Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Di-μ-iodido-bis[(*N*-morpholino-2-pyridylmethanimine-κ^2N,N')copper(I)] acetonitrile solvate

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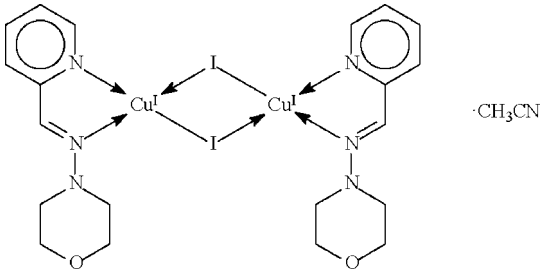
Received 10 August 2007; accepted 10 August 2007

Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.005 Å; R factor = 0.030; wR factor = 0.080; data-to-parameter ratio = 20.2.

In the crystal structure of the title compound, $[Cu_2I_2(C_{10}H_{13}N_3O)_2]\cdot CH_3CN$, the Schiff base chelates the Cu^I atom, which is linked to two I atoms in a tetrahedral geometry; the covalent Cu—I bond is only marginally shorter than the dative Cu—I bond. The dinuclear molecule lies about a centre of inversion and the solvent molecule on a twofold rotation axis.

Related literature

For the synthesis of the Schiff base ligand, see Wiley *et al.* (1959). There are only two reports of metal adducts of this ligand (not crystallographic studies): see Nasser-Eddine *et al.* (2004) for the copper(I) bromide adduct, and Nikolcheva *et al.* (2003) for the platinum(II) dichloride adduct.



Experimental

Crystal data

$[Cu_2I_2(C_{10}H_{13}N_3O)_2]\cdot C_2H_3N$
M_r = 804.40
Monoclinic, $C2/c$
a = 16.0206 (2) Å
b = 10.2820 (1) Å
c = 17.2086 (1) Å
β = 95.329 (1)°
V = 2822.41 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.73 mm^{-1}
T = 200 (2) K
0.24 × 0.18 × 0.05 mm

Data collection

Bruker SMART area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.468, T_{max} = 0.836
13511 measured reflections
3247 independent reflections
2897 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.080
S = 1.04
3247 reflections
161 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.01 e Å^{-3}
$\Delta\rho_{min}$ = −1.03 e Å^{-3}

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

The authors thank the Alzahra University Research Council, the University of Heidelberg and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2476).

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 doi:10.1107/S1600536807039670

supplementary materials

Acta Cryst. (2007). E**63**, m2377 [**doi:10.1107/S1600536807039670**]

Di-μ-iodido-bis[(*N*-morpholino-2-pyridylmethanimine-κ^2N,N')copper(I)] acetonitrile solvate

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Comment

Copper(I) iodide forms a large number of adducts with Schiff bases. However, there are no structural studies on the morpholine-2-pyridyl-methanimine. In the title compound, the Schiff base chelates to the copper(I) atom, which is linked to two iodine atoms in a tetrahedral geometry; the covalent Cu–I bond is only marginally shorter than the dative Cu–I bond (Table 1) in the crystal structure of $(C_{10}H_{13}N_3O)_2(CuI)_2{\cdot}CH_3CN$. The dinuclear molecule lies about a center-of-inversion whereas the solvent molecule lies on a twofold rotation axis.

Experimental

Copper(I) iodide (1 mmol) and morpholine-2-pyridylmethanimine (1 mmol) were dissolved in acetonitrile under a nitrogen atmosphere. The solvent was partially removed and diethyl ether vapor diffused into the concentrated solution. Orange crystals were obtained in 90% yield. Calc. for $C_{20}H_{26}Cu_2I_2N_6O_2$:*C* 31.47, H 3.43, N 11.01%. Found: C 31.45, H 3.40, N 11.06%.

Refinement

The carbon-bound hydrogen atoms were placed at calculated positions (C–H 0.93 – 0.99 Å), and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2 – 1.5 U_{eq}(C). The methyl group of the acetonitrile molecule is disordered over two equally occupied sites. The final difference Fourier map had a large peak/hole in the vicinity of the iodine atom.

Figures

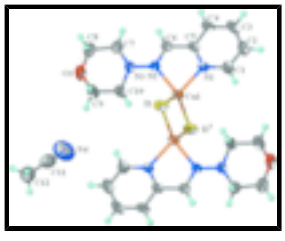

Fig. 1. Thermal ellipsoid plot of $(C_{10}H_{13}N_3O)_2(CuI)_2{\cdot}CH_3CN$ drawn at the 70% probability level; hydrogen atoms are shown as spheres of arbitrary radius. Symmetry code (i): $1-x, 1-y, 1-z$.

Di-μ-iodido-bis[(*N*-morpholino-2-pyridylmethanimine-κ$^2N,N'$)copper(I)] acetonitrile solvate

Crystal data

$[Cu_2I_2(C_{10}H_{13}N_3O)_2]{\cdot}C_2H_3N$	F_{000} = 1560
M_r = 804.40	D_x = 1.893 Mg m^{-3}

Monoclinic, $C2/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 8192 reflections
$a = 16.0206$ (2) Å	$\theta = 2.4$–27.5°
$b = 10.2820$ (1) Å	$\mu = 3.73$ mm^{-1}
$c = 17.2086$ (1) Å	$T = 200$ (2) K
$\beta = 95.329$ (1)°	Polyhedron, orange
$V = 2822.41$ (5) Å^3	0.24 × 0.18 × 0.05 mm
$Z = 4$	

Data collection

Bruker SMART area-detector diffractometer	3247 independent reflections
Radiation source: fine-focus sealed tube	2897 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.050$
$T = 200$(2) K	$\theta_{max} = 27.5^\circ$
φ and ω scans	$\theta_{min} = 2.4^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -20 \rightarrow 20$
$T_{min} = 0.468$, $T_{max} = 0.836$	$k = -13 \rightarrow 13$
13511 measured reflections	$l = -22 \rightarrow 22$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.030$	H-atom parameters constrained
$wR(F^2) = 0.080$	$w = 1/[\sigma^2(F_o^2) + (0.0356P)^2 + 6.1582P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} = 0.001$
3247 reflections	$\Delta\rho_{max} = 1.01$ e Å^{-3}
161 parameters	$\Delta\rho_{min} = -1.03$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Cu1	0.55574 (2)	0.41824 (4)	0.48182 (2)	0.03299 (11)	
I1	0.600440 (12)	0.60525 (2)	0.576912 (13)	0.03845 (9)	
O1	0.69807 (18)	0.6939 (3)	0.24161 (16)	0.0525 (7)	
N1	0.59867 (15)	0.2310 (2)	0.50200 (15)	0.0302 (5)	
N2	0.64685 (15)	0.4059 (2)	0.39882 (14)	0.0274 (5)	
N3	0.67489 (16)	0.5071 (2)	0.35742 (15)	0.0309 (5)	
N4	0.5000	0.9807 (8)	0.2500	0.135 (4)	
C1	0.5713 (2)	0.1401 (3)	0.5497 (2)	0.0395 (7)	

H1	0.5306	0.1643	0.5838	0.047*	
C2	0.5995 (2)	0.0129 (3)	0.5513 (2)	0.0433 (8)	
H2	0.5778	−0.0489	0.5853	0.052*	
C3	0.6595 (2)	−0.0229 (3)	0.5030 (2)	0.0421 (8)	
H3	0.6803	−0.1094	0.5034	0.051*	
C4	0.6886 (2)	0.0692 (3)	0.45417 (19)	0.0348 (7)	
H4	0.7303	0.0470	0.4207	0.042*	
C5	0.65638 (17)	0.1957 (3)	0.45415 (16)	0.0271 (6)	
C6	0.68527 (18)	0.2951 (3)	0.40212 (17)	0.0278 (6)	
H6	0.7307	0.2789	0.3717	0.033*	
C7	0.7327 (2)	0.4817 (3)	0.29850 (18)	0.0342 (6)	
H7A	0.7036	0.4332	0.2542	0.041*	
H7B	0.7803	0.4284	0.3211	0.041*	
C8	0.7643 (2)	0.6107 (3)	0.2703 (2)	0.0456 (8)	
H8A	0.7979	0.6544	0.3139	0.055*	
H8B	0.8014	0.5946	0.2284	0.055*	
C9	0.6470 (3)	0.7216 (4)	0.3030 (3)	0.0560 (10)	
H9A	0.6020	0.7828	0.2839	0.067*	
H9B	0.6814	0.7639	0.3466	0.067*	
C10	0.6084 (2)	0.5989 (3)	0.3320 (2)	0.0453 (9)	
H10A	0.5752	0.6197	0.3761	0.054*	
H10B	0.5704	0.5597	0.2897	0.054*	
C11	0.5000	1.0891 (7)	0.2500	0.0634 (17)	
C12	0.5000	1.2277 (6)	0.2500	0.0564 (14)	
H12A	0.4421	1.2595	0.2474	0.085*	0.50
H12B	0.5267	1.2595	0.2047	0.085*	0.50
H12C	0.5311	1.2595	0.2980	0.085*	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0308 (2)	0.02673 (19)	0.0425 (2)	0.00814 (14)	0.00937 (16)	−0.00119 (15)
I1	0.03060 (13)	0.03548 (13)	0.04833 (15)	0.00547 (8)	−0.00136 (9)	−0.01112 (8)
O1	0.0623 (16)	0.0444 (14)	0.0523 (15)	−0.0009 (12)	0.0136 (13)	0.0198 (12)
N1	0.0269 (12)	0.0278 (12)	0.0356 (13)	0.0044 (9)	0.0018 (10)	0.0041 (10)
N2	0.0281 (12)	0.0240 (11)	0.0303 (12)	0.0011 (9)	0.0040 (10)	0.0011 (9)
N3	0.0345 (13)	0.0273 (12)	0.0317 (12)	0.0028 (10)	0.0077 (10)	0.0042 (10)
N4	0.110 (6)	0.059 (5)	0.239 (12)	0.000	0.024 (7)	0.000
C1	0.0387 (17)	0.0377 (17)	0.0425 (18)	0.0016 (14)	0.0058 (14)	0.0101 (14)
C2	0.0465 (19)	0.0343 (17)	0.0476 (19)	−0.0028 (14)	−0.0036 (15)	0.0134 (15)
C3	0.0484 (19)	0.0254 (15)	0.0499 (19)	0.0067 (14)	−0.0098 (15)	0.0039 (13)
C4	0.0342 (16)	0.0285 (14)	0.0403 (17)	0.0102 (12)	−0.0027 (13)	−0.0026 (12)
C5	0.0261 (13)	0.0239 (13)	0.0300 (14)	0.0041 (10)	−0.0039 (11)	−0.0025 (10)
C6	0.0278 (13)	0.0258 (13)	0.0299 (14)	0.0032 (11)	0.0036 (11)	−0.0049 (11)
C7	0.0384 (16)	0.0348 (16)	0.0306 (15)	0.0002 (13)	0.0103 (12)	−0.0009 (12)
C8	0.049 (2)	0.046 (2)	0.0441 (19)	−0.0056 (15)	0.0149 (16)	0.0072 (15)
C9	0.064 (2)	0.0336 (18)	0.073 (3)	0.0077 (17)	0.020 (2)	0.0157 (18)
C10	0.0424 (19)	0.0364 (18)	0.059 (2)	0.0099 (14)	0.0142 (17)	0.0162 (15)

C11	0.044 (3)	0.056 (4)	0.091 (5)	0.000	0.015 (3)	0.000
C12	0.064 (4)	0.055 (3)	0.052 (3)	0.000	0.013 (3)	0.000

Geometric parameters (Å, °)

Cu1—N1	2.063 (2)	C3—H3	0.9500
Cu1—N2	2.139 (2)	C4—C5	1.399 (4)
Cu1—Cu1^{i}	2.5719 (7)	C4—H4	0.9500
Cu1—I1	2.5827 (4)	C5—C6	1.462 (4)
Cu1—I1^{i}	2.6211 (4)	C6—H6	0.9500
I1—Cu1^{i}	2.6211 (4)	C7—C8	1.516 (4)
O1—C8	1.415 (5)	C7—H7A	0.9900
O1—C9	1.423 (5)	C7—H7B	0.9900
N1—C5	1.344 (4)	C8—H8A	0.9900
N1—C1	1.345 (4)	C8—H8B	0.9900
N2—C6	1.293 (4)	C9—C10	1.510 (5)
N2—N3	1.361 (3)	C9—H9A	0.9900
N3—C7	1.459 (4)	C9—H9B	0.9900
N3—C10	1.459 (4)	C10—H10A	0.9900
N4—C11	1.114 (9)	C10—H10B	0.9900
C1—C2	1.383 (5)	C11—C12	1.425 (9)
C1—H1	0.9500	C12—H12A	0.9800
C2—C3	1.378 (5)	C12—H12B	0.9800
C2—H2	0.9500	C12—H12C	0.9800
C3—C4	1.376 (5)		
N1—Cu1—N2	79.59 (9)	C4—C5—C6	120.8 (3)
N1—Cu1—Cu1^{i}	142.87 (8)	N2—C6—C5	117.8 (3)
N2—Cu1—Cu1^{i}	137.20 (7)	N2—C6—H6	121.1
N1—Cu1—I1	121.31 (7)	C5—C6—H6	121.1
N2—Cu1—I1	107.53 (7)	N3—C7—C8	108.6 (3)
Cu1^{i}—Cu1—I1	61.128 (16)	N3—C7—H7A	110.0
N1—Cu1—I1^{i}	105.69 (7)	C8—C7—H7A	110.0
N2—Cu1—I1^{i}	115.04 (7)	N3—C7—H7B	110.0
Cu1^{i}—Cu1—I1^{i}	59.638 (16)	C8—C7—H7B	110.0
I1—Cu1—I1^{i}	120.766 (14)	H7A—C7—H7B	108.4
Cu1—I1—Cu1^{i}	59.234 (14)	O1—C8—C7	112.2 (3)
C8—O1—C9	109.0 (3)	O1—C8—H8A	109.2
C5—N1—C1	117.9 (3)	C7—C8—H8A	109.2
C5—N1—Cu1	112.77 (19)	O1—C8—H8B	109.2
C1—N1—Cu1	129.0 (2)	C7—C8—H8B	109.2
C6—N2—N3	121.2 (3)	H8A—C8—H8B	107.9
C6—N2—Cu1	112.01 (19)	O1—C9—C10	111.1 (3)
N3—N2—Cu1	125.65 (18)	O1—C9—H9A	109.4
N2—N3—C7	119.2 (2)	C10—C9—H9A	109.4
N2—N3—C10	112.6 (2)	O1—C9—H9B	109.4
C7—N3—C10	113.6 (3)	C10—C9—H9B	109.4
N1—C1—C2	123.1 (3)	H9A—C9—H9B	108.0

N1—C1—H1	118.5	N3—C10—C9	109.2 (3)
C2—C1—H1	118.5	N3—C10—H10A	109.8
C3—C2—C1	119.0 (3)	C9—C10—H10A	109.8
C3—C2—H2	120.5	N3—C10—H10B	109.8
C1—C2—H2	120.5	C9—C10—H10B	109.8
C4—C3—C2	118.7 (3)	H10A—C10—H10B	108.3
C4—C3—H3	120.7	N4—C11—C12	180.000 (3)
C2—C3—H3	120.7	C11—C12—H12A	109.5
C3—C4—C5	119.6 (3)	C11—C12—H12B	109.5
C3—C4—H4	120.2	H12A—C12—H12B	109.5
C5—C4—H4	120.2	C11—C12—H12C	109.5
N1—C5—C4	121.7 (3)	H12A—C12—H12C	109.5
N1—C5—C6	117.4 (2)	H12B—C12—H12C	109.5
N1—Cu1—I1—Cu1^{i}	−136.91 (9)	C5—N1—C1—C2	−0.2 (5)
N2—Cu1—I1—Cu1^{i}	134.79 (7)	Cu1—N1—C1—C2	172.5 (3)
I1^{i}—Cu1—I1—Cu1^{i}	0.0	N1—C1—C2—C3	1.1 (5)
N2—Cu1—N1—C5	−2.1 (2)	C1—C2—C3—C4	−0.7 (5)
Cu1^{i}—Cu1—N1—C5	171.24 (14)	C2—C3—C4—C5	−0.5 (5)
I1—Cu1—N1—C5	−106.40 (19)	C1—N1—C5—C4	−1.1 (4)
I1^{i}—Cu1—N1—C5	111.17 (19)	Cu1—N1—C5—C4	−174.9 (2)
N2—Cu1—N1—C1	−175.1 (3)	C1—N1—C5—C6	179.5 (3)
Cu1^{i}—Cu1—N1—C1	−1.8 (4)	Cu1—N1—C5—C6	5.7 (3)
I1—Cu1—N1—C1	80.6 (3)	C3—C4—C5—N1	1.5 (5)
I1^{i}—Cu1—N1—C1	−61.8 (3)	C3—C4—C5—C6	−179.2 (3)
N1—Cu1—N2—C6	−2.0 (2)	N3—N2—C6—C5	173.9 (2)
Cu1^{i}—Cu1—N2—C6	−176.06 (16)	Cu1—N2—C6—C5	5.5 (3)
I1—Cu1—N2—C6	117.78 (19)	N1—C5—C6—N2	−7.8 (4)
I1^{i}—Cu1—N2—C6	−104.5 (2)	C4—C5—C6—N2	172.8 (3)
N1—Cu1—N2—N3	−169.6 (2)	N2—N3—C7—C8	−171.4 (3)
Cu1^{i}—Cu1—N2—N3	16.3 (3)	C10—N3—C7—C8	52.2 (4)
I1—Cu1—N2—N3	−49.9 (2)	C9—O1—C8—C7	61.5 (4)
I1^{i}—Cu1—N2—N3	87.8 (2)	N3—C7—C8—O1	−56.1 (4)
C6—N2—N3—C7	18.0 (4)	C8—O1—C9—C10	−61.5 (4)
Cu1—N2—N3—C7	−175.4 (2)	N2—N3—C10—C9	167.4 (3)
C6—N2—N3—C10	154.8 (3)	C7—N3—C10—C9	−53.2 (4)
Cu1—N2—N3—C10	−38.6 (4)	O1—C9—C10—N3	57.0 (5)

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

Fig. 1