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Crystal structure of diaqua-bis(2,4-dinitrophenolato- $\kappa^2 O, O'$)copper(II) 1.5 hydrate, $C_{12}H_{13}CuN_4O_{13.5}$

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Abstract

$C_{24}H_{26}Cu_2N_8O_{27}$, triclinic, $P\bar{1}$ (no. 2), $a = 4.7705(1)$ Å, $b = 14.6219(5)$ Å, $c = 14.7418(3)$ Å, $\alpha = 61.225(3)^\circ$, $\beta = 85.342(2)^\circ$, $\gamma = 83.938(2)^\circ$, $V = 895.73(5)$ Å³, $Z = 1$, $R_{gt}(F) = 0.0485$, $wR_{ref}(F^2) = 0.1341$, $T = 170.0$ K.

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The molecular structure is shown in the figure (free water molecules are omitted). 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:	Brown plate
Size:	$0.18 \times 0.15 \times 0.04$ mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ :	2.54 mm^{-1}
Diffractometer, scan mode:	XtaLAB Synergy R, ω
$\theta_{\max}$, completeness:	75.4° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	10,333, 3542, 0.031
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3306
$N(\text{param})_{\text{refined}}$:	285
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3], Olex2 [4]

Source of material

2,4-Dinitrophenol (1.84 g, 0.01 mol) and sodium hydroxide (50% aqueous solution) were mixed in ethanol solution (30 ml) at room temperature, adding copper(II) chloride dihydrate (0.852 g, 0.005 mol), stirring the mixture for two days and the brown filtrate was extracted. Then cool it down to room temperature, evaporation crystallization under natural conditions, finally we obtained dark brown crystal.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cu1	0.57377 (7)	0.20131 (3)	0.35736 (3)	0.02492 (16)
O1	0.3196 (4)	0.21217 (16)	0.46119 (14)	0.0298 (4)
O2	0.6973 (4)	0.35691 (16)	0.33399 (15)	0.0340 (4)
O3	0.9004 (4)	0.43562 (17)	0.39834 (16)	0.0351 (4)
O4	0.6394 (5)	0.42687 (18)	0.71248 (17)	0.0396 (5)
O5	0.2814 (5)	0.34617 (17)	0.80504 (15)	0.0372 (5)
O6	0.8315 (4)	0.18917 (15)	0.25500 (14)	0.0285 (4)
O7	0.4570 (4)	0.04444 (16)	0.37072 (14)	0.0340 (4)
O8	0.2454 (5)	−0.02316 (18)	0.29713 (16)	0.0432 (5)
O9	0.3079 (4)	0.09091 (16)	−0.06113 (15)	0.0332 (4)
O10	0.6928 (4)	0.15707 (17)	−0.14458 (15)	0.0368 (5)
O11	0.8645 (4)	0.11957 (16)	0.46283 (14)	0.0327 (4)
H11a	0.7863 (9)	0.0924 (19)	0.5237 (2)	0.0490 (6)*
H11b	0.977 (4)	0.1612 (3)	0.4636 (16)	0.0490 (6)*
O12	0.2944 (4)	0.27891 (15)	0.24790 (15)	0.0304 (4)
H12a	0.243 (4)	0.3394 (9)	0.2434 (15)	0.0457 (6)*
H12b	0.140 (2)	0.2477 (12)	0.2651 (10)	0.0457 (6)*
N1	0.7272 (5)	0.37592 (17)	0.40552 (17)	0.0256 (4)
N2	0.4466 (5)	0.37042 (18)	0.73046 (17)	0.0303 (5)
N3	0.4113 (5)	0.03807 (17)	0.29238 (17)	0.0271 (5)
N4	0.5292 (5)	0.12998 (18)	−0.06853 (16)	0.0285 (5)
C1	0.3608 (5)	0.2523 (2)	0.52054 (19)	0.0241 (5)
C2	0.5516 (5)	0.32929 (19)	0.49966 (19)	0.0238 (5)
C3	0.5796 (5)	0.3666 (2)	0.56983 (19)	0.0254 (5)
H3	0.7112 (5)	0.4167 (2)	0.55573 (19)	0.0305 (6)*
C4	0.4147 (5)	0.3299 (2)	0.65894 (19)	0.0258 (5)
C5	0.2237 (5)	0.2549 (2)	0.68305 (19)	0.0275 (5)
H5	0.1108 (5)	0.2306 (2)	0.74520 (19)	0.0330 (6)*
C6	0.2019 (5)	0.2171 (2)	0.6156 (2)	0.0263 (5)
H6	0.0746 (5)	0.1648 (2)	0.6331 (2)	0.0316 (6)*
C7	0.7507 (5)	0.1733 (2)	0.18192 (19)	0.0244 (5)
C8	0.5504 (5)	0.10235 (19)	0.19409 (19)	0.0246 (5)
C9	0.4760 (5)	0.0893 (2)	0.1114 (2)	0.0260 (5)
H9	0.3392 (5)	0.0424 (2)	0.1211 (2)	0.0312 (6)*
C10	0.6045 (5)	0.1455 (2)	0.01644 (19)	0.0260 (5)
C11	0.8034 (6)	0.2157 (2)	−0.0003 (2)	0.0290 (5)
H11	0.8906 (6)	0.2533 (2)	−0.0669 (2)	0.0348 (6)*
C12	0.8720 (6)	0.2298 (2)	0.0802 (2)	0.0284 (5)
H12	1.0049 (6)	0.2789 (2)	0.0680 (2)	0.0340 (6)*
O13	0.2076 (8)	0.4887 (2)	0.1559 (2)	0.0650 (8)
H13a	0.253 (12)	0.523 (3)	0.185 (4)	0.0976 (12)*
H13b	0.121 (11)	0.534 (3)	0.103 (3)	0.0976 (12)*
O14 ^a	0.7862 (12)	0.5292 (5)	0.0345 (4)	0.0669 (16)
H14a ^a	0.7400 (12)	0.5605 (5)	−0.0285 (4)	0.100 (2)*
H14b ^a	0.6572 (12)	0.5581 (5)	0.0575 (4)	0.100 (2)*

^aOccupancy: 0.5.

Comment

In recent years, the preparation and characterization of metal complexes containing nitrophenolate have been a

hot topic. On the one hand, these compounds can be used as explosive components [5, 6]; on the other hand, phenolic ligands can be modified by substitution providing a large number of complex structures [7–10]. Hence, we chose 2,4-dinitrophenol and copper to synthesize a new complex.

The molecular structure of the title compound is shown in the figure. In the title complex, the central copper(II) is connected with six O atoms, two of them belong to the two water ligands, and the other four O atoms belong to the 2,4-dinitrophenolato ligands. The bond length of Cu–O range from 1.930 to 2.329 Å, which is a little bit longer than the reported copper complex [11].

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