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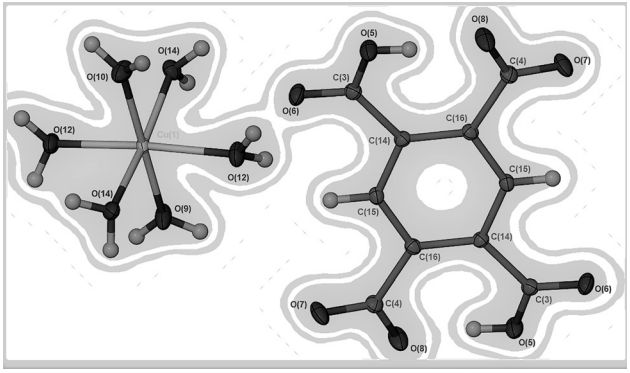
Synthesis and crystal structure of hexaaquacopper(II) 2,5-dicarboxyterephthalate, $C_{10}H_{16}O_{14}Cu$



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Abstract

$C_{10}H_{16}O_{14}Cu$, monoclinic, *I*2/*a* (no. 15), a = 7.2407(3) Å, b = 9.7565(4) Å, c = 21.1814(10) Å, β = 93.913(2)°, V = 1492.85(11) Å^3, Z = 4, $R_{gt}(F)$ = 0.0198, $wR_{ref}(F^2)$ = 0.0509, T = 200 K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

All commercially available starting materials were used without further purification. The titled compound was obtained from slow evaporation reaction of H_4B_4C (benzene-1,2,4,5-tetracarboxylic acid) (1 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (1 mmol), $Zn(CH_3COO)_2{\cdot}2H_2O$ (1 mmol) in 20 ml of MeOH–H_2O (1:1, *v*/*v*) mixture. The reaction mixture was heated for 15 min while stirring. Then it was allowed to cool down. The resulting solution was filtered, covered with parafilm and kept aside in a secluded area for nine days to allow for the growth of blue crystal of the titled compound. Elemental analysis (calcd.)(%) C, 27.95 (28.34), H, 3.25 (3.81), Cu, 14.99 (14.72).

Table 1: Data collection and handling.

Crystal:	Blue block
Size:	0.48 × 0.45 × 0.20 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.54 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
θ_{max}, completeness:	28.3°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6519, 1835, 0.015
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 $\sigma(I_{obs})$, 1729
$N(param)_{refined}$:	140
Programs:	Bruker [1, 2], SHELX [3, 4], PLATON [5], Mercury [6], X-Seed [7]

Experimental details

Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U(H) set to 1.2 U_{eq}(C).

The H atom of the hydroxyl group was allowed to rotate with a fixed angle around the C–O bond to best fit the experimental electron density (HFIX 147 in the SHELX program suite (Sheldrick, 2015)), with U_{iso}(H) set to 1.5U_{eq}(O).

Comment

Pyromellitic acid (H_4B_4C), the most symmetrical of the three benzenetetracarboxylic acids [8] is a member of benzenepolycarboxylic acid family and has found applications in the hydrogen-bond formation. The salts of this acid [9] are also of interest in gravimetric analysis,

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

Atom	x	y	z	U_{iso}*/U_{eq}
Cu1	0.250000	0.29898 (2)	0.500000	0.01188 (7)
O1	0.65983 (14)	0.62566 (9)	0.27809 (4)	0.0215 (2)
H1	0.691389	0.614701	0.240993	0.026*
O2	0.55354 (14)	0.49985 (9)	0.35314 (4)	0.0201 (2)
O3	0.83469 (15)	0.07623 (10)	0.40118 (4)	0.0230 (2)
O4	0.78501 (16)	−0.09129 (9)	0.33210 (4)	0.0272 (2)
O11	0.39511 (15)	0.28873 (10)	0.41953 (4)	0.0214 (2)
O12	0.250000	0.51463 (14)	0.500000	0.0233 (3)
O13	0.250000	0.08946 (14)	0.500000	0.0272 (3)
O14	0.50179 (14)	0.31353 (9)	0.55718 (4)	0.01793 (19)
C1	0.62713 (17)	0.50851 (12)	0.30253 (6)	0.0148 (2)
C2	0.68793 (16)	0.37840 (12)	0.27096 (5)	0.0128 (2)
C3	0.71930 (17)	0.26832 (13)	0.31202 (6)	0.0137 (2)
H3	0.697416	0.281195	0.355331	0.016*
C4	0.78085 (16)	0.14049 (12)	0.29336 (5)	0.0131 (2)
C5	0.80471 (18)	0.03509 (13)	0.34617 (6)	0.0164 (2)
H12	0.177 (2)	0.5666 (13)	0.5182 (8)	0.038 (5)*
H11A	0.434 (3)	0.3561 (14)	0.4001 (8)	0.042 (6)*
H11B	0.360 (3)	0.2287 (16)	0.3932 (8)	0.050 (6)*
H13	0.281 (3)	0.0390 (14)	0.4706 (5)	0.046 (6)*
H14A	0.483 (2)	0.3589 (15)	0.5901 (6)	0.030 (5)*
H14B	0.546 (3)	0.2377 (13)	0.5700 (9)	0.046 (6)*

heat stabilizers, detergent builders, catalysts and applied dyes [10]. The crystal structure of title compound is built from $[Cu(H_2O)_6]^{2+}$ dications and doubly deprotonated pyromellitic dianions, $(H_2B_4C)^{2-}$ which are linked to each other through hydrogen bonds and ionic interactions. Similar structures such as $\{Co(H_2O)_6\}\{C_{10}H_4O_8\}$ [10], $\{M(H_2O)_6\}\{C_6H_2(COO)_2(COOH)_2\}$ with M=Mn, Co, and Ni [11] and $[Mg(H_2O)_6](C_{10}H_4O_8)$ [12] have been reported.

The Cu^{2+} in the dication is surrounded by six water ligands exhibiting an octahedral geometry. The position occupied by the Cu^{2+} is on a twofold axis, with a pair of O atoms on the twofold axis while the other four O atoms are related to in pairs by this same axis. The Cu–O distances are in the range 2.044(14)–2.124(9) Å and the O–Cu–O angles are between 86.17(2)° and 179(3)°. The $(H_2B4C)^{2-}$ anion is located around a center of symmetry. The benzene ring internal angles are 117.90(11)° and 118.28(11)° for the substituted C atoms and 123.82(11)° for the unsubstituted. The exterior angles are 127.42(10)° and 114.83(10)°.

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

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