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trans-Bis(5-carboxy-2-methyl-1*H*-imidazole-4-carboxylato-κ^2N^3,O^4)copper(II)

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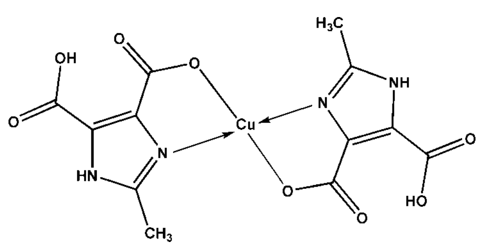
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.003 Å; R factor = 0.030; wR factor = 0.084; data-to-parameter ratio = 13.9.

In the title compound, [Cu($C_6H_5N_2O_4$)$_2$], the copper(II) atom lies on an inversion centre and is in an N_2O_2 four-coordinate environment with a nearly regular square-planar geometry. An extended network of intramolecular O—H···O and intermolecular N—H···O and C—H···O hydrogen bonds stabilizes the crystal structure.

Related literature

For the synthesis and crystal structure of metal complexes with *N*-heterocyclic carboxylic acids, see: Nie *et al.* (2007); Liang *et al.* (2002); Net *et al.* (1989); Zeng *et al.* (2008).



Experimental

Crystal data

[Cu($C_6H_5N_2O_4$)$_2$]
M_r = 401.78
Monoclinic, $P2_1/n$
a = 7.3780 (17) Å
b = 7.575 (2) Å
c = 12.863 (3) Å
β = 101.287 (13)°
V = 705.0 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.61 mm^{-1}
T = 292 K
0.30 × 0.25 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.638, T_{max} = 0.727
5198 measured reflections
1609 independent reflections
1472 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.084
S = 1.07
1609 reflections
116 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O2^{i}	0.86	2.01	2.822 (2)	157
C1—H1A···O4^{ii}	0.96	2.49	3.217 (2)	132
C1—H1B···O3^{iii}	0.96	2.58	3.285 (3)	130
C1—H1C···O1^{iv}	0.96	2.43	3.258 (3)	144
O3—H3A···O2	0.91	1.67	2.576 (2)	171

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x-\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$; (iv) $-x, -y+1, -z$.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL/PC* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL/PC*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2318).

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supporting information

Acta Cryst. (2009). E**65**, m637 [doi:10.1107/S1600536809016791]

trans-Bis(5-carboxy-2-methyl-1*H*-imidazole-4-carboxylato-$\kappa^2 N^3,O^4$)copper(II)

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S1. Comment

The study of metal complexes with *N*-heterocyclic carboxylic acids has been given considerable attention ((Nie *et al.*, 2007; Liang *et al.*, 2002; Net *et al.*, 1989; Zeng *et al.*, 2008). In this paper, we report on the synthesis and structure of the title compound, which was obtained by the hydrothermal reaction of $CuCl_2$ with 2-methyl-1*H*-imidazole-4,5-dicarboxylic acid.

Figure 1 shows the monomeric complex molecule along with the atom-labelling scheme. The copper(II) metal lies on an inversion centre and is in an N_2O_2 four-coordinate environment with a regular square-planar geometry. The Cu—O distance is 1.9633 (15) Å and the Cu—N distance is 1.9830 (14) Å. The five-membered chelating ring assumes an approximately planar conformation (maximum deviation -0.033 (1) Å for atom N1). The crystal structure is stabilized by an intramolecular O—H···O hydrogen bond, and by intermolecular N—H···O and C—H···O hydrogen interactions (Table 1), forming an extended three-dimensional network (Fig. 2).

S2. Experimental

Blue single crystals of title compound were obtained by hydrothermal treatment of $CuCl_2$ (1 mmol), 2-methyl-1*H*-imidazole-4,5-dicarboxylic acid (1 mmol) and water (5 ml) over 2 days at 388 K. Yield: 61% (based on $CuCl_2$).

S3. Refinement

The hydroxyl H atom was located from a difference Fourier map but not refined [$U_{iso}(H)=1.5U_{eq}(O)$]. All other H atoms were placed at calculated positions and refined as riding, with C—H = 0.96 Å, N—H = 0.86 Å, and with $U_{iso}(H)=1.5U_{eq}(C)$ and $U_{iso}(H)=1.2U_{eq}(N)$

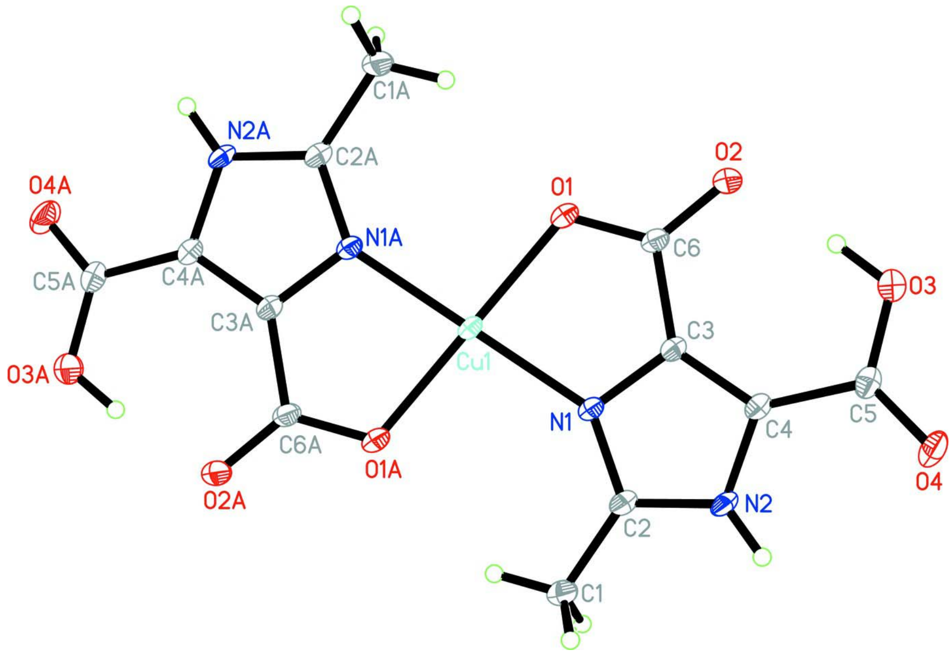

Figure 1

The molecular structure of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Atoms labelled with suffix A are generated by the symmetry operation (-*x*,1 - *y*,-*z*).

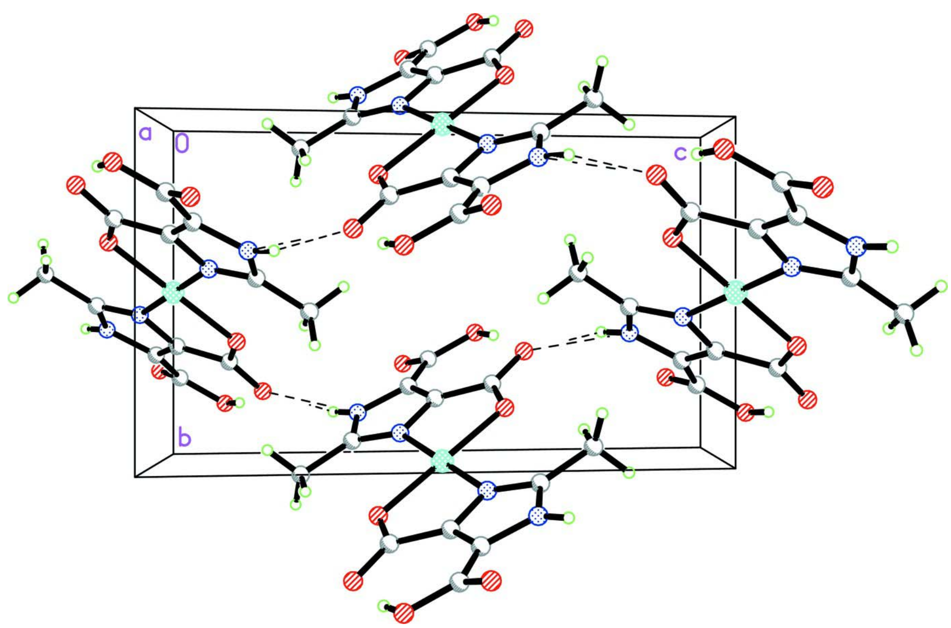

Figure 2

Packing diagram of the title compound viewed along the *a* axis. Intermolecular hydrogen bonds are shown as dashed lines.

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Crystal data

$[Cu(C_6H_5N_2O_4)_2]$
$M_r = 401.78$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.3780$ (17) Å
$b = 7.575$ (2) Å
$c = 12.863$ (3) Å
$\beta = 101.287$ (13)°
$V = 705.0$ (3) Å^3
$Z = 2$
$F(000) = 406$
$D_x = 1.893$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2111 reflections
$\theta = 3.1$–27.5°
$\mu = 1.61$ mm^{-1}
$T = 292$ K
Prism, blue
0.30 × 0.25 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min} = 0.638$, $T_{max} = 0.727$
5198 measured reflections
1609 independent reflections
1472 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
$h = -9\rightarrow9$
$k = -9\rightarrow8$
$l = -16\rightarrow16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.084$
$S = 1.07$
1609 reflections
116 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0506P)^2 + 0.3299P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.66$ e Å^{-3}

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	0.0000	0.5000	0.0000	0.02591 (14)
C1	0.3363 (3)	0.5580 (3)	0.26068 (15)	0.0322 (4)
H1A	0.4201	0.6562	0.2725	0.048*
H1B	0.3575	0.4828	0.3219	0.048*

H1C	0.2114	0.6005	0.2482	0.048*
C2	0.3672 (2)	0.4571 (3)	0.16737 (14)	0.0222 (4)
C3	0.3316 (2)	0.3269 (2)	0.01393 (13)	0.0211 (3)
C4	0.5089 (2)	0.2949 (2)	0.06414 (13)	0.0221 (4)
C5	0.6677 (3)	0.2144 (3)	0.02784 (15)	0.0267 (4)
C6	0.2221 (2)	0.2795 (3)	−0.09127 (14)	0.0241 (4)
N1	0.24525 (19)	0.4288 (2)	0.07763 (11)	0.0216 (3)
N2	0.5278 (2)	0.3781 (2)	0.16036 (11)	0.0226 (3)
H2A	0.6266	0.3796	0.2086	0.027*
O1	0.06229 (18)	0.3460 (2)	−0.11265 (10)	0.0328 (3)
O2	0.29058 (19)	0.1816 (2)	−0.15194 (10)	0.0325 (3)
O3	0.6306 (2)	0.1289 (2)	−0.06309 (11)	0.0362 (4)
H3A	0.5102	0.1358	−0.0972	0.054*
O4	0.8232 (2)	0.2331 (2)	0.07777 (12)	0.0396 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.01512 (18)	0.0374 (2)	0.02196 (19)	0.00490 (12)	−0.00433 (12)	−0.00595 (12)
C1	0.0263 (10)	0.0449 (12)	0.0231 (9)	0.0015 (9)	−0.0008 (7)	−0.0072 (8)
C2	0.0190 (8)	0.0268 (9)	0.0190 (8)	−0.0015 (7)	−0.0004 (6)	0.0029 (7)
C3	0.0172 (8)	0.0242 (9)	0.0205 (8)	−0.0010 (6)	0.0000 (6)	0.0001 (6)
C4	0.0189 (8)	0.0240 (9)	0.0217 (8)	0.0008 (7)	−0.0001 (6)	0.0029 (7)
C5	0.0210 (8)	0.0291 (10)	0.0294 (9)	0.0036 (7)	0.0034 (7)	0.0054 (7)
C6	0.0201 (8)	0.0304 (10)	0.0196 (8)	−0.0010 (7)	−0.0013 (6)	−0.0023 (7)
N1	0.0165 (7)	0.0275 (8)	0.0190 (7)	0.0003 (6)	−0.0013 (5)	−0.0017 (6)
N2	0.0172 (7)	0.0294 (8)	0.0184 (7)	0.0003 (6)	−0.0038 (5)	0.0025 (6)
O1	0.0206 (7)	0.0470 (9)	0.0263 (7)	0.0062 (6)	−0.0065 (5)	−0.0103 (6)
O2	0.0252 (7)	0.0441 (8)	0.0256 (7)	0.0036 (6)	−0.0010 (5)	−0.0113 (6)
O3	0.0273 (7)	0.0465 (9)	0.0342 (8)	0.0079 (6)	0.0047 (6)	−0.0077 (6)
O4	0.0196 (7)	0.0569 (10)	0.0390 (8)	0.0073 (7)	−0.0025 (6)	0.0007 (7)

Geometric parameters (Å, º)

Cu1—N1	1.9633 (15)	C3—N1	1.370 (2)
Cu1—N1^{i}	1.9633 (15)	C3—C6	1.478 (2)
Cu1—O1	1.9830 (14)	C4—N2	1.372 (2)
Cu1—O1^{i}	1.9830 (14)	C4—C5	1.475 (3)
C1—C2	1.478 (3)	C5—O4	1.208 (2)
C1—H1A	0.9600	C5—O3	1.318 (2)
C1—H1B	0.9600	C6—O2	1.252 (2)
C1—H1C	0.9600	C6—O1	1.262 (2)
C2—N1	1.335 (2)	N2—H2A	0.8600
C2—N2	1.346 (2)	O3—H3A	0.9117
C3—C4	1.363 (2)		
N1—Cu1—N1^{i}	180.0	C3—C4—N2	105.31 (15)
N1—Cu1—O1	83.56 (6)	C3—C4—C5	132.21 (17)

N1^{i}—Cu1—O1	96.44 (6)	N2—C4—C5	121.95 (16)
N1—Cu1—O1^{i}	96.44 (6)	O4—C5—O3	122.69 (18)
N1^{i}—Cu1—O1^{i}	83.56 (6)	O4—C5—C4	120.88 (18)
O1—Cu1—O1^{i}	180.00 (5)	O3—C5—C4	116.37 (16)
C2—C1—H1A	109.5	O2—C6—O1	125.06 (16)
C2—C1—H1B	109.5	O2—C6—C3	119.96 (16)
H1A—C1—H1B	109.5	O1—C6—C3	114.98 (16)
C2—C1—H1C	109.5	C2—N1—C3	107.11 (14)
H1A—C1—H1C	109.5	C2—N1—Cu1	142.56 (13)
H1B—C1—H1C	109.5	C3—N1—Cu1	109.91 (11)
N1—C2—N2	108.89 (16)	C2—N2—C4	109.26 (14)
N1—C2—C1	126.93 (17)	C2—N2—H2A	125.4
N2—C2—C1	124.18 (16)	C4—N2—H2A	125.4
C4—C3—N1	109.42 (15)	C6—O1—Cu1	114.58 (11)
C4—C3—C6	133.84 (17)	C5—O3—H3A	114.3
N1—C3—C6	116.72 (15)		

N1—C3—C4—N2	0.6 (2)	C4—C3—N1—C2	−1.1 (2)
C6—C3—C4—N2	178.80 (19)	C6—C3—N1—C2	−179.58 (16)
N1—C3—C4—C5	−170.85 (19)	C4—C3—N1—Cu1	173.21 (12)
C6—C3—C4—C5	7.3 (4)	C6—C3—N1—Cu1	−5.3 (2)
C3—C4—C5—O4	163.5 (2)	O1—Cu1—N1—C2	175.3 (2)
N2—C4—C5—O4	−6.8 (3)	O1^{i}—Cu1—N1—C2	−4.7 (2)
C3—C4—C5—O3	−13.6 (3)	O1—Cu1—N1—C3	4.36 (12)
N2—C4—C5—O3	176.04 (17)	O1^{i}—Cu1—N1—C3	−175.64 (12)
C4—C3—C6—O2	4.4 (3)	N1—C2—N2—C4	−0.7 (2)
N1—C3—C6—O2	−177.58 (17)	C1—C2—N2—C4	179.41 (18)
C4—C3—C6—O1	−174.8 (2)	C3—C4—N2—C2	0.0 (2)
N1—C3—C6—O1	3.2 (3)	C5—C4—N2—C2	172.59 (17)
N2—C2—N1—C3	1.1 (2)	O2—C6—O1—Cu1	−178.45 (16)
C1—C2—N1—C3	−179.04 (19)	C3—C6—O1—Cu1	0.7 (2)
N2—C2—N1—Cu1	−170.06 (15)	N1—Cu1—O1—C6	−2.89 (14)
C1—C2—N1—Cu1	9.8 (4)	N1^{i}—Cu1—O1—C6	177.11 (14)

Symmetry code: (i) −*x*, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O2^{ii}	0.86	2.01	2.822 (2)	157
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