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Crystal structure of (1,10-phenanthroline-κ^2N:N')-(μ_2-5-isonicotinamidoisophthalato-κ^2O^1,O^3)copper(II) monohydrate, $Cu(C_{12}H_8N_2)(C_{14}H_8N_2O_5) \cdot H_2O$

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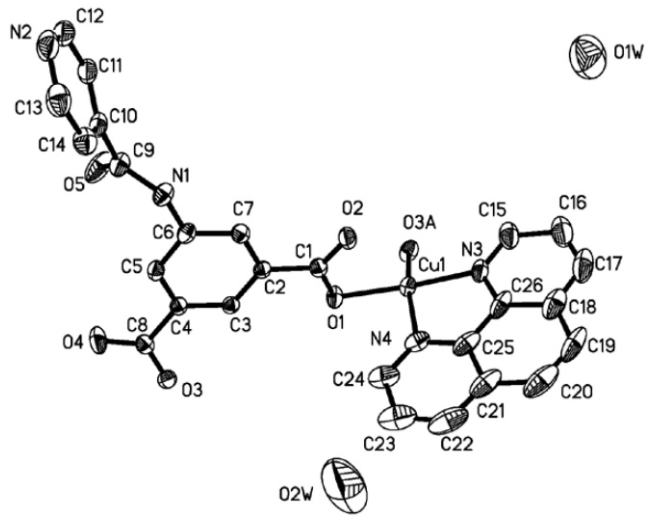

Abstract

$C_{26}H_{18}CuN_4O_6$, monoclinic, $C12/c1$ (no. 15), a = 27.347(5) Å, b = 13.317(3) Å, c = 13.523(3) Å, β = 110.869(2)°, V = 4601.8 Å^3, Z = 8, $R_{gt}(F)$ = 0.065, $wR_{ref}(F^2)$ = 0.159, T = 293 K.

Source of material

A mixture of $Cu(CH_3CO_2)_2 \cdot 2H_2O$ (0.040 g, 0.1 mmol), 5-isonicotinamidoisophthalic acid (iaip, 0.028 g, 0.1 mmol), NaOH (0.081 g, 0.2 mmol), phen (0.020 g, 0.1 mmol), CH_3CH_2OH (2 mL) and H_2O (8 mL) was sealed in a 15 ml Teflon-lined stainless steel reactor, which was heated at 413 K for 72 h and then cooled to room temperature. Blue needle-like crystals of the title compound were collected.

Experimental details

H atoms bonded to C atoms were placed geometrically and treated as riding. The water H atoms found from Fourier difference maps were refined with restraints for d(O—H) = 0.8499 - 0.8501 Å and U_{iso}(H) = 1.2 U_{eq}(O). The number of disordered water molecules was established by the thermogravimetric analysis.

Discussion

The rapid expanding field of crystal engineering of one-, two- and three-dimensional coordination architectures is of great current interests for their structural as well as for their potential applications as functional materials [1]. Among them, metal-organic frameworks (MOFs) with carboxylate-containing ligands have been extensively studied because the carboxylate groups can vary coordination modes resulting in formation of different structures. Particularly, copper complexes with the heterocycle have been investigated extensively to date [3,4]. It is well known that multicarboxylate with pyridine functional group have good coordination capacity as well as the amide group, a fascinating functional group with two different types of hydrogen bonding sites [5,6].

In the title complex, the central Cu(II) ion is four-coordinated by two N atoms from phen ligands, two carboxylate O atoms from two iaip ligands in a square planar coordination. Furthermore, carboxylate groups of the iaip ligand adopt the mono-dentate coordination mode to connect two Cu(II) atoms into an infinite chain, whereas the pyridyl group is not participating in coordination. There are weak π-π interactions between the aromatic groups within the chains between the central benzene rings and benzene and pyridyl rings of iaip with a centroid-centroid distances of 3.6315(6) Å, 3.7245(7) Å, respectively. Then the π-π interactions interlink the chains into layer, which are connected together through the N–H···O and O–H···O hydrogen bonding interactions to give the 3D supramolecular architecture.

Table 1. Data collection and handling.

Crystal:	blue needle, size 0.10 × 0.15 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	10.02 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX CCD, φ/ω
$2\theta_{max}$:	50.0°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11196, 4042
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3105
$N(param)_{refined}$:	341
Program:	SHELXTL [7]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(3)	8f		0.7625	0.6260	0.3179	0.041
H(5)	8f		0.8349	0.7331	0.6116	0.045
H(7)	8f		0.7318	0.5056	0.5635	0.045
H(11)	8f		0.8901	0.4977	0.9570	0.067
H(12)	8f		0.8907	0.5024	1.1266	0.078
H(13)	8f		0.7987	0.7346	1.0661	0.088
H(14)	8f		0.7892	0.7302	0.8866	0.074
H(15)	8f		0.6039	0.1643	0.1874	0.085

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Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(16)	8*f*		0.5208	0.0860	0.1321	0.111
H(17)	8*f*		0.4476	0.1813	0.0832	0.114
H(19)	8*f*		0.4032	0.3609	0.0640	0.099
H(20)	8*f*		0.4145	0.5235	0.0700	0.109
H(22)	8*f*		0.4764	0.6772	0.1092	0.110
H(23)	8*f*		0.5588	0.7354	0.1671	0.112
H(24)	8*f*		0.6295	0.6229	0.2103	0.085
H(1)	8*f*		0.7653	0.6110	0.7332	0.053
H(1WA)	8*f*	0.575	0.4196	0.2079	0.5929	0.154
H(1WB)	8*f*	0.575	0.4235	0.1479	0.6785	0.154
H(2WA)	8*f*	0.425	0.5120	0.9016	0.1744	0.120
H(2WB)	8*f*	0.425	0.5421	0.9241	0.1152	0.120

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cu(1)	8*f*		0.64040(2)	0.38788(4)	0.20276(4)	0.0293(3)	0.0424(4)	0.0378(3)	–0.0023(3)	0.0090(2)	–0.0053(3)
C(1)	8*f*		0.7042(2)	0.4817(3)	0.3584(4)	0.028(2)	0.036(3)	0.047(3)	0.006(2)	0.012(2)	–0.001(2)
C(2)	8*f*		0.7417(2)	0.5550(3)	0.4299(3)	0.025(2)	0.032(2)	0.041(2)	0.006(2)	0.009(2)	0.001(2)
C(3)	8*f*		0.7681(2)	0.6244(3)	0.3914(3)	0.031(2)	0.034(2)	0.036(2)	0.004(2)	0.010(2)	–0.002(2)
C(4)	8*f*		0.8027(2)	0.6913(3)	0.4592(4)	0.026(2)	0.035(2)	0.045(3)	0.003(2)	0.009(2)	–0.001(2)
C(5)	8*f*		0.8109(2)	0.6874(3)	0.5649(4)	0.030(2)	0.036(2)	0.043(3)	–0.000(2)	0.008(2)	–0.007(2)
C(6)	8*f*		0.7853(2)	0.6184(3)	0.6052(3)	0.028(2)	0.046(3)	0.038(2)	0.004(2)	0.011(2)	–0.003(2)
C(7)	8*f*		0.7501(2)	0.5526(3)	0.5369(3)	0.026(2)	0.041(3)	0.043(3)	0.001(2)	0.010(2)	0.002(2)
C(8)	8*f*		0.8307(2)	0.7705(3)	0.4198(4)	0.034(3)	0.037(3)	0.049(3)	0.001(2)	0.016(2)	–0.001(2)
C(9)	8*f*		0.8401(2)	0.6142(4)	0.7930(4)	0.043(3)	0.066(3)	0.041(3)	–0.006(3)	0.014(2)	–0.003(2)
C(10)	8*f*		0.8395(2)	0.6148(4)	0.9024(4)	0.041(3)	0.059(3)	0.042(3)	–0.015(2)	0.012(2)	–0.011(3)
C(11)	8*f*		0.8700(2)	0.5469(4)	0.9766(4)	0.050(3)	0.057(3)	0.054(3)	–0.017(3)	0.011(3)	–0.010(3)
C(12)	8*f*		0.8710(2)	0.5516(5)	1.0778(4)	0.066(4)	0.073(4)	0.048(3)	–0.027(3)	0.011(3)	0.000(3)
C(13)	8*f*		0.8170(3)	0.6849(5)	1.0426(5)	0.077(4)	0.082(4)	0.070(4)	–0.020(3)	0.036(3)	–0.028(3)
C(14)	8*f*		0.8117(2)	0.6838(4)	0.9352(4)	0.064(4)	0.063(4)	0.058(3)	–0.012(3)	0.022(3)	–0.007(3)
C(15)	8*f*		0.5730(3)	0.2040(5)	0.1675(4)	0.075(4)	0.091(4)	0.044(3)	–0.036(3)	0.017(3)	–0.009(3)
C(16)	8*f*		0.5235(3)	0.1571(6)	0.1346(5)	0.096(5)	0.117(5)	0.060(4)	–0.052(4)	0.021(4)	–0.007(4)
C(17)	8*f*		0.4807(3)	0.2138(7)	0.1072(5)	0.061(4)	0.161(6)	0.058(4)	–0.046(4)	0.016(3)	–0.002(4)
C(18)	8*f*		0.4818(2)	0.3181(7)	0.1118(4)	0.052(3)	0.153(5)	0.032(3)	–0.017(4)	0.014(3)	–0.001(3)
C(19)	8*f*		0.4377(2)	0.3869(7)	0.0852(5)	0.035(3)	0.174(6)	0.039(3)	–0.004(4)	0.013(2)	–0.005(4)
C(20)	8*f*		0.4448(3)	0.4822(7)	0.0898(5)	0.064(4)	0.170(6)	0.041(3)	0.026(4)	0.020(3)	0.007(4)
C(21)	8*f*		0.4935(3)	0.5304(7)	0.1216(4)	0.059(4)	0.148(5)	0.029(3)	0.026(4)	0.019(3)	0.009(3)
C(22)	8*f*		0.5046(3)	0.6307(7)	0.1286(5)	0.098(5)	0.132(5)	0.045(3)	0.053(4)	0.028(3)	0.009(4)
C(23)	8*f*		0.5525(3)	0.6651(6)	0.1609(5)	0.121(6)	0.098(5)	0.060(4)	0.049(5)	0.033(4)	–0.001(4)
C(24)	8*f*		0.5948(3)	0.5972(5)	0.1866(4)	0.087(4)	0.075(4)	0.053(3)	0.029(3)	0.027(3)	0.004(3)
C(25)	8*f*		0.5383(2)	0.4646(5)	0.1482(4)	0.049(3)	0.112(4)	0.028(2)	0.022(3)	0.015(2)	0.009(3)
C(26)	8*f*		0.5317(2)	0.3575(6)	0.1431(4)	0.036(3)	0.132(4)	0.025(2)	–0.008(3)	0.014(2)	0.000(3)
N(1)	8*f*		0.7932(2)	0.6140(3)	0.7152(3)	0.031(2)	0.064(3)	0.038(2)	–0.002(2)	0.013(2)	–0.001(2)
N(2)	8*f*		0.8464(2)	0.6194(5)	1.1121(4)	0.088(4)	0.094(4)	0.045(3)	–0.042(3)	0.024(3)	–0.016(3)
N(3)	8*f*		0.5766(2)	0.3030(3)	0.1705(3)	0.041(3)	0.066(3)	0.036(2)	–0.016(2)	0.009(2)	–0.007(2)
N(4)	8*f*		0.5878(2)	0.4993(3)	0.1788(3)	0.052(3)	0.061(3)	0.041(2)	0.019(2)	0.017(2)	0.004(2)
O(1)	8*f*		0.7000(1)	0.4806(2)	0.2615(2)	0.041(2)	0.054(2)	0.042(2)	–0.015(2)	0.012(2)	–0.011(2)
O(2)	8*f*		0.6776(1)	0.4260(3)	0.3934(3)	0.045(2)	0.051(2)	0.050(2)	–0.016(2)	0.014(2)	–0.001(2)
O(3)	8*f*		0.8159(1)	0.7825(2)	0.3200(3)	0.037(2)	0.051(2)	0.047(2)	–0.007(2)	0.009(2)	0.012(2)
O(4)	8*f*		0.8657(1)	0.8198(3)	0.4825(3)	0.063(3)	0.068(3)	0.056(2)	–0.035(2)	0.026(2)	–0.017(2)
O(5)	8*f*		0.8818(2)	0.6124(4)	0.7786(3)	0.036(2)	0.161(5)	0.051(2)	–0.004(3)	0.014(2)	0.004(3)
O(1W)	8*f*	0.575(8)	0.4387(3)	0.1650(5)	0.6361(6)	0.097(8)	0.109(7)	0.21(1)	–0.038(6)	0.092(8)	–0.044(7)
O(2W)	8*f*	0.425(8)	0.5111(3)	0.9252(5)	0.1154(6)	0.11(1)	0.099(9)	0.120(9)	0.030(7)	0.075(8)	–0.019(7)

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