

Cl(4)—Cu(2)—O(2)	93.54 (15)	N(3)—C(32)—C(33)	120.6 (7)
Cl(4)—Cu(2)—O(4)	105.53 (12)	C(32)—C(33)—C(34)	117.1 (7)
O(1)—Cu(2)—O(2)	163.44 (19)	C(33)—C(34)—C(35)	122.7 (7)
O(1)—Cu(2)—O(4)	75.21 (16)	C(31)—C(35)—C(34)	118.2 (7)
O(2)—Cu(2)—O(4)	88.61 (17)	Cu(1)—O(4)—Cu(2)	104.18 (18)
Cu(1)—O(1)—Cu(2)	104.95 (19)	Cu(1)—O(4)—C(41)	123.4 (4)
Cu(2)—O(1)—C(11)	132.5 (4)	Cu(2)—O(4)—C(41)	132.2 (4)
O(1)—C(11)—N(1)	117.1 (6)	O(4)—C(41)—N(4)	117.4 (6)
O(1)—C(11)—C(15)	127.0 (6)	O(4)—C(41)—C(45)	126.2 (6)
N(1)—C(11)—C(15)	115.9 (6)	N(4)—C(41)—C(45)	116.3 (6)
C(11)—N(1)—C(12)	123.5 (6)	C(41)—N(4)—C(42)	123.9 (6)
N(1)—C(12)—C(13)	120.0 (7)	N(4)—C(42)—C(43)	120.0 (6)
C(12)—C(13)—C(14)	118.6 (7)	C(42)—C(43)—C(44)	119.6 (7)
C(13)—C(14)—C(15)	121.4 (7)	C(43)—C(44)—C(45)	120.1 (7)
		C(41)—C(45)—C(44)	120.1 (6)

Table 3. Torsion angles ($^{\circ}$) with standard deviations

Cl(1)—Cu(1)—O(1)—Cu(2)	84.20 (18)	Cu(2)—O(1)—C(11)—N(1)	160.9 (4)
Cl(1)—Cu(1)—O(1)—C(11)	-98.2 (6)	Cu(2)—O(1)—C(11)—C(15)	-20.1 (9)
Cl(2)—Cu(1)—O(1)—Cu(2)	-91.39 (17)	O(1)—C(11)—N(1)—C(12)	176.4 (6)
Cl(2)—Cu(1)—O(1)—C(11)	86.2 (6)	C(15)—C(11)—N(1)—C(12)	-2.7 (10)
O(3)—Cu(1)—O(1)—Cu(2)	173.97 (21)	O(1)—C(11)—C(15)—C(14)	-177.4 (7)
O(3)—Cu(1)—O(1)—C(11)	-8.4 (6)	N(1)—C(11)—C(15)—C(14)	1.6 (10)
O(4)—Cu(1)—O(1)—Cu(2)	-2.40 (19)	C(11)—N(1)—C(12)—C(13)	2.0 (11)
O(4)—Cu(1)—O(1)—C(11)	175.2 (6)	N(1)—C(12)—C(13)—C(14)	-0.1 (11)
Cl(1)—Cu(1)—O(3)—C(31)	-75.9 (5)	C(12)—C(13)—C(14)—C(15)	-0.9 (11)
Cl(2)—Cu(1)—O(3)—C(31)	67.0 (5)	C(13)—C(14)—C(15)—C(11)	0.0 (11)
O(1)—Cu(1)—O(3)—C(31)	171.0 (5)	Cu(2)—O(2)—C(21)—N(2)	-162.2 (5)
O(4)—Cu(1)—O(3)—C(31)	-176.0 (6)	Cu(2)—O(2)—C(21)—C(25)	18.0 (10)
Cl(1)—Cu(1)—O(4)—Cu(2)	-111.30 (14)	O(2)—C(21)—N(2)—C(22)	179.7 (6)
Cl(1)—Cu(1)—O(4)—C(41)	73.6 (5)	C(25)—C(21)—N(2)—C(22)	-0.5 (10)
Cl(2)—Cu(1)—O(4)—Cu(2)	105.76 (15)	O(2)—C(21)—C(25)—C(24)	179.7 (7)
Cl(2)—Cu(1)—O(4)—C(41)	-69.4 (5)	N(2)—C(21)—C(25)—C(24)	0.0 (10)
O(1)—Cu(1)—O(4)—Cu(2)	1.99 (15)	C(21)—N(2)—C(22)—C(23)	-0.4 (11)
O(1)—Cu(1)—O(4)—C(41)	-173.1 (5)	N(2)—C(22)—C(23)—C(24)	1.7 (11)
O(3)—Cu(1)—O(4)—Cu(2)	-11.5 (8)	C(22)—C(23)—C(24)—C(25)	-2.3 (11)
O(3)—Cu(1)—O(4)—C(41)	173.4 (6)	C(23)—C(24)—C(25)—C(21)	1.4 (11)
Cl(3)—Cu(2)—O(1)—Cu(1)	115.08 (15)	Cu(1)—O(3)—C(31)—N(3)	166.8 (4)
Cl(3)—Cu(2)—O(1)—C(11)	-62.8 (5)	Cu(1)—O(3)—C(31)—C(35)	-13.9 (9)
Cl(4)—Cu(2)—O(1)—Cu(1)	102.98 (15)	O(3)—C(31)—N(3)—C(32)	-179.4 (6)
Cl(4)—Cu(2)—O(1)—C(11)	79.1 (5)	C(35)—C(31)—N(3)—C(32)	1.2 (10)
O(2)—Cu(2)—O(1)—Cu(1)	14.5 (8)	O(3)—C(31)—C(35)—C(34)	-178.7 (7)
O(2)—Cu(2)—O(1)—C(11)	-163.4 (6)	N(3)—C(31)—C(35)—C(34)	0.6 (10)
O(4)—Cu(2)—O(1)—Cu(1)	2.00 (15)	C(31)—N(3)—C(32)—C(33)	-2.6 (11)
O(4)—Cu(2)—O(1)—C(11)	-175.9 (5)	N(3)—C(32)—C(33)—C(34)	2.0 (11)
Cl(3)—Cu(2)—O(2)—C(21)	65.5 (5)	C(32)—C(33)—C(34)—C(35)	-0.2 (12)
Cl(4)—Cu(2)—O(2)—C(21)	-76.0 (5)	C(33)—C(34)—C(35)—C(31)	-1.1 (11)
O(1)—Cu(2)—O(2)—C(21)	166.5 (6)	Cu(1)—O(4)—C(41)—N(4)	173.8 (4)
O(4)—Cu(2)—O(2)—C(21)	178.5 (5)	Cu(1)—O(4)—C(41)—C(45)	-5.6 (9)
Cl(3)—Cu(2)—O(4)—Cu(1)	-88.94 (17)	Cu(2)—O(4)—C(41)—N(4)	0.2 (9)
Cl(3)—Cu(2)—O(4)—C(41)	85.6 (5)	Cu(2)—O(4)—C(41)—C(45)	-179.2 (5)
Cl(4)—Cu(2)—O(4)—Cu(1)	87.87 (17)	O(4)—C(41)—N(4)—C(42)	-179.9 (6)
Cl(4)—Cu(2)—O(4)—C(41)	-97.6 (5)	C(45)—C(41)—N(4)—C(42)	-0.5 (10)
O(1)—Cu(2)—O(4)—Cu(1)	-2.37 (18)	O(4)—C(41)—C(45)—C(44)	179.2 (7)
O(1)—Cu(2)—O(4)—C(41)	172.1 (6)	N(4)—C(41)—C(45)—C(44)	-0.2 (10)
O(2)—Cu(2)—O(4)—Cu(1)	-178.84 (21)	C(41)—N(4)—C(42)—C(43)	0.4 (10)
O(2)—Cu(2)—O(4)—C(41)	-4.4 (4)	N(4)—C(42)—C(43)—C(44)	0.5 (11)
Cu(1)—O(1)—C(11)—N(1)	-16.4 (9)	C(42)—C(43)—C(44)—C(45)	-1.3 (11)
Cu(1)—O(1)—C(11)—C(15)	162.7 (5)	C(43)—C(44)—C(45)—C(41)	1.1 (11)

The figures were produced using *SHELXTL PC* (SAXI, 1990) and molecular geometry calculations performed using *CALC* (Gould & Taylor, 1985). Program used to solve structure by automatic direct methods: *SHELXS86* (Sheldrick, 1986). Program used to refine structure: *SHELXL76* (Sheldrick, 1976). Reflection data were collected using the learnt profile method (Clegg, 1981) and corrected for absorption by the ψ -scan method (North, Phillips & Mathews, 1968). Refinement was by full-matrix least squares with all non-H atoms being allowed isotropic motion.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55759 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1013]

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Structure of [μ -3,10-Dimethyl-4,9-diaza-3,9-dodecadiene-2,11-dione dioximato(1-)]-(isothiocyanato)copper(II)

TAHIR TAHIROV AND TIAN-HUEY LU

Department of Physics, National Tsing Hua University, Hsinchu, Taiwan 300

HUNG LUH AND CHUNG-SUN CHUNG

Department of Chemistry, National Tsing Hua University, Hsinchu, Taiwan 300

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Abstract

The Cu^{II} ion is five coordinated in a distorted square-pyramidal geometry with the four N atoms equatorial and an N-bound isothiocyanate axial. It deviates from the best plane formed by the four N atoms towards the isothiocyanate N atom. The axial Cu—N distance is longer than the equatorial Cu—N distances. Comparison of the present work with other similar complexes reveals some steric effects of the monodentate ligand $-\text{NCS}^-$ on the coordination geometry.

Comment

The crystal structure of bis[μ -3,10-dimethyl-4,9-diaza-3,9-dodecadiene-2,11-dione dioximato(1-)]- μ -O,N,N',N''-dicopper(II) diperchlorate, $[\text{Cu}(\text{C}_{12}\text{H}_{21}\text{N}_4\text{O}_2)]_2^{2+} \cdot 2\text{ClO}_4^-$ has been reported (Wang, Wang, Wang & Chung, 1990). In order to study the

effect of the monodentate ligand on the O...O distances and the structures of this type of complex, we have prepared the title complex and studied its structure.

The ligand, 3,10-dimethyl-4,9-diaza-3,9-dodecadiene-2,11-dione dioxime, was prepared as described in the literature (Wang, Wang, Wang & Chung, 1990). Sufficient NaOH was added to a mixture of an acidic aqueous solution of this ligand and $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ to make this solution neutral. Addition of excess NaSCN solution gave reddish-brown crystals which were recrystallized from a methanol solution.

The coordination geometry about the copper ion is distorted square-pyramidal with the four N atoms equatorial and an N-bound isothiocyanate axial. The equatorial N atoms undergo a slightly tetrahedral distortion. The maximum deviation of these N atoms from the least-squares plane formed by the four N atoms is 0.166 (6) Å. The deviation of the copper ion from this plane towards the isothiocyanate N atom is 0.298 (2) Å. The equatorial Cu—N distances are shorter than the axial Cu—N distance [2.147 (4) Å]. An oxime proton is lost from the ligand in forming the complex and the remaining oxime proton forms an intramolecular hydrogen bond [$\text{O} \cdots \text{O}$ 2.461 (6) Å, $\text{O}—\text{H} \cdots \text{O}$ 166.5 (3)°]. It is interesting to note that in the absence of NCS^- , the copper(II) complex of the previously reported tetradentate ligand is a binuclear complex, bis[μ -3,10-dimethyl-4,9-diaza-3,9-dodecadiene-2,11-dione dioximato(1-)- μ -O, N,N',N'',N''']-dicopper(II) diperchlorate, $[\text{Cu}(\text{C}_{12}\text{H}_{21}\text{N}_4\text{O}_2)]_2^{2+} \cdot 2\text{ClO}_4^-$ (Wang, Wang, Wang & Chung, 1990). As in the title complex, the copper(II) coordination of this complex is distorted square pyramidal. The four Cu—N distances and the four N=C distances of this binuclear complex are slightly shorter than those of the title complex; however, the O...O and C—C distances in the five-membered rings of the binuclear complex are longer than those of the title complex. These results indicate that the monodentate ligand — NCS^- has a significant effect on the structure of this type of complex.

The ambidentate ligand NCS^- is N-bound and the N—Cu bond is slightly distorted from the normal to the N basal plane as evidenced by the N(5)—Cu—N(1), N(5)—Cu—N(2), N(5)—Cu—N(3) and N(5)—Cu—N(4) angles. The axial Cu—N distance is much longer than the Cu—N distances of the four-coordinated *trans*-[Cu(pyridine) $_2$ (NCS) $_2$] (Soldanova, Kabesova & Gazo, 1983).

The seven-membered ring is disordered at C(7) and C(7'). The fragment CuN(3)C(8)C(9)C(10)C(11)-C(12)N(4)O(2) is planar to ± 0.120 (8) Å and the fragment O(1)N(1)C(1)C(2)C(3)C(4)N(2)C(5) to ± 0.103 (9) Å. The deviation of the Cu atom from this plane is 0.266 (5) Å. The seven-membered ring is

in a skew form, the deviations of atoms C(6), C(7), C(7') and C(8) from the best plane formed by N(3)CuN(2)C(5) [planar to within 0.009 (9) Å] being 0.35 (1), -1.07 (1), -0.23 (2) and -0.70 (1) Å, respectively.

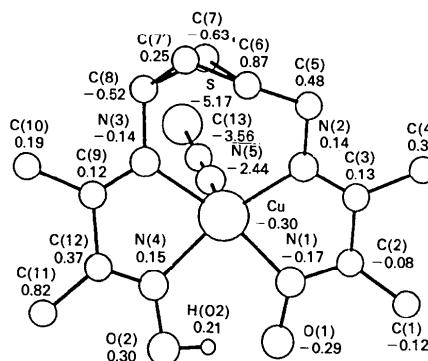


Fig. 1. A perspective view of the molecular structure with numbering scheme, showing displacements from the best plane formed by the four equatorial N atoms.

Experimental

Crystal data

[Cu(C₁₂H₂₁N₄O₂)(NCS)]
M_r = 374.95
 Orthorhombic
*Pn*2₁*a*
a = 7.5322 (7) Å
b = 12.652 (2) Å
c = 17.985 (4) Å
V = 1713.9 (5) Å³
Z = 4
D_x = 1.453 Mg m⁻³

Mo *K*α radiation
 λ = 0.7093 Å
 Cell parameters from 25 reflections
 θ = 9.75–12.45°
 μ = 1.41 mm⁻¹
T = 298 (3) K
 Square bipyramid
 0.56 × 0.47 × 0.44 mm
 Black

Data collection

Nonius CAD-4 (graphite monochromator) diffractometer
 $\theta/2\theta$ scans
 Absorption correction: empirical, based on ψ scan (North, Phillips & Mathews, 1968)
 T_{\min} = 0.6343, T_{\max} = 0.6391
 5140 measured reflections

2610 independent reflections
 1928 observed reflections [$I \geq 2.5\sigma(I)$]
 R_{int} = 0.015
 θ_{\max} = 29.9°
 h = 0 → 10
 k = 0 → 17
 l = 0 → 25
 3 standard reflections
 frequency: 60 min
 intensity variation: $\pm 2.0\%$

Refinement

Refinement on *F*
 Final *R* = 0.034
 wR = 0.036
 S = 1.20
 1928 reflections
 H-atom parameters not refined
 w = 1
 $(\Delta/\sigma)_{\max}$ = 0.151

$\Delta\rho_{\max}$ = 0.41 e Å⁻³
 $\Delta\rho_{\min}$ = -0.35 e Å⁻³
 Extinction coefficient: 0.42(2) (length in mm)
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2)

B_{eq} is the mean of the principal axes of the thermal ellipsoids.

	x	y	z	B_{eq}
Cu	0.54953 (7)	0.37260	0.11812 (3)	3.028 (24)
S	0.8669 (3)	0.41871 (16)	-0.11865 (10)	5.81 (8)
O(1)	0.7430 (7)	0.5309 (4)	0.2031 (3)	4.67 (19)
O(2)	0.8382 (5)	0.3486 (3)	0.22944 (20)	4.28 (17)
N(1)	0.5925 (6)	0.5080 (4)	0.16713 (24)	3.54 (17)
N(2)	0.3109 (6)	0.4444 (4)	0.10058 (23)	3.42 (17)
N(3)	0.5157 (5)	0.2181 (4)	0.08695 (21)	3.28 (17)
N(4)	0.7326 (5)	0.2989 (4)	0.18083 (22)	2.95 (15)
N(5)	0.6925 (6)	0.4036 (5)	0.01715 (25)	4.69 (23)
C(1)	0.4981 (10)	0.6918 (5)	0.1847 (4)	5.1 (3)
C(2)	0.4736 (8)	0.5811 (4)	0.1575 (3)	3.68 (22)
C(3)	0.3128 (8)	0.5422 (5)	0.1208 (3)	3.89 (23)
C(4)	0.1583 (10)	0.6158 (6)	0.1099 (4)	5.7 (3)
C(5)	0.1453 (9)	0.4019 (6)	0.0712 (4)	5.4 (3)
C(6)	0.1403 (8)	0.2854 (6)	0.0715 (5)	5.4 (3)
C(7)*	0.2754 (15)	0.2579 (9)	-0.0005 (6)	3.0 (4)
C(7')*	0.2202 (21)	0.2009 (18)	0.0324 (12)	8.2 (11)
C(8)	0.4020 (9)	0.1834 (7)	0.0244 (3)	5.6 (3)
C(9)	0.6233 (6)	0.1533 (4)	0.1191 (3)	3.10 (19)
C(10)	0.6347 (9)	0.0373 (5)	0.1024 (4)	5.2 (3)
C(11)	0.8598 (8)	0.1320 (5)	0.2242 (3)	4.29 (24)
C(12)	0.7410 (7)	0.1982 (4)	0.1767 (3)	2.82 (19)
C(13)	0.7645 (7)	0.4094 (5)	-0.0385 (3)	3.61 (20)

*Occupancy factor assumed to be 0.5.

Table 2. Selected distances (\AA) and angles ($^\circ$)

Cu—N(1)	1.954 (5)	N(4)—C(12)	1.279 (7)
Cu—N(2)	2.039 (4)	N(5)—C(13)	1.141 (7)
Cu—N(4)	2.011 (4)	C(1)—C(2)	1.496 (9)
Cu—N(3)	2.049 (5)	C(2)—C(3)	1.464 (9)
Cu—N(5)	2.147 (4)	C(3)—C(4)	1.503 (9)
S—C(13)	1.639 (6)	C(5)—C(6)	1.474 (11)
O(1)—N(1)	1.338 (6)	C(6)—C(7)	1.684 (13)
O(2)—N(4)	1.339 (5)	C(7)—C(8)	1.413 (13)
N(1)—C(2)	1.298 (7)	C(6)—C(7')	1.415 (20)
N(2)—C(3)	1.289 (8)	C(7')—C(8)	1.394 (18)
N(2)—C(5)	1.457 (8)	C(9)—C(10)	1.501 (8)
N(3)—C(8)	1.481 (7)	C(9)—C(12)	1.476 (7)
N(3)—C(9)	1.290 (7)	C(11)—C(12)	1.494 (8)
N(1)—Cu—N(2)	79.91 (18)	Cu—N(5)—C(13)	172.8 (5)
N(1)—Cu—N(3)	168.65 (17)	N(1)—C(2)—C(1)	122.5 (5)
N(1)—Cu—N(4)	92.29 (18)	N(1)—C(2)—C(3)	113.1 (5)
N(1)—Cu—N(5)	97.94 (20)	C(1)—C(2)—C(3)	124.4 (5)
N(2)—Cu—N(3)	105.86 (17)	N(2)—C(3)—C(2)	117.3 (5)
N(2)—Cu—N(4)	153.92 (16)	N(2)—C(3)—C(4)	123.3 (6)
N(2)—Cu—N(5)	103.29 (18)	C(2)—C(3)—C(4)	119.4 (6)
N(3)—Cu—N(4)	78.26 (17)	N(2)—C(5)—C(6)	112.9 (5)
N(3)—Cu—N(5)	90.31 (20)	C(5)—C(6)—C(7)	100.8 (6)
N(4)—Cu—N(5)	102.42 (18)	C(5)—C(6)—C(7')	138.0 (12)
Cu—N(1)—O(1)	123.3 (4)	C(6)—C(7)—C(8)	107.6 (7)
Cu—N(1)—C(2)	116.7 (4)	C(6)—C(7')—C(8)	126.1 (13)
O(1)—N(1)—C(2)	119.7 (5)	N(3)—C(8)—C(7)	115.7 (7)
Cu—N(2)—C(3)	112.0 (4)	N(3)—C(8)—C(7')	116.2 (8)
Cu—N(2)—C(5)	130.2 (4)	N(3)—C(9)—C(10)	124.6 (5)
C(3)—N(2)—C(5)	117.8 (5)	N(3)—C(9)—C(12)	116.6 (5)
Cu—N(3)—C(8)	124.2 (4)	C(10)—C(9)—C(12)	118.9 (5)
Cu—N(3)—C(9)	113.9 (3)	N(4)—C(12)—C(9)	113.3 (5)
C(8)—N(3)—C(9)	121.1 (5)	N(4)—C(12)—C(11)	123.7 (5)
Cu—N(4)—O(2)	123.8 (3)	C(9)—C(12)—C(11)	123.1 (5)
Cu—N(4)—C(12)	117.6 (3)	S—C(13)—N(5)	179.4 (5)
O(2)—N(4)—C(12)	118.5 (4)		

Method used to solve and refine structure: direct and Fourier methods, full-matrix least-squares refinement; H atoms by difference Fourier method and theoretical calculation. Program used: *NRCVAX* (Gabe, Le Page, White & Lee, 1987).

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Acta Cryst. (1993). **C49**, 803–805

{ N^2 -[2,2-Bis(diphenylphosphino)ethyl]- N^1, N^1 -dimethylethane-1,2-diamine}- $1\kappa^2 P, P': 2\kappa^2 N^1, N^2$ -tetracarbonyl- $1\kappa^4 C$ -dichloro- $2\kappa^2 Cl$ -molybdenumzinc(II), a Bimetal Complex with a Hetero-Bifunctional Ligand

ULRICH FLÖRKE AND HANS-JÜRGEN HAUPT

Anorganische und Analytische Chemie, Universität-GH Paderborn, Warburgerstrasse 100, D-4790 Paderborn, Germany

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Abstract

The two metal centres of the complex are Mo with an octahedral and Zn with a tetrahedral coordination sphere. Both metal atoms are members of chelate ring systems; the Mo atom belongs to a four-membered non-planar MoP_2C ring and the Zn atom is part of a five-membered diamine chelate ring.

Comment

In recent work we prepared new hetero-bifunctional ligands and their bimetal complexes (Haupt, Kleiberg & Flörke, 1993). With N^2 -[2,2-bis(diphenylphosphino)ethyl]- N^1, N^1 -dimethylethane-1,2-diamine (EPP-4C2N) as ligand, the compounds $[M(\text{CO})_4(\text{EPP-4C2N})M'\text{Cl}_2]$ with