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Crystal structure analysis of Crystal structure of dichloridobis(2-ethylimidazole) copper(II)

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Abstract : In the title compound, $C_{10}H_{16}Cl_2CuN_4$ was synthesized by the reaction of copper dichloride and 2-ethylimidazole. Cu^{2+}cation is coordinated by two Cl-anions and the N atoms of two ethylimidazole ligands, forming a distorted $Cu(N_2Cl_2)$ tetrahedron. The two ethylimidazole rings are almost perpendicular to one another, making a dihedral angle of 34.9(3)°. Crystal data were collected using CrysAlis CCD Oxford Diffraction X-ray diffractometer. The structure was solved by direct methods and refined on F2 by full-matrix least-squares procedures to the final R1 of 0.0370 using SHELXL programs.
Key Words : Imidazole, Copper (II) and crystal structure.

Introduction

Copper (II) complexes with Schiff base ligands have received much attention in recent years[1,2,3]. Some of the complexes have been found to have pharmacological and antitumor properties[4]. We have recently reported a few transition metal complexes[5,6,7].

Experimental

X-ray Structure Determination

Single crystal of the compound suitable for x-ray diffraction was obtained by slow evaporation method. Three dimensional intensity data were collected on a CrysAlis CCD[8] Oxford diffraction Xcalibur diffractometer with Eos detector using graphite monochromatized Mo-Kα radiation (λ= 0.71073 Å) at Department of chemistry, Department of Chemistry, Pondicherry University, Pondicherry 605 014, India. The structure was solved by direct methods and refined on F^2 by full-matrix least-squares procedures using the SHELXL programs[9]. All the non-hydrogen atoms were refined using isotropic and later anisotropic thermal parameters. The hydrogen atoms were included in the structure factor calculation at idealized positions by using a riding model, but not refined. Images were created with ORTEP-3[10]. The crystallographic data for the compound are listed in Table 1.

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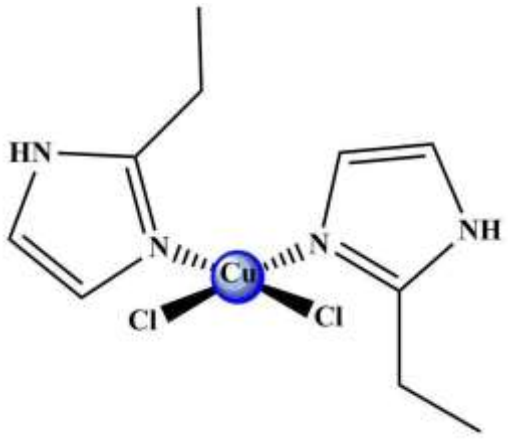
Table 1: Crystal data and structure refinement of the titled compound

Compound	**Parameters**
Empirical formula	C10 H16 Cl2 Cu N4
Formula weight	326.71
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P21/c
Unit cell dimensions	a = 8.7151(4) Å alpha = 90° b = 14.1628(6) Å beta = 102.979(4)° c = 11.9206(5) Å gamma = 90°.
Volume	1433.77(11) $Å^3$
Z, Calculated density	4, 1.514 Mg/m^3
Absorption coefficient	1.880 mm^{-1}
F(000)	668
Crystal size	0.25 x 0.30 x 0.20 mm
Theta range for data collection	3.58 to 25.00°.
Limiting indices	-10<=h<=8, -16<=k<=16, -14<=l<=14
Reflections collected / unique	5760 / 2495 [R(int) = 0.0255]
Completeness to theta = 25.00	98.80%
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2495 / 0 / 156
Goodness-of-fit on F^2	1.057
Final R indices [I>2sigma(I)]	R1 = 0.0403, wR2 = 0.1046
R indices (all data)	R1 = 0.0501, wR2 = 0.1102
Largest diff. peak and hole	0.685 and -0.664 e. $Å^{-3}$

Synthesis of the compound



Copper(II) complexes are known to play a significant role in naturally occurring biological systems or as pharmacological agents. The biological activity and the medical implications of copper(II) complexes are influenced by the manner in which the copper(II) ions coordinate with the ligand molecules. Cooperative interactions have commonly been observed in biological systems that perform an extraordinary array of catalytic transformations. An ethanol solution (10mL) of 2-ethylimidazole (2m) was slowly added at ambient temperature to a magnetically stirred ethanol solution (10 mL) of $CuCl_2.2H_2O$ (1 M). The whole reaction mixture was stirred for 5 hour and the resulting crystals were filtered, washed with water, and dried in desiccator over P_2O_5. It was then filtered to discard any insoluble precipitates. The X-ray-quality sky-blue plate-shaped single crystals were obtained from hot ethanol after seven days.

Results and Discussion

In the title compound (Fig.1), the copper atom has a slightly distorted tetrahedral geometry, being coordinated by the atoms N1 and N2 of two ethylimidazole molecules and two Cl^- anions. The two ethylimidazole rings (N1-C6-N4-C8-C7) and (N2-C1-C2-C3-N3) are perpendicular to one another with a dihedral angle of 34.9(3)°. The bond distance Cu-Cl and Cu-Cl are 2.267(11) Å and 1.966(3) Å, and the bond angles Cl-Cu-Cl and N-Cu-N are 144.13(5)° and 155.34(3)°. All bond lengths and bond angles in (I) are in the range of expected values. The latter two rings are coplanar with atoms C4 and C9 deviate from the two imidazole rings by -0.035 Å and -0.009 Å, respectively.

In the crystal, molecules are linked via pairs of N---H...Cl hydrogen bonds, forming chains propagating along the a axis. There are a number of π—π interactions present linking the ribbons and forming a three dimensional structure. The selected bond lengths and angles are listed in table 3 and 4, respectively.

Table 2: Hydrogen-bond geometry [Å]

D—H…A	D—H	H…A	D…A	D—H…A
N3-H3…Cl2	0.86	2.77	3.595(5)	162

Symmetry code: -1+x,y,z

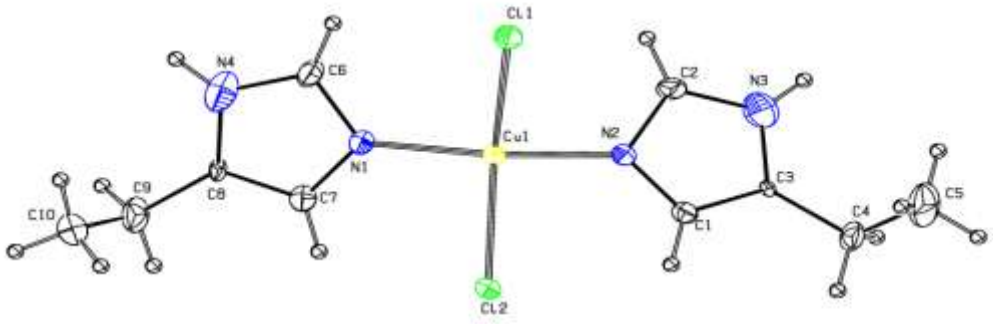


Fig.1. The molecular structure of the title compound, with the atom-numbering scheme. The displacement ellipsoids are drawn at 30% probability level. H atoms are shown as spheres of arbitrary radius.

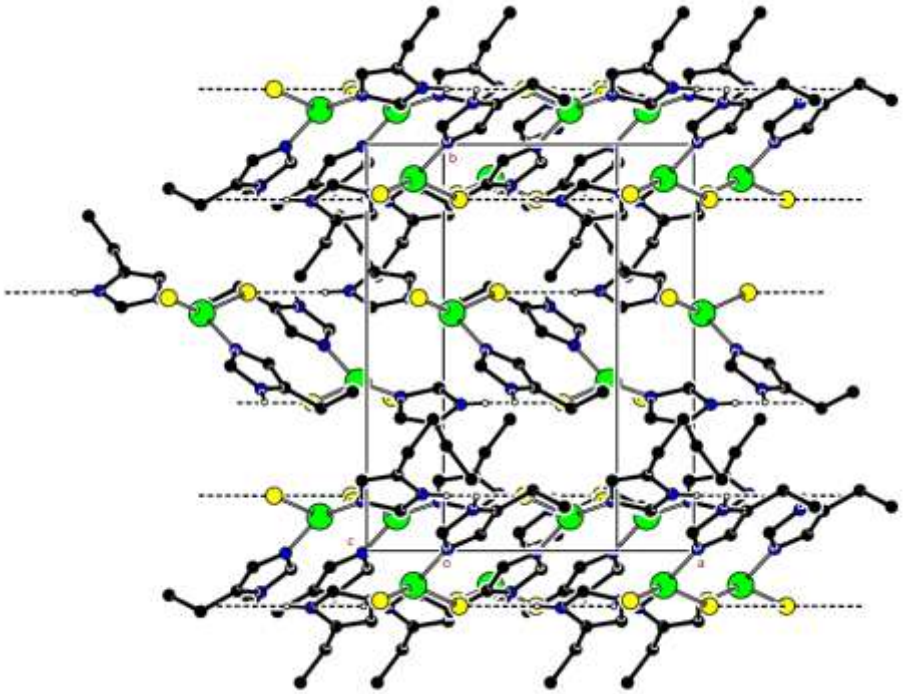


Fig.2. The crystal packing of the title compound, viewed along b axis, showing N---H...Cl hydrogen bonds chains parallel to c axis. Hydrogen bond omitted clarity (see Table 2 for details).

Table 3: Selected Bond lengths (Å) Table 4: Selected Bond angles (º)

Bond	lengths (Å)
C(1)-N(2)	1.319(5)
C(1)-C(3)	1.331(5)
C(1)-H(1)	0.93
C(2)-N(3)	1.361(6)
C(2)-N(2)	1.386(5)
C(2)-H(2)	0.93
C(3)-N(3)	1.363(6)
C(3)-C(4)	1.466(5)
C(4)-C(5)	1.491(8)
C(4)-H(4A)	0.97
C(4)-H(4B)	0.97
C(5)-H(5A)	0.96
C(5)-H(5B)	0.96
C(5)-H(5C)	0.96
C(6)-N(4)	1.359(6)
C(6)-N(1)	1.381(5)
C(6)-H(6)	0.93
C(7)-N(1)	1.316(5)
C(7)-C(8)	1.335(5)
C(7)-H(7)	0.93
C(8)-N(4)	1.348(6)
C(8)-C(9)	1.464(6)
C(9)-C(10)	1.500(8)
C(9)-H(9A)	0.97
C(9)-H(9B)	0.97
C(10)-H(10A)	0.96
C(10)-H(10B)	0.96

C(10)-H(10C)	0.96
N(1)-Cu(1)	1.966(3)
N(2)-Cu(1)	1.956(3)
N(3)-H(3)	0.86

Bond	**Angles (°)**
N(2)-C(1)-C(3)	111.9(3)
N(2)-C(1)-H(1)	124.1
C(3)-C(1)-H(1)	124.1
N(3)-C(2)-N(2)	108.4(4)
N(3)-C(2)-H(2)	125.8
N(2)-C(2)-H(2)	125.8
C(1)-C(3)-N(3)	107.3(3)
C(1)-C(3)-C(4)	126.7(3)
N(3)-C(3)-C(4)	126.0(3)
C(3)-C(4)-C(5)	112.7(4)
C(3)-C(4)-H(4A)	109.1
C(5)-C(4)-H(4A)	109.1
C(3)-C(4)-H(4B)	109.1
C(5)-C(4)-H(4B)	109.1
H(4A)-C(4)-H(4B)	107.8
C(4)-C(5)-H(5A)	109.5
C(4)-C(5)-H(5B)	109.5
H(5A)-C(5)-H(5B)	109.5
C(4)-C(5)-H(5C)	109.5
H(5A)-C(5)-H(5C)	109.5
H(5B)-C(5)-H(5C)	109.5
N(4)-C(6)-N(1)	108.1(4)
N(4)-C(6)-H(6)	126
N(1)-C(6)-H(6)	126
N(1)-C(7)-C(8)	111.6(4)
N(1)-C(7)-H(7)	124.2
C(8)-C(7)-H(7)	124.2
C(7)-C(8)-N(4)	107.1(4)
C(7)-C(8)-C(9)	126.8(4)
N(4)-C(8)-C(9)	126.1(4)
C(8)-C(9)-C(10)	113.3(4)

Conclusion

The crystal structure analysis of a novel copper dichloride and 2-ethylimidazole compound was studied using x-ray diffraction method. In the crystal, molecules are linked by N---H…Cl hydrogen bonds, forming chains along c axis. There are a number of π—π interactions present linking the ribbons and forming a three dimensional structure.

Acknowledgments

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