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The crystal structure of hexakis(1-propylimidazole- κ^1N)copper(II) dichloride, $C_{36}H_{60}Cl_2CuN_{12}$

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Abstract

$C_{36}H_{60}Cl_2CuN_{12}$, monoclinic, $P2_1/c$ (no. 14), $a = 8.3112(2)$ Å, $b = 15.9513(4)$ Å, $c = 17.1068(4)$ Å, $\beta = 99.671(2)^\circ$, $V = 2235.69(9)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0523$, $wR_{ref}(F^2) = 0.1606$, $T = 298(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Table 1 contains crystallographic data and

Table 1: Data collection and handling.

Crystal:	Blue block
Size:	$0.19 \times 0.18 \times 0.16$ mm
Wavelength:	Cu $K\alpha$ radiation (1.54178 Å)
μ :	2.09 mm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX II, φ and ω
θ_{max} , completeness:	66.0° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8002, 3880, 0.031
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3310
$N(\text{param})_{\text{refined}}$:	263
Programs:	Bruker [1], SHELX [2]

Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

In a typical experiment, copper chloride (1 mmol) and 1-propylimidazole (10 mmol) were dissolved in H_2O (25 mL), and then maintained for 6 h at 80°C with stirring. After the reaction was completed, the filtrate was left to slowly evaporate at room temperature for about four days, and then the blue rod crystals were filtered off. Yield: 54.5%. **Anal. Calcd.** for $C_{36}H_{60}Cl_2CuN_{12}$: C, 54.36; H, 7.60; N, 21.13; found: C, 54.39; H 7.52; N 21.18.

Experimental details

Hydrogen atoms were assigned with common isotropic displacement factors $U_{\text{iso}}(\text{H}) = 1.2$ times U_{eq} (C, imidazole ring and methylene) and $U_{\text{iso}}(\text{H}) = 1.5$ times U_{eq} (C, methyl carbon). All the H atoms were refined as riding on their parent atom.

Comment

Over the last decades, transition metal complexes have received much attention in the fields of catalysis, magnetism, medicine and material because their biocompatibility, various coordination modes, and catalytic properties [3–5]. Among these transition metal

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
Cu1	1.000000	0.500000	0.500000	0.0424 (3)
Cl1	0.34311 (8)	0.23789 (5)	0.18672 (5)	0.0611 (3)
N1	0.7656 (3)	0.47176 (16)	0.38734 (15)	0.0571 (6)
N2	0.5683 (4)	0.4642 (2)	0.2853 (2)	0.0857 (10)
N3	1.1108 (2)	0.58182 (13)	0.43467 (12)	0.0431 (5)
N4	1.1638 (3)	0.68223 (15)	0.35540 (14)	0.0550 (6)
N5	1.1232 (2)	0.40701 (13)	0.45399 (12)	0.0435 (5)
N6	1.2850 (3)	0.30316 (15)	0.43430 (15)	0.0563 (6)
C1	0.6735 (4)	0.5141 (2)	0.3341 (2)	0.0686 (9)
H1	0.677997	0.572144	0.329398	0.082*
C2	0.7166 (4)	0.3898 (2)	0.3731 (2)	0.0655 (8)
H2	0.761207	0.344017	0.402716	0.079*
C3	0.5970 (4)	0.3853 (2)	0.3111 (2)	0.0687 (9)
H3	0.543694	0.337120	0.289833	0.082*
C4 ^a	0.4812 (11)	0.4961 (8)	0.2043 (6)	0.117 (3)
H4A ^a	0.522219	0.550971	0.192984	0.140*
H4B ^a	0.498600	0.457833	0.162470	0.140*
C5 ^a	0.3009 (13)	0.5010 (10)	0.2099 (9)	0.179 (5)
H5A ^a	0.287869	0.522777	0.261381	0.215*
H5B ^a	0.253229	0.445406	0.204179	0.215*
C6 ^a	0.214 (2)	0.5579 (15)	0.1451 (10)	0.192 (7)
H6A ^a	0.099801	0.560769	0.148930	0.288*
H6B ^a	0.260183	0.613104	0.151236	0.288*
H6C ^a	0.225622	0.535911	0.094166	0.288*
C7	1.2756 (3)	0.58484 (19)	0.43375 (18)	0.0548 (7)
H7	1.352791	0.549337	0.462220	0.066*
C8	1.3086 (3)	0.6467 (2)	0.38562 (19)	0.0576 (7)
H8	1.410783	0.662038	0.375137	0.069*
C9	1.0478 (3)	0.64154 (18)	0.38679 (16)	0.0504 (6)
H9	0.937058	0.654276	0.375812	0.060*
C10 ^b	1.1326 (16)	0.7546 (3)	0.3014 (5)	0.084 (2)
H10A ^b	1.017823	0.756162	0.278271	0.101*
H10B ^b	1.195080	0.748428	0.258673	0.101*
C11 ^b	1.1789 (12)	0.8352 (3)	0.3445 (5)	0.130 (3)
H11A ^b	1.297104	0.838427	0.354227	0.156*
H11B ^b	1.139825	0.880649	0.308553	0.156*
C12 ^b	1.1248 (17)	0.8517 (5)	0.4179 (5)	0.194 (5)
H12A ^b	1.164468	0.905440	0.437623	0.291*
H12B ^b	1.007632	0.851524	0.409712	0.291*
H12C ^b	1.165814	0.809060	0.455648	0.291*
C13	1.1163 (4)	0.3951 (2)	0.37366 (16)	0.0554 (7)
H13	1.053504	0.426649	0.334101	0.066*
C14	1.2137 (4)	0.3311 (2)	0.36114 (18)	0.0600 (8)
H14	1.229418	0.309926	0.312335	0.072*
C15	1.2274 (3)	0.35043 (17)	0.48787 (16)	0.0514 (6)
H15	1.257194	0.344109	0.542421	0.062*
C16 ^c	1.4024 (8)	0.2337 (4)	0.4495 (8)	0.084 (2)
H16A ^c	1.486449	0.248519	0.493891	0.101*
H16B ^c	1.454646	0.226089	0.403419	0.101*
C17 ^c	1.3268 (11)	0.1542 (4)	0.4673 (7)	0.143 (3)
H17A ^c	1.402537	0.110037	0.459102	0.172*
H17B ^c	1.322256	0.154744	0.523596	0.172*
C18 ^c	1.1680 (12)	0.1279 (5)	0.4275 (9)	0.214 (6)
H18A ^c	1.141695	0.074445	0.447833	0.320*
H18B ^c	1.087816	0.168467	0.436658	0.320*

Table 2: (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
H18C ^c	1.168747	0.123399	0.371642	0.320*
C4' ^d	0.4037 (12)	0.4818 (9)	0.2431 (7)	0.117 (3)
H4'1 ^d	0.337798	0.506682	0.278720	0.140*
H4'2 ^d	0.351338	0.430512	0.221587	0.140*
C5' ^d	0.4211 (19)	0.5410 (11)	0.1781 (9)	0.179 (5)
H5'1 ^d	0.517191	0.525766	0.156058	0.215*
H5'2 ^d	0.438319	0.596990	0.200086	0.215*
C6' ^d	0.275 (2)	0.5424 (17)	0.1120 (10)	0.192 (7)
H6'1 ^d	0.293794	0.581778	0.072074	0.288*
H6'2 ^d	0.258372	0.487558	0.088967	0.288*
H6'3 ^d	0.179436	0.558840	0.133030	0.288*
C10 ^e	1.155 (8)	0.7605 (13)	0.309 (2)	0.084 (2)
H10C ^e	1.091742	0.750621	0.256466	0.101*
H10D ^e	1.264347	0.776844	0.301862	0.101*
C11 ^e	1.077 (5)	0.8314 (12)	0.349 (3)	0.130 (3)
H11C ^e	1.104128	0.823811	0.405777	0.156*
H11D ^e	0.959854	0.827299	0.334146	0.156*
C12 ^e	1.130 (6)	0.9192 (12)	0.329 (2)	0.194 (5)
H12D ^e	1.075663	0.959902	0.356188	0.291*
H12E ^e	1.245930	0.924701	0.344188	0.291*
H12F ^e	1.101397	0.928196	0.272428	0.291*
C16 ^f	1.378 (4)	0.2276 (12)	0.457 (4)	0.084 (2)
H16C ^f	1.410587	0.226946	0.514340	0.101*
H16D ^f	1.475542	0.227769	0.433379	0.101*
C17 ^f	1.278 (6)	0.1485 (11)	0.431 (4)	0.143 (3)
H17C ^f	1.170614	0.152956	0.445694	0.172*
H17D ^f	1.265615	0.142022	0.374213	0.172*
C18 ^f	1.368 (5)	0.0725 (14)	0.472 (4)	0.214 (6)
H18D ^f	1.306957	0.022528	0.456631	0.320*
H18E ^f	1.474257	0.068255	0.457646	0.320*
H18F ^f	1.379562	0.079153	0.528897	0.320*

^a Occupancy: 0.524(7), ^b Occupancy: 0.817(8), ^c Occupancy: 0.824(10), ^d Occupancy: 0.476(7), ^e Occupancy: 0.183(8), ^f Occupancy: 0.176(10).

complexes, copper complexes with their versatile structures, redox behavior and physicochemical properties have been found to be useful as active agents in chemotherapeutic, catalytic applications. So far, some thrilling work about inorganic-organic hybrid copper complexes have been reported [6–8]. In addition, imidazoles as multifunctional organic reagents could be used not only as organic base, solvent, but also as N-ligands in the synthesis of transition metal complexes, and some imidazole-based compounds, including some transition metal clusters have been reported [9–11]. However, despite the progress achieved, newly designed imidazole-based crystalline copper materials are long-sought-after yet still unmet.

Single crystal X-ray diffraction analysis reveals that the asymmetric unit of the title structure consist of half a copper(II) cation, three 1-propylimidazole N-ligands and one free chloride anion. The Cu(II) cation adopted a six-

coordinated mode with a twisted octahedral geometry conformation. The bond distances of Cu–N are in the range of 2.034(2) to 2.540(2) Å, which are similar with those of the reported tetrakis (N-methylimidazole-*N'*)-copper(I) perchlorate complex [12]. Meanwhile, the bond angles of N(5)–Cu(1)–N(3), N(5)–Cu(1)–N(1), and N(3)–Cu(1)–N(1) are 87.22(9), 87.07(8) and 93.27(8)°, respectively. Moreover, the disordered atom sets were (C4, C4'), (C5, C5'), (C6, C6'), (C10, C10'), (C11, C11'), (C12, C12'), (C16, C16'), (C17, C17'), and (C18, C18'), respectively.

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