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Crystal structure of dimethanolato- $k^2O:O$ -bis(1-((2-methyl-1*H*-benzo[*d*]imidazol-1-yl)methyl)-1*H*-benzo[*d*][1,2,3]triazole- κN)-bis(thiocyanato- κN) dicopper(II), $C_{34}H_{32}Cu_2N_{12}O_2S_2$

<https://doi.org/10.1515/ncrs-2021-0045>

Received January 29, 2021; accepted February 11, 2021;
published online March 4, 2021

Abstract

$C_{34}H_{32}Cu_2N_{12}O_2S_2$, triclinic, $P\bar{1}$ (no. 2), $a = 7.7970(7)$ Å, $b = 9.6110(9)$ Å, $c = 12.7629(12)$ Å, $\alpha = 71.544(8)^\circ$, $\beta = 79.322(8)^\circ$, $\gamma = 83.734(8)^\circ$, $Z = 1$, $V = 890.21(14)$ Å³, $R_{gt}(F) = 0.0634$, $wR_{ref}(F^2) = 0.1827$, $T = 291(2)$ K.

CCDC no.: 2062593

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.

Table 1: Data collection and handling.

Crystal:	Blue prism
Size:	0.20 × 0.20 × 0.16 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ :	2.99 mm ⁻¹
Diffractometer, scan mode:	Xcalibur, ω
θ_{max} , completeness:	67.3°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	6384, 6384
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 5176
$N(param)_{refined}$:	238
Programs:	CrysAlis ^{PRO} [1], SHELX [2]

Source of material

All starting materials are commercially available without further purification. The educt 1-[(2-methyl-1*H*-benzoimidazol-1-yl)methyl]-1*H*-benzotriazole (mbmb) was prepared according to the literature method [3]. The ligand mbmb (0.02 mmol, 0.0053 g) in methanol (6 mL) was added dropwise to a methanol solution (6 mL) of $Cu(CH_3COO)_2$ (0.02 mmol, 0.0040 g). Then an aqueous solution (2 mL) of KSCN (0.02 mmol, 0.0019 g) was added dropwise. The resulting solution was allowed to stand at room temperature. After three weeks blue crystals were obtained.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cu1	0.02074 (7)	0.36055 (6)	0.09413 (5)	0.0420 (3)
S1	−0.33898 (18)	0.13713 (16)	0.43127 (10)	0.0637 (4)
O1	0.1441 (4)	0.4783 (3)	−0.0435 (3)	0.0513 (8)
N1	0.1811 (4)	0.1822 (3)	0.1066 (3)	0.0396 (7)
N2	0.2964 (4)	−0.0393 (3)	0.1824 (3)	0.0395 (7)
N3	0.2365 (5)	−0.2865 (4)	0.2941 (3)	0.0538 (9)
N4	0.2750 (7)	−0.3960 (4)	0.2455 (4)	0.0747 (15)
N5	0.1417 (8)	−0.4756 (5)	0.2687 (5)	0.0816 (17)
N6	−0.1232 (5)	0.2728 (4)	0.2344 (3)	0.0466 (8)
C1	0.3238 (6)	−0.1359 (5)	0.0163 (4)	0.0502 (10)
H1	0.373452	−0.228724	0.048159	0.060*
C2	0.2901 (7)	−0.0905 (6)	−0.0921 (4)	0.0592 (12)
H2	0.319479	−0.154387	−0.135001	0.071*
C3	0.2134 (6)	0.0476 (7)	−0.1394 (4)	0.0589 (12)
H3	0.191608	0.073225	−0.212517	0.071*
C4	0.1695 (6)	0.1466 (5)	−0.0804 (4)	0.0496 (10)
H4	0.117216	0.238442	−0.112095	0.060*
C5	0.2052 (5)	0.1058 (4)	0.0278 (3)	0.0391 (8)
C6	0.2792 (5)	−0.0345 (4)	0.0745 (3)	0.0382 (8)
C7	0.2379 (5)	0.0932 (4)	0.1965 (3)	0.0393 (8)
C8	0.2481 (7)	0.1353 (5)	0.2972 (4)	0.0515 (10)
H8A	0.221029	0.238915	0.282529	0.077*
H8B	0.364040	0.112079	0.315303	0.077*
H8C	0.165793	0.082309	0.358857	0.077*
C9	0.3555 (6)	−0.1704 (5)	0.2663 (4)	0.0515 (10)
H9A	0.471056	−0.204001	0.237482	0.062*
H9B	0.363219	−0.145829	0.333311	0.062*
C10	0.0695 (6)	−0.2955 (4)	0.3497 (4)	0.0468 (9)
C11	0.0090 (7)	−0.4179 (5)	0.3343 (4)	0.0586 (12)
C12	−0.1600 (8)	−0.4617 (6)	0.3808 (5)	0.0689 (15)
H12	−0.200801	−0.543771	0.371584	0.083*
C13	−0.2638 (7)	−0.3800 (6)	0.4405 (5)	0.0625 (12)
H13	−0.377695	−0.406142	0.471613	0.075*
C14	−0.2020 (7)	−0.2578 (6)	0.4556 (4)	0.0574 (11)
H14	−0.276216	−0.205041	0.497094	0.069*
C15	−0.0362 (6)	−0.2127 (5)	0.4115 (4)	0.0505 (10)
H15	0.003875	−0.131303	0.422199	0.061*
C16	−0.2120 (5)	0.2167 (4)	0.3160 (3)	0.0404 (9)
C17	0.3248 (6)	0.4722 (6)	−0.0825 (5)	0.0689 (15)
H17A	0.383719	0.408008	−0.024043	0.103*
H17B	0.367619	0.568857	−0.104305	0.103*
H17C	0.346545	0.435490	−0.145766	0.103*

Experimental details

H atoms were generated geometrically and refined as riding atoms with C–H = 0.93 $\text{\AA}$ and the $U_{\text{iso}}(\text{H})$ = 1.2 times $U_{\text{eq}}(\text{C})$ for aromatic H atoms, with C–H = 0.97 $\text{\AA}$ and $U_{\text{iso}}(\text{H})$ = 1.2 times $U_{\text{eq}}(\text{C})$ for methylene H atoms, and with C–H = 0.96 $\text{\AA}$ and $U_{\text{iso}}(\text{H})$ = 1.5 times $U_{\text{eq}}(\text{C})$ for methyl H atoms.

Comment

In past the design and synthesis of coordination compounds have become a research hotspot in various fields, such as biochemistry [4, 5], food engineering [6] and medical [7]. *N*-containing heterocyclic ligands are widely used in the rational design and controlled synthesis of multifunctional complexes due to their abundant coordination and functional properties [8]. Benzotriazole and benzimidazole derivatives are excellent *N*-containing heterocyclic ligands, and they have been widely used as flexible ligands. Especially the incorporation of benzotriazole and benzimidazole can provide a new idea in the design of novel drug candidate molecules [9]. In addition, many Cu-containing complexes have extensive biological activities [10, 11].

The title compound contains a binuclear complex. As is shown in the Figure, each Cu(II) atom is four-coordinated by one N atom from a 1-[(2-methyl-1*H*-benzoimidazol-1-yl)methyl]-1*H*-benzotriazole ligand, one N atom from a thiocyanate ligand, two O atoms from two coordinated methanolato ligands (with the Cu1–N1 bond lengths of 1.990(3) $\text{\AA}$, the Cu1–N6 bond lengths of 1.919(4) $\text{\AA}$, the Cu1–O1 bond lengths of 1.909(3) $\text{\AA}$ and the Cu1–O1A bond lengths of 1.925(3) $\text{\AA}$). The bond angles around Cu(II) ion range from 76.38(13) to 170.34(14)°. All geometric parameters are in the expected ranges [12, 13]. In addition, the benzotriazole rings in adjacent molecules are parallel, with an average interplanar distance of 3.89 $\text{\AA}$, thus π – π interaction cannot be ruled out.

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was financially supported by the Key Scientific and Research Projects of the Education Department of Henan Province (No. 21A430024).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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