Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis{*N*-[5-(4-methoxyphenyl)-1,3,4-oxadiazol-2-yl]ethanimidamidato}copper(II)

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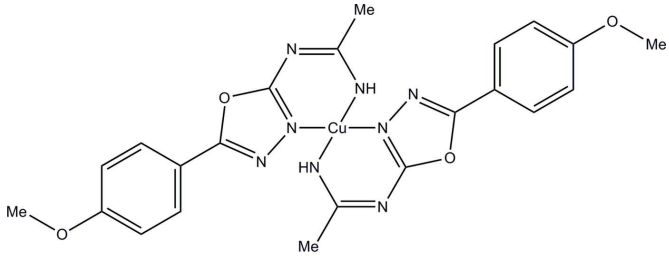
Received 16 February 2010; accepted 9 March 2010

Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.087; data-to-parameter ratio = 15.9.

The title compound, $[Cu(C_{11}H_{11}N_4O_2)_2]$, was prepared by solvothermal synthesis using 2-amino-5-(4-methoxyphenyl)-1,3,4-oxadiazole and copper sulfate pentahydrate in an acetonitrile solution. The Cu^{II} atom lies on an inversion center and is four-coordinated in a slightly distorted square-planar geometry by four N atoms of the ligands obtained from the formation of a bond between the amine N atom of the oxadiazole molecule and the nitrile C atom of the solvent. In the crystal structure an intermolecular N—H···N hydrogen bond links inversion-related molecules.

Related literature

For comparative bond lengths in similar coordination compounds, see: Cai, (2009). For applications of complexes formed by Schiff base ligands, see: Lu & Schauss (2002). For chemotherapeutic effects of 2,5-substituted-1,3,4-oxadiazole derivatives, see: Cao *et al.* (2002); Kadi *et al.* (2007); Zareef *et al.* (2006, 2007, 2008).



Experimental

Crystal data

$[Cu(C_{11}H_{11}N_4O_2)_2]$
M_r = 526.02
Triclinic, $P\overline{1}$
a = 4.9020 (6) Å
b = 11.2083 (14) Å
c = 11.5739 (14) Å
α = 111.501 (5)°
β = 99.274 (6)°
γ = 91.564 (5)°
V = 581.33 (12) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.99 mm^{-1}
T = 120 K
0.53 × 0.23 × 0.07 mm

Data collection

Bruker APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.707, T_{max} = 0.933
6654 measured reflections
2633 independent reflections
2417 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.087
S = 1.06
2633 reflections
166 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N8^{i}	0.88	2.42	2.983 (2)	123

Symmetry code: (i) $-x, -y, -z+1$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

This work was supported by the Laboratoire de Chimie des Materiaux, Faculté des Sciences, Université Mentouri. We would like to thank J.-Y. Saillard of Rennes 1 University for providing diffraction facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2228).

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supporting information

Acta Cryst. (2010). E**66**, m410 [doi:10.1107/S1600536810009050]

Bis{*N*-[5-(4-methoxyphenyl)-1,3,4-oxadiazol-2-yl]ethanimidamidato}copper(II)

Yacine Djebli, Salima Mosbah, Sihem Boufas, Leila Bencharif and Thierry Roisnel

S1. Comment

In recent years, there has been a considerable effort towards preparation of new materials containing polyfunctional organic ligands able to bind metallic ions by solvothermal synthesis. For example, with Schiff bases ligands, such complexes could be applied in different areas, including biochemistry, electrochemistry, and catalysis (Lu *et al.*, 2002).

The 2,5-substituted-1,3,4-oxadiazole derivatives are of significant interest due to their chemotherapeutic effects (Kadi *et al.*, 2007; Zareef *et al.*, 2008; Zareef *et al.*, 2007; Zareef *et al.*, 2006; Cao *et al.*, 2002). In this paper, we report the structure of one of these compounds with copper (II).

In the centrosymmetric title complex, the Cu (II) atom is located on an inversion center and is four-coordinated in a square planar geometry by four N atoms of the ligands obtained from the formation of a bond between *N*-amine of the oxadiazole molecule and *C*-nitrile of the solvent. All the coordinated bond lengths are typical and comparable with those in similar copper (II) complexes (Cai,2009). In the title compound, there is just one weak hydrogen bond N1-H1···N8 linking different inversion (-x, -y, -Z+1) related molecules.

S2. Experimental

5-(4-Methoxy-phenyl)-2amino-1, 3, 4-oxadiazole (0,2 mmole) and (0,1 mmole) copper sulfate pentahydrate were mixed in 5 ml of acetonitrile. The mixture was placed in a Teflon-lined stainless steel vessel, and heated to 160° C for 16 h. It was then cooled to room temperature over a period of 24 h, and washed using acetonitrile. Brown crystals suitable for X-Ray crystallography were obtained.

S3. Refinement

H atoms were placed at calculated positions (C-H = 0..88-0.98 Å) and were treated as riding on their parent atoms, with U_{iso}(H) set to 1.2-1.5 times U_{eq}(C).

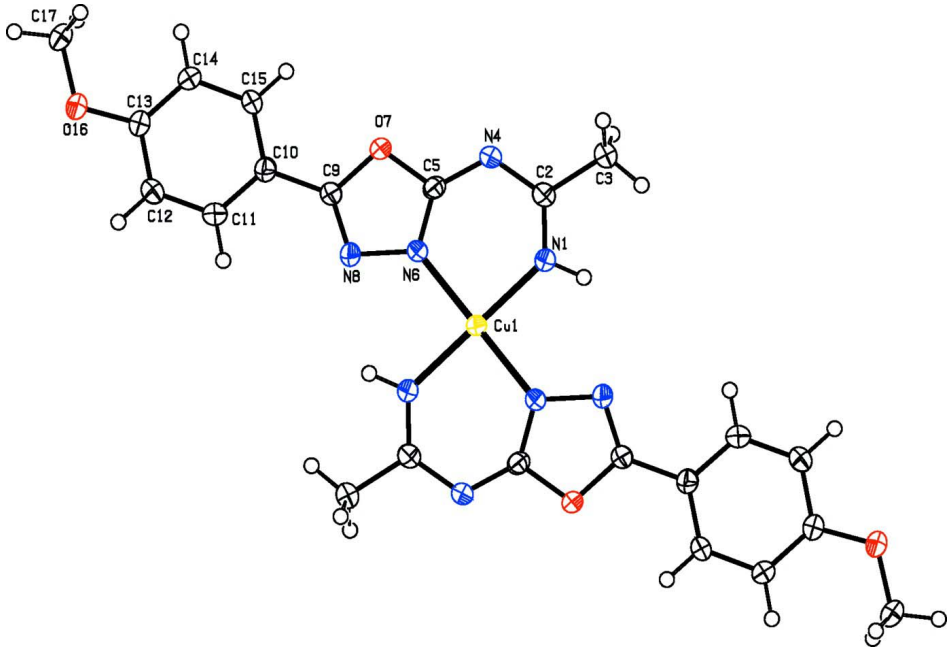

Figure 1

The molecular structure of the title compound in 30% probability displacement ellipsoids for non-H atoms.

Bis{*N*-[5-(4-methoxyphenyl)-1,3,4-oxadiazol-2- yl]ethanimidamidato}copper(II)

Crystal data

$[Cu(C_{11}H_{11}N_4O_2)_2]$
$M_r = 526.02$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 4.9020\ (6)$ Å
$b = 11.2083\ (14)$ Å
$c = 11.5739\ (14)$ Å
$\alpha = 111.501\ (5)°$
$\beta = 99.274\ (6)°$
$\gamma = 91.564\ (5)°$
$V = 581.33\ (12)$ Å^3
$Z = 1$
$F(000) = 271$
$D_x = 1.503$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2925 reflections
$\theta = 3.2–27.4°$
$\mu = 0.99$ mm^{-1}
$T = 120$ K
Plate, brown
0.53 × 0.23 × 0.07 mm

Data collection

Bruker APEXII diffractometer
Radiation source: Enraf-Nonius FR590
Graphite monochromator
CCD rotation images, thin slices scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
$T_{min} = 0.707$, $T_{max} = 0.933$
6654 measured reflections
2633 independent reflections
2417 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.2°$
$h = -6\rightarrow6$
$k = -14\rightarrow14$
$l = -15\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.087$
$S = 1.06$
2633 reflections
166 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
$w = 1/[\sigma^2(F_o^2) + (0.0355P)^2 + 0.2373P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cu1	0	0	0.5	0.02231 (12)
N1	0.1428 (3)	−0.09664 (16)	0.34848 (16)	0.0245 (4)
H1	0.0696	−0.1769	0.3098	0.029*
C2	0.3294 (4)	−0.06281 (19)	0.29405 (19)	0.0240 (4)
C3	0.3968 (5)	−0.1574 (2)	0.1722 (2)	0.0314 (5)
H3A	0.3138	−0.2438	0.1562	0.047*
H3B	0.5986	−0.1581	0.1794	0.047*
H3C	0.3216	−0.1314	0.1021	0.047*
N4	0.4749 (3)	0.05335 (16)	0.33696 (16)	0.0255 (4)
C5	0.4203 (4)	0.14395 (19)	0.44061 (19)	0.0229 (4)
N6	0.2379 (3)	0.14792 (15)	0.51450 (16)	0.0230 (4)
O7	0.5775 (3)	0.26005 (13)	0.48592 (13)	0.0244 (3)
N8	0.2771 (3)	0.26958 (16)	0.61315 (17)	0.0257 (4)
C9	0.4788 (4)	0.33114 (19)	0.59302 (19)	0.0234 (4)
C10	0.6076 (4)	0.46056 (19)	0.6693 (2)	0.0251 (4)
C11	0.5447 (5)	0.5265 (2)	0.7888 (2)	0.0368 (5)
H11	0.417	0.4865	0.8202	0.044*
C12	0.6665 (5)	0.6494 (2)	0.8615 (2)	0.0385 (6)
H12	0.6239	0.6931	0.9429	0.046*
C13	0.8519 (4)	0.70962 (19)	0.8158 (2)	0.0266 (4)
C14	0.9179 (5)	0.6449 (2)	0.6975 (2)	0.0309 (5)
H14	1.0455	0.6849	0.6661	0.037*
C15	0.7950 (5)	0.5211 (2)	0.6259 (2)	0.0321 (5)
H15	0.8404	0.4767	0.5452	0.038*
O16	0.9576 (3)	0.83100 (14)	0.89471 (14)	0.0336 (4)
C17	1.1494 (5)	0.8972 (2)	0.8518 (2)	0.0321 (5)
H17A	1.3138	0.8496	0.8384	0.048*
H17B	1.205	0.9841	0.9154	0.048*
H17C	1.0601	0.903	0.7722	0.048*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.02216 (19)	0.01886 (19)	0.0244 (2)	−0.00107 (13)	0.00942 (14)	0.00450 (14)

N1	0.0251 (8)	0.0203 (8)	0.0251 (9)	−0.0016 (6)	0.0080 (7)	0.0039 (7)
C2	0.0251 (10)	0.0244 (10)	0.0225 (10)	0.0037 (8)	0.0058 (8)	0.0083 (8)
C3	0.0365 (12)	0.0275 (11)	0.0263 (11)	−0.0012 (9)	0.0110 (9)	0.0039 (9)
N4	0.0290 (9)	0.0231 (9)	0.0238 (9)	−0.0008 (7)	0.0106 (7)	0.0057 (7)
C5	0.0225 (9)	0.0206 (10)	0.0261 (10)	−0.0003 (7)	0.0048 (8)	0.0094 (8)
N6	0.0233 (8)	0.0199 (8)	0.0238 (8)	0.0002 (6)	0.0094 (7)	0.0040 (7)
O7	0.0277 (7)	0.0197 (7)	0.0247 (7)	−0.0024 (5)	0.0093 (6)	0.0056 (6)
N8	0.0266 (9)	0.0187 (8)	0.0279 (9)	−0.0001 (7)	0.0097 (7)	0.0027 (7)
C9	0.0247 (10)	0.0208 (10)	0.0243 (10)	0.0022 (8)	0.0081 (8)	0.0064 (8)
C10	0.0257 (10)	0.0203 (10)	0.0274 (11)	0.0015 (8)	0.0068 (8)	0.0060 (8)
C11	0.0446 (13)	0.0299 (12)	0.0344 (12)	−0.0070 (10)	0.0200 (11)	0.0061 (10)
C12	0.0540 (15)	0.0298 (12)	0.0281 (12)	−0.0058 (10)	0.0201 (11)	0.0021 (10)
C13	0.0294 (10)	0.0205 (10)	0.0265 (10)	−0.0013 (8)	0.0053 (9)	0.0053 (8)
C14	0.0354 (12)	0.0251 (11)	0.0305 (11)	−0.0060 (9)	0.0127 (9)	0.0061 (9)
C15	0.0397 (12)	0.0252 (11)	0.0257 (11)	−0.0046 (9)	0.0157 (9)	−0.0004 (9)
O16	0.0432 (9)	0.0231 (8)	0.0287 (8)	−0.0081 (6)	0.0108 (7)	0.0021 (6)
C17	0.0365 (12)	0.0225 (11)	0.0343 (12)	−0.0059 (9)	0.0070 (10)	0.0075 (9)

Geometric parameters (Å, º)

Cu1—N6^{i}	1.9403 (16)	C9—C10	1.454 (3)
Cu1—N6	1.9403 (16)	C10—C15	1.386 (3)
Cu1—N1^{i}	1.9451 (17)	C10—C11	1.398 (3)
Cu1—N1	1.9451 (17)	C11—C12	1.380 (3)
N1—C2	1.311 (3)	C11—H11	0.95
N1—H1	0.88	C12—C13	1.394 (3)
C2—N4	1.346 (3)	C12—H12	0.95
C2—C3	1.515 (3)	C13—O16	1.361 (2)
C3—H3A	0.98	C13—C14	1.390 (3)
C3—H3B	0.98	C14—C15	1.388 (3)
C3—H3C	0.98	C14—H14	0.95
N4—C5	1.329 (3)	C15—H15	0.95
C5—N6	1.324 (3)	O16—C17	1.436 (3)
C5—O7	1.370 (2)	C17—H17A	0.98
N6—N8	1.405 (2)	C17—H17B	0.98
O7—C9	1.377 (2)	C17—H17C	0.98
N8—C9	1.289 (2)		

N6^{i}—Cu1—N6	180.00 (6)	N8—C9—C10	127.92 (19)
N6^{i}—Cu1—N1^{i}	87.39 (7)	O7—C9—C10	119.18 (17)
N6—Cu1—N1^{i}	92.61 (7)	C15—C10—C11	118.54 (19)
N6^{i}—Cu1—N1	92.61 (7)	C15—C10—C9	121.09 (19)
N6—Cu1—N1	87.39 (7)	C11—C10—C9	120.36 (19)
N1^{i}—Cu1—N1	180	C12—C11—C10	120.5 (2)
C2—N1—Cu1	131.13 (14)	C12—C11—H11	119.7
C2—N1—H1	114.4	C10—C11—H11	119.7
Cu1—N1—H1	114.4	C11—C12—C13	120.2 (2)
N1—C2—N4	125.48 (18)	C11—C12—H12	119.9

N1—C2—C3	120.38 (18)	C13—C12—H12	119.9
N4—C2—C3	114.13 (17)	O16—C13—C14	124.78 (19)
C2—C3—H3A	109.5	O16—C13—C12	115.32 (19)
C2—C3—H3B	109.5	C14—C13—C12	119.90 (19)
H3A—C3—H3B	109.5	C15—C14—C13	119.2 (2)
C2—C3—H3C	109.5	C15—C14—H14	120.4
H3A—C3—H3C	109.5	C13—C14—H14	120.4
H3B—C3—H3C	109.5	C10—C15—C14	121.6 (2)
C5—N4—C2	118.09 (17)	C10—C15—H15	119.2
N6—C5—N4	133.31 (18)	C14—C15—H15	119.2
N6—C5—O7	109.26 (17)	C13—O16—C17	117.41 (17)
N4—C5—O7	117.43 (17)	O16—C17—H17A	109.5
C5—N6—N8	108.50 (16)	O16—C17—H17B	109.5
C5—N6—Cu1	124.23 (14)	H17A—C17—H17B	109.5
N8—N6—Cu1	126.65 (13)	O16—C17—H17C	109.5
C5—O7—C9	104.03 (14)	H17A—C17—H17C	109.5
C9—N8—N6	105.28 (16)	H17B—C17—H17C	109.5
N8—C9—O7	112.90 (17)		

N6^{i}—Cu1—N1—C2	−177.96 (19)	N6—N8—C9—O7	0.9 (2)
N6—Cu1—N1—C2	2.04 (19)	N6—N8—C9—C10	−178.20 (19)
Cu1—N1—C2—N4	1.8 (3)	C5—O7—C9—N8	−1.4 (2)
Cu1—N1—C2—C3	−178.05 (14)	C5—O7—C9—C10	177.76 (17)
N1—C2—N4—C5	−2.6 (3)	N8—C9—C10—C15	−170.3 (2)
C3—C2—N4—C5	177.23 (17)	O7—C9—C10—C15	10.7 (3)
C2—N4—C5—N6	−2.5 (3)	N8—C9—C10—C11	9.8 (3)
C2—N4—C5—O7	177.53 (17)	O7—C9—C10—C11	−169.16 (19)
N4—C5—N6—N8	179.1 (2)	C15—C10—C11—C12	0.2 (4)
O7—C5—N6—N8	−0.9 (2)	C9—C10—C11—C12	180.0 (2)
N4—C5—N6—Cu1	7.6 (3)	C10—C11—C12—C13	0.7 (4)
O7—C5—N6—Cu1	−172.39 (12)	C11—C12—C13—O16	179.4 (2)
N1^{i}—Cu1—N6—C5	174.14 (16)	C11—C12—C13—C14	−1.1 (4)
N1—Cu1—N6—C5	−5.86 (16)	O16—C13—C14—C15	−179.9 (2)
N1^{i}—Cu1—N6—N8	4.22 (16)	C12—C13—C14—C15	0.6 (3)
N1—Cu1—N6—N8	−175.78 (16)	C11—C10—C15—C14	−0.6 (3)
N6—C5—O7—C9	1.4 (2)	C9—C10—C15—C14	179.6 (2)
N4—C5—O7—C9	−178.66 (17)	C13—C14—C15—C10	0.2 (4)
C5—N6—N8—C9	0.0 (2)	C14—C13—O16—C17	0.6 (3)
Cu1—N6—N8—C9	171.26 (14)	C12—C13—O16—C17	−179.9 (2)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N8^{i}	0.88	2.42	2.983 (2)	123

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