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The crystal structure of bis(benzoato- κ^2O,O')-(2,9-dimethyl-1,10-phenanthroline- κ^2N,N')-copper(II), $C_{28}H_{22}CuN_2O_4$

Table 1: Data collection and handling.

Crystal:	Blue block
Size:	0.24 × 0.23 × 0.21 mm
Wavelength:	Cu K α radiation (1.54184 Å)
μ :	1.60 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
$\theta_{\max}$, completeness:	71.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	5189, 2261, 0.017
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2084
$N(\text{param})_{\text{refined}}$:	160
Programs:	Bruker [1], Diamond [2], Olex2 [3], SHELX [4, 5]

Abstract

$C_{28}H_{22}CuN_2O_4$, monoclinic, $C2/c$ (no. 15), $a = 17.2971(3)$ Å, $b = 14.4409(3)$ Å, $c = 9.5617(2)$ Å, $\beta = 90.580(2)^\circ$, $V = 2388.25(8)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0536$, $wR_{\text{ref}}(F^2) = 0.1406$, $T = 293$ K.

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The molecular structure is shown in the figure (30% probability). Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was prepared by mixing of a water solution of $Cu(NO_3)_2$ (0.5 mmol) and sodium benzoate (1 mmol) with an ethanol-water (5:5 mL) solution of 2,9-dimethyl-1,10-phenanthroline (0.5 mmol). The resulting solution was refluxed for 30 min at 323 K. Blue single crystals were obtained by slow evaporation at room temperature after three days.

Experimental details

The H atoms were placed in geometrically idealized positions and refined to in the riding model on their respective parent atoms with $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(\text{aromatic})$ and $U_{\text{iso}}(H) = 1.5U_{\text{eq}}(\text{methyl})$.

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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	0.5000	0.26335 (4)	0.7500	0.0649 (3)
O1	0.62345 (13)	0.25848 (14)	0.6096 (2)	0.0696 (6)
O2	0.51926 (10)	0.17467 (15)	0.60237 (19)	0.0625 (5)
N1	0.55255 (10)	0.36993 (14)	0.85393 (18)	0.0422 (4)
C1	0.61861 (13)	0.13741 (16)	0.4426 (2)	0.0433 (5)
C2	0.69016 (14)	0.1566 (2)	0.3892 (3)	0.0549 (6)
H2	0.7182	0.2067	0.4240	0.066*
C3	0.72118 (16)	0.1027 (2)	0.2846 (3)	0.0659 (8)
H3	0.7697	0.1169	0.2492	0.079*
C4	0.6813 (2)	0.0293 (3)	0.2333 (4)	0.0778 (9)
H4	0.7022	−0.0073	0.1631	0.093*
C5	0.6099 (3)	0.0094 (3)	0.2857 (5)	0.1088 (17)
H5	0.5823	−0.0410	0.2511	0.131*
C6	0.5784 (2)	0.0638 (3)	0.3899 (4)	0.0861 (12)
H6	0.5295	0.0501	0.4241	0.103*
C7	0.58647 (13)	0.19482 (17)	0.5588 (2)	0.0459 (5)
C8	0.52918 (12)	0.45348 (16)	0.8047 (2)	0.0406 (5)
C9	0.55809 (14)	0.53758 (18)	0.8547 (2)	0.0485 (5)
C10	0.61643 (15)	0.5328 (2)	0.9587 (3)	0.0564 (6)
H10	0.6379	0.5868	0.9953	0.068*
C11	0.64104 (14)	0.4488 (2)	1.0048 (3)	0.0577 (7)
H11	0.6801	0.4456	1.0722	0.069*
C12	0.60824 (13)	0.36659 (19)	0.9520 (2)	0.0496 (6)
C13	0.63521 (18)	0.2736 (2)	1.0009 (3)	0.0699 (8)
H13A	0.5916	0.2375	1.0297	0.105*
H13B	0.6704	0.2810	1.0784	0.105*
H13C	0.6610	0.2424	0.9259	0.105*
C14	0.52833 (17)	0.62223 (19)	0.8001 (3)	0.0590 (7)
H14	0.5478	0.6782	0.8331	0.071*

Comment

Transition metal compounds with N-donor ligand have been attracting interest due to their versatile architectures and desirable characteristics, e.g., optical [6, 7], magnetic [8], catalytic [9], and electrical properties [10]. The phenanthroline and its derivatives as an organic ligand, is still a hot topic, and numerous related transition metal compounds have been prepared and investigated [11–14]. For example, Ruan et al. [11] recently reported a dinuclear europium complex based on 2-phenylimidazo [4,5-*f*] [1,10] phenanthroline, and found that it is the first convenient luminescent sensor for clinafloxacin and the limit of detection is 0.21 μM. The title compound, containing the benzoate anion and 2,9-dimethyl-1,10-phenanthroline (DMPHEN) ligand, was recently successfully synthesized and its crystal structure is reported here. Reference survey

showed this structure is isostructural to the Co-complex [8, 12], Mn-complex [13], but different to the structure obtained by the use of CuCl₂ as the reactant instead of Cu(NO₃)₂ [14].

The central Cu(II) atom is six-coordinated in a distorted octahedral geometry by four O atoms from two benzoate anions and two N atoms from one DMPHEN molecule. Crystallographically, the Cu is located on a twofold rotation axis through the middle of the DMPHEN ligands, which makes only a half of the DMPHEN molecule and one benzoate anion to be crystallographically independent as given in the figure. The Cu–N bond length is about 2.0407(19) Å, and two Cu–O distances significantly differ from each other [1.937(2) and 2.5347(19) Å]. The crystal structure is stabilized by weak intermolecular C–H⋯O and C–H⋯N hydrogen bonds, and also by π–π stacking interactions between adjacent dmphen rings and the shortest centroid-centroid distance is about 3.47 Å.

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