catena-Poly[[[triaquacopper(II)]-μ2-pyrazine-2,3-dicarboxylato] monohydrate]

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The Cu atom in the title complex, $\{[Cu(C_6H_2N_2O_4)(H_2O)_3]\cdot H_2O\}_n$ or $\{[Cu(L)(H_2O)_3]\cdot H_2O\}_n$ (L is pyrazine-2,3-dicarboxylate), displays octahedral coordination formed by the ligand L and three coordinated water molecules. The ligand L is tridentate, with one N atom of the pyrazine ring and one O atom of one carboxylate group forming a chelate ring, whereas one O atom from the second carboxylate group is coordinated to another Cu atom. The ligand L links molecules to form an infinite chain parallel to the [101] direction. The chains are further linked through O—H···O and O—H···N hydrogen bonds involving the water molecules to build up a three-dimensional network.

Related literature

For related literature, see: Gokel *et al.* (2004); Shan *et al.* (2001); Starosta & Leciejewicz (2005); Takusagawa & Shimada (1973); Tombul *et al.* (2007); Ptasiewicz-Bak & Leciejewicz (1997*a*,*b*).
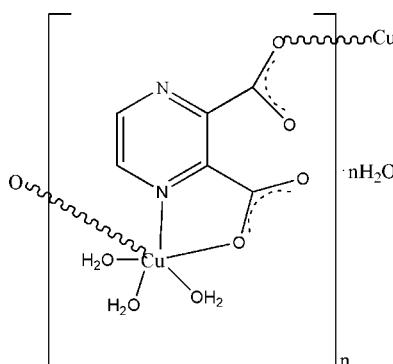
Experimental

Crystal data

$[Cu(C_6H_2N_2O_4)(H_2O)_3]\cdot H_2O$
$M_r = 301.70$
Monoclinic, Cc
$a = 8.4254$ (4) Å
$b = 18.0692$ (8) Å
$c = 7.4187$ (3) Å
$\beta = 114.4120$ (10)°
$V = 1028.45$ (8) Å³
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 2.16$ mm⁻¹
$T = 298$ (2) K
$0.28 \times 0.25 \times 0.17$ mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min} = 0.519$, $T_{max} = 0.660$
3079 measured reflections
1683 independent reflections
1654 reflections with $I > 2\sigma(I)$
$R_{int} = 0.067$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.089$
$S = 1.11$
1683 reflections
156 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max} = 0.67$ e Å⁻³
$\Delta\rho_{min} = -1.08$ e Å⁻³
Absolute structure: Flack (1983), 758 Friedel pairs
Flack parameter: −0.04 (2)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5A···O7[i]	0.84	2.30	2.953 (4)	134
O5—H5B···O8	0.86	1.79	2.630 (6)	168
O6—H6A···O4[ii]	0.85	1.98	2.837 (4)	176
O6—H6A···O3[ii]	0.85	2.57	3.170 (5)	128
O6—H6B···O4[iii]	0.84	1.87	2.691 (4)	167
O7—H7A···N2[iii]	0.83	1.98	2.817 (5)	177
O7—H7B···O1[iv]	0.83	1.90	2.719 (6)	166
O8—H8A···O4[iv]	0.85	2.02	2.864 (6)	176
O8—H8B···O1[v]	0.85	2.11	2.898 (6)	155

Symmetry codes: (i) $x, -y, z + \frac{1}{2}$; (ii) $x + \frac{1}{2}, y - \frac{1}{2}, z$; (iii) $x + \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$; (iv) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$; (v) $x - \frac{1}{2}, y - \frac{1}{2}, z$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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supporting information

catena-Poly[[[triaquacopper(II)]-μ_2-pyrazine-2,3-dicarboxylato] monohydrate]

Wei-Ping Wu, Feng-Chun Zeng, Yu Wu and Jian Peng

S1. Comment

Transition metal complexes with bipyridine derivatives are suitable models for the study of excited state dynamics. In addition, they are of interest for the development of light-energy conversion devices and optical sensors (Gokel *et al.*, 2004; Shan *et al.*, 2001). Since the single-crystal X-ray analysis of pyrazine-2,3 dicarboxylic acid was first determined (Takusagawa & Shimada, 1973), a variety of metal-organic compounds of pyrazine-2,3-dicarboxylic acid have been characterized crystallographically, due to growing interest in supramolecular chemistry (Tombul *et al.*, 2007). These include the calcium (Ptasiewicz-Bak & Leciejewicz, 1997*a*; Starosta & Leciejewicz, 2005) and magnesium (Ptasiewicz-Bak & Leciejewicz, 1997*b*) complexes. In this paper, we report the synthesis and crystal structure of the title complex,(I).

The Cu^{II} ion displays octahedral coordination formed by the one *L* ligand and three coordinated water molecules. The ligand *L* is tridentate with the N atom of the pyridine ring and one O atom of one carboxylate forming a chelate ring whereas one O atom from the second carboxylate is coordinated to another Cu atom (Fig. 1). Then the ligand *L* links molecules to form an infinite chain parallel to the [1 O 1] direction. The chains are further linked through O—H···O and O—H···N involving the water molecules to build up a three dimensionnal network (Table 1).

S2. Experimental

L (0.031 g, 0.018 mmol), CuSO4 (0.018 g, 0.016 mmol) and NaOH(0.048 mmol,0.12 mmol), were added in a mixed solvent of ethanol, the mixture was heated for three hours under reflux. during the process stirring and influx were required. The resultant was then filtered to give a pure solution which was infiltrated by diethyl ether freely in a closed vessel, two weeks later some single crystals of the size suitable for X-Ray diffraction analysis.

S3. Refinement

All H atoms attached to C atoms were fixed geometrically and treated as riding with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C). H atoms of water molecule were located in difference Fourier maps and included in the subsequent refinement using restraints (O—H= 0.85 (1)Å and H···H= 1.39 (2) Å) with U_{iso}(H) = 1.5U_{eq}(O). In the last stage of refinement, they were treated as riding on their parent O atoms.
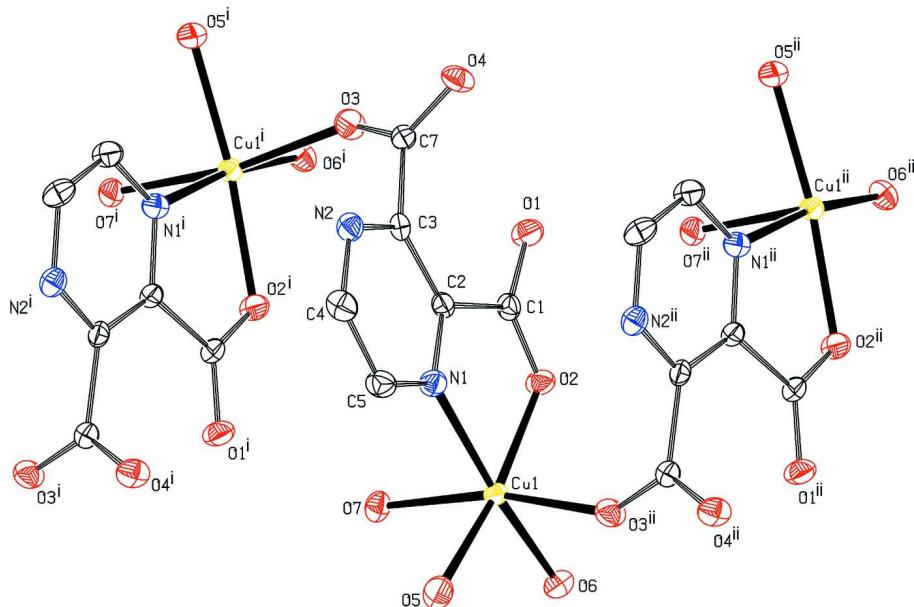
Figure 1

View of compound (I) with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity. [Symmetry codes: (i) $x - 1/2$, $1/2 - y$, $z - 1/2$; (ii) $1/2 + x$, $1/2 - y$, $1/2 + z$]

catena-**Poly[[[triaquacopper(II)]-*µ*₂-pyrazine-2,3-dicarboxylato] monohydrate]**

Crystal data
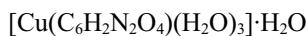
[Cu(C₆H₂N₂O₄)(H₂O)₃]·H₂O	$F(000) = 612$
$M_r = 301.70$	$D_x = 1.949$ Mg m⁻³
Monoclinic, Cc	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: C -2yc	Cell parameters from 1683 reflections
$a = 8.4254$ (4) Å	$\theta = 2.3–25.2°$
$b = 18.0692$ (8) Å	$\mu = 2.16$ mm⁻¹
$c = 7.4187$ (3) Å	$T = 298$ K
$\beta = 114.412$ (1)°	Block, blue
$V = 1028.45$ (8) Å³	$0.28 \times 0.25 \times 0.17$ mm
$Z = 4$	

Data collection

Bruker APEXII area-detector diffractometer	3079 measured reflections
Radiation source: fine-focus sealed tube	1683 independent reflections
Graphite monochromator	1654 reflections with $I > 2\sigma(I)$
φ and ω scan	$R_{int} = 0.067$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)	$\theta_{max} = 25.2°$, $\theta_{min} = 2.3°$
$T_{min} = 0.519$, $T_{max} = 0.660$	$h = -10 \rightarrow 9$
	$k = -21 \rightarrow 20$
	$l = -8 \rightarrow 8$

Refinement

Refinement on F^2	Hydrogen site location: inferred from
Least-squares matrix: full	neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.036$	H-atom parameters constrained
$wR(F^2) = 0.089$	$w = 1/[\sigma^2(F_o^2) + (0.0632P)^2]$
$S = 1.11$	where $P = (F_o^2 + 2F_c^2)/3$
1683 reflections	$(\Delta/\sigma)_{max} < 0.001$
156 parameters	$\Delta\rho_{max} = 0.67$ e Å^{-3}
2 restraints	$\Delta\rho_{min} = -1.08$ e Å^{-3}
Primary atom site location: structure-invariant	Absolute structure: Flack (1983), 758 Friedel
direct methods	pairs
Secondary atom site location: difference Fourier	Absolute structure parameter: −0.04 (2)
map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	y	z	U_{iso}*/U_{eq}
Cu1	0.89418 (3)	0.118643 (19)	0.89933 (3)	0.02206 (17)
N1	0.6873 (5)	0.18186 (18)	0.9306 (5)	0.0277 (8)
N2	0.4368 (5)	0.2821 (2)	0.9255 (5)	0.0289 (10)
O1	0.9124 (6)	0.34477 (15)	0.9145 (8)	0.0380 (8)
O2	0.9816 (4)	0.22606 (16)	0.9101 (5)	0.0311 (7)
O3	0.5382 (5)	0.40933 (18)	0.6974 (4)	0.0347 (8)
O4	0.6390 (5)	0.42851 (17)	1.0176 (4)	0.0346 (7)
O5	0.7715 (5)	0.01798 (16)	0.8841 (6)	0.0428 (8)
H5A	0.8168	−0.0080	0.9872	0.064*
H5B	0.6618	0.0110	0.8202	0.064*
O6	1.1011 (4)	0.07381 (17)	0.8605 (4)	0.0314 (7)
H6A	1.1120	0.0292	0.9021	0.047*
H6B	1.1000	0.0780	0.7471	0.047*
O7	0.7584 (5)	0.12285 (15)	0.5794 (5)	0.0302 (8)
H7A	0.8141	0.1510	0.5379	0.039 (15)*
H7B	0.6501	0.1250	0.5249	0.033 (16)*
C1	0.8817 (8)	0.2773 (2)	0.9095 (8)	0.0247 (9)
C2	0.7074 (6)	0.2543 (2)	0.9108 (6)	0.0229 (9)
C3	0.5804 (6)	0.3047 (2)	0.9043 (6)	0.0232 (9)
C4	0.4209 (6)	0.2097 (3)	0.9474 (6)	0.0328 (10)
H4	0.3237	0.1927	0.9642	0.039*
C5	0.5417 (6)	0.1592 (2)	0.9461 (6)	0.0322 (10)
H5	0.5227	0.1089	0.9561	0.039*

C7	0.5890 (6)	0.3869 (2)	0.8690 (7)	0.0226 (9)
O8	0.4396 (6)	−0.0183 (3)	0.7257 (8)	0.0755 (15)
H8A	0.3537	0.0099	0.6642	0.113*
H8B	0.4043	−0.0601	0.7463	0.113*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0207 (2)	0.0188 (2)	0.0264 (2)	0.0014 (2)	0.00942 (17)	−0.00095 (19)
N1	0.028 (2)	0.0247 (17)	0.0301 (16)	−0.0003 (15)	0.0116 (15)	0.0023 (14)
N2	0.023 (3)	0.0329 (18)	0.031 (2)	−0.0011 (14)	0.0125 (18)	−0.0029 (15)
O1	0.0243 (17)	0.0237 (14)	0.065 (2)	−0.0024 (14)	0.0179 (14)	0.0017 (17)
O2	0.0231 (17)	0.0236 (14)	0.0479 (16)	0.0006 (13)	0.0160 (14)	−0.0014 (13)
O3	0.0441 (19)	0.0313 (16)	0.0264 (15)	−0.0016 (15)	0.0122 (13)	0.0034 (12)
O4	0.049 (2)	0.0275 (14)	0.0286 (14)	−0.0017 (14)	0.0175 (14)	−0.0071 (12)
O5	0.0312 (17)	0.0303 (16)	0.0567 (19)	−0.0052 (14)	0.0080 (14)	0.0110 (15)
O6	0.0317 (16)	0.0314 (14)	0.0324 (13)	0.0075 (14)	0.0145 (12)	0.0045 (13)
O7	0.0225 (18)	0.0338 (18)	0.0320 (19)	−0.0006 (11)	0.0088 (15)	0.0049 (11)
C1	0.020 (2)	0.0256 (18)	0.0268 (16)	−0.004 (2)	0.0080 (14)	−0.0001 (18)
C2	0.022 (2)	0.0225 (19)	0.0215 (17)	−0.0037 (16)	0.0063 (15)	−0.0012 (14)
C3	0.019 (2)	0.027 (2)	0.0194 (16)	−0.0031 (16)	0.0039 (14)	−0.0017 (15)
C4	0.029 (3)	0.035 (2)	0.042 (3)	−0.009 (2)	0.023 (2)	−0.0048 (16)
C5	0.034 (3)	0.0257 (19)	0.039 (2)	−0.0028 (19)	0.0162 (18)	−0.0010 (17)
C7	0.020 (2)	0.025 (2)	0.024 (2)	0.0004 (15)	0.0112 (17)	−0.0018 (14)
O8	0.031 (2)	0.062 (3)	0.102 (3)	−0.0071 (18)	−0.004 (2)	0.021 (2)

Geometric parameters (Å, º)

Cu1—O6	2.048 (3)	O5—H5A	0.8422
Cu1—O2	2.066 (3)	O5—H5B	0.8559
Cu1—O5	2.072 (3)	O6—H6A	0.8539
Cu1—O3[i]	2.098 (3)	O6—H6B	0.8410
Cu1—O7	2.169 (4)	O7—H7A	0.8328
Cu1—N1	2.175 (4)	O7—H7B	0.8323
N1—C2	1.335 (5)	C1—C2	1.530 (7)
N1—C5	1.343 (7)	C2—C3	1.391 (6)
N2—C4	1.332 (6)	C3—C7	1.515 (5)
N2—C3	1.347 (6)	C4—C5	1.369 (7)
O1—C1	1.244 (5)	C4—H4	0.9300
O2—C1	1.249 (6)	C5—H5	0.9300
O3—C7	1.232 (6)	O8—H8A	0.8483
O3—Cu1[ii]	2.098 (3)	O8—H8B	0.8472
O4—C7	1.255 (5)		
O6—Cu1—O2	93.81 (13)	Cu1—O6—H6B	116.5
O6—Cu1—O5	94.56 (15)	H6A—O6—H6B	113.7
O2—Cu1—O5	171.41 (14)	Cu1—O7—H7A	107.6
O6—Cu1—O3[i]	84.14 (13)	Cu1—O7—H7B	120.6

O2—Cu1—O3[i]	98.28 (13)	H7A—O7—H7B	117.6
O5—Cu1—O3[i]	84.50 (14)	O1—C1—O2	126.5 (6)
O6—Cu1—O7	87.38 (13)	O1—C1—C2	117.0 (5)
O2—Cu1—O7	91.59 (12)	O2—C1—C2	116.5 (4)
O5—Cu1—O7	86.87 (14)	N1—C2—C3	121.0 (4)
O3[i]—Cu1—O7	167.38 (13)	N1—C2—C1	115.6 (4)
O6—Cu1—N1	171.52 (13)	C3—C2—C1	123.2 (4)
O2—Cu1—N1	77.94 (13)	N2—C3—C2	120.8 (4)
O5—Cu1—N1	93.62 (15)	N2—C3—C7	115.3 (4)
O3[i]—Cu1—N1	98.85 (14)	C2—C3—C7	123.9 (4)
O7—Cu1—N1	90.86 (14)	N2—C4—C5	122.8 (5)
C2—N1—C5	118.1 (4)	N2—C4—H4	118.6
C2—N1—Cu1	111.0 (3)	C5—C4—H4	118.6
C5—N1—Cu1	130.5 (3)	N1—C5—C4	120.3 (4)
C4—N2—C3	116.9 (4)	N1—C5—H5	119.8
C1—O2—Cu1	117.8 (3)	C4—C5—H5	119.8
C7—O3—Cu1[ii]	144.4 (3)	O3—C7—O4	123.9 (4)
Cu1—O5—H5A	114.2	O3—C7—C3	118.7 (4)
Cu1—O5—H5B	124.0	O4—C7—C3	117.3 (4)
H5A—O5—H5B	113.7	H8A—O8—H8B	110.3
Cu1—O6—H6A	107.0		

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