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Tetraaquabis(3-pyridinecarboxamide-κN)-copper(II) bis(2,4,6-trinitrophenolate)

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.047
wR factor = 0.141
Data-to-parameter ratio = 11.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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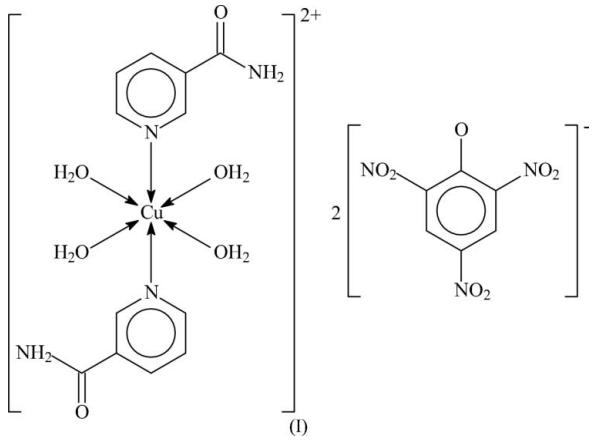
The Cu atom in the title compound, $[Cu(C_6H_6N_2O)_2(H_2O)_4]\cdot(C_6H_2N_3O_7)_2$, exists in an all *trans*-O_4N_2Cu octahedron; the anion interacts indirectly with the cation through the coordinated water molecules. Hydrogen bonds link the cations and anions into a three-dimensional network.

Comment

The picrate (2,4,6-trinitrophenolate) ion binds directly to copper in only a small number of examples such as the pyridine adduct (Simonov *et al.*, 1985), the 4,4′-bipyridine adduct (Liang *et al.*, 2001) and the diaqua complex that crystallizes with benzo-15-crown-5 (Ji *et al.*, 1998). With *N*-heterocycles, the nature of the substituents affects the binding ability of the anion; this is reflected in the title complex with 3-pyridinecarboxamide (nicotinamide) whose cation consists of a tetraaquacopper unit that has the two ligands in a *trans* configuration in its octahedral coordination (Fig. 1). The two anions in the title compound, (I), interact indirectly with the metal through the coordinated water molecules, and the extensive hydrogen bonds (Table 2) give rise to a three-dimensional network.



(I)

The compound is not isostructural with the Zn analog (Zeng *et al.*, 2002) although it has similar hydrogen bonds linking the cations and anions.

Experimental

An aqueous ethanol solution (1:4 *v*/*v*, 10 ml) containing copper(II) nitrate hexahydrate (0.149 g, 0.5 mmol) was mixed with an ethanol solution (10 ml) of nicotinamide (0.122 g, 1.0 mmol). To the mixture was added an ethanol solution (10 ml) of picric acid (0.299 g, 1.0 mmol). The pH of the mixture was adjusted to about 5 by the addition of drops of dilute nitric acid. Blue crystals separated from the solution after several days in 60% yield.

Crystal data

$[Cu(C_6H_6N_2O)_2(H_2O)_4]\cdot(C_6H_2N_3O_7)_2$	$Z = 2$
$M_r = 836.07$	$D_x = 1.740$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 7.154$ (1) Å	Cell parameters from 25 reflections
$b = 14.360$ (2) Å	$\theta = 3.8$–$16.7°$
$c = 15.704$ (2) Å	$\mu = 0.79$ mm^{-1}
$\alpha = 84.85$ (1)°	$T = 295$ (2) K
$\beta = 88.61$ (1)°	Block, blue
$\gamma = 83.31$ (1)°	0.50 × 0.40 × 0.30 mm
$V = 1595.7$ (4) Å^3	

Data collection

Siemens P4 four-circle diffractometer	$R_{int} = 0.014$
ω scans	$\theta_{max} = 25.0°$
Absorption correction: ψ scan (North *et al.*, 1968)	$h = -8 \rightarrow 8$
$T_{min} = 0.693$, $T_{max} = 0.797$	$k = -16 \rightarrow 17$
6306 measured reflections	$l = 0 \rightarrow 18$
5613 independent reflections	3 standard reflections every 97 reflections
4260 reflections with $I > 2\sigma(I)$	intensity decay: 4.4%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0855P)^2 + 0.8133P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.048$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.141$	$\Delta\rho_{max} = 1.15$ e Å^{-3}
$S = 1.06$	$\Delta\rho_{min} = -1.04$ e Å^{-3}
5613 reflections	
476 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Cu1—O1*w*	2.013 (2)	Cu1—O4*w*	2.321 (2)
Cu1—O2*w*	2.302 (2)	Cu1—N1	2.040 (3)
Cu1—O3*w*	2.024 (2)	Cu1—N3	2.034 (3)
O1*w*—Cu1—O2*w*	94.6 (1)	O2*w*—Cu1—N3	87.7 (1)
O1*w*—Cu1—O3*w*	175.9 (1)	O3*w*—Cu1—O4*w*	85.2 (1)
O1*w*—Cu1—O4*w*	90.7 (1)	O3*w*—Cu1—N1	89.0 (1)
O1*w*—Cu1—N1	90.5 (1)	O3*w*—Cu1—N3	92.1 (1)
O1*w*—Cu1—N3	88.6 (1)	O4*w*—Cu1—N1	90.2 (1)
O2*w*—Cu1—O3*w*	89.4 (1)	O4*w*—Cu1—N3	92.6 (1)
O2*w*—Cu1—O4*w*	174.6 (1)	N1—Cu1—N3	177.1 (1)
O2*w*—Cu1—N1	89.6 (1)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*w*—H1*w*2···O1^{i}	0.85	1.84	2.686 (3)	172
O1*w*—H1*w*1···O3	0.85	1.97	2.731 (3)	149
O2*w*—H2*w*1···O1^{ii}	0.85	1.94	2.780 (4)	170
O2*w*—H2*w*2···O14^{iii}	0.85	2.28	2.841 (4)	124
O3*w*—H3*w*1···O2^{iv}	0.85	1.87	2.720 (3)	178
O3*w*—H3*w*2···O10	0.85	1.86	2.705 (3)	176
O4*w*—H4*w*2···O2^{v}	0.85	1.99	2.826 (4)	168
O4*w*—H4*w*1···O10	0.85	2.14	2.933 (4)	156
N2—H2*n*1···O3^{i}	0.85	2.36	3.161 (4)	157
N2—H2*n*2···O11	0.85	2.20	3.014 (4)	161
N4—H4*n*1···O7^{vi}	0.85	2.21	3.020 (4)	159
N4—H4*n*2···O9	0.85	2.31	3.133 (5)	162

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

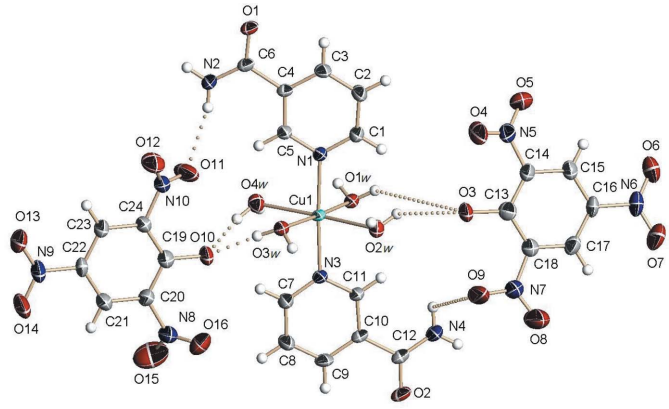


Figure 1
The asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms as spheres of arbitrary radii. Dotted lines indicate hydrogen bonds.

The aromatic rings of both picrate ions were refined as rigid hexagons of 1.39 Å sides. All H atoms were positioned geometrically (C—H = 0.93 Å, N—H = 0.85 Å and O—H = 0.85 Å) and were included in the refinement in the riding-model approximation, with U_{iso}(H)= U_{eq}(C,N,O). The water H atoms were rotated to fit the electron density. In the final difference Fourier map the largest peak and deepest hole are 1 Å from C13, but was otherwise featureless.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *LEAST SQUARES* in *XSCANS*; data reduction: *REDUCE* in *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXL97*.

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