# An ethylene cross-bridged pentaazamacrocycle and its $Cu^{2+}$ complex: constrained ligand topology and excellent kinetic stability

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## Supplementary Information

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## 1. Experimental Section

### 1.1. General.

15aneN5 was synthesized according to a modified literature procedure.[1] Glyoxal (40% wt in water), methyl iodide (99%), sodium borohydride (98%), $NH_4PF_6$, and $CuCl_2 . 2H_2O$ were purchased from Aldrich Chemical Co. All solvents were of reagent grade and were dried, when necessary, by accepted procedures. Elemental analyses were performed by on a Perkin-Elmer EA2400 elemental analyzer. Electrospray mass spectra were collected on a Shimadzu LCMS 2020 Electrospray Mass Spectrometer. NMR spectra were obtained on a Varian Bruker AVANCE II 300 MHz NMR Spectrometer instrument. UV-Visible spectra were obtained on a Beckman Coulter DU800 UV-Vis Spectrometer.

### 1.2 Syntheses.

**Synthesis of (5) 1,4,7,10,13-pentaazatetracyclo[8.5.2.0$^{4,17}$.0$^{13,16}$]heptadecane**:

To a 100 ml round bottom flask was added 2.00 g (9.29 mmol) of 15aneN5, 50 mL of dry MeOH, and a stir bar. The 15aneN5 dissolved with stirring and was then cooled to an ice-water bath temperature (~5

°C). 1 eq of glyoxal from a 40% by mass in water glyoxal solution (9.29 mmol, 1.348 g of solution) was dissolved in 10 mL of dry MeOH and added to an addition funnel. The glyoxal solution was added to the stirring, cooled 15aneN5 dropwise over ~30 minutes and open to the air. Upon complete addition, a stopper was placed in the round bottom flask and the reaction was allowed to warm to room temperature and stirred overnight. Next, the solvent was removed under vacuum to yield a yellow oil, which was dried under a high vacuum for 30 minutes to remove remaining solvents. The residue was dissolved in stirred 80 mL diethyl ether for 60 minutes, and then filtered through packed Celite to remove any insoluble polymers. The solvent was removed by evaporation and the pale yellow oil product dried overnight. Yield: 1.87 g (85%). ESI-MS$^+$: m/z = 238 (LH$^+$). Elemental Analysis: Calc. (Found) for $C_{12}H_{23}N_5 \cdot H_2O \cdot 0.2C_4H_{10}O$: C 56.90 (57.17), H 10.07 (10.05), N 25.92 (26.05). $^1$H NMR (300 MHz, $CDCl_3$, 298 K) *See manuscript for discussion of likelihood of more than one isomers being present. Integration was normalized to 23H for $^1$H spectrum as all likely isomers would have that in common. Integration is given as an indication of the isomeric mixture, although no quantification of isomer number or ratio has been attempted*. $\delta$ [ppm] = 3.69 (d, 0.308H), 3.60-3.45 (m, 1.189H), 3.34 (m, 0.811H), 3.22 (m, 1.014H), 3.16-2.83 (m, 10.136H), 2.83-2.58 (m, 5.368H), 2.58-2.36 (m, 2.560H), 2.35 (d, 0.412H), 2.31 (m, 0.843H), 2.01 (d, 0.341H). $^{13}$C NMR (75.47, MHz, $CDCl_3$, 298 K) $\delta$ [ppm] = 85.183, 80.897, 79.310, 78.341, 59.616, 54.492, 54.242, 51.000, 50.886, 50.535, 49.849, 49.214, 48.748, 48.600, 47.673, 47.390.

**Synthesis of (3) 4,7,13-trimethyl-1,4,7,10,13-pentaazabicyclo[8.5.2]heptadecane**:
To a 100 ml round bottom flask was added 0.729 g (3.07 mmol) of **5**, 25 mL of dry MeCN, and a stir bar. Separately, 10 eq (30.70 mmol, 4.358 g) iodomethane was dissolved in 20 mL dry MeCN. The iodomethane solution was added to the stirring solution of **5** and the solution stirred at room temperature for 5 days. Air was bubbled through the solution in a chemical fume hood to evaporate the excess iodomethane with the outlet air bubbled through a water/$Et_3N$ solution to trap out the iodomethane. The solution was then evaporated to dryness. The white solid residue was broken up and stirred with 25 ml DCM (to remove any mono- or di-methylated compound) for 30 minutes and the remaining white solid filtered off. This solid was then used without purification (and is believed to contain multiple constitutional and methylation isomers) in the following synthetic step. The solid was suspended in 120 mL of 95% EtOH in a 500 ml round bottom flask. 20 eq (61.40 mmol, 2.323 g) of sodium borohydride was added in portions over 5 minutes and then the reaction stirred under nitrogen for 5 days at room temperature. Excess sodium borohydride was decomposed with 6 M HCl to a pH = 2. The solution was then evaporated to dryness. The residue was dissolved in 100 mL 30% aqueous KOH and extracted with chloroform. The chloroform layers were collected, dried over sodium sulfate, and evaporated to the brown oil product **3**. Yield: 0.230 g (32%). ESI-MS$^+$: m/z = 284 (LH$^+$). Elemental Analysis: Calc. (Found) for $C_{15}H_{33}N_5 \cdot CHCl_3$: C 47.70 (47.62), H 8.51 (8.89), N 17.39 (17.50). $^1$H NMR (300 MHz, $CDCl_3$, 298 K) *See manuscript for discussion of likelihood of more than one isomers being present. Integration was normalized to 33H for $^1$H spectrum as all likely isomers would have that in common. Integration is given as an indication of the isomeric mixture, although no quantification of isomer number or ratio has been attempted*. $\delta$ [ppm] = 3.72-3.29 (m, 3.00H), 3.29-3.13 (m, 1.72H), 3.11-2.70 (m, 10.41H), 2.67 (s, 1.87 H), 2.64-2.24 (m, 11.32H), 2.22 (s, 3.50H), 2.18-2.04 (m, 1.18H). $^{13}$C NMR (75.47, MHz, $CDCl_3$, 298 K) $\delta$ [ppm] = 53.566, 53.430, 49.082, 47.184, 43.164, 42.619.

**Synthesis of [Cu(3)]($PF_6$)$_2$:**
In a 4 dram vial and open to the air, 0.525 g (1.50 mmol) of **3** was dissolved in 5 mL dry MeOH. Separately, 0.256 g (1.50 mmol) of $CuCl_2 \cdot 2H_2O$ was dissolved in 5 mL dry MeOH. The $CuCl_2$ solution was then added dropwise by pipet to the stirring ligand solution. A dark green solution with some brown precipitate formed. The reaction was left to stir overnight at room temperature. Any undissolved solids were removed by filtration and the stirred dark green filtrate* was treated with dropwise addition of $NH_4PF_6$ (0.900 g, 5.52 mmol) which had been dissolved in 5 mL dry MeOH. A dark blue precipitate formed immediately. The suspension was stored overnight at -10 °C to complete the precipitation. The dark blue product was filtered on a glass frit, washed with MeOH and diethyl ether, and dried under vacuum. Yield: 0.374 g (39%). ESI-MS$^+$: m/z = 178 [CuL$^{2+}$] and m/z = 381 [CuL(MeOH)]$^+$. $\lambda_{max}$ ($H_2O$) = 575nm ($\varepsilon$ = 285 $M^{-1}$ $cm^{-1}$). Elemental Analysis: Calc. (Found) for [Cu($C_{15}H_{33}N_5$)]($PF_6$)$_2$ $\cdot$ 0.3$NH_4PF_6$: C 26.27 (26.40), H 5.03 (4.86), N 10.82 (10.65).

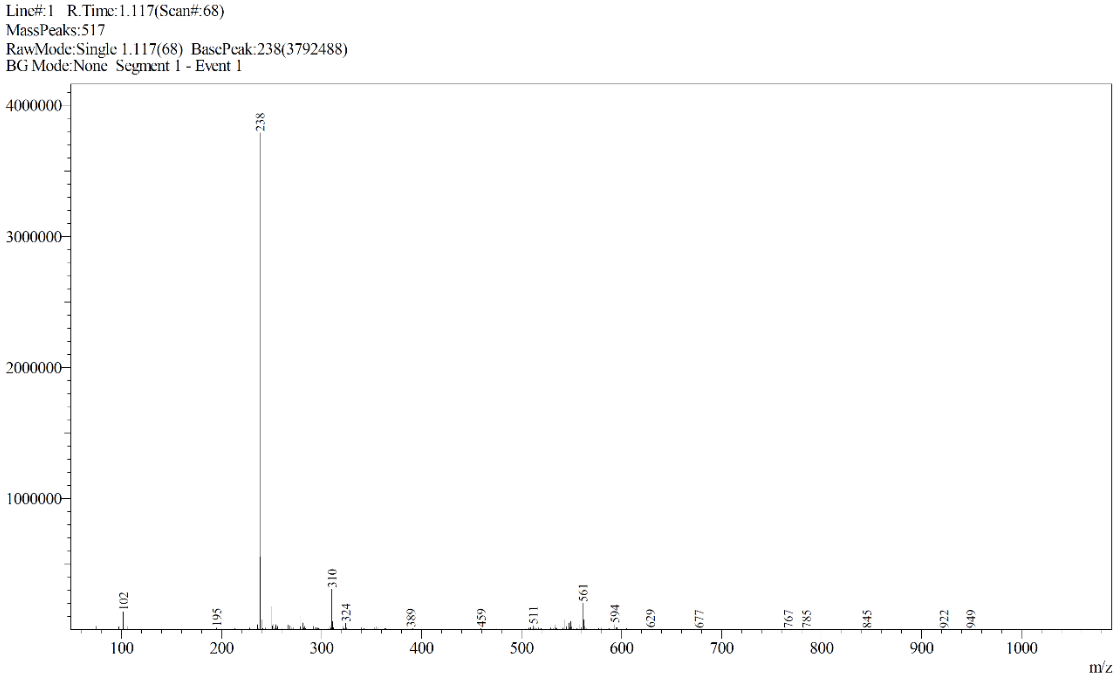
*Crystals suitable for X-ray diffraction were obtained from evaporating a portion of the dark green filtrate to dryness, without adding any $NH_4PF_6$. The resulting blue-violet solid was dissolved in $MeNO_2$ open to the air, and diethyl ether diffusion into this solution caused the formation of violet crystals.

### 1.3. Acid Decomplexation Studies.

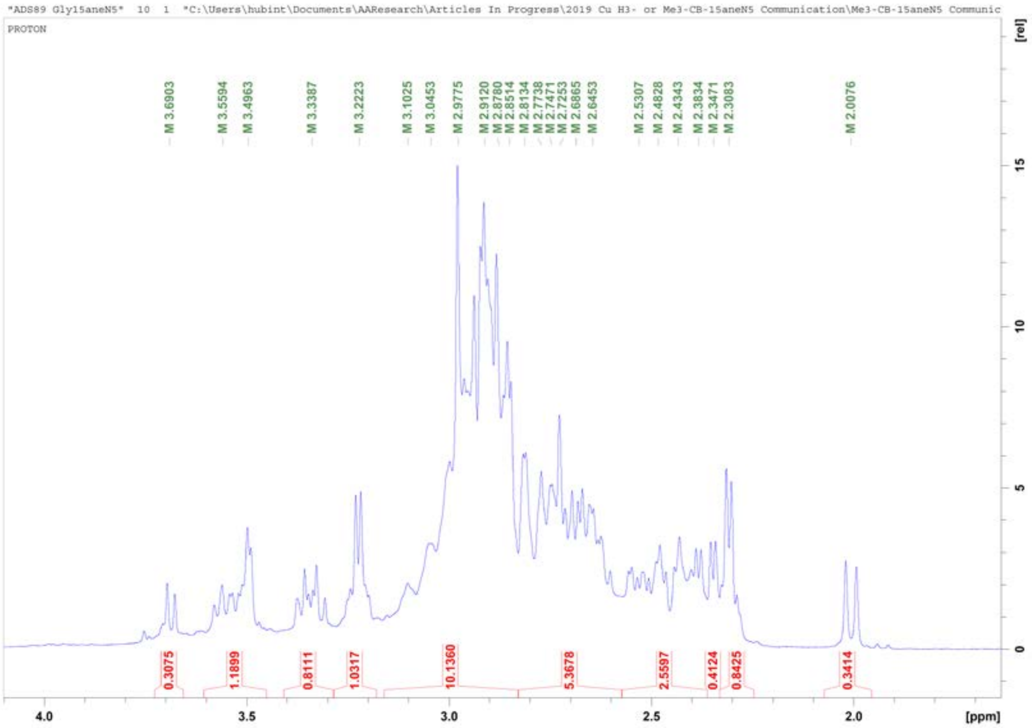
[Cu(**3**)]($PF_6$)$_2$ was used at 1 mM. The complex's lone d−d absorption was recorded on a Beckman Coulter DU800 UV-Vis Spectrometer in 5 M HCl at and 50 °C over time. A relatively fast (~16 h) process occurs, resulting in a shift from $\lambda_{max}$ = 571 nm ($\varepsilon$ = 397 $M^{-1}$ $cm^{-1}$) to $\lambda_{max}$ = 701 nm ($\varepsilon$ = 162 $M^{-1}$ $cm^{-1}$). No further shift of $\lambda_{max}$ occurs after this time, only a decrease in absorption as the complex slowly decomposes over the multiple days of the experiment. Pseudo-first-order conditions applied to the slow process after the solution reaches its final $\lambda_{max}$ = 701 nm allowed the calculation of the half-life from the slope of the linear ln(absorbance) versus time plot. The pseudo first order rate constant k = 0.00547 $h^{-1}$ was calculated, giving a half-life (0.693/k) of $t_{1/2}$ = 126 h = 5.3 d.

The nature of the fast initial process resulting in the shift of $\lambda_{max}$ is not completely understood. Simple binding of $Cl^-$ by the complex as a cause was ruled out by a parallel experiment in 2.5 M NaCl (~ saturated in our hands) instead of 5 M HCl, where $\lambda_{max}$ = 575 nm ($\varepsilon$ = 285 $M^{-1}$ $cm^{-1}$) and does not shift or decrease in intensity. It is possible that a protonation occurs in the fast step prior to decomposition and that this protonation, followed by interaction of the protonated complex with chloride that causes the wavelength shift. An additional experiment in 5 M $HClO_4$ was carried out to test this hypothesis. Similar to the 5 M HCl experiment, a relatively fast process occurs over the first ~23 hours of the experiment, followed by slow decomposition of the complex. In $HClO_4$, however, there is no shift of $\lambda_{max}$, which is at 564 nm. The fast process includes a reduction of $\varepsilon$ = 268 $M^{-1}$ $cm^{-1}$ to $\varepsilon$ = 195 $M^{-1}$ $cm^{-1}$ within the first ~23 hours. After this time, the change in the spectrum is minimal with the $\varepsilon$ being reduced only to ($\varepsilon$ = 185 $M^{-1}$ $cm^{-1}$) over the next ~5 days. It appears a "fast" protonation process occurs in both acids, but that $Cl^-$ assists the decomposition whereas $ClO_4^-$ does not. The $t_{1/2}$ of the "slow" decomposition step calculated in 5 M $HClO_4$, 47.6 d, is much longer than in 5 M HCl, an observation also true for the cross-bridged tetraazamacrocycle $Cu^{2+}$ complexes.[2]

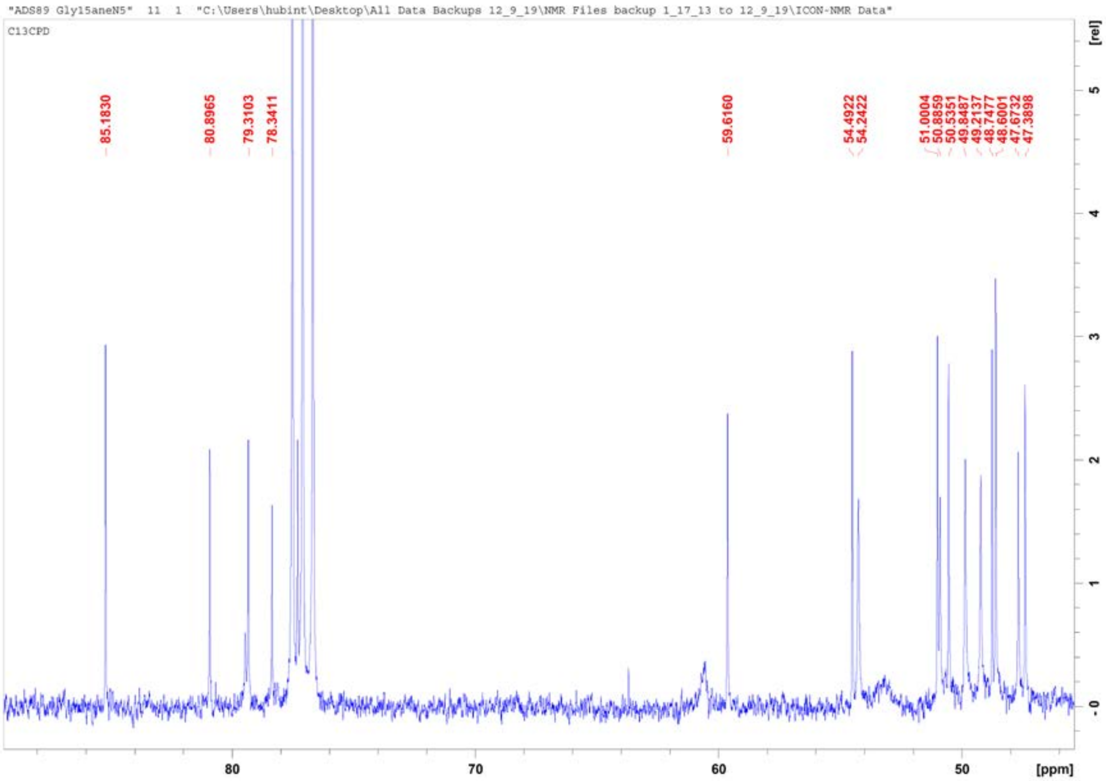
## 2. Mass Spectra and $^{1}$H and $^{13}$C NMR Spectra of 5, 3, and Cu(3)$^{2+}$



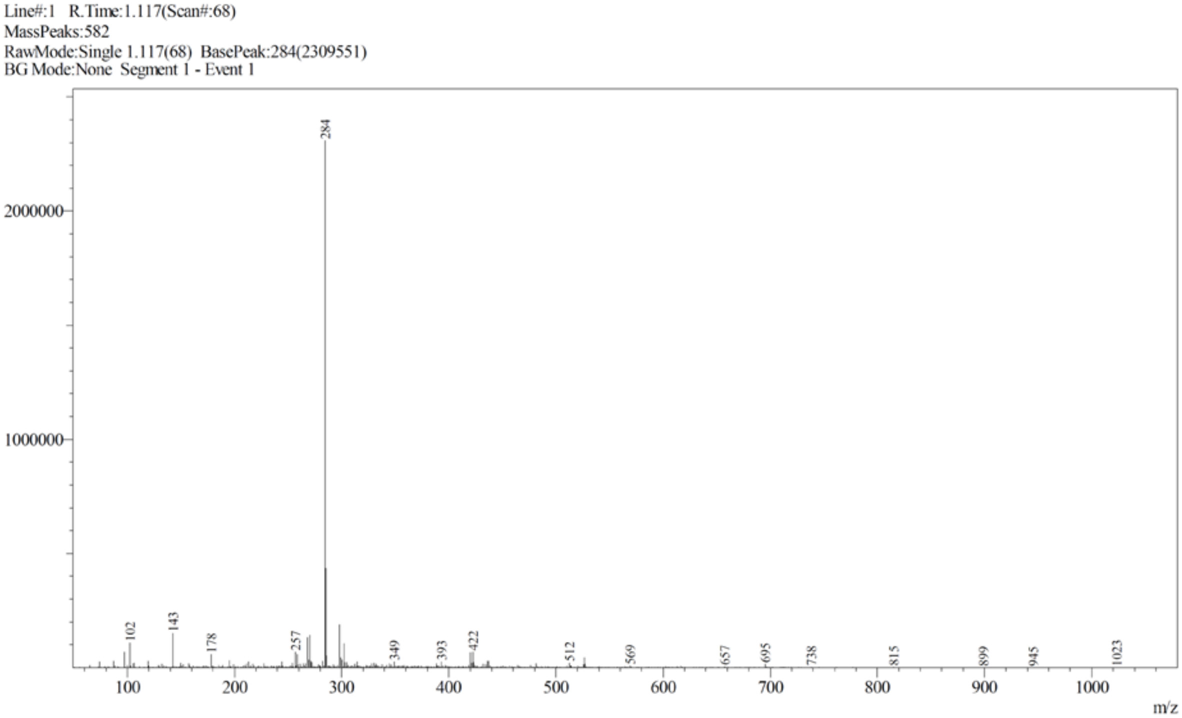
ESI-MS$^{+}$ mass spectrum of **5**.



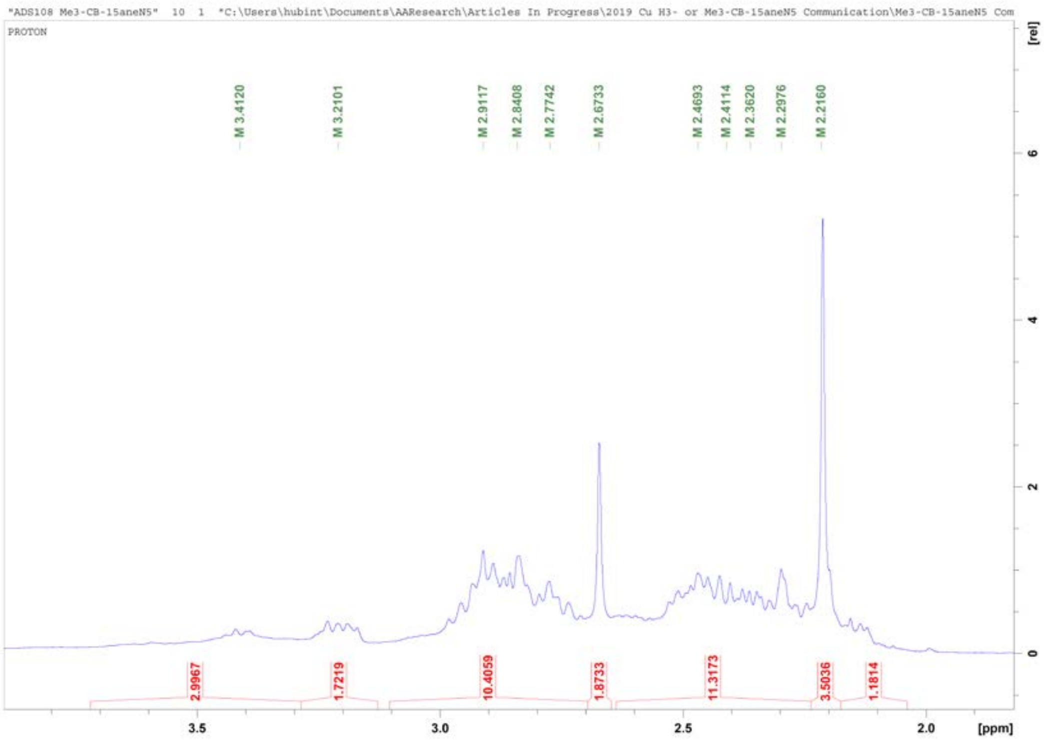
$^{1}$H NMR spectrum of **5** (300 MHz, CDCl$_3$, 298 K).



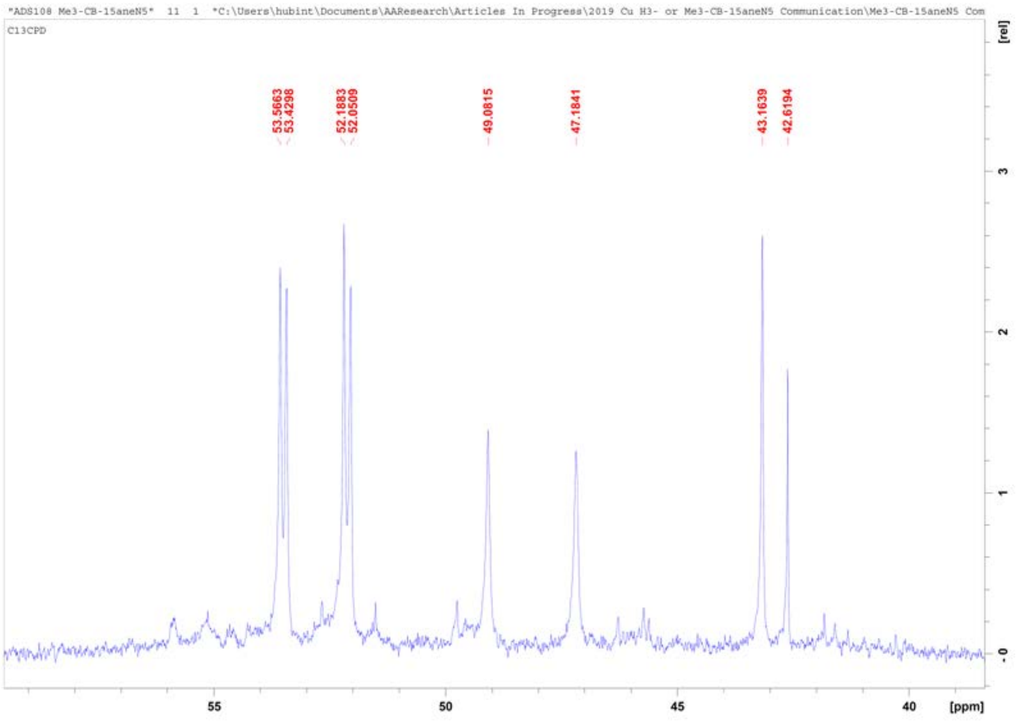
$^{13}$C NMR spectrum of **5** (75 MHz, $CDCl_3$, 298 K).



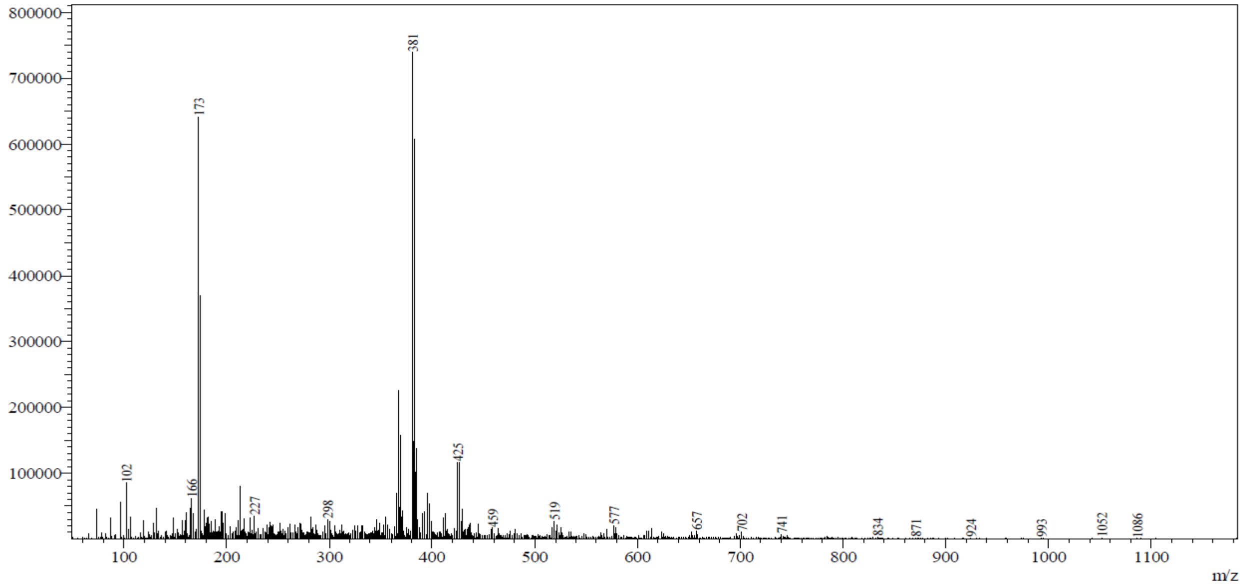
ESI-MS$^+$ mass spectrum of **3**.



$^{1}$H NMR spectrum of **3** (300 MHz, CDCl$_3$, 298 K).

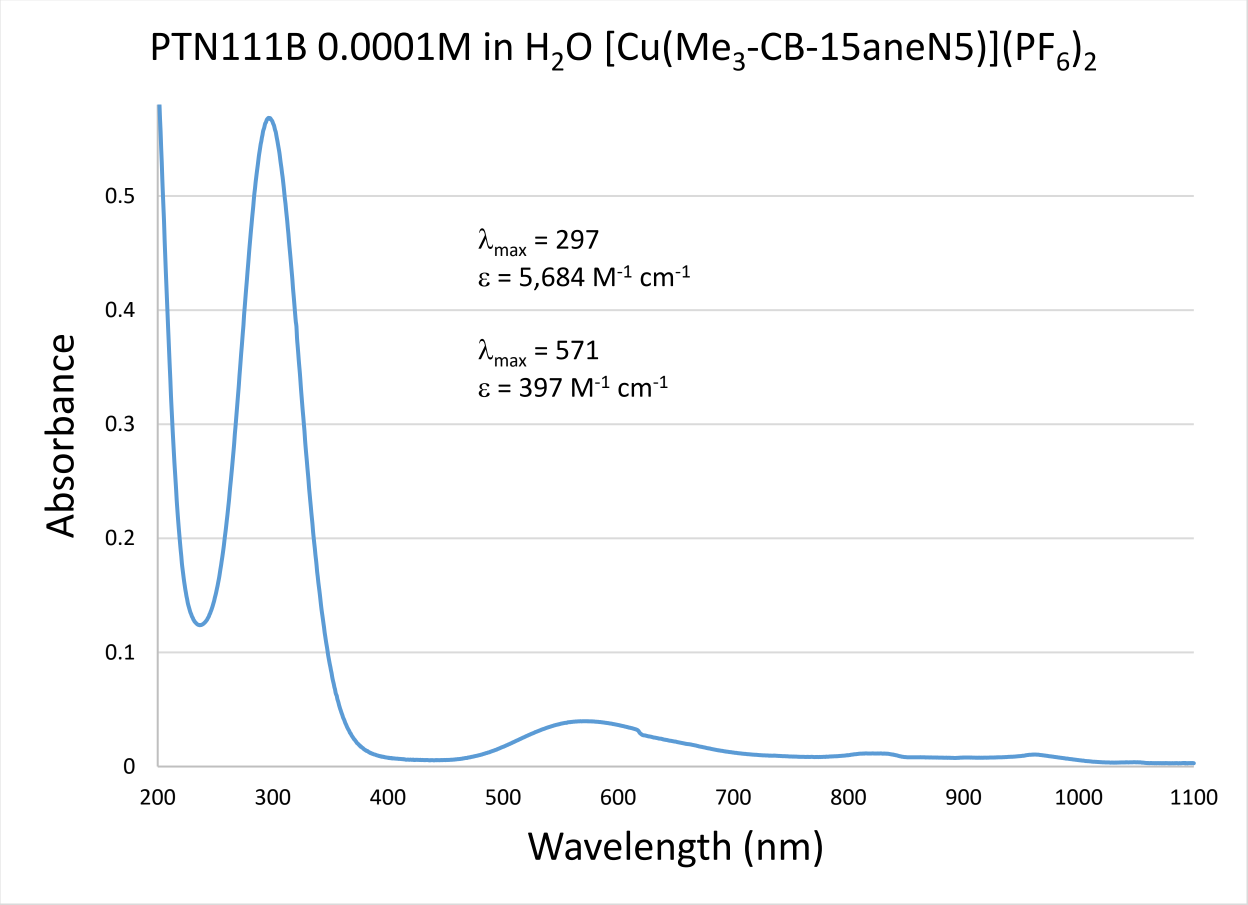


$^{13}$C NMR spectrum of **3** (75 MHz, CDCl$_3$, 298 K).



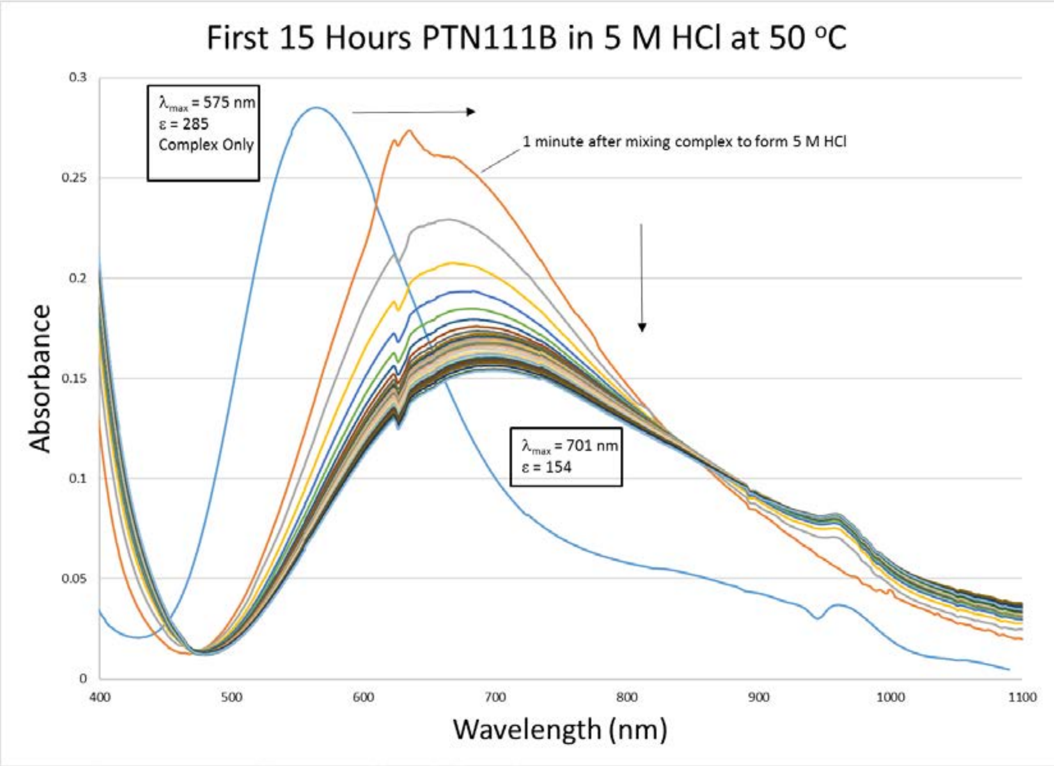
ESI-MS$^+$ mass spectrum of Cu(**3**)$^{2+}$.

## 3. UV-Vis spectrum of Cu(3)$^{2+}$ in $H_2O$

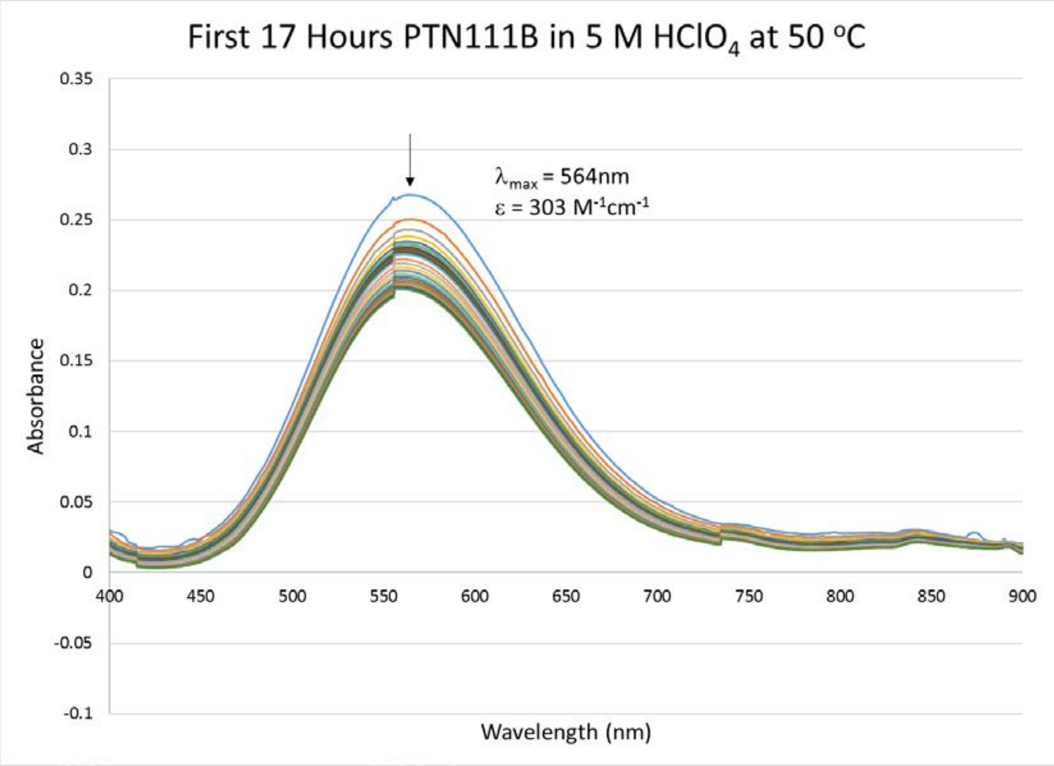


## 4. UV-Visible changes during first 15 Hours in (a) 5M HCl and (b) 5M $HClO_4$

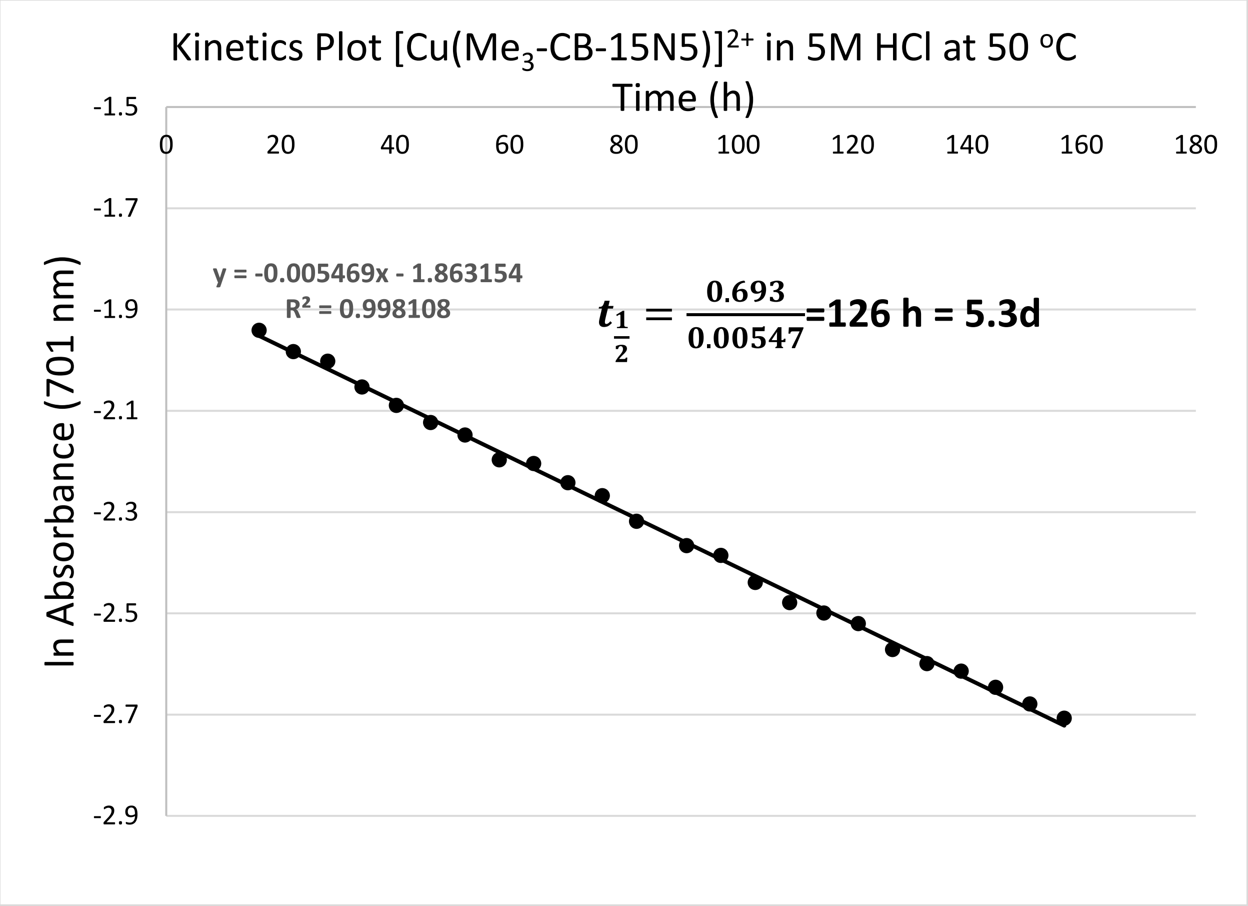
(a)



(b)



**4. Kinetics of dissociation plot**



## 5. X-Ray Crystallography

### 5.1 X-Ray Crystallography of 6’ (CCDC 1981500)

Chemist Code or Formula: AS02B
ALS Code SWOSU233
Crystal Size: 0.100 x 0.0558 x 0.036
Crystal Color & Habit: colorless plates

Intensity data were collected at 150K on a D8 goniostat equipped with a Bruker PHOTON-II detector at Beamline 12.2.1 at the Advanced Light Source (Lawrence Berkeley National Laboratory) using synchrotron radiation tuned to λ=0.7288Å. For data collection 1-s frames were measured in shutterless mode. The data frames were collected using the program APEX3 and processed using the program SAINT routine within APEX3. The data were corrected for absorption and beam corrections based on the multi-scan technique as implemented in SADABS.

**Acknowledgements, References and Notes:**

(1) Crystallographic data were collected through the SCrALS (Service Crystallography at the Advanced Light Source) program at Beamline 12.2.1 at the Advanced Light Source (ALS), Lawrence Berkeley National Laboratory. The Advanced Light Source, is a DOE Office of Science User Facility under contract no. DE-AC02-05CH11231.

(2) APEX3 v2016.9.0 and SAINT v8.38A data collection and data processing programs, respectively. Bruker Analytical X-ray Instruments, Inc., Madison, WI; SADABS v2016.2 semi-empirical absorption and beam correction program. G.M. Sheldrick, University of Göttingen, Germany.

Table S1. Crystal data and structure refinement for **6’**.

| | | |
|---|---|---|
| Identification code | **6’** (AS02B) | |
| Empirical formula | C15.62 H35.50 I2 N5 O1.62 | |
| Chemical formula | $C_{15}H_{31}N_5$, 2I, $H_2O$, 0.625($CH_3OH$) | |
| Formula weight | 573.29 | |
| Temperature | 150(2) K | |
| Wavelength | 0.7288 Å | |
| Crystal system | Monoclinic | |
| Space group | C2/c | |
| Unit cell dimensions | a = 16.5537(9) Å | $\alpha$= 90° |
| | b = 18.0400(9) Å | $\beta$= 97.514(2)° |
| | c = 15.0137(7) Å | $\gamma$ = 90° |
| Volume | 4445.0(4) Å$^3$ | |
| Z | 8 | |
| Density (calculated) | 1.713 Mg/m$^3$ | |
| Absorption coefficient | 3.006 mm$^{-1}$ | |
| F(000) | 2266 | |
| Crystal size | 0.100 × 0.056 × 0.036 mm$^3$ | |
| Theta range for data collection | 2.112 to 29.100° | |
| Index ranges | $-22 \leq h \leq 22$, $-24 \leq k \leq 24$, $-19 \leq l \leq 20$ | |
| Reflections collected | 50133 | |
| Independent reflections | 5526 [R(int) = 0.0532] | |
| Completeness to theta = 25.930° | 99.9 % | |
| Absorption correction | Semi-empirical from equivalents | |
| Max. and min. transmission | 0.7458 and 0.6624 | |
| Refinement method | Full-matrix least-squares on F$^2$ | |
| Data / restraints / parameters | 5526 / 6 / 242 | |
| Goodness-of-fit on F$^2$ | 1.076 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0258, wR2 = 0.0532 | |
| R indices (all data) | R1 = 0.0307, wR2 = 0.0560 | |
| Largest diff. peak and hole | 0.615 and -0.869 e.Å$^{-3}$ | |

Table S2. Atomic coordinates (x $10^4$) and equivalent isotropic displacement parameters (Å$^2$x $10^3$) for **6’**. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| I(2) | 5000 | 5665(1) | 2500 | 25(1) |
| I(3) | 0 | 3000(1) | 7500 | 28(1) |
| I(1) | 1883(1) | 3690(1) | 3259(1) | 32(1) |
| N(5) | 3695(1) | 7065(1) | 4590(1) | 20(1) |
| N(3) | 4283(1) | 5962(1) | 5383(1) | 20(1) |
| N(4) | 5909(1) | 7039(1) | 5015(1) | 24(1) |
| N(1) | 2366(1) | 6127(1) | 3765(1) | 24(1) |
| N(2) | 2597(1) | 5969(1) | 5289(1) | 23(1) |
| C(12) | 3827(1) | 6259(1) | 4586(2) | 19(1) |
| C(11) | 3006(1) | 5863(1) | 4511(2) | 20(1) |
| C(8) | 4435(2) | 7506(1) | 4816(2) | 22(1) |
| C(5) | 5163(1) | 5860(1) | 5434(2) | 24(1) |
| C(9) | 3196(2) | 7290(1) | 3760(2) | 25(1) |
| C(7) | 5176(2) | 7276(1) | 4358(2) | 24(1) |
| C(6) | 5712(2) | 6511(1) | 5749(2) | 25(1) |
| C(3) | 3089(2) | 5704(1) | 6106(2) | 26(1) |
| C(2) | 1812(2) | 5600(2) | 5044(2) | 31(1) |
| C(14) | 6509(2) | 6688(2) | 4476(2) | 31(1) |
| O(1W) | 702(2) | 4446(2) | 6173(2) | 79(1) |
| C(15) | 6301(2) | 7710(2) | 5483(2) | 31(1) |
| C(10) | 2349(2) | 6968(1) | 3745(2) | 27(1) |
| C(4) | 3919(2) | 6079(1) | 6205(2) | 25(1) |
| C(13) | 2502(2) | 5820(2) | 2872(2) | 33(1) |
| C(1) | 1577(2) | 5824(2) | 4064(2) | 33(1) |
| C(17) | -234(4) | 4091(3) | 3452(4) | 60(2) |
| O(16) | 143(2) | 4258(2) | 4315(3) | 52(1) |

Table S3. Bond lengths [Å] and angles [°] for **6’**.

| | | | |
|---|---|---|---|
| N(5)-C(9) | 1.460(3) | N(5)-C(8) | 1.464(3) |
| N(5)-C(12) | 1.470(3) | N(3)-C(12) | 1.432(3) |
| N(3)-C(4) | 1.457(3) | N(3)-C(5) | 1.461(3) |
| N(4)-C(14) | 1.501(3) | N(4)-C(15) | 1.503(3) |
| N(4)-C(7) | 1.522(3) | N(4)-C(6) | 1.524(3) |
| N(1)-C(13) | 1.495(3) | N(1)-C(11) | 1.515(3) |
| N(1)-C(10) | 1.518(3) | N(1)-C(1) | 1.537(3) |
| N(2)-C(11) | 1.438(3) | N(2)-C(3) | 1.462(3) |
| N(2)-C(2) | 1.463(3) | C(12)-C(11) | 1.526(3) |
| C(12)-H(12) | 1.0000 | C(11)-H(11) | 1.0000 |
| C(8)-C(7) | 1.539(3) | C(8)-H(8A) | 0.9900 |
| C(8)-H(8B) | 0.9900 | C(5)-C(6) | 1.521(3) |
| C(5)-H(5A) | 0.9900 | C(5)-H(5B) | 0.9900 |
| C(9)-C(10) | 1.515(4) | C(9)-H(9A) | 0.9900 |
| C(9)-H(9B) | 0.9900 | C(7)-H(7A) | 0.9900 |
| C(7)-H(7B) | 0.9900 | C(6)-H(6A) | 0.9900 |
| C(6)-H(6B) | 0.9900 | C(3)-C(4) | 1.522(4) |
| C(3)-H(3A) | 0.9900 | C(3)-H(3B) | 0.9900 |
| C(2)-C(1) | 1.526(4) | C(2)-H(2A) | 0.9900 |
| C(2)-H(2B) | 0.9900 | C(14)-H(14A) | 0.9800 |
| C(14)-H(14B) | 0.9800 | C(14)-H(14C) | 0.9800 |
| O(1W)-H(1D) | 0.944 | O(1W)-H(1C) | 0.9362 |
| C(15)-H(15A) | 0.9800 | C(15)-H(15B) | 0.9800 |
| C(15)-H(15C) | 0.9800 | C(10)-H(10A) | 0.9900 |
| C(10)-H(10B) | 0.9900 | C(4)-H(4A) | 0.9900 |
| C(4)-H(4B) | 0.9900 | C(13)-H(13A) | 0.9800 |
| C(13)-H(13B) | 0.9800 | C(13)-H(13C) | 0.9800 |
| C(1)-H(1A) | 0.9900 | C(1)-H(1B) | 0.9900 |
| C(17)-O(16) | 1.396(7) | C(17)-H(17A) | 0.9800 |
| C(17)-H(17B) | 0.9800 | C(17)-H(17C) | 0.9800 |
| O(16)-H(16) | 0.881(19) | | |

| | | | |
|---|---|---|---|
| C(9)-N(5)-C(8) | 114.20(18) | C(9)-N(5)-C(12) | 109.86(18) |
| C(8)-N(5)-C(12) | 114.70(18) | C(12)-N(3)-C(4) | 115.02(19) |
| C(12)-N(3)-C(5) | 120.22(19) | C(4)-N(3)-C(5) | 119.57(19) |
| C(14)-N(4)-C(15) | 108.42(19) | C(14)-N(4)-C(7) | 107.22(19) |
| C(15)-N(4)-C(7) | 109.66(19) | C(14)-N(4)-C(6) | 110.21(19) |
| C(15)-N(4)-C(6) | 106.61(19) | C(7)-N(4)-C(6) | 114.59(19) |
| C(13)-N(1)-C(11) | 111.96(19) | C(13)-N(1)-C(10) | 111.0(2) |
| C(11)-N(1)-C(10) | 109.82(18) | C(13)-N(1)-C(1) | 111.3(2) |
| C(11)-N(1)-C(1) | 102.18(18) | C(10)-N(1)-C(1) | 110.3(2) |

| | | | |
|---|---|---|---|
| C(11)-N(2)-C(3) | 111.62(19) | C(11)-N(2)-C(2) | 103.46(19) |
| C(3)-N(2)-C(2) | 116.1(2) | N(3)-C(12)-N(5) | 115.37(19) |
| N(3)-C(12)-C(11) | 104.68(18) | N(5)-C(12)-C(11) | 109.36(18) |
| N(3)-C(12)-H(12) | 109.1 | N(5)-C(12)-H(12) | 109.1 |
| C(11)-C(12)-H(12) | 109.1 | N(2)-C(11)-N(1) | 101.54(18) |
| N(2)-C(11)-C(12) | 112.75(19) | N(1)-C(11)-C(12) | 115.96(18) |
| N(2)-C(11)-H(11) | 108.7 | N(1)-C(11)-H(11) | 108.7 |
| C(12)-C(11)-H(11) | 108.7 | N(5)-C(8)-C(7) | 116.24(19) |
| N(5)-C(8)-H(8A) | 108.2 | C(7)-C(8)-H(8A) | 108.2 |
| N(5)-C(8)-H(8B) | 108.2 | C(7)-C(8)-H(8B) | 108.2 |
| H(8A)-C(8)-H(8B) | 107.4 | N(3)-C(5)-C(6) | 118.1(2) |
| N(3)-C(5)-H(5A) | 107.8 | C(6)-C(5)-H(5A) | 107.8 |
| N(3)-C(5)-H(5B) | 107.8 | C(6)-C(5)-H(5B) | 107.8 |
| H(5A)-C(5)-H(5B) | 107.1 | N(5)-C(9)-C(10) | 109.02(19) |
| N(5)-C(9)-H(9A) | 109.9 | C(10)-C(9)-H(9A) | 109.9 |
| N(5)-C(9)-H(9B) | 109.9 | C(10)-C(9)-H(9B) | 109.9 |
| H(9A)-C(9)-H(9B) | 108.3 | N(4)-C(7)-C(8) | 113.61(19) |
| N(4)-C(7)-H(7A) | 108.8 | C(8)-C(7)-H(7A) | 108.8 |
| N(4)-C(7)-H(7B) | 108.8 | C(8)-C(7)-H(7B) | 108.8 |
| H(7A)-C(7)-H(7B) | 107.7 | C(5)-C(6)-N(4) | 115.7(2) |
| C(5)-C(6)-H(6A) | 108.4 | N(4)-C(6)-H(6A) | 108.4 |
| C(5)-C(6)-H(6B) | 108.4 | N(4)-C(6)-H(6B) | 108.4 |
| H(6A)-C(6)-H(6B) | 107.4 | N(2)-C(3)-C(4) | 109.43(19) |
| N(2)-C(3)-H(3A) | 109.8 | C(4)-C(3)-H(3A) | 109.8 |
| N(2)-C(3)-H(3B) | 109.8 | C(4)-C(3)-H(3B) | 109.8 |
| H(3A)-C(3)-H(3B) | 108.2 | N(2)-C(2)-C(1) | 103.2(2) |
| N(2)-C(2)-H(2A) | 111.1 | C(1)-C(2)-H(2A) | 111.1 |
| N(2)-C(2)-H(2B) | 111.1 | C(1)-C(2)-H(2B) | 111.1 |
| H(2A)-C(2)-H(2B) | 109.1 | N(4)-C(14)-H(14A) | 109.5 |
| N(4)-C(14)-H(14B) | 109.5 | H(14A)-C(14)-H(14B) | 109.5 |
| N(4)-C(14)-H(14C) | 109.5 | H(14A)-C(14)-H(14C) | 109.5 |
| H(14B)-C(14)-H(14C) | 109.5 | H(1D)-O(1W)-H(1C) | 101.9 |
| N(4)-C(15)-H(15A) | 109.5 | N(4)-C(15)-H(15B) | 109.5 |
| H(15A)-C(15)-H(15B) | 109.5 | N(4)-C(15)-H(15C) | 109.5 |
| H(15A)-C(15)-H(15C) | 109.5 | H(15B)-C(15)-H(15C) | 109.5 |
| C(9)-C(10)-N(1) | 111.5(2) | C(9)-C(10)-H(10A) | 109.3 |
| N(1)-C(10)-H(10A) | 109.3 | C(9)-C(10)-H(10B) | 109.3 |
| N(1)-C(10)-H(10B) | 109.3 | H(10A)-C(10)-H(10B) | 108.0 |
| N(3)-C(4)-C(3) | 108.7(2) | N(3)-C(4)-H(4A) | 109.9 |
| C(3)-C(4)-H(4A) | 109.9 | N(3)-C(4)-H(4B) | 109.9 |
| C(3)-C(4)-H(4B) | 109.9 | H(4A)-C(4)-H(4B) | 108.3 |
| N(1)-C(13)-H(13A) | 109.5 | N(1)-C(13)-H(13B) | 109.5 |
| H(13A)-C(13)-H(13B) | 109.5 | N(1)-C(13)-H(13C) | 109.5 |

| | | | |
|---|---|---|---|
| H(13A)-C(13)-H(13C) | 109.5 | H(13B)-C(13)-H(13C) | 109.5 |
| C(2)-C(1)-N(1) | 104.9(2) | C(2)-C(1)-H(1A) | 110.8 |
| N(1)-C(1)-H(1A) | 110.8 | C(2)-C(1)-H(1B) | 110.8 |
| N(1)-C(1)-H(1B) | 110.8 | H(1A)-C(1)-H(1B) | 108.8 |
| O(16)-C(17)-H(17A) | 109.5 | O(16)-C(17)-H(17B) | 109.5 |
| H(17A)-C(17)-H(17B) | 109.5 | O(16)-C(17)-H(17C) | 109.5 |
| H(17A)-C(17)-H(17C) | 109.5 | H(17B)-C(17)-H(17C) | 109.5 |
| C(17)-O(16)-H(16) | 106(3) | | |

Table S4. Anisotropic displacement parameters (Å$^2$x 10$^3$) for **6’**. The anisotropic displacement factor exponent takes the form: -2$\pi^2$[ h$^2$a*$^2$U$^{11}$ + ... + 2 h k a* b* U$^{12}$ ]

| | U$^{11}$ | U$^{22}$ | U$^{33}$ | U$^{23}$ | U$^{13}$ | U$^{12}$ |
|---|---|---|---|---|---|---|
| I(2) | 32(1) | 25(1) | 19(1) | 0 | 7(1) | 0 |
| I(3) | 32(1) | 30(1) | 20(1) | 0 | 1(1) | 0 |
| I(1) | 35(1) | 34(1) | 29(1) | 1(1) | 9(1) | -2(1) |
| N(5) | 20(1) | 16(1) | 23(1) | 3(1) | 2(1) | 0(1) |
| N(3) | 17(1) | 22(1) | 22(1) | 5(1) | 4(1) | 2(1) |
| N(4) | 20(1) | 25(1) | 29(1) | 1(1) | 6(1) | -3(1) |
| N(1) | 18(1) | 26(1) | 28(1) | 1(1) | 1(1) | 0(1) |
| N(2) | 18(1) | 24(1) | 28(1) | 3(1) | 9(1) | 0(1) |
| C(12) | 17(1) | 19(1) | 21(1) | 1(1) | 4(1) | 0(1) |
| C(11) | 19(1) | 18(1) | 24(1) | 2(1) | 5(1) | 1(1) |
| C(8) | 23(1) | 20(1) | 24(1) | 0(1) | 3(1) | -2(1) |
| C(5) | 19(1) | 22(1) | 31(1) | 3(1) | 4(1) | 2(1) |
| C(9) | 27(1) | 23(1) | 26(1) | 6(1) | 1(1) | 0(1) |
| C(7) | 25(1) | 23(1) | 24(1) | 2(1) | 5(1) | -4(1) |
| C(6) | 24(1) | 27(1) | 25(1) | 2(1) | 4(1) | -2(1) |
| C(3) | 30(1) | 26(1) | 26(1) | 5(1) | 11(1) | 4(1) |
| C(2) | 20(1) | 31(1) | 43(2) | 6(1) | 11(1) | 1(1) |
| C(14) | 22(1) | 35(2) | 37(1) | -6(1) | 10(1) | -3(1) |
| O(1W) | 94(2) | 52(2) | 101(2) | 15(2) | 53(2) | -2(2) |
| C(15) | 29(2) | 33(2) | 33(1) | -5(1) | 5(1) | -9(1) |
| C(10) | 26(1) | 24(1) | 30(1) | 4(1) | -1(1) | 6(1) |
| C(4) | 30(1) | 22(1) | 22(1) | 1(1) | 5(1) | 0(1) |
| C(13) | 34(2) | 38(2) | 27(1) | -4(1) | 0(1) | -3(1) |
| C(1) | 19(1) | 35(2) | 44(2) | 1(1) | 4(1) | -4(1) |
| C(17) | 57(4) | 48(3) | 75(4) | 5(3) | 14(3) | 6(3) |
| O(16) | 34(2) | 50(2) | 73(3) | 5(2) | 16(2) | 0(2) |

Table S5. Hydrogen coordinates (x $10^4$) and isotropic displacement parameters (Å$^2$x $10^3$) for **6’**.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(12) | 4106 | 6125 | 4056 | 23 |
| H(11) | 3097 | 5320 | 4431 | 24 |
| H(8A) | 4593 | 7483 | 5475 | 27 |
| H(8B) | 4304 | 8029 | 4659 | 27 |
| H(5A) | 5284 | 5715 | 4830 | 28 |
| H(5B) | 5318 | 5438 | 5841 | 28 |
| H(9A) | 3443 | 7108 | 3236 | 30 |
| H(9B) | 3165 | 7837 | 3727 | 30 |
| H(7A) | 5015 | 6861 | 3941 | 28 |
| H(7B) | 5336 | 7698 | 3998 | 28 |
| H(6A) | 6230 | 6313 | 6062 | 30 |
| H(6B) | 5448 | 6798 | 6193 | 30 |
| H(3A) | 3157 | 5160 | 6074 | 32 |
| H(3B) | 2810 | 5817 | 6635 | 32 |
| H(2A) | 1404 | 5777 | 5422 | 37 |
| H(2B) | 1867 | 5056 | 5106 | 37 |
| H(14A) | 6264 | 6246 | 4171 | 46 |
| H(14B) | 7000 | 6547 | 4876 | 46 |
| H(14C) | 6653 | 7042 | 4027 | 46 |
| H(1D) | 404 | 4891 | 6181 | 118 |
| H(1C) | 465 | 4153 | 6584 | 118 |
| H(15A) | 6491 | 8043 | 5039 | 47 |
| H(15B) | 6765 | 7554 | 5916 | 47 |
| H(15C) | 5903 | 7969 | 5800 | 47 |
| H(10A) | 2001 | 7136 | 3195 | 32 |
| H(10B) | 2105 | 7154 | 4270 | 32 |
| H(4A) | 3858 | 6616 | 6312 | 30 |
| H(4B) | 4275 | 5867 | 6724 | 30 |
| H(13A) | 3028 | 5993 | 2719 | 50 |
| H(13B) | 2065 | 5988 | 2412 | 50 |
| H(13C) | 2501 | 5277 | 2898 | 50 |
| H(1A) | 1148 | 6209 | 4010 | 39 |
| H(1B) | 1376 | 5391 | 3696 | 39 |
| H(17A) | -267 | 4540 | 3082 | 90 |
| H(17B) | -784 | 3901 | 3486 | 90 |
| H(17C) | 86 | 3714 | 3183 | 90 |
| H(16) | 669(12) | 4190(40) | 4310(30) | 77 |

### 5.2 X-Ray Crystallograpy of $H_33^{3+}$ (CCDC 1917395)

SCrALS Code SWOSU070D
Chemist Code: DD08 (from MeCN-ether)
Crystal Size: 0.11 x 0.05 x 0.01
Crystal Habit: colorless blade

Intensity data were collected at 150K on a D8 goniostat equipped with a Bruker PHOTON100 CMOS detector at Beamline 11.3.1 at the Advanced Light Source (Lawrence Berkeley National Laboratory) using synchrotron radiation tuned to λ=0.7749Å. For data collection frames were measured for a duration of 2-s at 0.50 intervals of ω. The data frames were collected using the program APEX3 and processed using the program SAINT routine within APEX3. The data were corrected for absorption and beam corrections based on the multi-scan technique as implemented in SADABS.

**Acknowledgements, References and Notes**:

(1) Crystallographic data were collected through the SCrALS (Service Crystallography at Advanced Light Source) program at the Small-Crystal Crystallography Beamline 11.3.1 at the Advanced Light Source (ALS), Lawrence Berkeley National Laboratory. The ALS is supported by the U.S. Department of Energy, Office of Energy Sciences Materials Sciences Division, under contract DE-AC02-05CH11231.

(2) APEX3 v2016.1.0 and SAINT v8.37A data collection and data processing programs, respectively. Bruker Analytical X-ray Instruments, Inc., Madison, WI; SADABS v2014.5 semi-empirical absorption and beam correction program. G.M. Sheldrick, University of Göttingen, Germany.

Table S6. Crystal data and structure refinement for $\mathbf{H_33^{3+}}$.

| | | |
|---|---|---|
| Identification code | $\mathbf{H_33^{3+}}$ (DD08) | |
| Empirical formula | C17 H39 Br F12 N6 P2 | |
| Chemical formula | $C_{15}H_{36}N_5$, 2($PF_6$), $CH_3CN$, Br | |
| Formula weight | 697.39 | |
| Temperature | 150(2) K | |
| Wavelength | 0.7749 Å | |
| Crystal system | Orthorhombic | |
| Space group | $P2_12_12_1$ | |
| Unit cell dimensions | a = 7.9823(8) Å | $\alpha$= 90° |
| | b = 13.9805(14) Å | $\beta$= 90° |
| | c = 24.662(3) Å | $\gamma$ = 90° |
| Volume | 2752.2(5) $Å^3$ | |
| Z | 4 | |
| Density (calculated) | 1.683 $Mg/m^3$ | |
| Absorption coefficient | 2.114 $mm^{-1}$ | |
| F(000) | 1424 | |
| Crystal size | 0.110 x 0.050 x 0.010 $mm^3$ | |
| Theta range for data collection | 2.401 to 29.832° | |
| Index ranges | -10<=h<=10, -17<=k<=17, -31<=l<=31 | |
| Reflections collected | 48044 | |
| Independent reflections | 6085 [R(int) = 0.0635] | |
| Completeness to theta = 27.706° | 99.9 % | |
| Absorption correction | Semi-empirical from equivalents | |
| Max. and min. transmission | 0.9281 and 0.7831 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 6085 / 3 / 350 | |
| Goodness-of-fit on $F^2$ | 1.069 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0411, wR2 = 0.1072 | |
| R indices (all data) | R1 = 0.0486, wR2 = 0.1105 | |
| Absolute structure parameter | 0.039(3) | |
| Largest diff. peak and hole | 1.414 and -0.568 e.$Å^{-3}$ | |

Table S7. Atomic coordinates (x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) for **$H_3 3^{3+}$**. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Br(1) | 6864(1) | 3332(1) | 5493(1) | 31(1) |
| C(1) | 4919(7) | 3523(4) | 7246(2) | 32(1) |
| C(2) | 4661(8) | 2520(4) | 7017(2) | 35(1) |
| C(3) | 2253(6) | 2839(4) | 6404(2) | 26(1) |
| C(4) | 1733(6) | 3300(3) | 5888(2) | 24(1) |
| C(5) | 1692(6) | 4609(3) | 5263(2) | 20(1) |
| C(6) | 2752(6) | 4386(4) | 4777(2) | 23(1) |
| C(7) | 4742(6) | 5759(3) | 4982(2) | 22(1) |
| C(8) | 5798(6) | 5903(3) | 5493(2) | 24(1) |
| C(9) | 6254(6) | 5685(4) | 6471(2) | 26(1) |
| C(10) | 5793(6) | 5115(4) | 6971(2) | 25(1) |
| C(11) | 7708(7) | 3789(4) | 6886(3) | 32(1) |
| C(12) | 4374(7) | 1622(4) | 6160(3) | 38(1) |
| C(13) | 5417(8) | 4507(5) | 4303(2) | 37(1) |
| C(14) | 2176(6) | 4964(3) | 6206(2) | 22(1) |
| C(15) | 3310(6) | 5822(3) | 6124(2) | 21(1) |
| C(16) | -434(7) | 7428(4) | 6161(3) | 34(1) |
| C(17) | -600(9) | 8141(4) | 6576(3) | 48(2) |
| N(1) | 5932(5) | 4089(3) | 6867(2) | 23(1) |
| N(2) | 4081(5) | 2558(3) | 6441(2) | 25(1) |
| N(3) | 2429(5) | 4258(3) | 5776(2) | 18(1) |
| N(4) | 4533(5) | 4727(3) | 4823(2) | 22(1) |
| N(5) | 5076(5) | 5500(3) | 6008(2) | 18(1) |
| N(6) | -312(7) | 6850(4) | 5834(3) | 48(1) |
| P(1) | 4602(2) | 8736(1) | 5956(1) | 32(1) |
| F(1) | 5034(5) | 7886(2) | 6369(2) | 45(1) |
| F(2) | 3389(5) | 8038(3) | 5631(2) | 64(1) |
| F(3) | 6129(4) | 8415(3) | 5580(2) | 46(1) |
| F(4) | 4185(4) | 9584(2) | 5536(2) | 45(1) |
| F(5) | 5811(6) | 9430(3) | 6282(2) | 57(1) |
| F(6) | 3095(6) | 9060(3) | 6331(2) | 75(2) |
| P(2) | 663(2) | 5958(1) | 7675(1) | 26(1) |
| F(7) | 1622(6) | 6114(4) | 8233(2) | 71(1) |
| F(8) | -898(4) | 5544(3) | 7998(1) | 45(1) |
| F(9) | 1400(6) | 4925(3) | 7603(2) | 65(1) |
| F(10) | -305(5) | 5851(3) | 7116(1) | 45(1) |
| F(11) | 2222(4) | 6397(3) | 7353(1) | 45(1) |
| F(12) | -76(6) | 7025(3) | 7729(2) | 61(1) |

Table S8. Bond lengths [Å] and angles [°] for **$H_{3}3^{3+}$**.

| | | | |
|---|---|---|---|
| C(1)-N(1) | 1.467(7) | C(1)-C(2) | 1.525(8) |
| C(1)-H(1A) | 0.9900 | C(1)-H(1B) | 0.9900 |
| C(2)-N(2) | 1.496(7) | C(2)-H(2A) | 0.9900 |
| C(2)-H(2B) | 0.9900 | C(3)-C(4) | 1.486(7) |
| C(3)-N(2) | 1.514(6) | C(3)-H(3A) | 0.9900 |
| C(3)-H(3B) | 0.9900 | C(4)-N(3) | 1.475(6) |
| C(4)-H(4A) | 0.9900 | C(4)-H(4B) | 0.9900 |
| C(5)-N(3) | 1.480(6) | C(5)-C(6) | 1.500(7) |
| C(5)-H(5A) | 0.9900 | C(5)-H(5B) | 0.9900 |
| C(6)-N(4) | 1.504(6) | C(6)-H(6A) | 0.9900 |
| C(6)-H(6B) | 0.9900 | C(7)-N(4) | 1.504(6) |
| C(7)-C(8) | 1.531(7) | C(7)-H(7A) | 0.9900 |
| C(7)-H(7B) | 0.9900 | C(8)-N(5) | 1.503(6) |
| C(8)-H(8A) | 0.9900 | C(8)-H(8B) | 0.9900 |
| C(9)-N(5) | 1.501(6) | C(9)-C(10) | 1.513(7) |
| C(9)-H(9A) | 0.9900 | C(9)-H(9B) | 0.9900 |
| C(10)-N(1) | 1.462(6) | C(10)-H(10A) | 0.9900 |
| C(10)-H(10B) | 0.9900 | C(11)-N(1) | 1.479(6) |
| C(11)-H(11A) | 0.9800 | C(11)-H(11B) | 0.9800 |
| C(11)-H(11C) | 0.9800 | C(12)-N(2) | 1.499(7) |
| C(12)-H(12A) | 0.9800 | C(12)-H(12B) | 0.9800 |
| C(12)-H(12C) | 0.9800 | C(13)-N(4) | 1.495(6) |
| C(13)-H(13A) | 0.9800 | C(13)-H(13B) | 0.9800 |
| C(13)-H(13C) | 0.9800 | C(14)-N(3) | 1.462(6) |
| C(14)-C(15) | 1.516(7) | C(14)-H(14A) | 0.9900 |
| C(14)-H(14B) | 0.9900 | C(15)-N(5) | 1.508(6) |
| C(15)-H(15A) | 0.9900 | C(15)-H(15B) | 0.9900 |
| C(16)-N(6) | 1.146(8) | C(16)-C(17) | 1.435(10) |
| C(17)-H(17A) | 0.9800 | C(17)-H(17B) | 0.9800 |
| C(17)-H(17C) | 0.9800 | N(2)-H(2) | 0.82(4) |
| N(4)-H(4) | 0.82(4) | N(5)-H(5) | 0.82(4) |
| P(1)-F(6) | 1.585(4) | P(1)-F(5) | 1.587(4) |
| P(1)-F(2) | 1.591(4) | P(1)-F(3) | 1.595(4) |
| P(1)-F(1) | 1.602(4) | P(1)-F(4) | 1.609(4) |
| P(2)-F(9) | 1.570(4) | P(2)-F(8) | 1.588(3) |
| P(2)-F(7) | 1.589(4) | P(2)-F(10) | 1.590(4) |
| P(2)-F(11) | 1.599(3) | P(2)-F(12) | 1.609(4) |

| | | | |
|---|---|---|---|
| N(1)-C(1)-C(2) | 109.6(4) | N(1)-C(1)-H(1A) | 109.8 |
| C(2)-C(1)-H(1A) | 109.8 | N(1)-C(1)-H(1B) | 109.8 |
| C(2)-C(1)-H(1B) | 109.8 | H(1A)-C(1)-H(1B) | 108.2 |

|  |  |  |  |
|---|---|---|---|
| N(2)-C(2)-C(1) | 111.1(4) | N(2)-C(2)-H(2A) | 109.4 |
| C(1)-C(2)-H(2A) | 109.4 | N(2)-C(2)-H(2B) | 109.4 |
| C(1)-C(2)-H(2B) | 109.4 | H(2A)-C(2)-H(2B) | 108.0 |
| C(4)-C(3)-N(2) | 115.7(4) | C(4)-C(3)-H(3A) | 108.4 |
| N(2)-C(3)-H(3A) | 108.4 | C(4)-C(3)-H(3B) | 108.4 |
| N(2)-C(3)-H(3B) | 108.4 | H(3A)-C(3)-H(3B) | 107.4 |
| N(3)-C(4)-C(3) | 116.7(4) | N(3)-C(4)-H(4A) | 108.1 |
| C(3)-C(4)-H(4A) | 108.1 | N(3)-C(4)-H(4B) | 108.1 |
| C(3)-C(4)-H(4B) | 108.1 | H(4A)-C(4)-H(4B) | 107.3 |
| N(3)-C(5)-C(6) | 113.0(4) | N(3)-C(5)-H(5A) | 109.0 |
| C(6)-C(5)-H(5A) | 109.0 | N(3)-C(5)-H(5B) | 109.0 |
| C(6)-C(5)-H(5B) | 109.0 | H(5A)-C(5)-H(5B) | 107.8 |
| C(5)-C(6)-N(4) | 114.0(4) | C(5)-C(6)-H(6A) | 108.8 |
| N(4)-C(6)-H(6A) | 108.8 | C(5)-C(6)-H(6B) | 108.8 |
| N(4)-C(6)-H(6B) | 108.8 | H(6A)-C(6)-H(6B) | 107.7 |
| N(4)-C(7)-C(8) | 113.6(4) | N(4)-C(7)-H(7A) | 108.8 |
| C(8)-C(7)-H(7A) | 108.8 | N(4)-C(7)-H(7B) | 108.8 |
| C(8)-C(7)-H(7B) | 108.8 | H(7A)-C(7)-H(7B) | 107.7 |
| N(5)-C(8)-C(7) | 115.9(4) | N(5)-C(8)-H(8A) | 108.3 |
| C(7)-C(8)-H(8A) | 108.3 | N(5)-C(8)-H(8B) | 108.3 |
| C(7)-C(8)-H(8B) | 108.3 | H(8A)-C(8)-H(8B) | 107.4 |
| N(5)-C(9)-C(10) | 112.1(4) | N(5)-C(9)-H(9A) | 109.2 |
| C(10)-C(9)-H(9A) | 109.2 | N(5)-C(9)-H(9B) | 109.2 |
| C(10)-C(9)-H(9B) | 109.2 | H(9A)-C(9)-H(9B) | 107.9 |
| N(1)-C(10)-C(9) | 110.9(4) | N(1)-C(10)-H(10A) | 109.5 |
| C(9)-C(10)-H(10A) | 109.5 | N(1)-C(10)-H(10B) | 109.5 |
| C(9)-C(10)-H(10B) | 109.5 | H(10A)-C(10)-H(10B) | 108.0 |
| N(1)-C(11)-H(11A) | 109.5 | N(1)-C(11)-H(11B) | 109.5 |
| H(11A)-C(11)-H(11B) | 109.5 | N(1)-C(11)-H(11C) | 109.5 |
| H(11A)-C(11)-H(11C) | 109.5 | H(11B)-C(11)-H(11C) | 109.5 |
| N(2)-C(12)-H(12A) | 109.5 | N(2)-C(12)-H(12B) | 109.5 |
| H(12A)-C(12)-H(12B) | 109.5 | N(2)-C(12)-H(12C) | 109.5 |
| H(12A)-C(12)-H(12C) | 109.5 | H(12B)-C(12)-H(12C) | 109.5 |
| N(4)-C(13)-H(13A) | 109.5 | N(4)-C(13)-H(13B) | 109.5 |
| H(13A)-C(13)-H(13B) | 109.5 | N(4)-C(13)-H(13C) | 109.5 |
| H(13A)-C(13)-H(13C) | 109.5 | H(13B)-C(13)-H(13C) | 109.5 |
| N(3)-C(14)-C(15) | 110.8(4) | N(3)-C(14)-H(14A) | 109.5 |
| C(15)-C(14)-H(14A) | 109.5 | N(3)-C(14)-H(14B) | 109.5 |
| C(15)-C(14)-H(14B) | 109.5 | H(14A)-C(14)-H(14B) | 108.1 |
| N(5)-C(15)-C(14) | 110.3(4) | N(5)-C(15)-H(15A) | 109.6 |
| C(14)-C(15)-H(15A) | 109.6 | N(5)-C(15)-H(15B) | 109.6 |
| C(14)-C(15)-H(15B) | 109.6 | H(15A)-C(15)-H(15B) | 108.1 |
| N(6)-C(16)-C(17) | 179.2(7) | C(16)-C(17)-H(17A) | 109.5 |

|  |  |  |  |
|---|---|---|---|
| C(16)-C(17)-H(17B) | 109.5 | H(17A)-C(17)-H(17B) | 109.5 |
| C(16)-C(17)-H(17C) | 109.5 | H(17A)-C(17)-H(17C) | 109.5 |
| H(17B)-C(17)-H(17C) | 109.5 | C(10)-N(1)-C(1) | 112.1(4) |
| C(10)-N(1)-C(11) | 110.2(4) | C(1)-N(1)-C(11) | 110.8(4) |
| C(2)-N(2)-C(12) | 111.1(4) | C(2)-N(2)-C(3) | 111.3(5) |
| C(12)-N(2)-C(3) | 110.4(4) | C(2)-N(2)-H(2) | 107.9 |
| C(12)-N(2)-H(2) | 107.9 | C(3)-N(2)-H(2) | 107.9 |
| C(14)-N(3)-C(4) | 115.1(4) | C(14)-N(3)-C(5) | 109.9(4) |
| C(4)-N(3)-C(5) | 108.1(4) | C(13)-N(4)-C(6) | 108.4(4) |
| C(13)-N(4)-C(7) | 111.6(4) | C(6)-N(4)-C(7) | 115.4(4) |
| C(13)-N(4)-H(4) | 107.0 | C(6)-N(4)-H(4) | 107.0 |
| C(7)-N(4)-H(4) | 107.0 | C(9)-N(5)-C(8) | 109.7(4) |
| C(9)-N(5)-C(15) | 112.9(4) | C(8)-N(5)-C(15) | 114.0(4) |
| C(9)-N(5)-H(5) | 106.6 | C(8)-N(5)-H(5) | 106.6 |
| C(15)-N(5)-H(5) | 106.6 | F(6)-P(1)-F(5) | 89.4(3) |
| F(6)-P(1)-F(2) | 90.4(3) | F(5)-P(1)-F(2) | 179.8(3) |
| F(6)-P(1)-F(3) | 179.6(3) | F(5)-P(1)-F(3) | 90.1(2) |
| F(2)-P(1)-F(3) | 90.0(2) | F(6)-P(1)-F(1) | 90.3(2) |
| F(5)-P(1)-F(1) | 90.0(2) | F(2)-P(1)-F(1) | 89.8(2) |
| F(3)-P(1)-F(1) | 89.8(2) | F(6)-P(1)-F(4) | 90.4(2) |
| F(5)-P(1)-F(4) | 90.1(2) | F(2)-P(1)-F(4) | 90.1(2) |
| F(3)-P(1)-F(4) | 89.53(19) | F(1)-P(1)-F(4) | 179.3(2) |
| F(9)-P(2)-F(8) | 90.9(2) | F(9)-P(2)-F(7) | 92.6(3) |
| F(8)-P(2)-F(7) | 89.7(2) | F(9)-P(2)-F(10) | 89.8(2) |
| F(8)-P(2)-F(10) | 91.05(19) | F(7)-P(2)-F(10) | 177.5(3) |
| F(9)-P(2)-F(11) | 90.3(2) | F(8)-P(2)-F(11) | 178.8(2) |
| F(7)-P(2)-F(11) | 90.2(2) | F(10)-P(2)-F(11) | 89.0(2) |
| F(9)-P(2)-F(12) | 178.0(2) | F(8)-P(2)-F(12) | 90.5(2) |
| F(7)-P(2)-F(12) | 88.8(3) | F(10)-P(2)-F(12) | 88.8(2) |
| F(11)-P(2)-F(12) | 88.3(2) |  |  |

Table S9. Anisotropic displacement parameters ($Å^2$x $10^3$) for **$H_3$3$^{3+}$**. The anisotropic displacement factor exponent takes the form: $-2\pi^2$[ $h^2a^{*2}U^{11}$ + ... + 2 h k a* b* $U^{12}$ ]

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| Br(1) | 29(1) | 29(1) | 35(1) | 1(1) | -1(1) | 5(1) |
| C(1) | 35(3) | 37(3) | 23(3) | 4(2) | -1(2) | 0(2) |
| C(2) | 36(3) | 33(3) | 36(3) | 16(2) | -4(2) | 0(2) |
| C(3) | 18(3) | 22(2) | 37(3) | 3(2) | 6(2) | -2(2) |
| C(4) | 21(2) | 23(2) | 29(2) | 0(2) | 2(2) | -6(2) |
| C(5) | 17(2) | 23(2) | 21(2) | 0(2) | -5(2) | -1(2) |
| C(6) | 22(3) | 23(2) | 22(2) | -1(2) | -3(2) | -3(2) |
| C(7) | 22(2) | 18(2) | 27(3) | 3(2) | 2(2) | 1(2) |
| C(8) | 20(2) | 18(2) | 33(3) | 2(2) | -1(2) | -3(2) |
| C(9) | 25(2) | 20(2) | 34(3) | -5(2) | -9(2) | -1(2) |
| C(10) | 26(2) | 24(3) | 24(3) | -5(2) | -6(2) | 5(2) |
| C(11) | 26(3) | 28(3) | 41(3) | -3(2) | -7(2) | 6(2) |
| C(12) | 34(3) | 24(3) | 55(4) | -5(3) | 5(3) | 0(2) |
| C(13) | 36(3) | 50(4) | 26(3) | -7(3) | 14(2) | -6(3) |
| C(14) | 22(3) | 23(2) | 20(2) | -1(2) | 1(2) | 1(2) |
| C(15) | 18(2) | 21(2) | 23(2) | -5(2) | 1(2) | 4(2) |
| C(16) | 21(3) | 34(3) | 48(4) | 15(3) | 0(2) | 2(2) |
| C(17) | 50(4) | 34(4) | 59(4) | 10(3) | -1(3) | 0(3) |
| N(1) | 23(2) | 22(2) | 25(2) | -1(2) | -2(2) | 3(2) |
| N(2) | 24(2) | 19(2) | 32(2) | 5(2) | 3(2) | -3(2) |
| N(3) | 16(2) | 16(2) | 21(2) | 1(2) | 2(2) | 3(2) |
| N(4) | 23(2) | 24(2) | 20(2) | 1(2) | 0(2) | 1(2) |
| N(5) | 18(2) | 13(2) | 22(2) | -4(2) | -1(2) | 1(2) |
| N(6) | 33(3) | 53(4) | 58(4) | 4(3) | -3(2) | 7(3) |
| P(1) | 27(1) | 21(1) | 49(1) | 10(1) | 10(1) | 3(1) |
| F(1) | 54(2) | 27(2) | 56(2) | 15(2) | 3(2) | 6(2) |
| F(2) | 57(3) | 56(2) | 80(3) | 21(2) | -18(2) | -29(2) |
| F(3) | 39(2) | 39(2) | 59(2) | 5(2) | 18(2) | 11(2) |
| F(4) | 38(2) | 36(2) | 61(2) | 23(2) | 16(2) | 10(1) |
| F(5) | 75(3) | 29(2) | 67(3) | -5(2) | -5(2) | -6(2) |
| F(6) | 67(3) | 76(3) | 82(3) | 38(2) | 46(3) | 38(3) |
| P(2) | 22(1) | 35(1) | 20(1) | -6(1) | 3(1) | -4(1) |
| F(7) | 56(2) | 131(4) | 27(2) | 0(2) | -7(2) | -48(3) |
| F(8) | 39(2) | 67(3) | 30(2) | -1(2) | 8(2) | -21(2) |
| F(9) | 73(3) | 47(2) | 75(3) | 15(2) | 16(2) | 25(2) |
| F(10) | 45(2) | 61(2) | 28(2) | -7(2) | -4(2) | -12(2) |
| F(11) | 33(2) | 67(2) | 34(2) | -4(2) | 11(1) | -18(2) |
| F(12) | 75(3) | 41(2) | 66(3) | -12(2) | 32(2) | 6(2) |

Table S10. Hydrogen coordinates (x $10^4$) and isotropic displacement parameters ($Å^2$x $10^3$) for **$H_3$3$^{3+}$**.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(1A) | 3819 | 3836 | 7301 | 38 |
| H(1B) | 5492 | 3483 | 7601 | 38 |
| H(2A) | 5728 | 2160 | 7038 | 42 |
| H(2B) | 3820 | 2177 | 7239 | 42 |
| H(3A) | 1563 | 2258 | 6458 | 31 |
| H(3B) | 2000 | 3283 | 6706 | 31 |
| H(4A) | 2051 | 2872 | 5585 | 29 |
| H(4B) | 496 | 3351 | 5887 | 29 |
| H(5A) | 1537 | 5311 | 5287 | 24 |
| H(5B) | 573 | 4316 | 5214 | 24 |
| H(6A) | 2235 | 4684 | 4453 | 27 |
| H(6B) | 2756 | 3685 | 4720 | 27 |
| H(7A) | 3621 | 6043 | 5041 | 27 |
| H(7B) | 5276 | 6107 | 4678 | 27 |
| H(8A) | 6909 | 5607 | 5433 | 28 |
| H(8B) | 5977 | 6597 | 5545 | 28 |
| H(9A) | 6236 | 6376 | 6560 | 31 |
| H(9B) | 7408 | 5517 | 6359 | 31 |
| H(10A) | 6546 | 5294 | 7273 | 30 |
| H(10B) | 4630 | 5270 | 7079 | 30 |
| H(11A) | 8131 | 3855 | 7257 | 47 |
| H(11B) | 8369 | 4194 | 6642 | 47 |
| H(11C) | 7800 | 3120 | 6772 | 47 |
| H(12A) | 5565 | 1457 | 6180 | 57 |
| H(12B) | 4035 | 1675 | 5779 | 57 |
| H(12C) | 3711 | 1121 | 6337 | 57 |
| H(13A) | 4953 | 4903 | 4012 | 56 |
| H(13B) | 5264 | 3830 | 4214 | 56 |
| H(13C) | 6615 | 4644 | 4344 | 56 |
| H(14A) | 2417 | 4667 | 6562 | 26 |
| H(14B) | 992 | 5175 | 6206 | 26 |
| H(15A) | 2891 | 6212 | 5817 | 25 |
| H(15B) | 3297 | 6225 | 6454 | 25 |
| H(17A) | 400 | 8548 | 6579 | 72 |
| H(17B) | -723 | 7829 | 6929 | 72 |
| H(17C) | -1590 | 8535 | 6503 | 72 |
| H(2) | 4630(30) | 2970(20) | 6283(8) | 30 |
| H(4) | 4980(20) | 4404(16) | 5061(12) | 27 |
| H(5) | 5039(5) | 4920(30) | 5968(3) | 21 |

## 5.3 X-Ray Crystallograpy of Cu(3)$^{2+}$ (CCDC 1917396)

SCrALS Code SWOSU059
Chemist Code: GB08A (from $MeNO_2$-ether)
Crystal Size: 0.04 x 0.02 x 0.002
Crystal Habit: violet plate

Intensity data were collected at 150K on a D8 goniostat equipped with a Bruker PHOTON100 CMOS detector at Beamline 11.3.1 at the Advanced Light Source (Lawrence Berkeley National Laboratory) using synchrotron radiation tuned to λ=0.7749Å. For data collection frames were measured for a duration of 3-s at 0.5o intervals of ω. The data frames were collected using the program APEX3 and processed using the program SAINT routine within APEX3. The data were corrected for absorption and beam corrections based on the multi-scan technique as implemented in SADABS.

**Acknowledgements, References and Notes**:

(1) Crystallographic data were collected through the SCrALS (Service Crystallography at Advanced Light Source) program at the Small-Crystal Crystallography Beamline 11.3.1 at the Advanced Light Source (ALS), Lawrence Berkeley National Laboratory. The ALS is supported by the U.S. Department of Energy, Office of Energy Sciences Materials Sciences Division, under contract DE-AC02-05CH11231.

(2) APEX3 v2016.1.0 and SAINT v8.37A data collection and data processing programs, respectively. Bruker Analytical X-ray Instruments, Inc., Madison, WI; SADABS v2014.5 semi-empirical absorption and beam correction program. G.M. Sheldrick, University of Göttingen, Germany.

Table S11. Crystal data and structure refinement for **Cu(3)**$^{2+}$.

| | | |
|---|---|---|
| Identification code | **Cu(3)**$^{2+}$ (GB08A) | |
| Empirical formula | C15 H33 Cl2 Cu N5 O1.79 | |
| Chemical formula | $C_{15}H_{33}N_5Cu$, 1.55Cl, 0.45($ClO_4$) | |
| Formula weight | 446.58 | |
| Temperature | 150(2) K | |
| Wavelength | 0.7749 Å | |
| Crystal system | Monoclinic | |
| Space group | $P2_1/n$ | |
| Unit cell dimensions | a = 8.8067(14) Å | α = 90° |
| | b = 15.792(2) Å | β = 90.357(4)° |
| | c = 14.030(2) Å | γ = 90° |
| Volume | 1951.1(5) $Å^3$ | |
| Z | 4 | |
| Density (calculated) | 1.520 $Mg/m^3$ | |
| Absorption coefficient | 1.783 $mm^{-1}$ | |
| F(000) | 941 | |
| Crystal size | 0.040 x 0.020 x 0.002 $mm^3$ | |
| θ range for data collection | 2.117 to 29.076° | |
| Index ranges | $-11 \leq h \leq 11$, $-19 \leq k \leq 19$, $-17 \leq l \leq 17$ | |
| Reflections collected | 20954 | |
| Independent reflections | 4026 [R(int) = 0.1643] | |
| Completeness to θ = 27.706° | 100.0 % | |
| Absorption correction | Semi-empirical from equivalents | |
| Max. and min. transmission | 0.9281 and 0.7057 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 4026 / 28 / 268 | |
| Goodness-of-fit on $F^2$ | 1.042 | |
| Final R indices [I>2σ(I)] | R1 = 0.0787, wR2 = 0.1748 | |
| R indices (all data) | R1 = 0.1734, wR2 = 0.2109 | |
| Largest diff. peak and hole | 1.111 and -0.875 $eÅ^{-3}$ | |

Table S12. Atomic coordinates [x10$^4$] and equivalent isotropic displacement parameters [Å$^2$x10$^3$] for **Cu(3)$^{2+}$**. U(eq) is defined as one third of the trace of the orthogonalized U$_{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Cu(1) | 7408(1) | 2526(1) | 9150(1) | 26(1) |
| N(1) | 5986(7) | 3290(4) | 8421(4) | 30(2) |
| N(2) | 5750(7) | 1633(4) | 9045(4) | 28(2) |
| N(3) | 8942(7) | 1565(4) | 9229(4) | 31(2) |
| N(4) | 9118(7) | 3263(4) | 8630(4) | 32(2) |
| N(5) | 7259(7) | 3478(4) | 10254(4) | 28(2) |
| C(1) | 5043(10) | 2698(5) | 7846(6) | 41(2) |
| C(2) | 4503(9) | 1985(5) | 8448(5) | 39(2) |
| C(3) | 5123(10) | 1305(5) | 9945(6) | 44(2) |
| C(4) | 6604(10) | 967(5) | 8518(6) | 38(2) |
| C(5) | 8076(10) | 773(5) | 9027(6) | 45(2) |
| C(6) | 9853(9) | 1453(5) | 10117(5) | 40(2) |
| C(7) | 9956(9) | 1801(5) | 8430(5) | 35(2) |
| C(8) | 10452(9) | 2703(5) | 8527(6) | 40(2) |
| C(9) | 8501(9) | 3528(5) | 7681(5) | 38(2) |
| C(10) | 6902(9) | 3874(5) | 7798(6) | 42(2) |
| C(11) | 9399(10) | 3987(5) | 9279(6) | 42(2) |
| C(12) | 8828(9) | 3809(5) | 10271(6) | 41(2) |
| C(13) | 5128(10) | 3754(5) | 9146(5) | 39(2) |
| C(14) | 6146(9) | 4122(5) | 9906(6) | 37(2) |
| C(15) | 6830(10) | 3166(6) | 11205(5) | 42(2) |
| Cl(1) | 7435(3) | 6584(3) | 8987(2) | 83(2) |
| Cl(1A) | 7430(20) | 7195(11) | 9263(12) | 24(4) |
| Cl(1B) | 7000(30) | 5911(18) | 8550(20) | 48(8) |
| Cl(2) | 2590(10) | 9722(4) | 7923(4) | 21(2) |
| Cl(2A) | 2478(14) | 9741(8) | 7980(9) | 70(6) |
| O(1) | 2503(16) | 10144(7) | 8742(8) | 56(5) |
| O(2) | 2699(14) | 8967(7) | 8049(8) | 42(4) |
| O(3) | 1116(17) | 9842(10) | 7671(11) | 114(6) |
| O(4) | 3330(20) | 10047(9) | 7365(10) | 100(8) |
| H(1A) | 5649 | 2473 | 7310 | 50 |
| H(1B) | 4160 | 3005 | 7575 | 50 |
| H(2A) | 3676 | 2187 | 8865 | 47 |
| H(2B) | 4086 | 1533 | 8033 | 47 |
| H(3A) | 4379 | 860 | 9805 | 65 |
| H(3B) | 4627 | 1766 | 10291 | 65 |
| H(3C) | 5946 | 1070 | 10337 | 65 |
| H(4A) | 6817 | 1166 | 7863 | 46 |

| | | | | |
|---|---|---|---|---|
| H(4B) | 5981 | 447 | 8473 | 46 |
| H(5A) | 7860 | 478 | 9633 | 53 |
| H(5B) | 8698 | 392 | 8627 | 53 |
| H(6A) | 10562 | 981 | 10036 | 60 |
| H(6B) | 9173 | 1333 | 10650 | 60 |
| H(6C) | 10425 | 1973 | 10250 | 60 |
| H(7A) | 9411 | 1724 | 7816 | 42 |
| H(7B) | 10857 | 1426 | 8430 | 42 |
| H(8A) | 11123 | 2762 | 9091 | 48 |
| H(8B) | 11036 | 2871 | 7956 | 48 |
| H(9A) | 8482 | 3036 | 7244 | 46 |
| H(9B) | 9162 | 3968 | 7401 | 46 |
| H(10A) | 6947 | 4444 | 8091 | 50 |
| H(10B) | 6406 | 3927 | 7166 | 50 |
| H(11A) | 8884 | 4497 | 9023 | 50 |
| H(11B) | 10502 | 4105 | 9307 | 50 |
| H(12A) | 9504 | 3391 | 10585 | 49 |
| H(12B) | 8859 | 4338 | 10650 | 49 |
| H(13A) | 4387 | 3367 | 9446 | 47 |
| H(13B) | 4552 | 4218 | 8835 | 47 |
| H(14A) | 6699 | 4615 | 9644 | 44 |
| H(14B) | 5522 | 4321 | 10445 | 44 |
| H(15A) | 6911 | 3629 | 11668 | 63 |
| H(15B) | 7512 | 2705 | 11394 | 63 |
| H(15C) | 5782 | 2958 | 11186 | 63 |

Table S13. Bond lengths [Å] and angles [°] for **Cu(3)$^{2+}$**.

| | | | |
|---|---|---|---|
| Cu(1)-N(1) | 2.013(6) | Cu(1)-N(3) | 2.034(6) |
| Cu(1)-N(2) | 2.035(6) | Cu(1)-N(4) | 2.042(6) |
| Cu(1)-N(5) | 2.162(6) | N(1)-C(13) | 1.467(9) |
| N(1)-C(1) | 1.486(9) | N(1)-C(10) | 1.508(9) |
| N(2)-C(3) | 1.475(9) | N(2)-C(2) | 1.486(9) |
| N(2)-C(4) | 1.491(9) | N(3)-C(7) | 1.485(9) |
| N(3)-C(6) | 1.488(9) | N(3)-C(5) | 1.491(10) |
| N(4)-C(8) | 1.479(9) | N(4)-C(11) | 1.481(9) |
| N(4)-C(9) | 1.494(9) | N(5)-C(15) | 1.474(9) |
| N(5)-C(12) | 1.477(9) | N(5)-C(14) | 1.493(9) |
| C(1)-C(2) | 1.487(11) | C(4)-C(5) | 1.507(11) |
| C(7)-C(8) | 1.497(11) | C(9)-C(10) | 1.521(11) |
| C(11)-C(12) | 1.509(11) | C(13)-C(14) | 1.505(11) |
| Cl(2A)-O(2) | 1.242(12) | Cl(2A)-O(1) | 1.243(11) |
| Cl(2A)-O(4) | 1.246(13) | Cl(2A)-O(3) | 1.283(14) |

| | | | |
|---|---|---|---|
| N(1)-Cu(1)-N(3) | 152.3(2) | N(1)-Cu(1)-N(2) | 86.3(2) |
| N(3)-Cu(1)-N(2) | 87.9(2) | N(1)-Cu(1)-N(4) | 86.2(3) |
| N(3)-Cu(1)-N(4) | 87.3(3) | N(2)-Cu(1)-N(4) | 154.1(2) |
| N(1)-Cu(1)-N(5) | 84.7(2) | N(3)-Cu(1)-N(5) | 121.5(2) |
| N(2)-Cu(1)-N(5) | 119.2(2) | N(4)-Cu(1)-N(5) | 84.7(2) |
| C(13)-N(1)-C(1) | 113.7(6) | C(13)-N(1)-C(10) | 112.0(6) |
| C(1)-N(1)-C(10) | 111.7(6) | C(13)-N(1)-Cu(1) | 105.6(4) |
| C(1)-N(1)-Cu(1) | 104.1(4) | C(10)-N(1)-Cu(1) | 109.2(5) |
| C(3)-N(2)-C(2) | 109.6(6) | C(3)-N(2)-C(4) | 111.7(6) |
| C(2)-N(2)-C(4) | 111.0(6) | C(3)-N(2)-Cu(1) | 117.0(5) |
| C(2)-N(2)-Cu(1) | 108.0(5) | C(4)-N(2)-Cu(1) | 99.2(4) |
| C(7)-N(3)-C(6) | 109.8(6) | C(7)-N(3)-C(5) | 112.1(6) |
| C(6)-N(3)-C(5) | 109.4(6) | C(7)-N(3)-Cu(1) | 100.0(5) |
| C(6)-N(3)-Cu(1) | 119.2(5) | C(5)-N(3)-Cu(1) | 106.0(5) |
| C(8)-N(4)-C(11) | 113.1(6) | C(8)-N(4)-C(9) | 111.4(6) |
| C(11)-N(4)-C(9) | 113.0(6) | C(8)-N(4)-Cu(1) | 106.4(5) |
| C(11)-N(4)-Cu(1) | 109.9(5) | C(9)-N(4)-Cu(1) | 102.3(5) |
| C(15)-N(5)-C(12) | 110.4(6) | C(15)-N(5)-C(14) | 110.6(6) |
| C(12)-N(5)-C(14) | 112.1(6) | C(15)-N(5)-Cu(1) | 115.7(5) |
| C(12)-N(5)-Cu(1) | 101.4(4) | C(14)-N(5)-Cu(1) | 106.4(4) |
| N(1)-C(1)-C(2) | 110.3(6) | N(2)-C(2)-C(1) | 111.5(7) |
| N(2)-C(4)-C(5) | 110.1(6) | N(3)-C(5)-C(4) | 110.9(6) |
| N(3)-C(7)-C(8) | 110.3(6) | N(4)-C(8)-C(7) | 110.3(7) |
| N(4)-C(9)-C(10) | 109.7(6) | N(1)-C(10)-C(9) | 109.9(6) |
| N(4)-C(11)-C(12) | 111.6(6) | N(5)-C(12)-C(11) | 111.5(6) |

| | | | |
|---|---|---|---|
| N(1)-C(13)-C(14) | 112.1(7) | N(5)-C(14)-C(13) | 110.9(6) |
| O(2)-Cl(2A)-O(1) | 115.7(11) | O(2)-Cl(2A)-O(4) | 109.9(11) |
| O(1)-Cl(2A)-O(4) | 112.9(11) | O(2)-Cl(2A)-O(3) | 107.1(11) |
| O(1)-Cl(2A)-O(3) | 103.8(11) | O(4)-Cl(2A)-O(3) | 106.6(12) |

Table S14. Anisotropic displacement parameters [Å$^2$x10$^3$] for **Cu(3)$^{2+}$**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+ ... +2hka^*b^*U_{12}]$

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| Cu(1) | 21(1) | 26(1) | 31(1) | 0(1) | 1(1) | 2(1) |
| N(1) | 27(4) | 37(4) | 25(3) | 3(3) | 3(3) | 5(3) |
| N(2) | 22(4) | 34(4) | 29(3) | -1(3) | 1(3) | 6(3) |
| N(3) | 26(4) | 33(4) | 34(4) | -3(3) | -3(3) | 1(3) |
| N(4) | 34(4) | 32(4) | 30(4) | -1(3) | 10(3) | -2(3) |
| N(5) | 24(4) | 34(4) | 27(3) | 0(3) | -2(3) | 1(3) |
| C(1) | 35(5) | 53(6) | 36(5) | 2(4) | -10(4) | 8(4) |
| C(2) | 31(5) | 53(6) | 35(5) | 0(4) | -4(4) | 3(4) |
| C(3) | 41(6) | 45(5) | 46(5) | 12(4) | 6(4) | -8(4) |
| C(4) | 40(6) | 36(5) | 39(5) | -8(4) | 1(4) | -3(4) |
| C(5) | 45(6) | 36(5) | 52(6) | 0(4) | 0(4) | 8(4) |
| C(6) | 34(5) | 47(5) | 39(5) | 1(4) | 1(4) | 12(4) |
| C(7) | 36(5) | 42(5) | 27(4) | -5(4) | 9(4) | 3(4) |
| C(8) | 27(5) | 51(6) | 43(5) | -6(4) | 6(4) | 4(4) |
| C(9) | 38(5) | 39(5) | 39(5) | 9(4) | 5(4) | -6(4) |
| C(10) | 29(5) | 49(5) | 48(5) | 12(4) | 2(4) | 3(4) |
| C(11) | 38(6) | 36(5) | 52(6) | -5(4) | 6(4) | -13(4) |
| C(12) | 30(5) | 37(5) | 55(6) | -14(4) | -3(4) | -10(4) |
| C(13) | 46(6) | 33(5) | 39(5) | 5(4) | 0(4) | 19(4) |
| C(14) | 39(5) | 24(4) | 47(5) | -4(4) | 2(4) | 11(4) |
| C(15) | 44(6) | 57(6) | 25(4) | -2(4) | 3(4) | 4(4) |
| Cl(1) | 22(2) | 187(5) | 41(2) | -4(2) | 2(1) | -7(2) |
| Cl(2) | 32(4) | 20(4) | 10(3) | -5(3) | 2(2) | 0(3) |
| Cl(2A) | 23(6) | 65(10) | 122(12) | 28(8) | -14(6) | -5(6) |
| O(1) | 114(13) | 30(8) | 25(7) | -17(6) | 18(7) | 11(7) |
| O(2) | 57(10) | 35(8) | 34(7) | -18(6) | 6(6) | 9(7) |
| O(3) | 158(14) | 90(10) | 93(10) | -20(8) | 19(9) | 10(10) |
| O(4) | 180(20) | 59(11) | 64(13) | 11(9) | 68(13) | -2(12) |

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