2Cl_2) δ 8.57 (d, J = 8.2 Hz, 2H), 8.49 (d, J = 8.3 Hz, 2H), 7.12 (d, J = 7.5 Hz, 4H), 7.08 (d, J = 7.4 Hz, 2H), 3.44 – 3.39 (m, 4H), 3.06 – 3.00 (m, 8H), 1.61 – 1.53 (m, 8H), 1.39 (q, J = 7.4 Hz, 8H), 1.26 (d, J = 6.9 Hz, 12H), 1.17 (d, J = 6.9 Hz, 12H), 1.00 (t, J = 7.3 Hz, 12H).

^{13}C-NMR (151 MHz, CD_2Cl_2) δ 166.63, 158.91, 149.65, 147.32, 142.84, 138.75, 125.00, 123.37, 122.93, 122.18, 59.46, 59.44, 59.42, 28.76, 24.24, 24.01, 23.71, 20.10, 13.71.

EA: Calc. for $C_{50}H_{74}Cu_2N_8O_2$: C, 63.46; H, 7.88; N, 11.84; Found: C, 63.55; H, 7.88; N, 11.92.

Synthesis of [Cu_2(**NBDA**)(CNXyl)$_2$] (**4**)

Inside a glovebox, a vial was charged with H_2**NBDA** (45.0 mg, 0.084 mmol, 1 equiv.) and THF (1.5 ml). To this, a solution of mesitylcopper(I) (31.2 mg, 0.171 mmol, 2.05 equiv.) in THF (1 mL) was added and the solution became dark yellow. A solution of xylyl isocyanide (21.8 mg, 0.166 mmol, 2.0 equiv.) in THF (0.5 mL) was added to the reaction mixture leading to an immediate color change to dark red. The solution was stirred for 44 h at 22 °C, with a pale reddish precipitate forming. The solvent was subsequently removed under vacuum and the resulting solid was triturated with diethyl ether (10 ml) and then pentane (2 x 10 mL). After removal of volatiles under vacuum, the product was obtained as a dark orange powder. Further purification was achieved by layering a concentrated THF solution of the product (ca. 5 mL) with pentane (15 mL) at 22 °C to give orange-red crystals. Yield: 71 mg, 92%.

^{1}H-NMR (600 MHz, CD_2Cl_2) δ 8.75 – 8.67 (m, 4H), 7.24 – 7.10 (m, 4H), 7.06 – 6.99 (m, 8H), 3.28 (dt, J = 6.7 Hz, 4H), 2.09 (s, 12H), 1.07 (d, J = 6.9, 1.6 Hz, 12H), 0.87 (d, J = 6.9 Hz, 12H).

^{13}C-NMR (151 MHz, CD_2Cl_2) δ 165.82, 160.62, 149.09, 146.05, 142.73, 141.12, 135.70, 130.13, 128.51, 126.42, 125.91, 125.13, 123.88, 123.08, 122.97, 117.77, 117.65, 28.83, 24.07, 23.51, 18.90.

EA: Calc. for $C_{52}H_{56}Cu_2N_6O_2$: C, 67.58; H, 6.11; N, 9.09; Found: C, 67.34; H, 6.02; N, 8.99.

Reduction of **1** with KC_8

Inside a glovebox, a solution of **1** (4.2 mg, 4 µmol, 1 equiv.) in THF (0.3 mL) was added to a stirring suspension of KC_8 (0.6 mg, 4 µmol, 1 equiv.) in THF (0.3 mL). An immediate appearance of a dark green color was observed. The solution was filtered through glass wool and the solvent then removed under vacuum. The product was then triturated with diethyl ether (1 ml) and dried under vacuum to give a dark green solid. An NMR spectrum in proteo-THF showed no signals other than solvent.

Supplementary Figures and Tables

Figure S1: IR spectrum of ligand H_2**NBDA**.

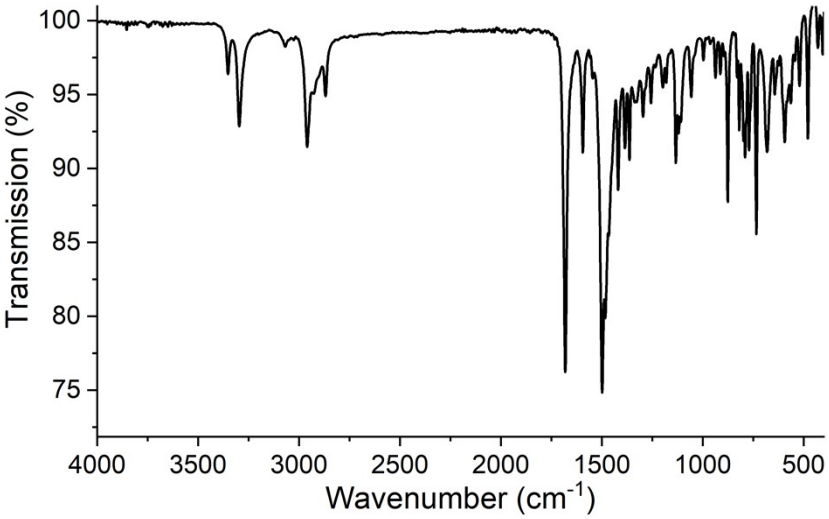


Figure S2: IR spectrum of complex **1**.

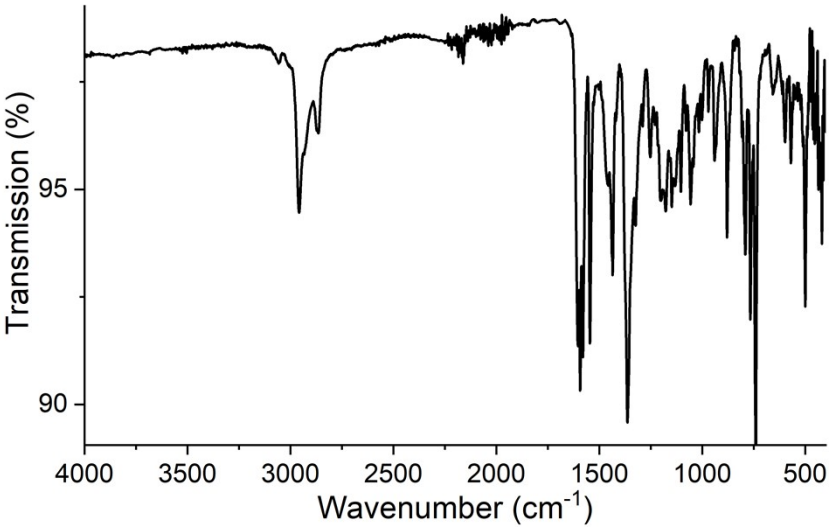


Figure S3: IR spectrum of complex **2**.

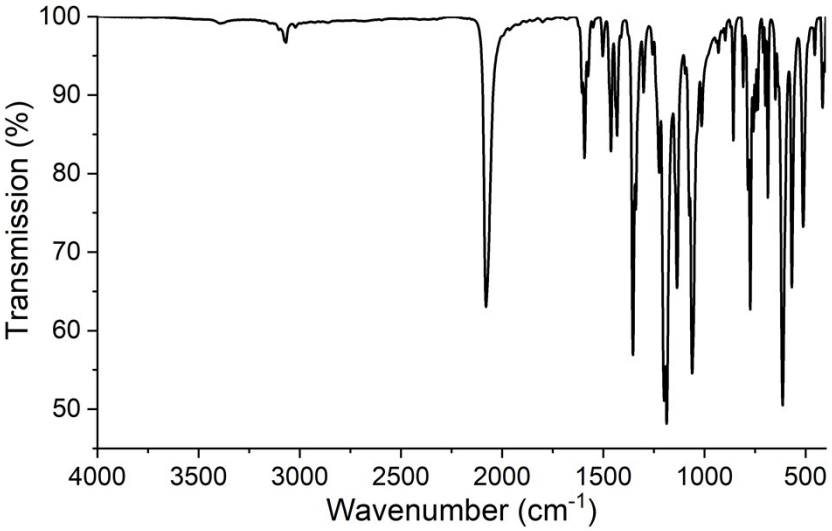


Figure S4: IR spectrum of complex **3**.

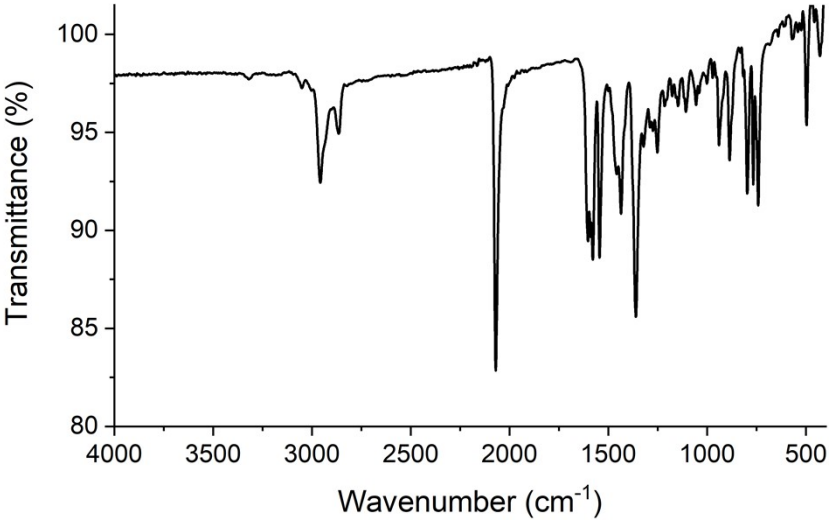


Figure S5: IR spectrum of complex **4**.

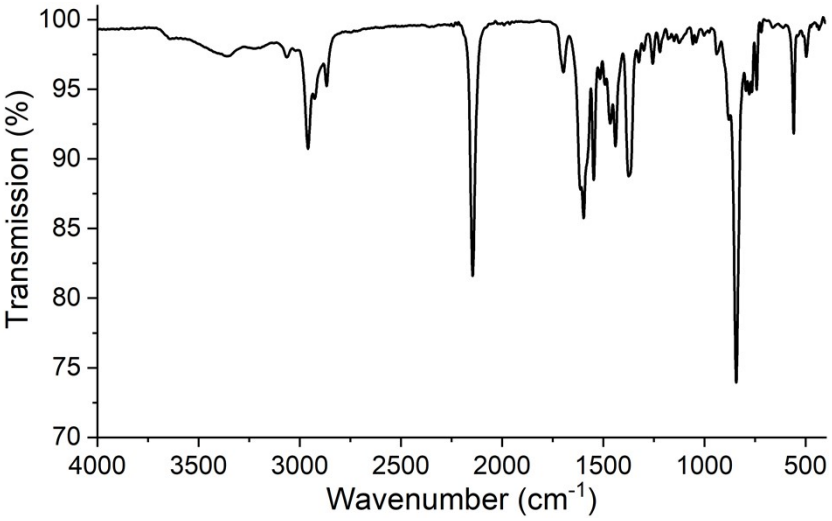


Table S1. Natural charges for copper atoms and ligands in the selected complexes. Charges are per atom (for copper) or per ligand (for bridging ligands).

	Calculated natural charge		
Complex	Cu	Naphthyridine ligand	Bridging ligand
$[Cu_2(\mathbf{NBDA})(\mu\text{-}Cl)]^-$ (**1**)	0.52	–1.53	–0.51
$[Cu_2(DPFN)(\mu\text{-}N_3)]^+$ (**2**)	0.53	0.58	–0.63
$[Cu_2(\mathbf{NBDA})(\mu\text{-}N_3)]^-$ (**3**)	0.60	–1.54	–0.65
$[Cu_2(\mathbf{NBDA})(XylNC)_2]$ (**4**)	0.49	–1.32	0.17
$[Cu_2(DPFN)(\mu\text{-}Cl)]^+$	0.45	0.58	–0.49
$[Cu_2(py\text{-}np)(\mu\text{-}Cl)_2]$	0.47	0.22	–0.58

Figure S6: highest occupied and unoccupied MOs of complex **1** from DFT calculation.

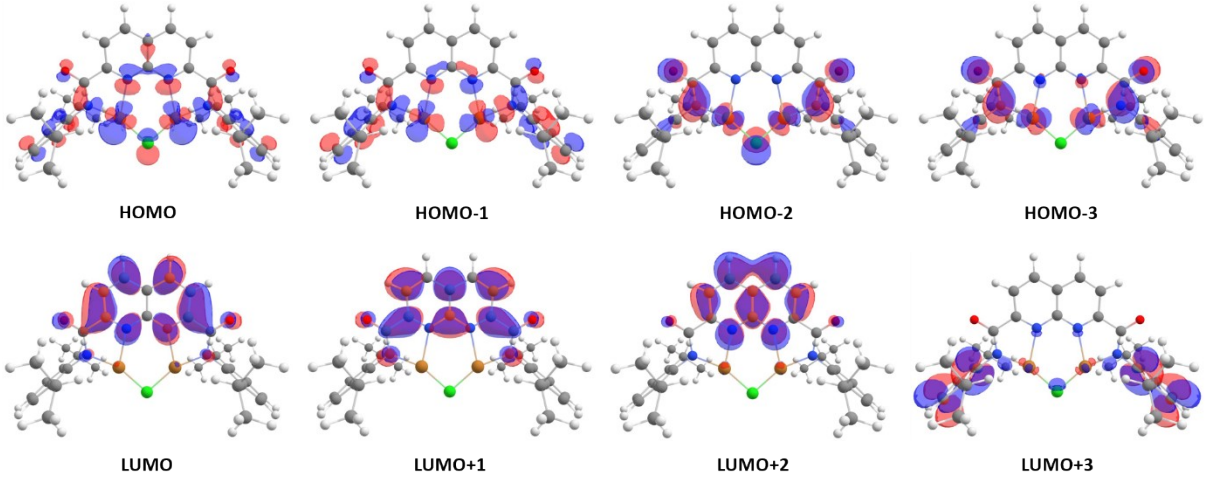


Figure S7: highest occupied and unoccupied MOs of complex **2** from DFT calculation.

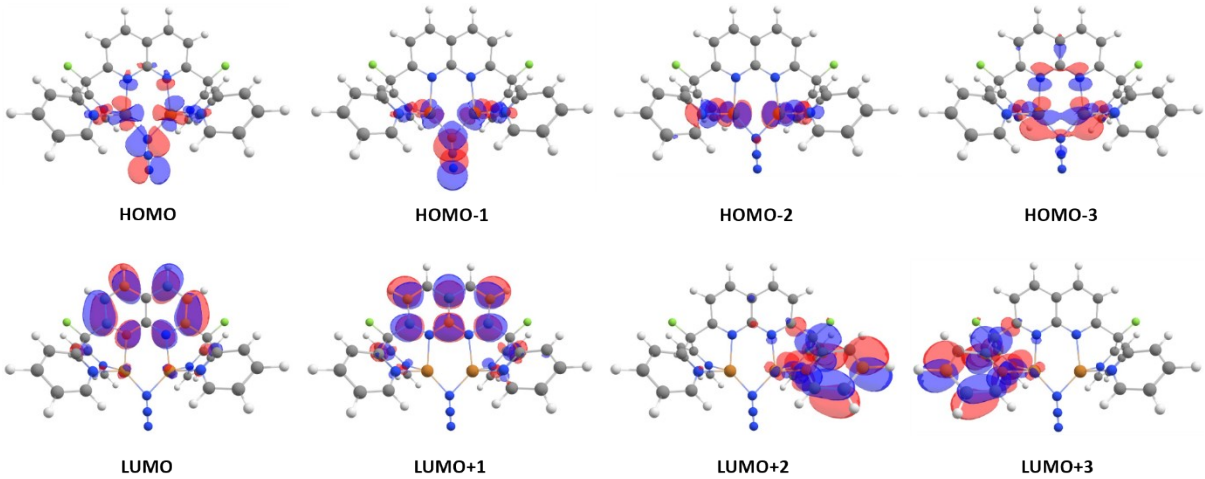


Figure S8: highest occupied and unoccupied MOs of complex **3** from DFT calculation.

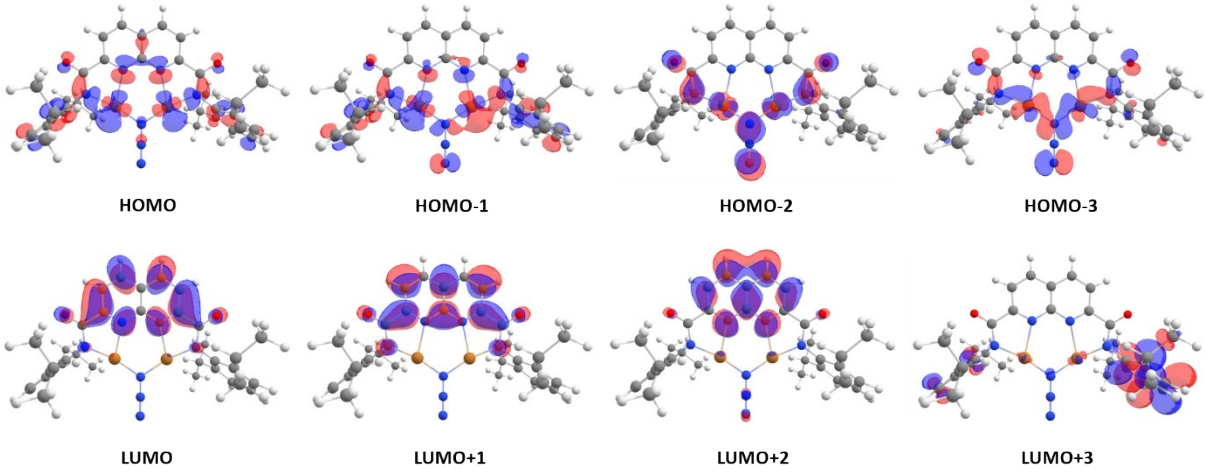


Figure S9: highest occupied and unoccupied MOs of complex **4** from DFT calculation.

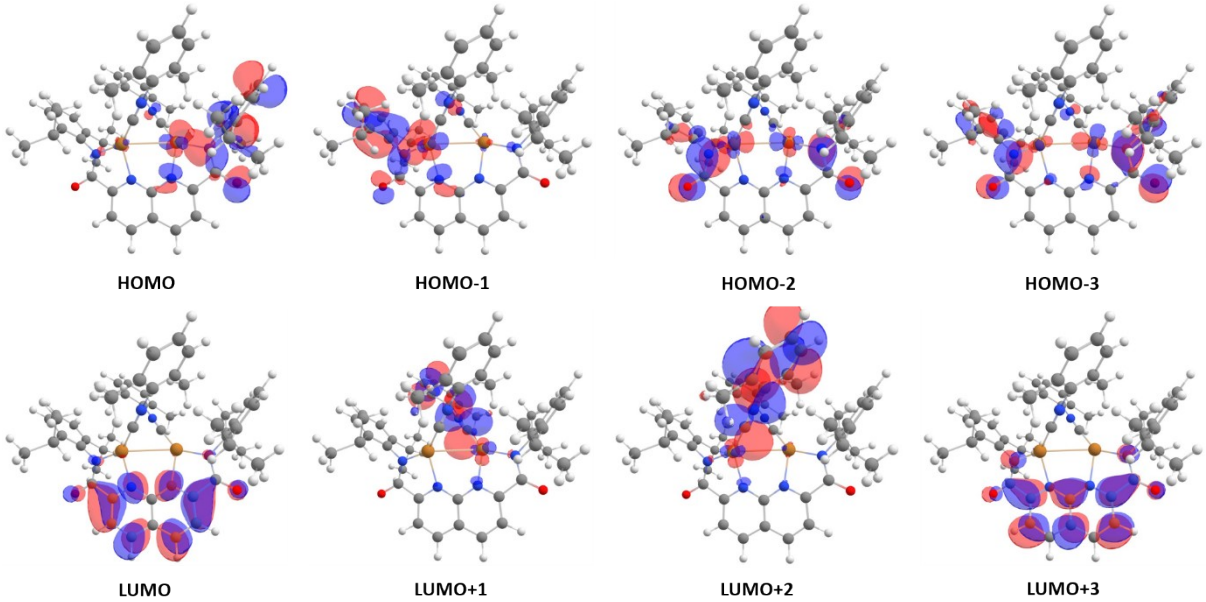


Figure S10: highest occupied and unoccupied MOs of $[Cu_2(DPFN)(Cl)]^+$ from DFT calculation.

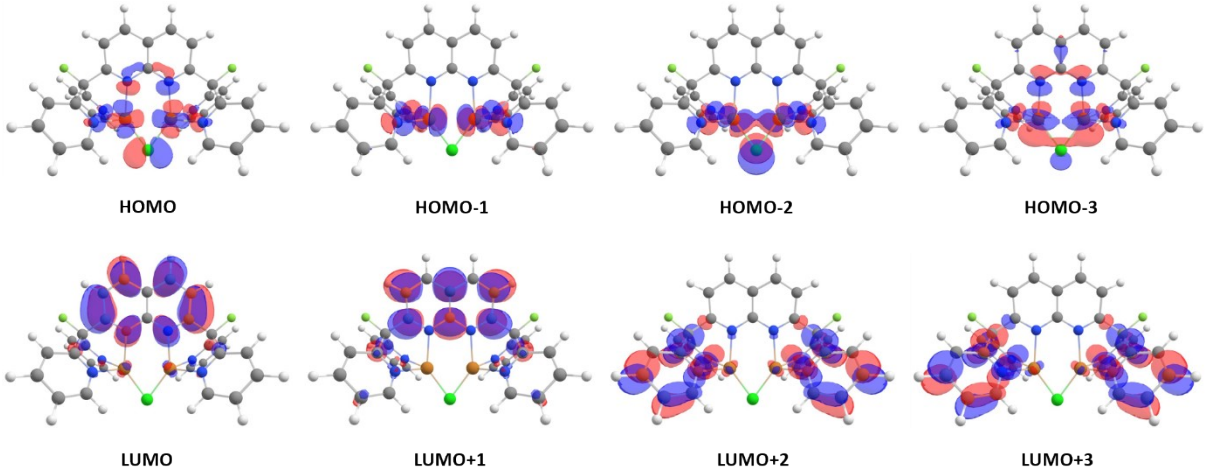


Figure S11: highest occupied and unoccupied MOs of $[Cu_2(bp\text{-}np)(Cl)_2]$ from DFT calculation.

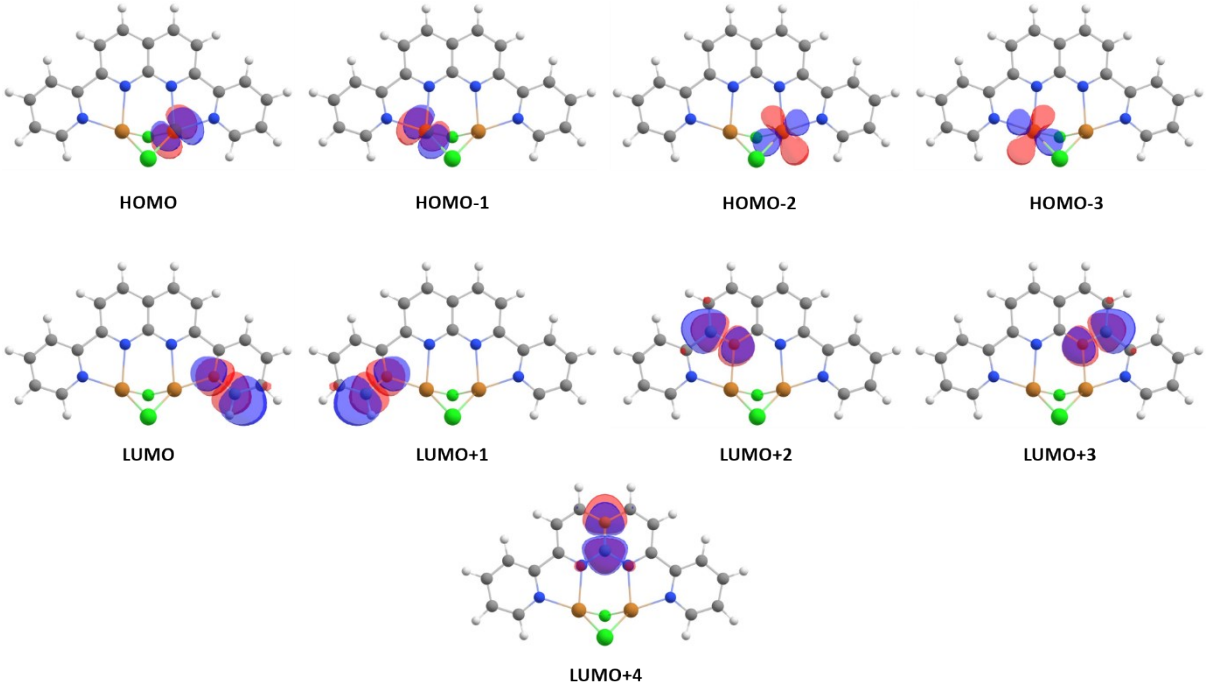
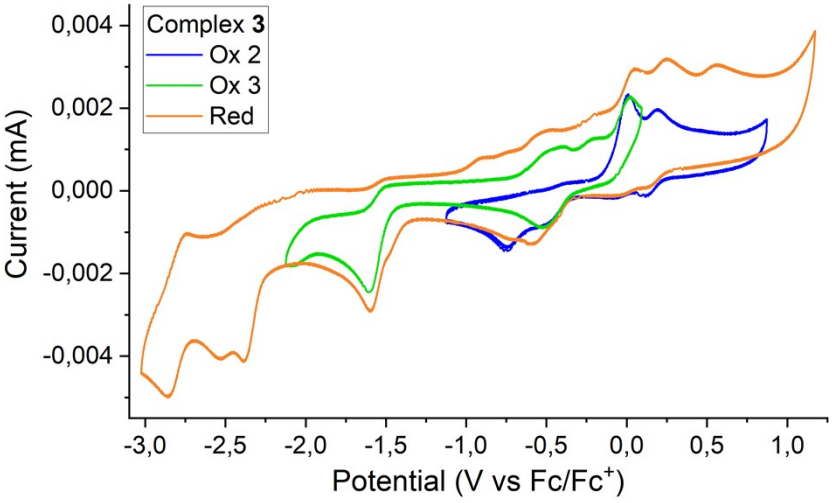


Figure S12: CV of complex **2** with different scan ranges: –1.2 V to 0.7 V vs ferrocene (blue); –2.2 V to 0.05 V vs ferrocene (green); –3.0 V to 1.15 V vs ferrocene (orange).



NMR spectra

Figure S13: ^{1}H-NMR spectrum of H_2**NDBA** (in dichloromethane-d_2)

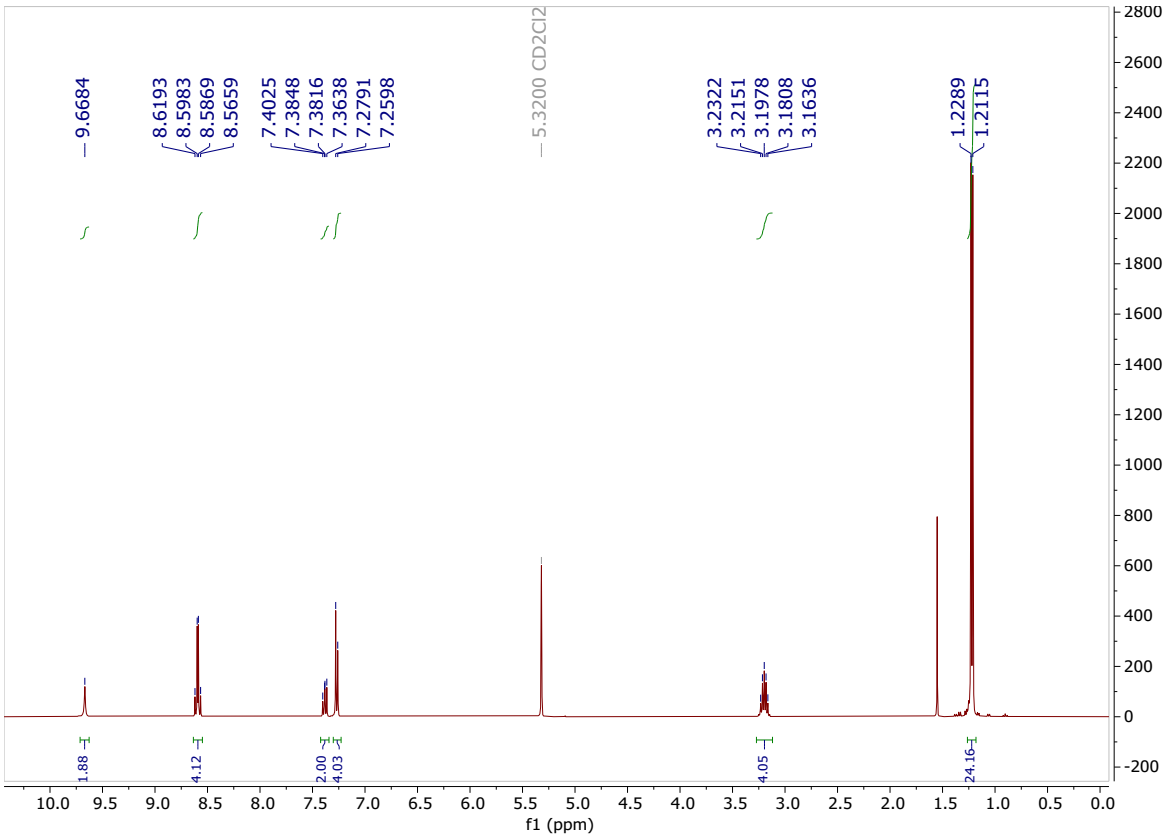


Figure S14: ^{13}C-NMR spectrum of H_2**NBDA** (in dichloromethane-d_2)

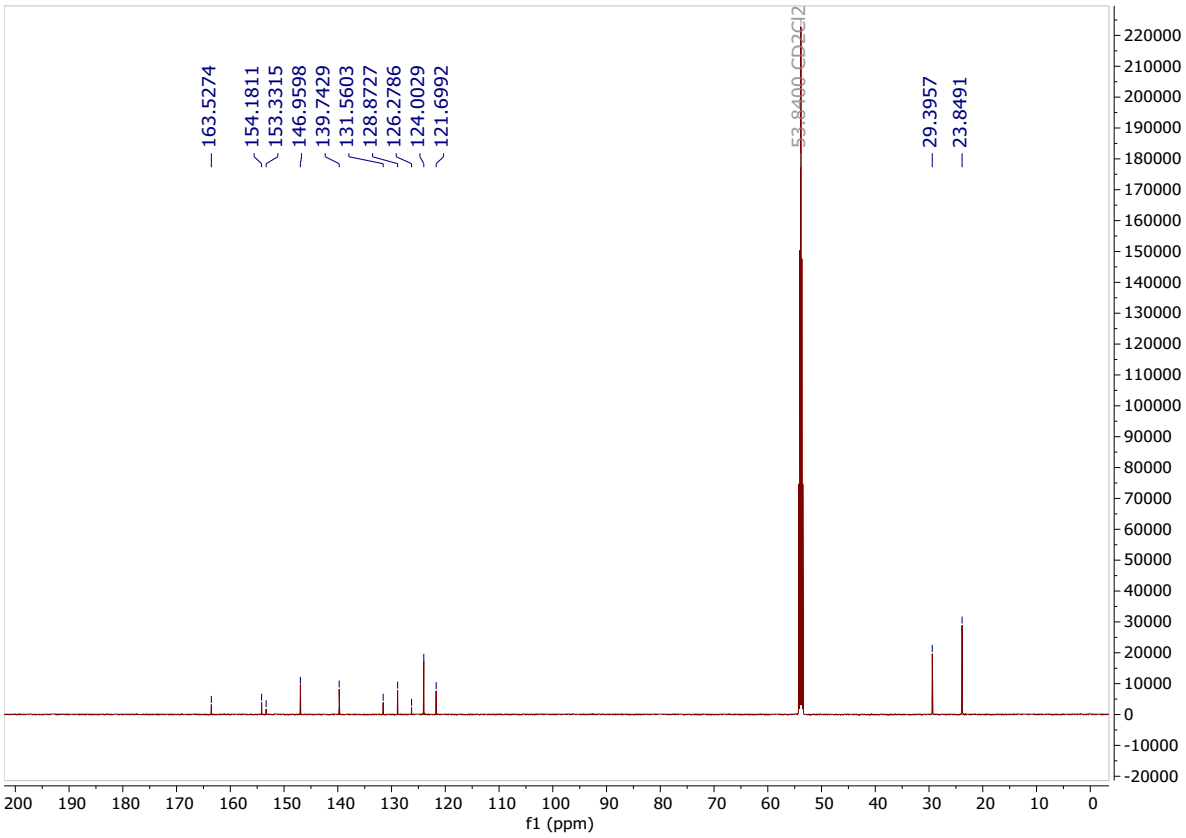


Figure S15: COSY spectrum of H_2**NBDA** (dichloromethane-d_2)

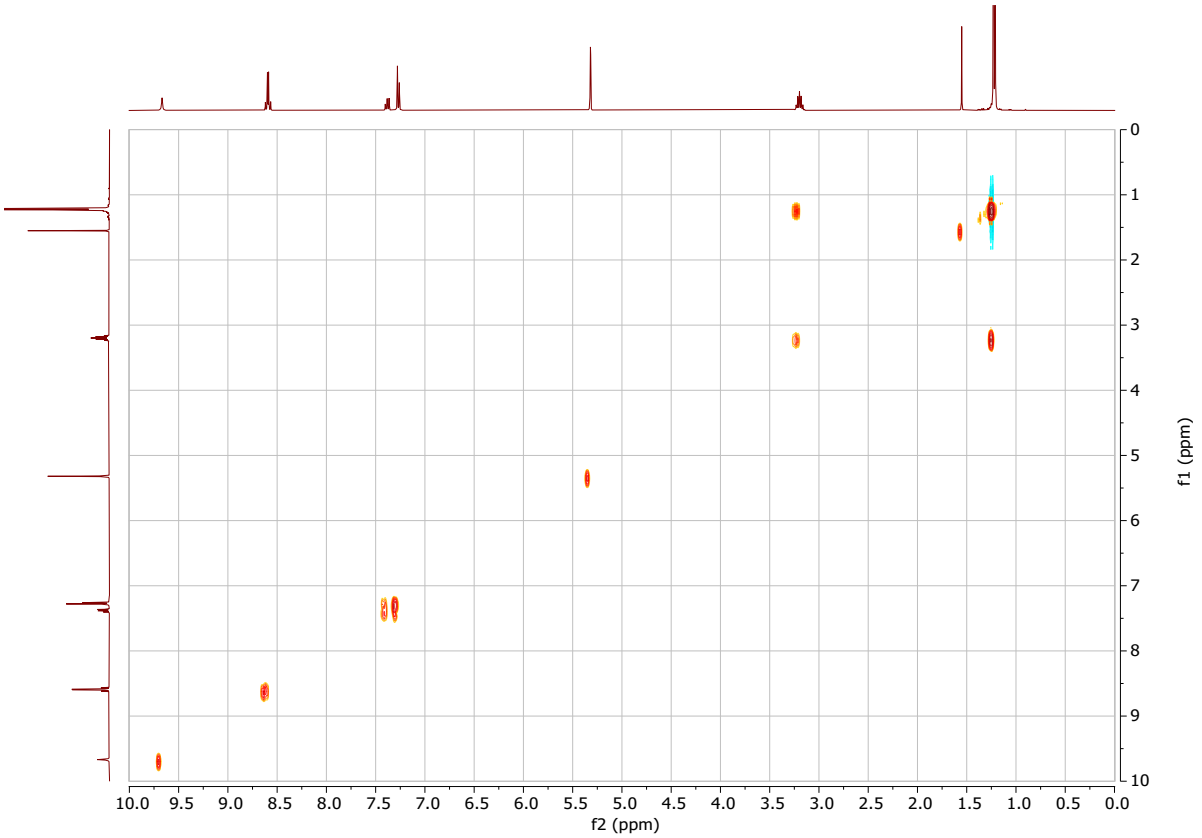


Figure S16: ^{1}H-NMR spectrum of **1** (dichloromethane-d_2)

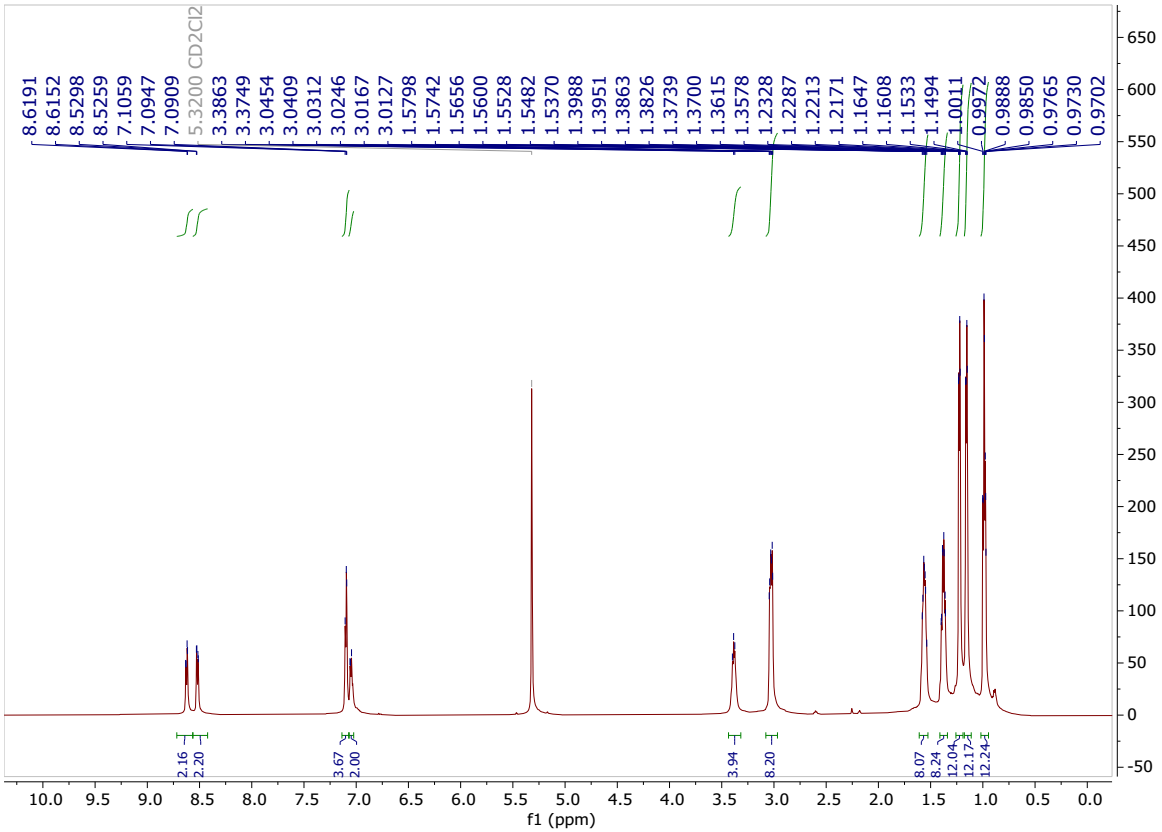


Figure S17: ^{13}C-NMR spectrum of **1** (dichloromethane-d_2)

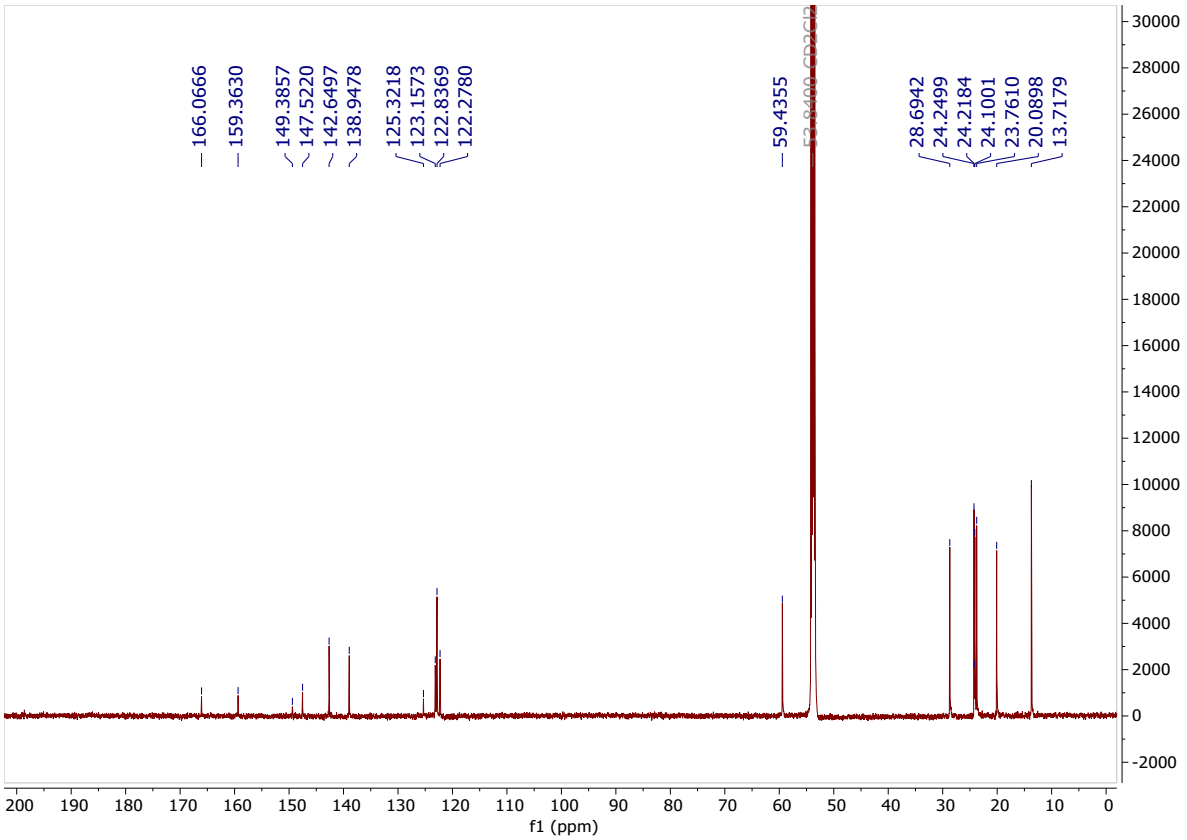


Figure S18: COSY spectrum of **1** (dichloromethane-d_2)

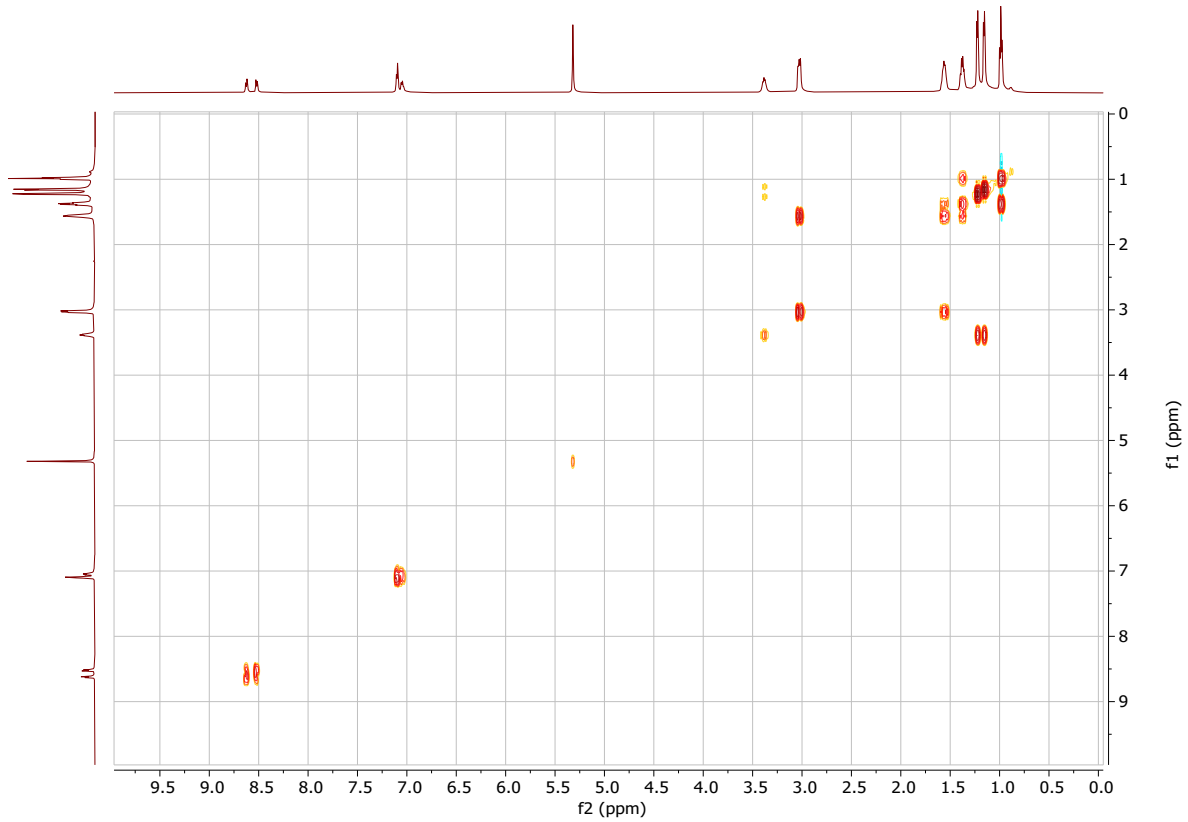


Figure S19: ^{1}H-NMR spectrum of **2** (THF-d_8)

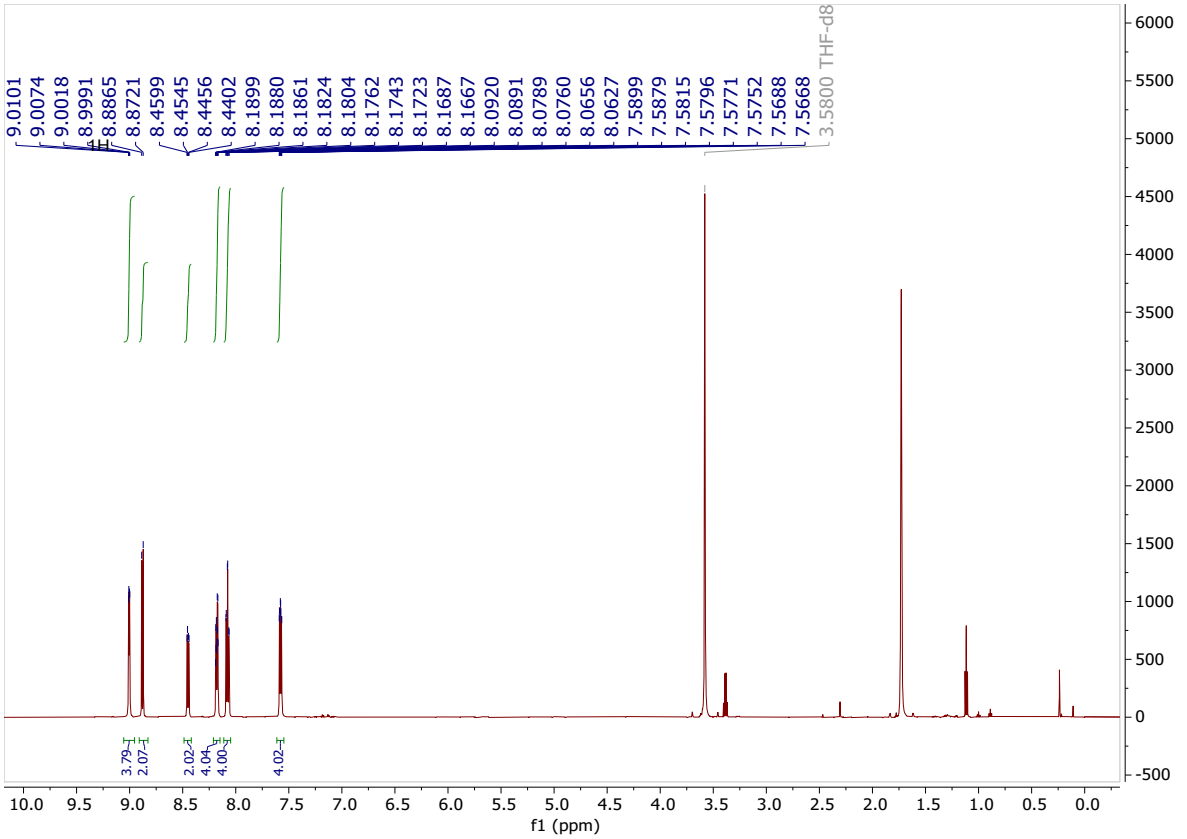


Figure S20: ^{13}C-NMR spectrum of **2** (THF-d_8)

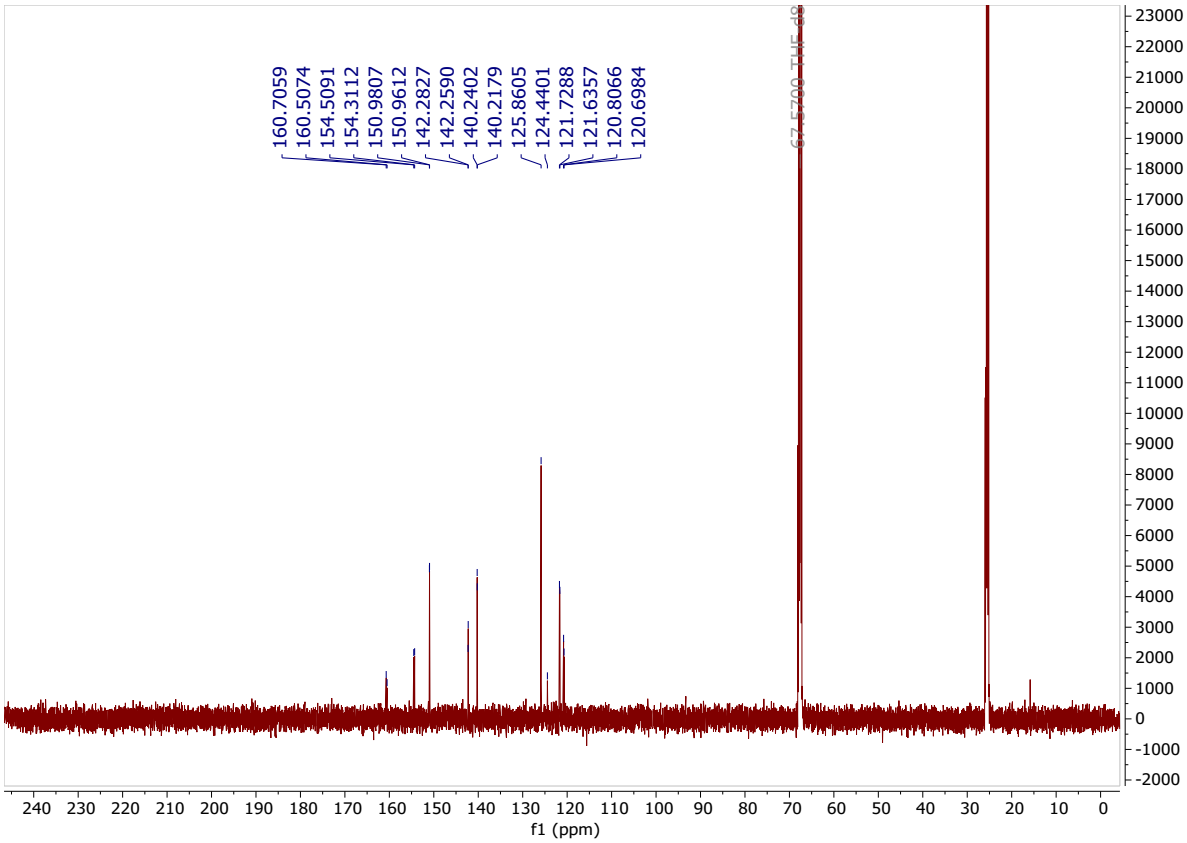


Figure S21: ^{19}F-NMR spectrum of **2** (THF-d_8)

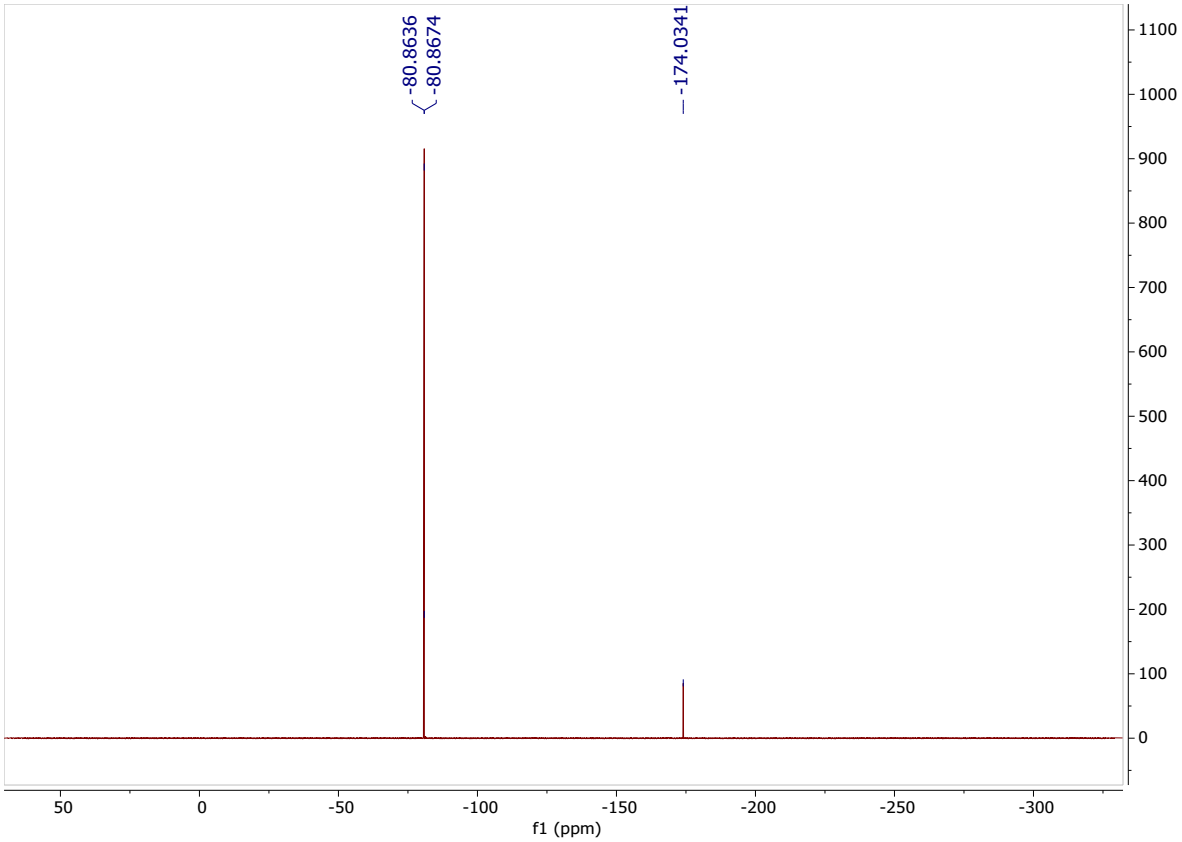


Figure S22: ^{1}H-NMR spectrum of **3** (dichloromethane-d_2)

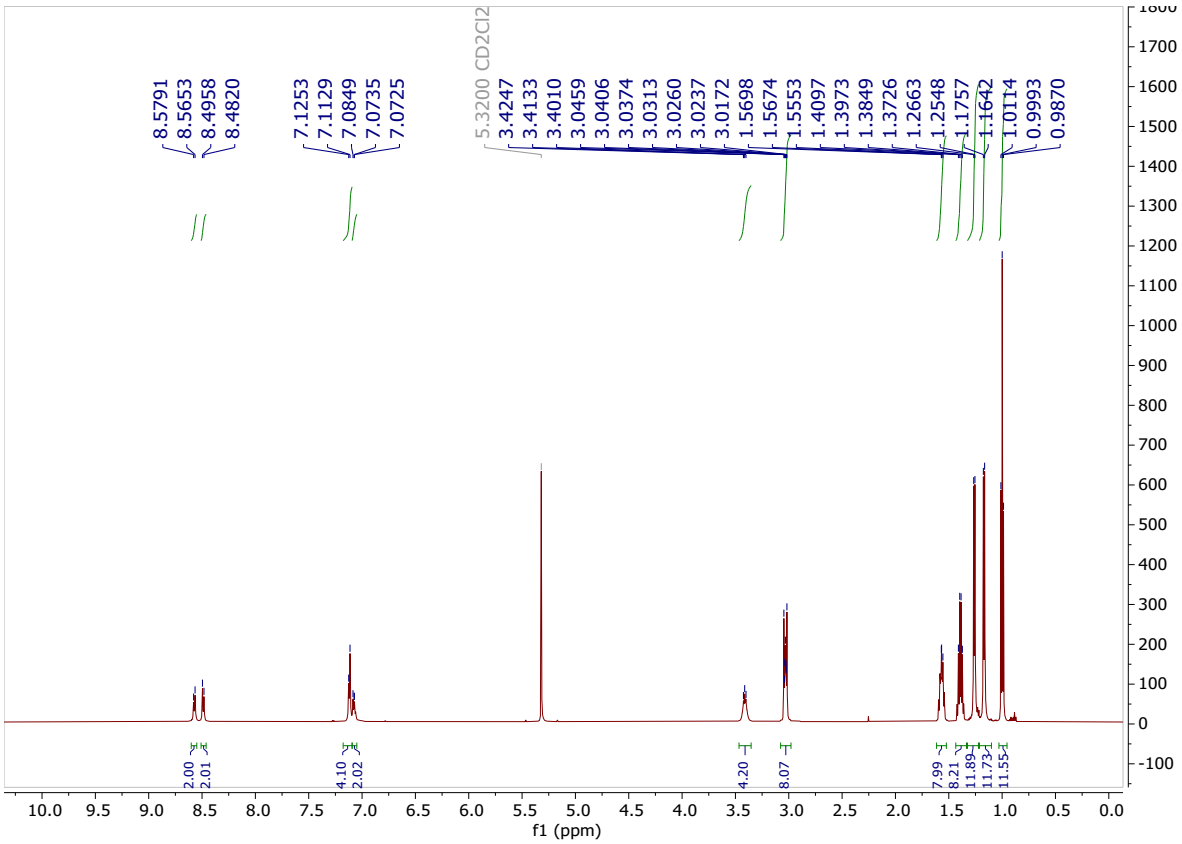


Figure S23: ^{13}C-NMR spectrum of **3** (dichloromethane-d_2)

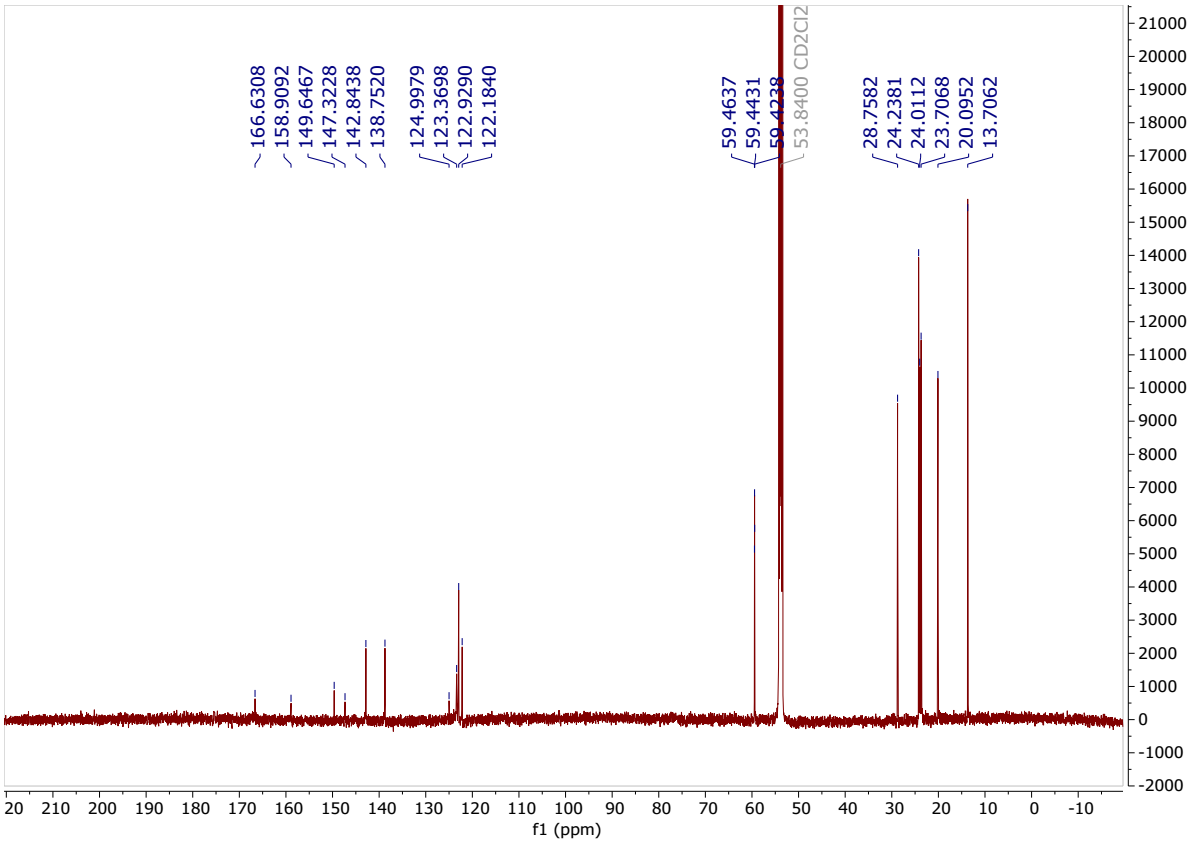


Figure S24: COSY spectrum of **3** (dichloromethane-d_2)

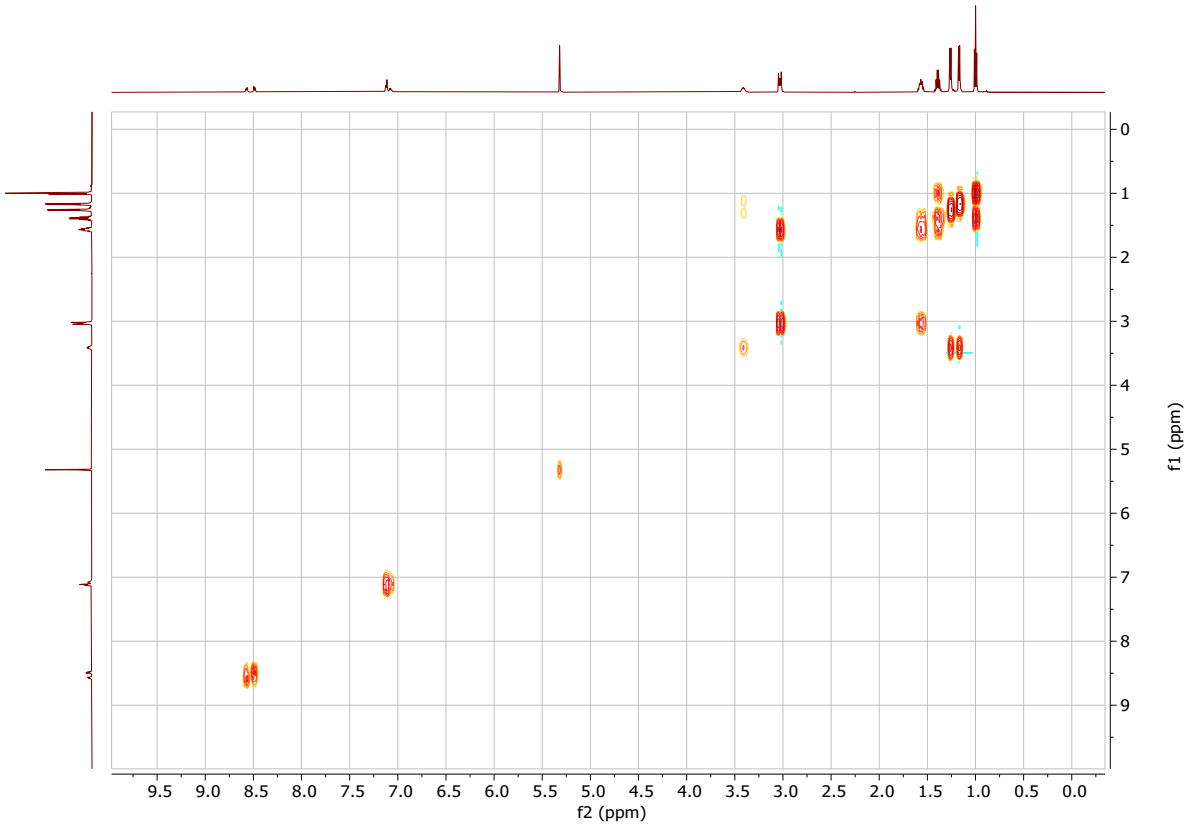


Figure S25: ^{1}H-NMR spectrum of **4** (dichloromethane-d_2)

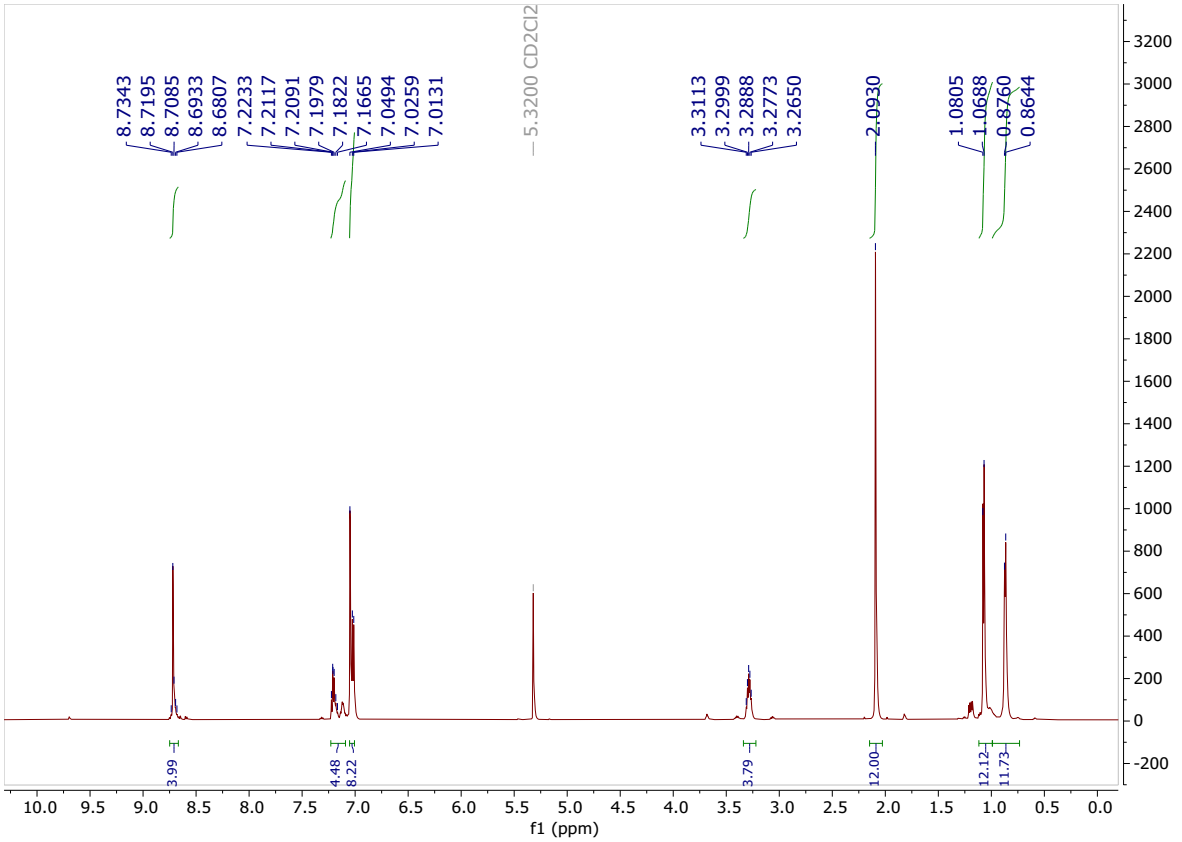


Figure S26: ^{13}C-NMR spectrum of **4** (dichloromethane-d_2)

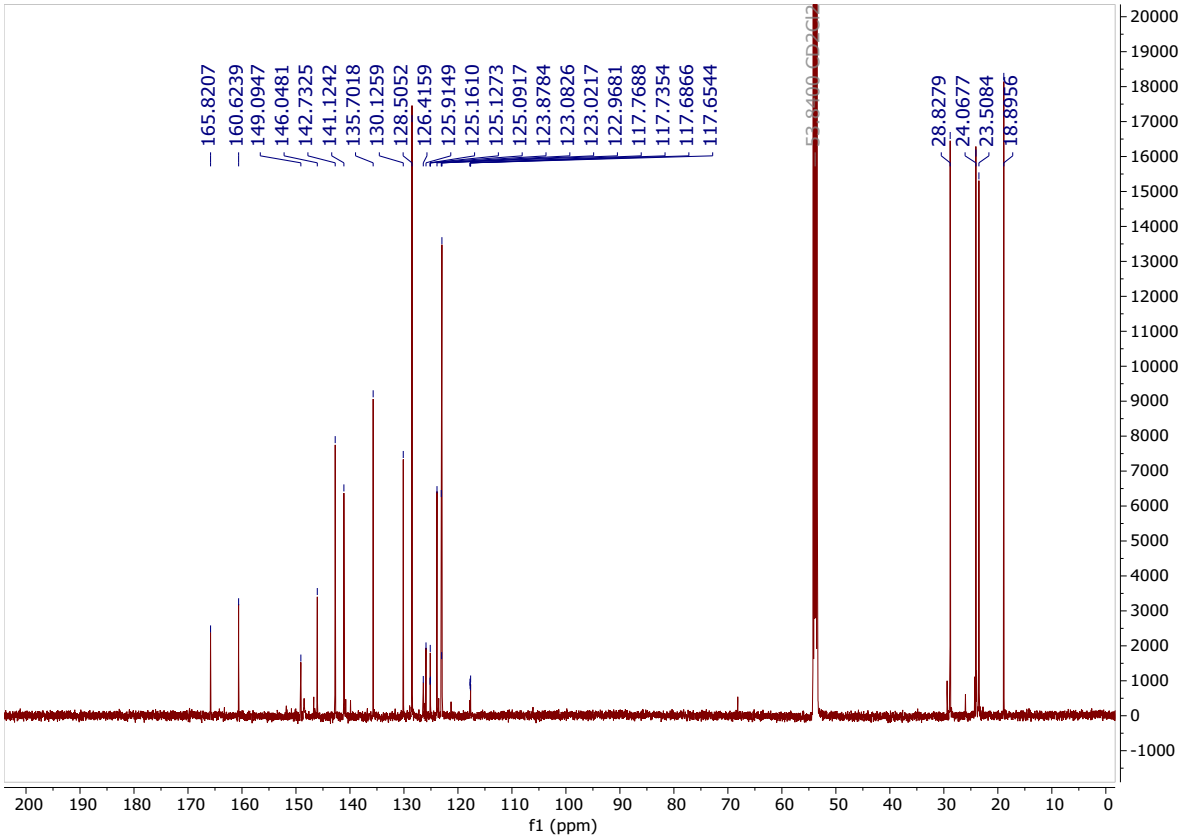
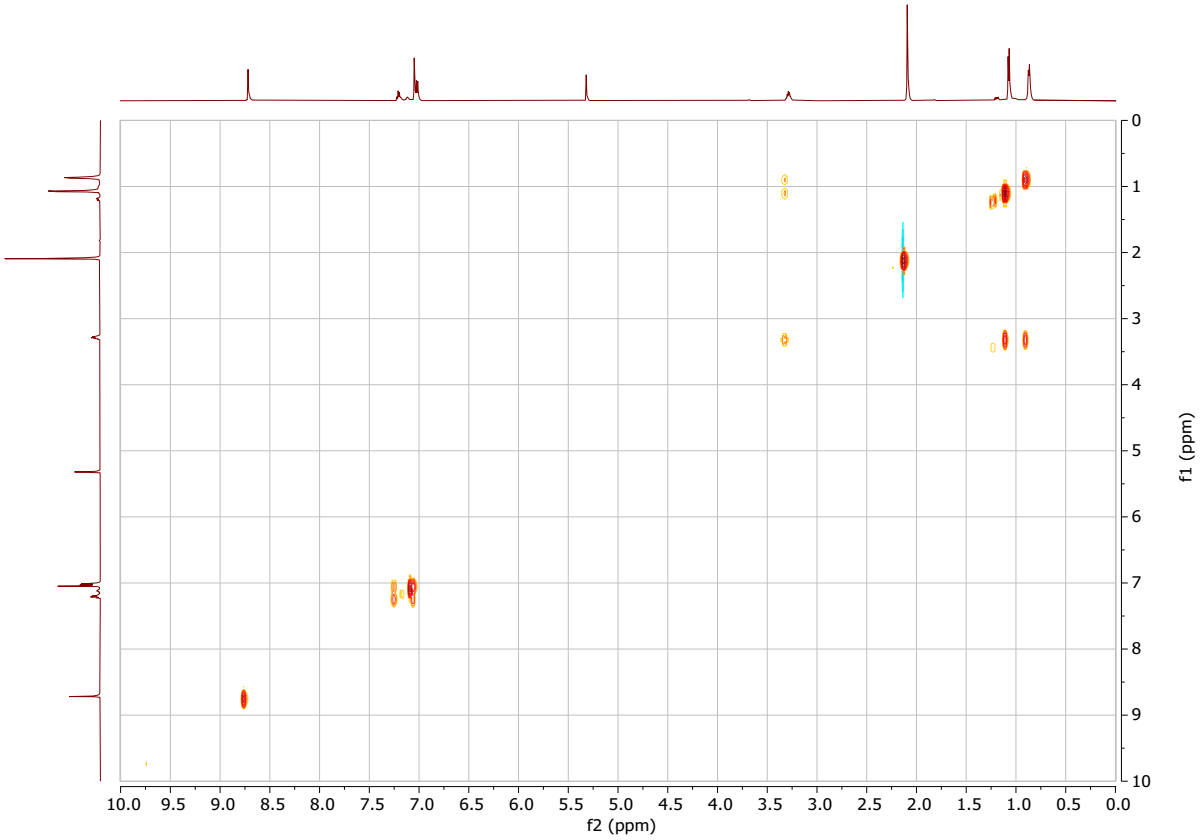


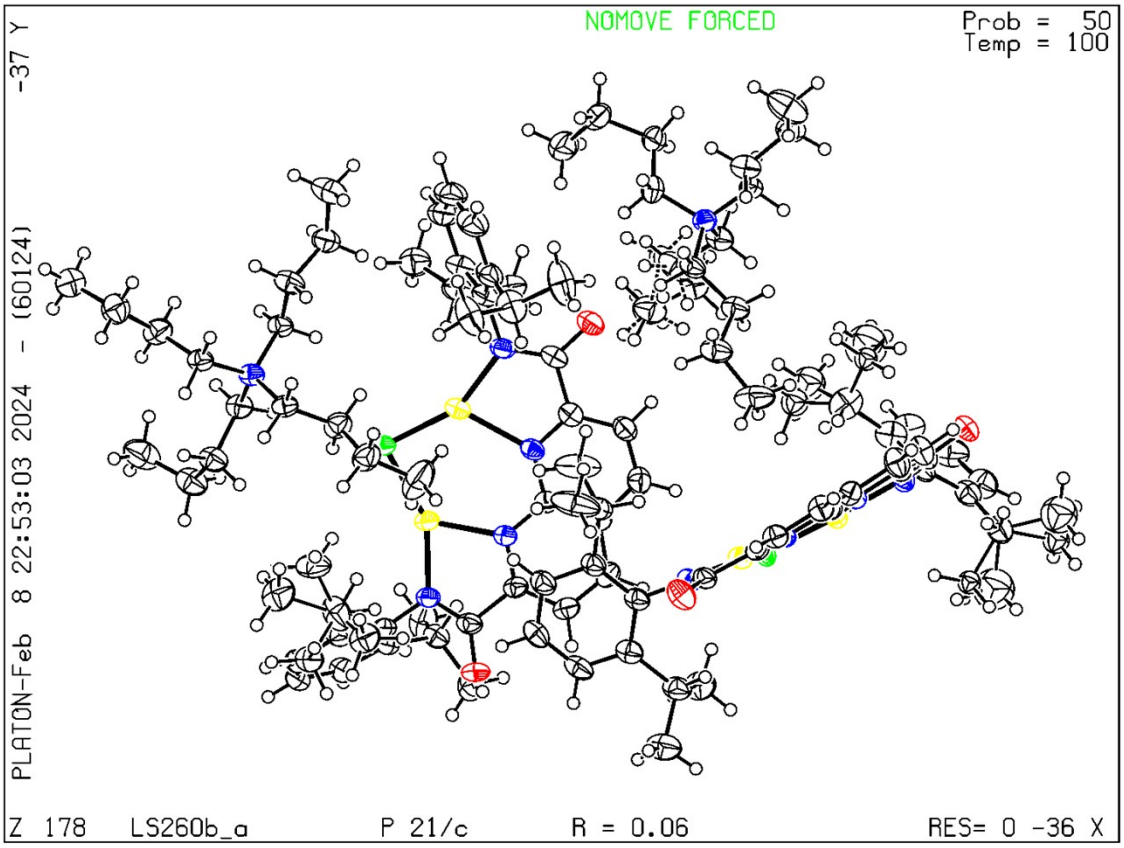
Figure S27: COSY spectrum of **4** (dichloromethane-d_2)



Solid-state molecular structures

Complex **1**

Identification code	$(NBu_4)[Cu_2(NBDA)(Cl)]$	LS260b_a
Empirical formula	C50 H74 Cl Cu2 N5 O2	
Formula weight	939.67	
Temperature	100(2) K	
Wavelength	0.7288 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 16.8395(12) Å	α= 90°.
	b = 28.680(2) Å	β= 90.644(3)°.
	c = 20.3614(14) Å	γ = 90°.
Volume	9833.2(12) $Å^3$	
Z	8	
Density (calculated)	1.269 Mg/m^3	
Absorption coefficient	1.028 mm^{-1}	
F(000)	4000	
Crystal size	0.080 x 0.065 x 0.060 mm^3	
Theta range for data collection	1.913 to 26.184°.	
Index ranges	-20<=h<=20, -34<=k<=34, -24<=l<=24	
Reflections collected	130939	
Independent reflections	18228 [R(int) = 0.0647]	
Completeness to theta = 25.930°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.941 and 0.810	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	18228 / 52 / 1100	
Goodness-of-fit on F^2	1.087	
Final R indices [I>2sigma(I)]	R1 = 0.0642, wR2 = 0.1706	
R indices (all data)	R1 = 0.0856, wR2 = 0.1901	
Extinction coefficient	n/a	
Largest diff. peak and hole	1.400 and -1.531 $e.Å^{-3}$	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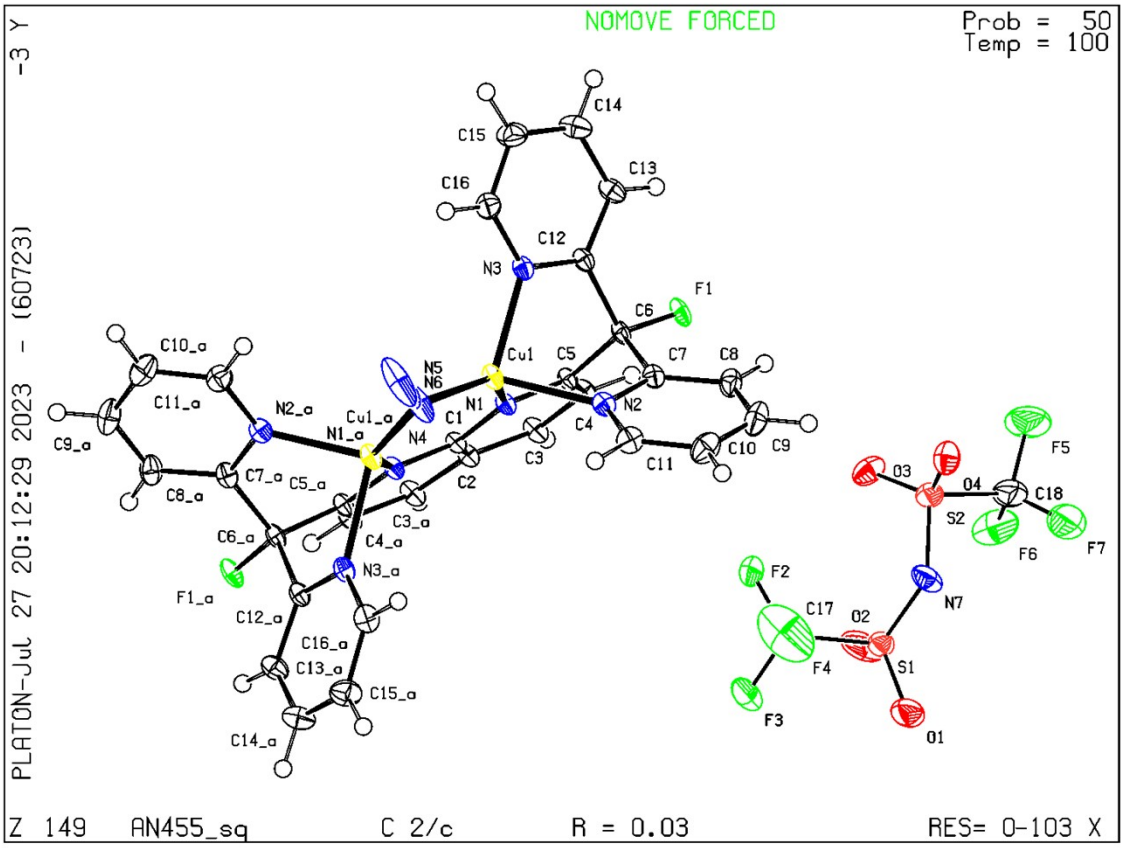
Temp = 100
-37 Y
PLATON-Feb 8 22:53:03 2024 - (60124)
Z 178 LS260b_a P 21/c R = 0.06 RES= 0 -36 X

Complex **2**

Identification code	$[Cu_2(DPFN)(N_3)](NTf_2)$	AN455_sq
Empirical formula	C40 H36 Cu2 F8 N10 O6 S2	
Formula weight	1095.99	
Temperature	100(2) K	
Wavelength	0.7288 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 15.4030(5) Å	α= 90°.
	b = 11.8625(4) Å	β= 105.5322(11)°.
	c = 24.8301(8) Å	γ = 90°.
Volume	4371.2(2) $Å^3$	
Z	4	
Density (calculated)	1.665 Mg/m^3	
Absorption coefficient	1.243 mm^{-1}	
F(000)	2224	
Crystal size	0.120 x 0.060 x 0.060 mm^3	
Theta range for data collection	2.254 to 34.136°.	
Index ranges	-23<=h<=22, -18<=k<=18, -38<=l<=38	
Reflections collected	45408	
Independent reflections	8312 [R(int) = 0.0544]	
Completeness to theta = 25.930°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.929 and 0.836	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8312 / 0 / 332	
Goodness-of-fit on F^2	1.034	
Final R indices [I>2sigma(I)]	R1 = 0.0348, wR2 = 0.0868	
R indices (all data)	R1 = 0.0465, wR2 = 0.0930	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.617 and -0.463 $e.Å^{-3}$	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Temp = 100
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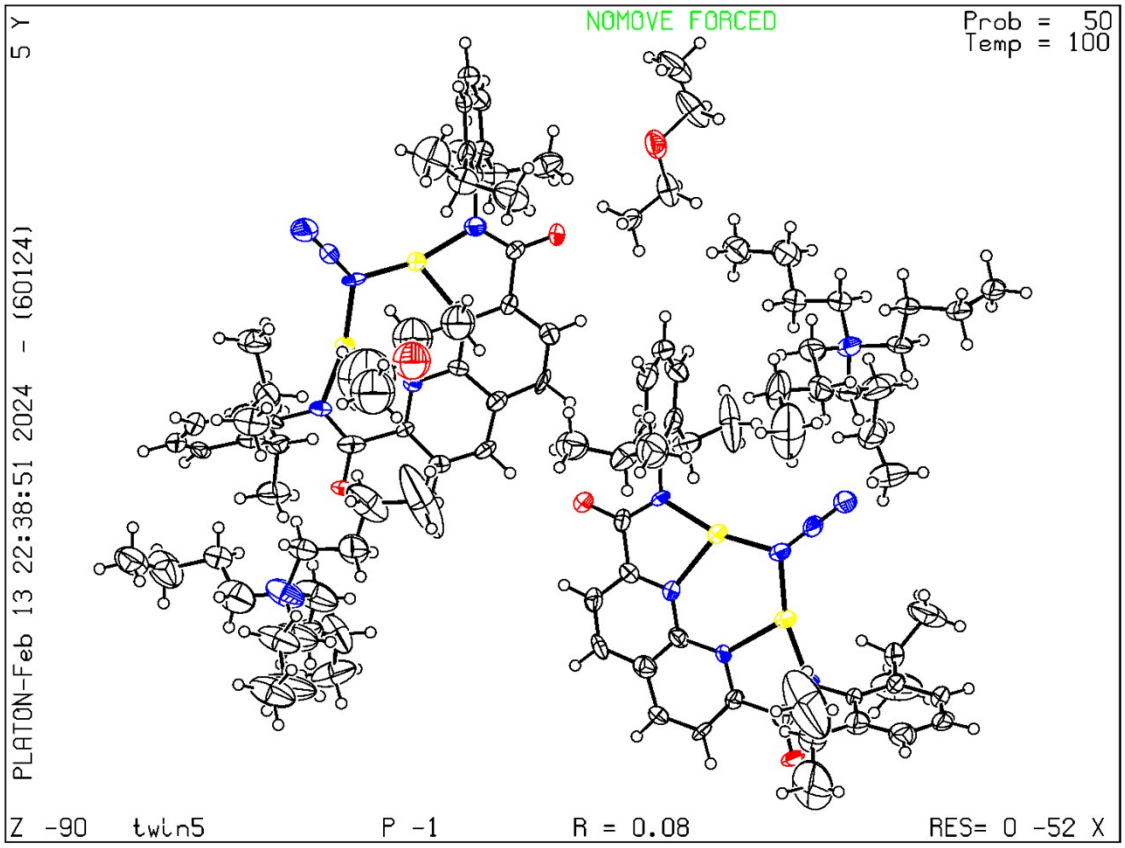
PLATON-Jul 27 20:12:29 2023 - (60723)
Z 149 AN455_sq C 2/c R = 0.03 RES= 0-103 X
C14
C15
C13
C16
C12
N3
F1
C6
C10_a
N5
N6
Cu1
C5
C7
C8
C11_a
N2_a
Cu1_a
N1
N2
C9_a
N1_a
C1
C4
C10
C9
N4
C3
C11
C7_a
C5_a
C2
C8_a
C3_a
C6_a
C4_a
N3_a
F1_a
C12_a
C16_a
C13_a
C15_a
C14_a
O3
O4
C18
F5
S2
F6
F7
F2
N7
C17
O2
F4
S1
F3
O1

Complex **3**

Identification code	$(NBu_4)[Cu_2(NBDA)(N_3)]$	twin5
Empirical formula	C104 H158 Cu4 N16 O5	
Formula weight	1966.61	
Temperature	100(2) K	
Wavelength	0.7288 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 14.6398(12) Å	α= 62.392(4)°.
	b = 20.0226(18) Å	β= 86.684(4)°.
	c = 21.2387(19) Å	γ = 86.526(4)°.
Volume	5503.5(9) $Å^3$	
Z	2	
Density (calculated)	1.187 Mg/m^3	
Absorption coefficient	0.874 mm^{-1}	
F(000)	2100	
Crystal size	0.120 x 0.120 x 0.040 mm^3	
Theta range for data collection	1.430 to 23.207°.	
Index ranges	-15<=h<=15, -18<=k<=21, 0<=l<=22	
Reflections collected	25195	
Independent reflections	10717 [R(int) = 0.0617]	
Completeness to theta = 23.207°	73.3 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.966 and 0.626	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	10717 / 245 / 1183	
Goodness-of-fit on F^2	1.030	
Final R indices [I>2sigma(I)]	R1 = 0.0795, wR2 = 0.2090	
R indices (all data)	R1 = 0.1034, wR2 = 0.2291	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.755 and -0.849 $e.Å^{-3}$	

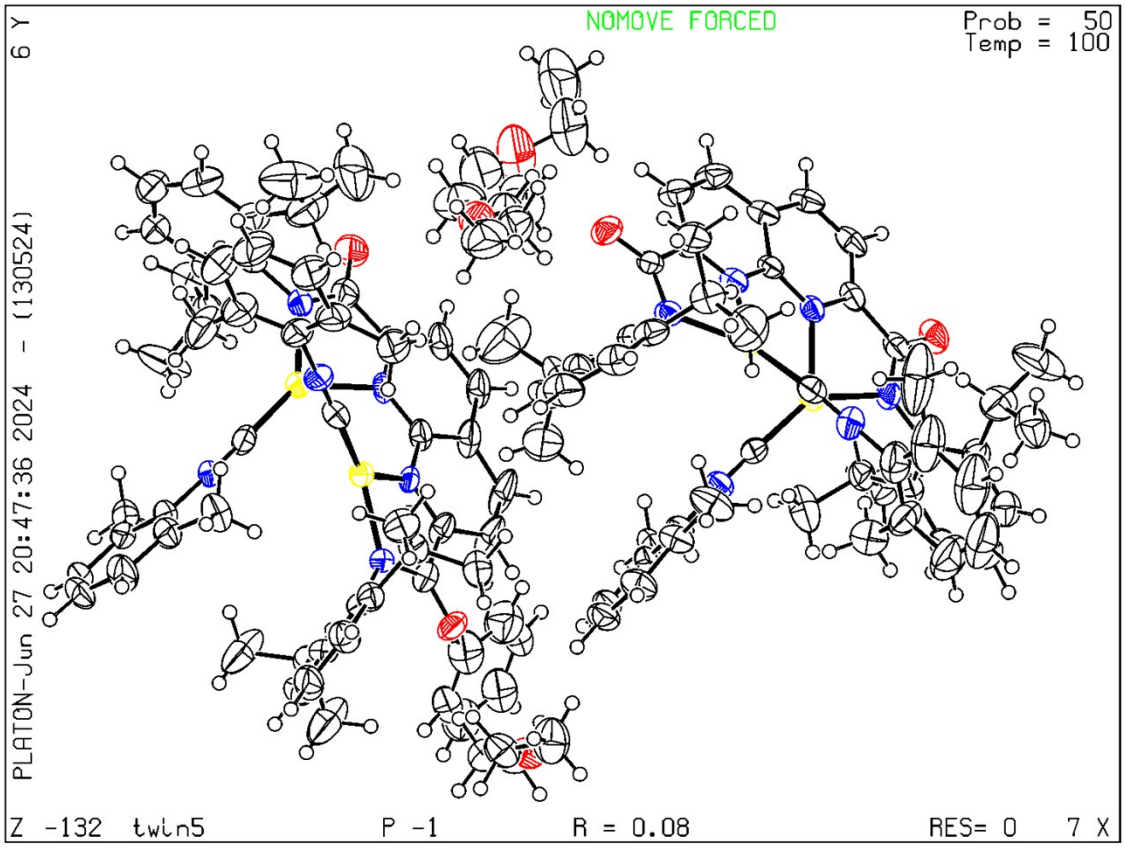


Notes on refinement:

- There is a solvent void in the structure which contains Q1 and Q2, 1.87 and 1.05 respectively, and symmetry generated version too. They do not fit the geometry of either ether or benzene and were left unmodelled.

Complex **4**

Identification code	$[Cu_2(NBDA)(XylylNC)_2]$	twin5
Empirical formula	C116.40 H139.40 Cu4 N12 O6.50	
Formula weight	2064.75	
Temperature	100(2) K	
Wavelength	1.54184 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 15.7286(2) Å	α= 86.084(2)°.
	b = 17.0404(2) Å	β= 76.810(2)°.
	c = 21.5628(6) Å	γ = 89.9820(10)°.
Volume	5613.00(19) $Å^3$	
Z	2	
Density (calculated)	1.222 Mg/m^3	
Absorption coefficient	1.300 mm^{-1}	
F(000)	2180	
Crystal size	0.290 x 0.220 x 0.140 mm^3	
Theta range for data collection	3.162 to 74.648°.	
Index ranges	-19<=h<=17, -21<=k<=21, -26<=l<=26	
Reflections collected	135790	
Independent reflections	31939 [R(int) = 0.0546]	
Completeness to theta = 67.684°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.00000 and 0.93562	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	31939 / 57 / 1304	
Goodness-of-fit on F^2	1.043	
Final R indices [I>2sigma(I)]	R1 = 0.0789, wR2 = 0.2409	
R indices (all data)	R1 = 0.0879, wR2 = 0.2499	
Extinction coefficient	n/a	
Largest diff. peak and hole	1.866 and -0.812 e.$Å^{-3}$	



Notes on refinement:

- The crystals were collected at a synchrotron beamline and even then they were still weakly diffracting. The diffraction pattern showed twinned spots with streaking and start to run into each other around 0.93 angstrom. The data were cut at 0.93 angstroms where the I/I(sigma) > 2, therefore on average the reflections were unobserved from this point.
- A full sphere of data was collected for this sample. The data had to be integrated as a twin and a large number of reflections were rejected for being partial overlap, this was made worse by the streaking of the reflection. A number of parameters were changed in the integration to try and improve this, such as "Minimum Common Volume %" and "Separation Factor", but no improvement could be found.

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