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Crystal structure of (5,5'-bitetrazole-1,1'-diolate)-bis(1,10-phenanthroline)-copper(II), $C_{26}H_{16}CuN_{12}O_2$

Table 1: Data collection and handling.

Crystal:	Blue block
Size:	$0.22 \times 0.20 \times 0.18$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.87 mm^{-1}
Diffractometer, scan mode:	Bruker-AXS SMART APEX-II, φ and ω
θ_{max} , completeness:	25.2° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	16114, 4751, 0.090
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2912
$N(\text{param})_{\text{refined}}$:	370
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

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Abstract

$C_{26}H_{16}CuN_{12}O_2$, monoclinic, $C2/c$ (no. 15), $a = 18.800(4)$ Å, $b = 21.224(4)$ Å, $c = 14.436(3)$ Å, $\beta = 112.094(6)^\circ$, $V = 5337.1(18)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0468$, $wR_{\text{ref}}(F^2) = 0.1287$, $T = 296(2)$ 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.

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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>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.6383(3)	0.25305(19)	0.7718(3)	0.0538(11)
H1	0.6510	0.2725	0.8337	0.065*
C2	0.6005(3)	0.1960(2)	0.7557(4)	0.0582(12)
H2	0.5873	0.1780	0.8058	0.070*
C3	0.5826(3)	0.1662(2)	0.6674(4)	0.0616(13)
H3	0.5571	0.1277	0.6564	0.074*
C4	0.6027(2)	0.19340(18)	0.5921(3)	0.0495(11)
C5	0.6389(2)	0.25224(17)	0.6128(3)	0.0429(10)
C6	0.6582(2)	0.28382(17)	0.5379(3)	0.0407(9)
C7	0.6421(2)	0.25570(19)	0.4447(3)	0.0506(11)
C8	0.6070(3)	0.1952(2)	0.4264(4)	0.0670(14)
H8	0.5975	0.1757	0.3653	0.080*
C9	0.5869(3)	0.1654(2)	0.4960(4)	0.0673(14)
H9	0.5627	0.1264	0.4813	0.081*
C10	0.6636(3)	0.2897(2)	0.3751(3)	0.0617(13)
H10	0.6542	0.2733	0.3119	0.074*
C11	0.6980(3)	0.3468(2)	0.4017(4)	0.0626(13)
H11	0.7131	0.3693	0.3570	0.075*
C12	0.7105(2)	0.37120(19)	0.4949(3)	0.0513(11)
H12	0.7333	0.4107	0.5110	0.062*
C13	0.5064(3)	0.3804(2)	0.6276(3)	0.0609(12)
H13	0.5036	0.3366	0.6265	0.073*
C14	0.4402(3)	0.4143(3)	0.6126(4)	0.0754(15)
H14	0.3942	0.3935	0.6019	0.090*
C15	0.4428(3)	0.4777(3)	0.6135(4)	0.0739(15)
H15	0.3987	0.5008	0.6045	0.089*

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C16	0.5123(3)	0.5091(2)	0.6281(3)	0.0609(13)
C17	0.5771(2)	0.4703(2)	0.6436(3)	0.0492(11)
C18	0.6491(3)	0.49943(18)	0.6583(3)	0.0484(10)
C19	0.6548(3)	0.5651(2)	0.6542(3)	0.0611(13)
C20	0.5864(4)	0.6022(2)	0.6375(4)	0.0776(16)
H20	0.5888	0.6459	0.6344	0.093*
C21	0.5205(4)	0.5748(3)	0.6267(4)	0.0782(16)
H21	0.4780	0.6000	0.6177	0.094*
C22	0.7259(4)	0.5900(2)	0.6641(4)	0.0799(17)
H22	0.7313	0.6333	0.6593	0.096*
C23	0.7866(4)	0.5521(2)	0.6805(4)	0.0798(16)
H23	0.8338	0.5689	0.6866	0.096*
C24	0.7780(3)	0.4873(2)	0.6883(4)	0.0651(13)
H24	0.8206	0.4615	0.7015	0.078*
C25	0.8660(3)	0.3470(2)	0.9385(3)	0.0543(11)
C26	0.8362(3)	0.4090(2)	0.9437(3)	0.0534(11)
Cu1	0.69785(3)	0.36917(2)	0.69988(4)	0.04497(18)
N1	0.65715(19)	0.28129(14)	0.7022(2)	0.0443(8)
N2	0.69153(19)	0.34107(14)	0.5626(2)	0.0438(8)
N3	0.57369(19)	0.40710(15)	0.6434(2)	0.0470(8)
N4	0.7120(2)	0.46143(15)	0.6778(3)	0.0493(9)
N5	0.8566(2)	0.31968(16)	0.8509(3)	0.0526(9)
N6	0.8863(3)	0.26154(18)	0.8706(3)	0.0801(13)
N7	0.9130(3)	0.2554(2)	0.9681(4)	0.0997(16)
N8	0.9011(3)	0.3076(2)	1.0117(3)	0.0805(13)
N9	0.7624(2)	0.42367(16)	0.8944(3)	0.0531(9)
N10	0.7511(3)	0.48365(19)	0.9124(3)	0.0729(12)
N11	0.8180(3)	0.5045(2)	0.9729(4)	0.0872(14)
N12	0.8722(2)	0.45896(19)	0.9940(3)	0.0764(13)
O1	0.82586(17)	0.34247(13)	0.7609(2)	0.0566(8)
O2	0.70627(17)	0.38588(13)	0.8390(2)	0.0563(8)

Source of material

1*H*,1'*H*-[5,5'-bitetrazole]-1,1'-diol (BTO) was synthesized according to the method using the procedure reported in the literature [5]. The title complex was obtained as follows: BTO (50.00 mg) and 1,10-phenanthroline (Phen, 105.95 mg) were dissolved in 50 mL of C₂H₅OH, heated to reflux 30 min, and then added the solution of (55.67 mg) in 5 mL water. The bright green precipitate formed after a week, which was filtered and dried under vacuum, yielding 160.0 mg (97.4%). IR (KBr, v/cm⁻¹): 3414, 3235, 1629, 1397, 1146, 614; **Elemental Analysis** (%) calcd. for C₂₆H₁₆CuN₁₂O₂: C, 52.75; H, 2.72; N, 28.39; found: C, 52.81; H, 2.80; N, 28.43.

Experimental details

A crystal of dimension 0.22 × 0.20 × 0.18 mm³ was mounted on a MiTeGen MicroMesh using a small amount of Cargille immersion oil. Data were collected on a Bruker three-circle diffractometer equipped with a Bruker-AXS SMART APEX II CCD detector. The crystal was irradiated using graphite monochromated MoK α radiation (λ = 0.71073). An Oxford Cobra low-temperature device was used to keep the crystal at a constant temperature of 296(2) K during data collection.

The structure was solved by SHELXT [2] and refined by the full-matrix least-squares method on F^2 with anisotropic thermal parameters for all non-hydrogen atoms. The hydrogen atoms were placed geometrically and refined using a riding model with common isotropic displacement factors $U_{iso}(H)$ = 1.2 (parent atom). Difference Fourier maps indicate some residual electron density located in the proximity of the 2-fold rotation axis. This electron density can be ascribed to a small molecule of highly disordered solvent, which could not be modelled. The PLATON SQUEEZE [6] procedure was applied in this case.

Comment

The title compound crystallizes in the monoclinic space group $C2/c$. The asymmetric unit is made up of one Cu²⁺ cation, one BTO²⁻ anion and two coordinated Phen molecules. In crystal structure, Cu(1) is coordinated by one adjacent BTO²⁻ anion [Cu(1)–O(1) = 2.301(3) Å and Cu(1)–O(2) = 1.986(3) Å] and two phenanthroline molecules [Cu(1)–N(1) = 2.319 Å, Cu(1)–N(1) = 2.319 Å, Cu(1)–N(2) = 2.030(3) Å, Cu(1)–N(3) = 2.309(3) Å and Cu(1)–N(4) = 2.018 Å]. The distances of Cu–O are close to those in [Cu(DOBT)(H₂O)₂] [2.299(18) and 2.003(5) Å] [7]. The torsion angles of N(8)–C(25)–C(26)–N(12) and N(5)–C(25)–C(26)–N(12) are 57.3(8)° and –126.5(5)° found in the BTO²⁻ anion, respectively, indicating that the five membered 6 π electron aromatic tetrazole rings are not coplanar.

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