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Crystal structure of [aqua-(4-iodopyridine-2,6-dicarboxylato- $\kappa^3 O,N,O'$)-(1,10-phenanthroline- $\kappa^2 N,N'$)copper(II)] dihydrate, $C_{19}H_{16}O_7N_3CuI$

Table 1: Data collection and handling.

Crystal:	Blue block
Size:	0.30 × 0.28 × 0.25 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	2.61 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
θ_{max} , completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	71,115, 3611, 0.041
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 3385
$N(\text{param})_{\text{refined}}$:	287
Programs:	CrysAlis ^{PRO} [1], Shelx [2, 3], Olex2 [4]

<https://doi.org/10.1515/ncrs-2021-0119>

Received March 27, 2021; accepted April 29, 2021;

published online May 12, 2021

Abstract

$\text{C}_{19}\text{H}_{16}\text{O}_7\text{N}_3\text{CuI}$, orthorhombic, *Pbca* (no. 61), $a = 12.5772(2)$ Å, $b = 17.3473(3)$ Å, $c = 18.8445(3)$ Å, $Z = 8$, $V = 4111.50(12)$ Å³, $R_{\text{gt}}(F) = 0.0310$, $wR_{\text{ref}}(F^2) = 0.0775$, $T = 289$ K.

CCDC no.: 2073699

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

Source of material

The mixtures of 4-iodopyridine-2,6-dicarboxylic acid (ipydCH₂) (29.2 mg, 0.1 mmol), 1,10-phenanthroline (phen) (18.0 mg, 0.1 mmol), Cu(OAc)₂·H₂O (20.0 mg, 0.1 mmol) and 4 mL deionized water were placed in a 23 mL Teflon liner stainless steel reactor. The vessel was heated to 343 K for three days, then cooled slowly to room

temperature. Blue crystals were obtained, and further crystals were filtered off, washed with distilled water, and dried under ambient conditions. Yield 45% (based on ipydcH_2).

Experimental details

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

Comment

Extensive efforts have been focused on the synthesis of coordination polymers (CPs), using predesigned organic ligands and central metal ions, because such materials can exhibit a variety of physicochemical properties in photoluminescence [5, 6], magnetism [7, 8], catalysis [9, 10], gas storage [11], photochromism [12, 13] and so on. In this field, the appropriate choice of well-designed organic ligands and central metal ions is one of the most effective ways to construct CPs. At present, versatile multicarboxyl compounds have been extensively used in the preparation of a variety of CPs. In this context, ipydc as nitrogen-containing heteroaryldicarboxylate ligand has three coordination sites, but was much less used so far.

The crystal structure consists of a Cu^{2+} ion, one ipydc anion, one phen ligand, one coordinated water molecule, and two lattice water molecules (see the Figure). The

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

Atom	x	y	z	U_{iso}^*/U_{eq}
C1	0.3923 (3)	0.2680 (2)	0.71127 (17)	0.0335 (7)
C2	0.4533 (2)	0.32214 (18)	0.75967 (17)	0.0277 (7)
C3	0.4287 (3)	0.33256 (19)	0.83002 (17)	0.0313 (7)
H3	0.3690	0.3092	0.8497	0.038*
C4	0.4939 (3)	0.37825 (18)	0.87093 (17)	0.0301 (7)
C5	0.5796 (3)	0.41508 (19)	0.84006 (17)	0.0327 (7)
H5	0.6235	0.4469	0.8669	0.039*
C6	0.5991 (2)	0.40389 (17)	0.76848 (17)	0.0281 (7)
C7	0.6896 (3)	0.44162 (19)	0.72712 (18)	0.0328 (7)
C8	0.3959 (3)	0.4519 (2)	0.6071 (2)	0.0406 (8)
H8	0.3953	0.4611	0.6557	0.049*
C9	0.3210 (3)	0.4893 (2)	0.5646 (2)	0.0474 (9)
H9	0.2732	0.5238	0.5849	0.057*
C10	0.3181 (3)	0.4755 (2)	0.4937 (2)	0.0479 (9)
H10	0.2672	0.4995	0.4654	0.058*
C11	0.3920 (3)	0.4250 (2)	0.46348 (19)	0.0378 (8)
C12	0.4664 (3)	0.39081 (18)	0.50973 (18)	0.0296 (7)
C13	0.5464 (3)	0.34126 (19)	0.48244 (17)	0.0301 (7)
C14	0.6934 (3)	0.2662 (2)	0.5052 (2)	0.0463 (9)
H14	0.7399	0.2436	0.5374	0.056*
C15	0.7094 (3)	0.2535 (3)	0.4333 (2)	0.0560 (11)
H15	0.7666	0.2239	0.4178	0.067*
C16	0.6407 (3)	0.2847 (2)	0.3857 (2)	0.0509 (10)
H16	0.6507	0.2762	0.3374	0.061*
C17	0.5550 (3)	0.3295 (2)	0.40883 (18)	0.0386 (8)
C18	0.4773 (4)	0.3632 (2)	0.3634 (2)	0.0502 (10)
H18	0.4806	0.3540	0.3149	0.060*
C19	0.3995 (4)	0.4078 (2)	0.3894 (2)	0.0504 (10)
H19	0.3491	0.4281	0.3584	0.060*
Cu1	0.57833 (3)	0.33163 (2)	0.63048 (2)	0.02950 (12)
I1	0.46300 (2)	0.38753 (2)	0.97973 (2)	0.05055 (11)
N1	0.53724 (19)	0.35685 (15)	0.72974 (13)	0.0268 (6)
N2	0.4677 (2)	0.40378 (16)	0.58110 (14)	0.0311 (6)
N3	0.6145 (2)	0.30925 (16)	0.52996 (14)	0.0331 (6)
O1	0.43356 (18)	0.25274 (14)	0.65288 (12)	0.0363 (5)
O2	0.3077 (2)	0.24108 (19)	0.73382 (14)	0.0623 (9)
O3	0.70235 (18)	0.41844 (14)	0.66492 (12)	0.0381 (5)
O4	0.7407 (2)	0.49330 (15)	0.75803 (13)	0.0480 (6)
O5	0.67300 (19)	0.24161 (14)	0.66128 (13)	0.0424 (6)
H5A	0.6346	0.2017	0.6724	0.064*
H5B	0.7054	0.2527	0.7007	0.064*
O6	0.3897 (3)	0.09118 (16)	0.64852 (19)	0.0636 (8)
H6A	0.3893	0.1402	0.6487	0.095*
H6B	0.3437	0.0786	0.6795	0.095*
O7	0.6110 (3)	0.0948 (2)	0.6863 (3)	0.1082 (17)
H7A	0.6428	0.0611	0.7112	0.162*
H7B	0.5545	0.0755	0.6690	0.162*

central Cu^{2+} ion is six-coordinated and exhibits an octahedral geometry with the CuO_3N_3 chromophore. In contrast to our expectations a mononuclear complex was obtained. The Cu–N bond lengths associated with central Cu^{2+} ion range from 1.986(3) to 2.090(3) $\text{\AA}$ and the Cu–O distances is

in the range of 2.048(2)–2.317(2) $\text{\AA}$. The bond angles about the Cu^{2+} ion range from $75.78(9)^\circ$ to $177.63(11)^\circ$. These values match with the previously reported Cu(II) compound [14].

There are O–H...O hydrogen bonding interactions between lattice water molecules, coordinated water molecules and carboxylate oxygen atoms. In addition, there are obvious π – π stacking interactions between the phen ligands. The centroid–centroid distances are 3.613(3) and 3.651(2) $\text{\AA}$, respectively. In addition, the structure also contains halogen bond interaction, in which the distance between I atom and the O atom of lattice water molecule is 3.345(4) $\text{\AA}$, which is shorter than the sum of the van der Waals radii (ca. 3.5 $\text{\AA}$). All noncovalent interactions mentioned above contribute to the formation of the supramolecular architecture.

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

Research funding: PhD Startup Fund of HAUST (13480061).

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

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