Guo-Xi Wang* and Zheng Xing

The crystal structure of dichlorido- μ_2 -dichlorido- $(\eta^2$ -1,4-bis(4-vinylbenzyl)-1,4-diazabicyclo[2.2.2]octane-1,4-diium)dicopper(I), $C_{24}H_{30}N_2Cu_2Cl_4$

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Abstract

$C_{24}H_{30}N_2Cu_2Cl_4$, triclinic, $P\bar{1}$ (no. 2), $a = 7.204(4)$ Å, $b = 8.593(5)$ Å, $c = 21.112(12)$ Å, $\alpha = 89.153(7)^\circ$, $\beta = 85.294(7)^\circ$, $\gamma = 84.604(6)^\circ$, $V = 1296.7(12)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0535$, $wR_{ref}(F^2) = 0.1471$, $T = 296(2)$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The educt 1,4-bis(4-vinylbenzyl)-1,4-diazabicyclo[2.2.2]octane-1,4-diium chloride (0.1 mmol), copper(I) chloride (0.3 mmol), methanol (1.5 mL) and pure water (0.4 mL) were placed in the heat-resistant glass tube. The mixture was stirred, and then the tube was frozen with liquid nitrogen, and finally

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.18 × 0.17 × 0.16 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	2.07 mm ^{−1}
Diffraction, scan mode:	Bruker Apex, φ and ω
θ_{max} , completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	20772, 4567, 0.055
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 3021
$N(param)_{refined}$:	338
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

evacuated under vacuum and sealed. The tube was allowed to stand under hydrothermal reaction conditions for 4 days to obtain quality yellow crystals.

Experimental details

H-atoms are positioned geometrically (Table 2). The largest difference electron density peak is found between the Cu atoms.

Comment

The use of 4-vinylbenzyl derivatives of heterocyclic compounds as ligands in d¹⁰metal η^2 -olefin-complexes are of interest in coordination and organometallic chemistry. They have several of potential applications, such as fluorescence, dielectric, catalytic organic reaction and so on [5–9]. In 1983, Thompson and his coworker reported the synthesis

*Corresponding author: Guo-Xi Wang, Zhenjiang Key Laboratory of Functional Chemistry and Institute of Medicine, Zhenjiang College, Zhenjiang Jiangsu 212028, P.R. China, e-mail: aydxwqx@163.com

Zheng Xing: Zhenjiang Key Laboratory of Functional Chemistry and Institute of Medicine and Chemical Engineering, Zhenjiang College, Zhenjiang Jiangsu 212028, P.R. China

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	0.58550(9)	0.81257(8)	0.34333(3)	0.0475(2)
Cu2	0.69272(11)	0.65681(10)	0.20477(5)	0.0787(3)
Cl2	0.66043(18)	0.55044(15)	0.33670(7)	0.0474(4)
Cl1	0.28066(19)	0.85704(18)	0.32683(9)	0.0682(5)
Cl3	0.7505(2)	0.88745(18)	0.22295(8)	0.0610(4)
Cl4	0.6727(2)	0.44493(19)	0.15778(8)	0.0644(5)
N1	0.8161(6)	0.7733(5)	0.79798(19)	0.0367(10)
N2	0.8289(5)	0.6289(4)	0.69253(18)	0.0316(9)
C1	0.6543(9)	1.0067(6)	0.3874(3)	0.0543(15)
H1A	0.5590	1.0331	0.4188	0.065*
H1B	0.6565	1.0594	0.3486	0.065*
C2	0.7904(8)	0.8914(7)	0.3981(3)	0.0465(14)
H2	0.9114	0.9028	0.3746	0.056*
C3	0.8009(7)	0.7997(6)	0.4582(2)	0.0390(12)
C4	0.9721(7)	0.7616(6)	0.4823(3)	0.0445(13)
H4	1.0798	0.7927	0.4603	0.053*
C5	0.9862(7)	0.6780(7)	0.5385(3)	0.0477(14)
H5	1.1028	0.6532	0.5538	0.057*
C6	0.8272(7)	0.6313(6)	0.5720(2)	0.0349(12)
C7	0.6573(8)	0.6655(6)	0.5471(2)	0.0453(14)
H7	0.5501	0.6310	0.5682	0.054*
C8	0.6436(8)	0.7501(7)	0.4916(3)	0.0488(14)
H8	0.5269	0.7742	0.4763	0.059*
C9	0.8413(7)	0.5335(6)	0.6311(2)	0.0404(13)
H9A	0.9593	0.4686	0.6276	0.048*
H9B	0.7417	0.4646	0.6340	0.048*
C10	0.9759(7)	0.7409(6)	0.6902(3)	0.0430(13)
H10A	1.0970	0.6863	0.6776	0.052*
H10B	0.9495	0.8221	0.6590	0.052*
C11	0.9805(7)	0.8129(7)	0.7540(3)	0.0474(14)
H11A	0.9781	0.9256	0.7492	0.057*
H11B	1.0958	0.7753	0.7722	0.057*
C12	0.8577(8)	0.5156(6)	0.7463(2)	0.0436(13)
H12A	0.9825	0.4619	0.7407	0.052*
H12B	0.7683	0.4380	0.7462	0.052*
C13	0.8333(7)	0.6002(6)	0.8096(2)	0.0387(12)
H13A	0.7219	0.5706	0.8340	0.046*
H13B	0.9401	0.5710	0.8338	0.046*
C14	0.6455(7)	0.7216(6)	0.7059(2)	0.0416(13)
H14A	0.5471	0.6516	0.7102	0.050*
H14B	0.6219	0.7916	0.6703	0.050*
C15	0.6406(7)	0.8157(6)	0.7656(2)	0.0381(12)
H15A	0.6301	0.9263	0.7552	0.046*
H15B	0.5326	0.7944	0.7938	0.046*
C16	0.8176(9)	0.8638(7)	0.8596(3)	0.0559(16)
H16A ^a	0.9432	0.8497	0.8735	0.067*
H16B ^a	0.7906	0.9741	0.8503	0.067*
H16C ^b	0.8183	0.9744	0.8497	0.067*
H16D ^b	0.9313	0.8307	0.8797	0.067*
C17 ^a	0.692(2)	0.8233(18)	0.9107(6)	0.045(3)
C18 ^a	0.732(2)	0.7171(16)	0.9563(6)	0.055(3)
H18 ^a	0.8506	0.6629	0.9542	0.066*
C19 ^a	0.6061(19)	0.6862(15)	1.0056(6)	0.059(3)
H19 ^a	0.6422	0.6166	1.0374	0.071*
C20 ^a	0.423(2)	0.7578(16)	1.0087(5)	0.056(3)

Table 2 (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C21 ^a	0.373(2)	0.8696(15)	0.9620(6)	0.055(3)
H21 ^a	0.2533	0.9202	0.9625	0.066*
C22 ^a	0.513(2)	0.9008(18)	0.9147(6)	0.054(4)
H22 ^a	0.4845	0.9772	0.8844	0.064*
C23 ^a	0.2832(16)	0.7225(14)	1.0628(6)	0.075(3)
H23 ^a	0.3306	0.6634	1.0961	0.090*
C24 ^a	0.1033(18)	0.7647(17)	1.0687(7)	0.097(4)
H24A ^a	0.0477	0.8240	1.0370	0.116*
H24B ^a	0.0311	0.7354	1.1047	0.116*
C17A ^b	0.641(2)	0.838(3)	0.9084(9)	0.022(7)
C18A ^b	0.468(3)	0.924(3)	0.9065(10)	0.031(7)
H18A ^b	0.4483	0.9975	0.8744	0.037*
C19A ^b	0.324(2)	0.900(3)	0.9527(11)	0.070(13)
H19A ^b	0.2085	0.9579	0.9515	0.084*
C20A ^b	0.353(2)	0.791(3)	1.0008(9)	0.046(8)
C21A ^b	0.526(3)	0.704(2)	1.0026(8)	0.032(6)
H21A ^b	0.5454	0.6310	1.0348	0.039*
C22A ^b	0.670(2)	0.728(3)	0.9564(10)	0.043(9)
H22A ^b	0.7853	0.6706	0.9577	0.051*
C23A ^b	0.191(4)	0.775(4)	1.0482(13)	0.075(3)
H23A ^b	0.0722	0.7986	1.0345	0.090*
C24A ^b	0.201(5)	0.735(4)	1.1036(15)	0.097(4)
H24C ^b	0.3173	0.7105	1.1193	0.116*
H24D ^b	0.0924	0.7284	1.1302	0.116*

^aOccupancy: 0.699(13), ^bOccupancy: 0.301(13).

of the first stable copper (I) olefin coordination complex [10]. Recent results of structure investigations of copper(I) π -complexes with 4-vinylbenzyl derivatives could conduct previously unknown inorganic moieties [11–17].

X-ray crystal analysis of the title complex shows that there are two different coordination environments for the copper atoms in the crystal structure. The asymmetric unit consists of two copper(I) atoms, two μ_2 chloride, two chloride and one organic ligand. The central Cu1 atom coordinates with the olefin group of the ligand, two μ_2 chloride atoms and one chloride atom to form a twist tetrahedral structure (see the figure). The Cu2 atom coordinates with two μ_2 chloride atoms and one chloride atom to accommodate the deformed triangular geometry. The length of C1–C2 is 1.360(8) Å and the distant of C23–C24 is 1.226(12) Å. Compared with the coordinated olefin moiety, the distance of the uncoordinated olefin moiety is slightly longer [18, 19].

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