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Crystal structure of aqua-bis(3,4-dimethoxybenzoato- $\kappa^1\text{O}$)-(2,2'-bipyridine- $\kappa^2\text{N},\text{N}'$) copper(II), $\text{C}_{28}\text{H}_{26}\text{CuN}_2\text{O}_9$

Table 1: Data collection and handling.

Crystal:	Blue block
Size:	$3.1 \times 2.9 \times 2.7$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.89 mm^{-1}
Diffractometer, scan mode:	CCD Area Detector, φ and ω
θ_{max} , completeness:	28.3° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8180, 3161, 0.065
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2831
$N(\text{param})_{\text{refined}}$:	188
Programs:	Bruker [10], SHELX [11]

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Abstract

$\text{C}_{28}\text{H}_{26}\text{CuN}_2\text{O}_9$, monoclinic, $C2/c$, $a = 12.3146(11)$ Å, $b = 11.1769(10)$ Å, $c = 19.3856(17)$ Å, $\beta = 101.012(1)^\circ$, $V = 2619.1(4)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0350$, $wR_{\text{ref}}(F^2) = 0.1005$, $T = 296(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

3,4-Dimethoxybenzoic acid (364 mg, 2 mmol), copper(II) acetate (199 mg, 1 mmol) and 2,2'-bipyridine (156 mg,

1 mmol) were added to DMF (15 mL). The reaction mixture was heated for 4 h and the reaction mixture was filtered. The resulting blue filtration was allowed to evaporate slowly for about 1 week, obtaining blue block crystals. Yield: 68% (base on Cu).

Experimental details

Hydrogen atoms were assigned with isotropic displacement factors $U_{\text{iso}}(\text{H}) = 1.2$ times U_{eq} (C, pyridyl and benzene ring), and $U_{\text{iso}}(\text{H}) = 1.5$ times U_{eq} (O, water and methyl group), and refined riding on their parent atom.

Comment

The complexes from carboxylates are among the most investigated complexes in the field of coordination chemistry because carboxylates may act as chelate ligands [1, 2]. Diamondoid networks have attracted great attention due to their robust structural topologies and potential applications in functional materials [3–6]. On the other hand, pyridine ligands, such as 2,2'-bipyridine can rotate in a narrow range to meet the requirement of coordination geometries of metal ions in the assembly process. 3,4-Dimethoxybenzoate is a common organic ligand for studies on metal carboxylate complexes owing to their interesting structural and chemical properties [7].

In this structure, the asymmetric unit consists of one half of the Cu(II) title complex (cf. the figure). Each Cu(II) is coordinated by two O atoms of the 3,4-dimethoxybenzoate ligands and by the two N atoms from the 2,2'-bipyridine molecule.

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
Cu1	1.000000	0.04769(2)	0.250000	0.02846(11)
O1	1.00180(10)	0.12579(11)	0.12579(7)	0.0395(3)
O2	0.89787(11)	0.16841(11)	0.20333(7)	0.0393(3)
O3	0.88198(12)	0.44266(12)	−0.06468(8)	0.0490(4)
O4	0.72277(13)	0.55443(13)	−0.02947(8)	0.0541(4)
N1	1.10352(11)	−0.08721(13)	0.27982(7)	0.0299(3)
C1	0.92671(13)	0.18405(14)	0.14431(9)	0.0300(3)
C2	0.86684(13)	0.28052(14)	0.09848(9)	0.0287(3)
C3	0.77860(14)	0.34127(15)	0.11693(9)	0.0329(3)
H3	0.754267	0.320379	0.157861	0.040*
C4	0.72602(14)	0.43298(15)	0.07505(10)	0.0343(4)
H4	0.666122	0.472523	0.087447	0.041*
C5	0.76335(14)	0.46494(15)	0.01492(10)	0.0341(4)
C6	0.85235(14)	0.40267(15)	−0.00446(9)	0.0325(4)
C7	0.90301(13)	0.31136(15)	0.03708(9)	0.0310(3)
H7	0.961577	0.270092	0.024151	0.037*
C8	0.96963(16)	0.38302(19)	−0.08805(11)	0.0481(5)
H8A	1.034541	0.384925	−0.051664	0.072*
H8B	0.984863	0.422239	−0.129227	0.072*
H8C	0.948966	0.301431	−0.099129	0.072*
C10	0.6279(2)	0.6166(2)	−0.01803(14)	0.0679(7)
H10A	0.570509	0.560268	−0.014137	0.102*
H10B	0.602741	0.669535	−0.056776	0.102*
H10C	0.645973	0.662155	0.024594	0.102*
C11	1.21062(14)	−0.07709(17)	0.31032(10)	0.0366(4)
H11	1.240245	−0.001085	0.320218	0.044*
C12	1.27819(15)	−0.17501(19)	0.32747(10)	0.0431(4)
H12	1.351867	−0.165452	0.349075	0.052*
C13	1.23450(15)	−0.28729(18)	0.31200(10)	0.0417(4)
H13	1.278958	−0.354755	0.321902	0.050*
C14	1.12331(15)	−0.29904(16)	0.28143(10)	0.0368(4)
H14	1.092264	−0.374388	0.271389	0.044*
C15	1.05952(12)	−0.19722(14)	0.26617(8)	0.0282(3)
O5	0.000000	0.4254(2)	0.250000	0.0634(6)
H5A	0.0513(19)	0.3892(10)	0.2777(14)	0.152(17)*

Moreover, the bond lengths O—Cu and N—Cu are 1.9458(12) Å and 1.9877(17) Å, which is smaller to those reported in the literature [8, 9], showing the strong stability.

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