Refinement

Refinement on F
$R = 0.031$
$wR = 0.038$
$S = 0.99$
2442 reflections
226 parameters
H atoms: see below
$w = 1/[\sigma^2(F^2) + (0.020F)^2 + 0.075]$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.343$ e Å^{-3}
$\Delta\rho_{min} = -0.240$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Zn···Cu	3.0757 (6)	Cu—N2	1.971 (3)
Zn—Cl1	2.208 (1)	O1—C1	1.339 (5)
Zn—Cl2	2.208 (1)	O2—C17	1.333 (5)
Zn—O1	2.004 (3)	N1—C7	1.286 (5)
Zn—O2	2.010 (3)	N1—C8	1.477 (5)
Cu—O1	1.942 (3)	N2—C10	1.497 (5)
Cu—O2	1.938 (3)	N2—C11	1.282 (6)
Cu—N1	1.960 (3)		
Cl1—Zn—Cl2	116.46 (5)	O1—Cu—N1	91.2 (1)
Cl1—Zn—O1	108.6 (1)	O1—Cu—N2	169.9 (1)
Cl1—Zn—O2	112.6 (1)	O2—Cu—N1	168.3 (1)
Cl2—Zn—O1	119.54 (9)	O2—Cu—N2	92.2 (1)
Cl2—Zn—O2	117.82 (9)	N1—Cu—N2	98.7 (1)
O1—Zn—O2	75.0 (1)	Zn—O1—Cu	102.4 (1)
O1—Cu—O2	78.1 (1)	Zn—O2—Cu	102.3 (1)

Ring H atoms were placed geometrically 0.95 Å from their parent atoms, while the other H atoms were taken from a difference map. For all H atoms, a riding model was used with $U_{iso}(H) = 1.3U_{eq}(C)$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1993). Cell refinement: *CAD-4 EXPRESS*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *MolEN*. Program(s) used to refine structure: *MolEN*. Molecular graphics: *ORTEP-3* (Farrugia, 1997). Software used to prepare material for publication: *MolEN*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1278). Services for accessing these data are described at the back of the journal.

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{[μ-Bis(salicylidene)-1,3-propane-diaminato](3-methylpyridine)copper(II)}-diiodozinc(II)†

Filiz Ercan,[a] Cengiz Arıcı,[a] Abdülkadir Akay,[b] Orhan Atakol[b] and Dinçer Ülkü[a]

[a]*Department of Physics Engineering, Hacettepe University, Beytepe 06532, Ankara, Turkey, and* [b]*Department of Chemistry, Ankara University, Tandogan 06100, Ankara, Turkey. E-mail: dulku@eti.cc.hun.edu.tr*

Abstract

The title complex, $[CuZnI_2(C_{17}H_{16}N_2O_2)(C_6H_7N)]$, consists of a doubly oxygen-bridged heteronuclear dimeric complex. The Cu atom has a distorted square-pyramidal environment involving two O atoms and two N atoms of the bis(salicylidene)-1,3-propanediaminate $(SALPD^{2-})$ ligand, and an N atom of the 3-methylpyridine group. The Cu—O distances in the coordination plane are 1.973 (3) and 1.974 (2) Å, and the average Cu—N distance is 2.088 (4) Å. In the bridging plane, the Cu—O—Zn, O—Zn—O, Zn—O—Cu and O—Cu—O angles are 102.5 (1), 76.7 (1), 102.7 (1) and 77.9 (1)°, respectively. The dihedral angle between the coordination plane around the Cu atom and the 3-methylpyridine group is 88.4 (1)°. The Cu···Zn distance is 3.1013 (7) Å.

Comment

Binuclear and trinuclear metal complexes based on Schiff base ligands are of interest because of the magnetic superexchange interactions between the bridged metal ions. These polynuclear complexes have been the subject of considerable interest in our laboratory,

† Alternative name: diiodo-2$\kappa^2 I$-(3-methylpyridine-1κN)-μ-{2,2′-[1,3-propanediylbis(nitrilomethylidyne)]diphenolato}-1$\kappa^4 O,N,N',O'$:2κ^2-O,O'-copper(II)zinc(II).

e.g. $[Cu_2(C_{11}H_{10}ClNO_2)_2]$ (Tahir *et al.*, 1996), $[Cu_2-(C_{14}H_{11}NO_2)_2]$ (Ülkü, Ercan, Atakol, Ercan & Gencer, 1997), $Ni_3\{(SALPD)(CH_3CO_2)[(CH_3)_2SO]\}_2$ (Ülkü, Ercan, Atakol & Dinçer, 1997), $[Cd\{Ni(SALPD)(CH_3-CO_2)[(CH_3)_2CHNO]\}_2]$ (Ülkü, Tahir *et al.*, 1997), $[Cu_2-(C_{12}H_{13}NO_2)_2]$ (Atakol *et al.*, 1997), $[Cu_2(C_{12}H_{13}-NO_2)_2]$ (Ülkü *et al.*, 1998), $[Ni_2\{Cu(SALPD)(NO_2)_2-[(CH_3)_2CHNO]\}_2]\cdot2[(CH_3)_2CHNO]$ (Tahir *et al.*, 1998), $[Mn\{Ni(SALPD)(CH_3CO_2)[(CH_3)_2CHNO]\}_2]$ (Ercan & Atakol, 1998), $[Cd\{Cu(SALPD)(CH_3CO_2)\}_2]\cdot C_4H_8O_2$ (Ercan *et al.*, 1998) and $[Cu_3\{(SALPD)(CH_3CO_2)\}_2]\cdot C_4H_8O_2$ (Atakol *et al.*, 1999) [SALPD is N,N'-bis-(salicylidene)-1,3-propanediaminate]. The structure and magnetic properties of the doubly oxygen-bridged homonuclear dimeric copper(II) complexes have been reported previously (Kato *et al.*, 1964; Barclay & Hoskins, 1965; Butcher & Sinn, 1976; Kato & Muto, 1988). We report here a new heteronuclear dimeric complex, (I). The magnetic superexchange interactions depend on the immediate environment of the bridge, as well as on the ligand arrangement about the metal atoms.

(I)
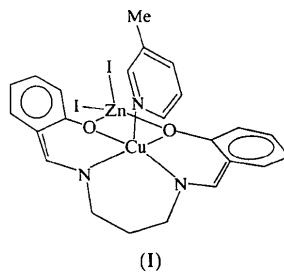
The coordination polyhedron around the copper ion in this hetero-dinuclear complex can be described as a square pyramid. The value of τ, which represents the relative amount of trigonality, is equal to 0 in a regular square pyramid; $\tau = (\beta - \alpha)/60°$, α and β being the two largest angles around the central atom (Addison & Rao, 1984; Uhlenbrock *et al.*, 1996). The value of τ is 0.036 for the Cu^{II} ion. The copper ion is coordinated in the four equatorial positions by the two N and two O atoms of the $SALPD^{2-}$ ligand. The axial position of this square-pyramidal polyhedron is occupied by an N atom of the 3-methylpyridine group. A minor distortion of the square pyramid is revealed by the bond angles between apical and equatorial donor atoms: N3—Cu—N1 99.3 (2), N3—Cu—N2 97.9 (2), N3—Cu—O1 96.9 (1) and N3—Cu—O2 93.0 (1)°. The average of these bond angles is 6.8° greater than the 90° angle in a regular square pyramid. The Cu—O1—Zn—O2 bridging plane is approximately planar. The Cu atom is located 0.2393 (6) Å from the best coordination plane, involving atoms N1, N2, O1 and O2. The bond angles within the bridging plane, Cu—O2—Zn and O1—Cu—O2, are 102.7 (1) and 77.9 (1)°,

respectively. The 3-methylpyridine group (N3 and C18–C22) and the coordination plane make a dihedral angle of 88.4 (1)°. The Cu—O1—Zn—O2 bridging plane and the coordination plane around the Cu atom make a dihedral angle of 11.4 (4)°. There are reports in the literature that this dihedral angle influences the magnetic properties of the system. The Cu$\cdots$Zn distance [3.1013 (7) Å] in the bridging plane is rather long for a direct interaction. The six-membered Cu—N1—C8—C9—C10—N2 chelate ring has a boat conformation. The distances of the two *para*-positioned boat atoms, Cu and C9B, from the N1/C8/C10/N2 plane are 0.702 (1) and 0.547 (1) Å, respectively. The C9 boat atom is disordered. For clarity, only one component of the disordered C9 atom is shown in Fig. 1. The atom with suffix A has an occupancy of 0.4 and that with suffix B has an occupancy of 0.6. A comparision of the dihedral angle (κ) between the bridging plane and the coordination plane around the Cu atom, along with the related distance ranges and bridging angle (φ), can be found in Table 2 for the five dimeric copper complexes recently studied in this laboratory. The bond lengths and angles within the ligands show no unusual values. The magnetic properties are currently under investigation.
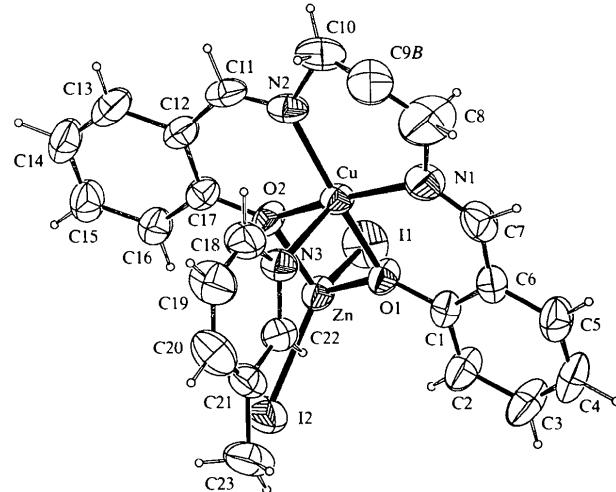
Fig. 1. *PLATON* (Spek, 1998) drawing of (I) with the atom-numbering scheme. For clarity, only one component of the disordered C9 atom is shown; the C atom of the other component carries suffix A instead of B. The displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small circles with arbitrary radii.

Experimental

To a solution of N,N'-bis(salicylidene)-1,3-propanediamine (0.282 g, 1 mmol) in hot ethanol (50 ml), ammonia solution (10 ml, 20%) was added and the mixture heated to boiling point. A solution of $CuCl_2\cdot2H_2O$ (0.170 g, 1 mmol) in hot methanol (20 ml) was then added and the resulting mixture set aside. After 3 h, the copper complex was filtered off and dried

at 353 K. This complex (0.343 g, 1 mmol) was dissolved in hot dioxane (50 ml) and 3-methylpyridine (0.7 ml) was added, followed by ZnI_2 (0.320 g, 1 mmol) in hot methanol (20 ml). The resulting mixture was set aside for 4 d and the green crystals which formed were filtered off and dried in air.

Crystal data

[CuZnI₂(C₁₇H₁₆N₂O₂)-
(C₆H₇N)]
$M_r = 756.18$
Triclinic
$P\bar{1}$
$a = 8.9034\,(12)$ Å
$b = 10.0168\,(11)$ Å
$c = 16.7090\,(12)$ Å
$\alpha = 73.579\,(2)°$
$\beta = 74.837\,(3)°$
$\gamma = 65.376\,(2)°$
$V = 1281.6\,(2)$ Å³
$Z = 2$
$D_x = 1.960$ Mg m⁻³
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10.45–18.03°$
$\mu = 4.20$ mm⁻¹
$T = 295$ K
Prism
$0.40 \times 0.30 \times 0.20$ mm
Green

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (Fair, 1990)
$T_{min} = 0.382$, $T_{max} = 0.432$
4888 measured reflections
4256 independent reflections

3672 reflections with $I > 3\sigma(I)$
$R_{int} = 0.011$
$\theta_{max} = 25.68°$
$h = -10 \rightarrow 0$
$k = -12 \rightarrow 10$
$l = -19 \rightarrow 19$
3 standard reflections frequency: 120 min intensity decay: -4.1%

Refinement

Refinement on F
$R = 0.032$
$wR = 0.039$
$S = 0.92$
3672 reflections
288 parameters
H-atom parameters constrained

$w = 1/[\sigma F^2 + (0.02F)^2 + 0.45]$
$(\Delta/\sigma)_{max} = 0.0006$
$\Delta\rho_{max} = 0.87$ e Å⁻³
$\Delta\rho_{min} = -0.15$ e Å⁻³
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Cu—Zn	3.1013 (7)	O1—C1	1.339 (5)
I1—Zn	2.5381 (8)	O2—C17	1.342 (6)
I2—Zn	2.5337 (6)	N1—C7	1.274 (7)
Zn—O1	2.005 (2)	N1—C8	1.483 (7)
Zn—O2	1.997 (3)	N2—C10	1.489 (6)
Cu—O1	1.973 (3)	N2—C11	1.274 (6)
Cu—O2	1.974 (2)	C8—C9A	1.35 (1)
Cu—N1	1.986 (4)	C9A—C9B	1.05 (2)
Cu—N2	1.999 (4)	C9A—C10	1.48 (2)
Cu—N3	2.279 (5)	C9B—C10	1.48 (1)
I1—Zn—I2	117.28 (2)	C7—N1—C8	116.7 (5)
I1—Zn—O1	111.9 (1)	Cu—N2—C10	121.7 (3)
I1—Zn—O2	110.9 (1)	Cu—N2—C11	123.6 (3)
I2—Zn—O1	114.4 (1)	C10—N2—C11	114.7 (4)
I2—Zn—O2	119.0 (1)	Cu—N3—C18	125.1 (3)
O1—Zn—O2	76.7 (1)	Cu—N3—C22	118.0 (4)
O1—Cu—O2	77.9 (1)	C18—N3—C22	116.9 (5)
O1—Cu—N1	90.3 (2)	O1—C1—C2	119.8 (3)

O1—Cu—N2	161.8 (2)	O1—C1—C6	121.0 (4)
O1—Cu—N3	96.9 (1)	N1—C7—C6	129.5 (4)
O2—Cu—N1	163.9 (2)	N1—C8—C9A	115.8 (7)
O2—Cu—N2	90.6 (1)	N1—C8—C9B	123.4 (7)
O2—Cu—N3	93.0 (1)	C8—C9A—C9B	67 (1)
N1—Cu—N2	97.8 (2)	C8—C9A—C10	122 (1)
N1—Cu—N3	99.3 (2)	C9B—C9A—C10	70 (1)
N2—Cu—N3	97.9 (2)	C8—C9B—C9A	68 (1)
Zn—O1—Cu	102.5 (1)	C8—C9B—C10	122 (1)
Zn—O1—C1	127.3 (2)	N2—C10—C9A	115.9 (6)
Cu—O1—C1	130.1 (2)	N2—C10—C9B	114.1 (5)
Zn—O2—Cu	102.7 (1)	N2—C11—C12	128.5 (4)
Zn—O2—C17	128.9 (2)	O2—C17—C12	120.4 (3)
Cu—O2—C17	128.2 (2)	O2—C17—C16	120.4 (4)
Cu—N1—C7	124.5 (3)	N3—C18—C19	122.9 (5)
Cu—N1—C8	118.8 (4)	N3—C22—C21	124.3 (5)

Table 2. *Structural data and bridging (φ) and dihedral (κ) angles (Å, °) for five homo- and hetero-dinuclear Cu complexes*

Complex	Cu—O bridge	Cu⋯M(Cu,Zn)	φ	κ
(I)	1.941 (5)–1.950 (4)	2.994 (2)	100.6 (2)	5 (1)
(II)	1.930 (1)–1.982 (9)	3.073 (2)	103.5 (4)	11.3 (8)
(III)	1.885 (3)–1.976 (3)	3.021 (2)	102.8 (1)	2.9 (9)–8.0 (3)
(IV)	1.938 (3)–1.955 (3)	3.047 (6)	103.4 (1)	12.7 (2)
(V)	1.973 (3)–1.974 (2)	3.1013 (7)	102.5 (1)–102.7 (1)	11.4 (4)

Notes: (I) is [Cu₂(C₁₁H₁₀ClNO₂)₂] (Tahir *et al.*, 1996); (II) is [Cu₂-(C₁₄H₁₁NO₂)₂] (Ülkü, Ercan, Atakol, Ercan & Gencer, 1997); (III) is [Cu₂(C₁₂H₁₃NO₂)₂] (Atakol *et al.*, 1997); (IV) is [Cu₂(C₁₂H₁₃-NO₂)₂] (Ülkü *et al.*, 1998); (V) is [Cu{Zn(C₂₃H₂₁I₂N₃O₂)}] (title compound).

Atoms C9A and C9B were refined isotropically with their H atoms ignored. H atoms were placed geometrically 0.95 Å from their parent C atoms and a riding model was used with $U_{iso}(H) = 1.3U_{eq}(C)$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *MolEN*. Program(s) used to refine structure: *MolEN*. Molecular graphics: *PLATON* (Spek, 1998). Software used to prepare material for publication: *MolEN*.

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Acta Cryst. (1999). C55, 928–930

{[μ-Bis(salicylidene)-1,3-propane-diaminato]bis(3,5-dimethylpyridine)-nickel(II)}diiodozinc(II)†

CENGIZ ARICI,[a] FILIZ ERCAN,[a] ORHAN ATAKOL,[b] ABDÜLKADIR AKAY[b] AND DINÇER ÜLKÜ[a]

[a]*Department of Engineering Physics, Hacettepe University, Beytepe 06532, Ankara, Turkey, and* [b]*Department of Chemistry, Faculty of Science, University of Ankara, Beşevler 06100, Ankara, Turkey. E-mail: dulku@eti.cc.hun.edu.tr*

Abstract

The structure of the title compound, $[NiZnI_2(C_{17}H_{16}N_2O_2)(C_7H_9N)_2]$, consists of doubly oxygen-bridged heteronuclear dimeric complexes. The compound is of interest because of the magnetic superexchange interactions that occur between doubly oxygen-bridged metal ions. The Ni atom has an irregular octahedral environment involving two O and two N atoms from the bis(salicylidene)-1,3-propanediaminate ligand in the equatorial plane and the N atoms from the two 3,5-dimethylpyridine groups in the apical positions. The coordination around the Zn atom is distorted tetrahedral,

with an average Zn—O distance of 2.001 (2) Å and an average Zn—I distance of 2.5499 (5) Å. In the bridged plane, the Ni—O1—Zn, O1—Zn—O2, Zn—O2—Ni and O2—Ni—O1 angles are 98.82 (9), 81.2 (1), 99.8 (1) and 79.6 (1)°, respectively. The Ni···Zn distance is 3.0753 (7) Å.

Comment

The syntheses and structures of oxygen-bridged binuclear and trinuclear linear homo- or heterometal complexes based on Schiff base ligands, such as $[Cu_2(C_{11}H_{10}ClNO_2)_2]$ (Tahir *et al.*, 1996), $[Cu_2(C_{14}H_{11}-NO_2)_2]$ (Ülkü, Ercan, Atakol, Ercan & Gencer, 1997), $[Cd\{(CH_3CO_2)(SALPD)Ni[(CH_3)_2CHNO]\}_2]$ (Ülkü, Tahir *et al.*, 1997), $[Ni_3(SALPD)_2(CH_3CO_2)_2\{(CH_3)_2SO\}_2]$ (Ülkü, Ercan, Atakol & Dinçer, 1997), $[Cu_2(C_{12}H_{13}-NO_2)_2]$ (Atakol *et al.*, 1997), $[Cu_2(C_{12}H_{13}NO_2)_2]$ (Ülkü *et al.*, 1998) and $[CuNi_2(NO_2)_2(SALPD)_2\{(CH_3)_2CH-NO\}_2]·2(CH_3)_2CHNO$ (Tahir *et al.*, 1998) [SALPD is N,N'-bis(salicylidene)-1,3-propanediaminate], have been the subject of considerable interest in this laboratory due to the magnetic superexchange interactions which occur between their bridged metal ions. Structural details of doubly oxygen-bridged homonuclear dimeric copper(II) complexes have been reported previously (Kato *et al.*, 1964; Barclay & Hoskins, 1965; Butcher & Sinn, 1976; Kato & Muto, 1988). We describe here the structure of a new heteronuclear dimeric complex, $[NiZnI_2(SALPD)-(C_7H_9N)_2]$, (I).

(I)
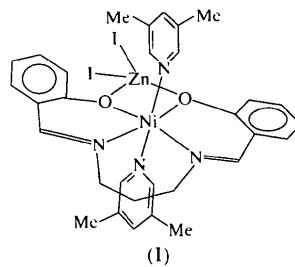
The magnetic properties of (I) are currently under investigation. Magnetic superexchange interactions depend on the immediate environment of the bridge, as well as on the ligand arrangement about the metal atoms. The coordination around the nickel ion in the title hetero-dinuclear complex is an irregular octahedron involving two bridging O and two N atoms from the SALPD²⁻ ligand in the equatorial plane. In the axial positions of this irregular octahedron lie atoms N3 and N4 from the two 3,5-dimethylpyridine groups. The bond angles around the central Ni ion are in the range 79.6 (1) (O1—Ni—O2) to 98.7 (1)° (N1—Ni—N2). The Ni atom is located −0.0087 (5) Å from the coordination plane (O1, O2, N1, N2). This coordination plane and the bridging plane around the Ni atom (Ni, O1, Zn, O2)