O—C(2)—N(1)	124.0 (3)	O′—C(2′)—N(1′)	124.6 (3)
O—C(2)—N(2)	119.0 (3)	O′—C(2′)—N(2′)	120.4 (3)
N(1)—C(2)—N(2)	117.0 (3)	N(1′)—C(2′)—N(2′)	115.0 (3)
		C(2′)—O′···As	162.7 (2)

Symmetry code: (i) $x, \frac{3}{2} - y, \frac{1}{2} + z$.

H atoms were refined using a riding model or rigid methyl groups. Slow convergence of the methyl group at C(3′) may indicate rotational disorder. Owing to crystal loss, the preferred method of absorption correction (ψ scans) was not available and we were forced to use a ΔF method. This may lead to systematic errors in U values. The additional 18 parameters were taken into account in calculating e.s.d.'s. The matrix 001/201/010 generates a metrically orthorhombic C-centred cell with a = 9.055, b = 34.397 and c = 18.329 Å. However, a high $R_{\rm int}$ value of 0.42 is obtained when the transformed intensities are merged in *mmm*, indicating that the true symmetry is no higher than monoclinic.

Data collection: *DIF*4 (Stoe & Cie, 1992*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1992*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993*b*). Molecular graphics: *XP* (Siemens, 1994). Software used to prepare material for publication: *SHELXL*93.

The authors thank the Fonds der Chemischen Industrie for financial support and Mr A. Weinkauf for technical assistance.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1265). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 31–37.

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.

Pinchuk, V. A., Müller, C., Fischer, A., Thönnessen, H., Jones, P. G., Schmutzler, R., Markovskii, L. N., Shermolovich, Yu. G. & Pinchuk, A. M. (1995). *Z. Anorg. Allg. Chem.* **621**, 2001–2011.

Pinchuk, V. A., Neda, I., Müller, C., Fischer, A., Jones, P. G., Shermolovich, Yu. G. & Schmutzler, R. (1994). *Chem. Ber.* **127**, 1395–1397.

Roesky, H. W., Djarrah, H., Amirzadeh-Asl, D. & Sheldrick, W. S. (1981). *Chem. Ber.* **114**, 1554–1558.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1993*a*). *SHELXA. Empirical Absorption Correction Program*. University of Göttingen, Germany.

Sheldrick, G. M. (1993*b*). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Siemens (1994). *XP. Molecular Graphics Program*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Stoe & Cie (1992*a*). *DIF4. Diffractometer Control Program*. Version 7.09. Stoe & Cie, Darmstadt, Germany.

Stoe & Cie (1992*b*). *REDU4. Data Reduction Program*. Version 7.03. Stoe & Cie, Darmstadt, Germany.

Vogt, R., Jones, P. G. & Schmutzler, R. (1993). *Chem. Ber.* **126**, 1271–1281.

Acta Cryst. (1996). **C52**, 2752–2754

A μ-Oxamidato-Bridged Dinuclear Copper(II) Complex

ZHONG-NING CHEN,[a] QUI JUN,[a] WEN-XIA TANG,[a]* JIAN CHEN[b] AND PEI-JU ZHENG[b]

[a]*State Key Laboratory of Coordination Chemistry, Institute of Coordination Chemistry, Nanjing University, Nanjing 210093, People's Republic of China, and* [b]*Analysis Center, Fudan University, Shanghai 200433, People's Republic of China*

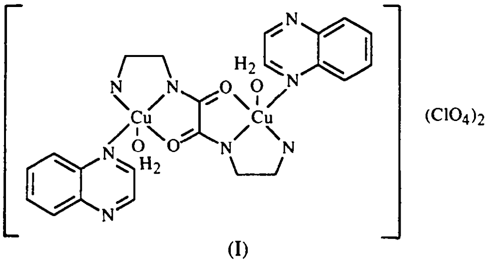
(*Received 14 April 1994; accepted 3 May 1996*)

Abstract

The oxen ligand [oxen = N,N'-bis(2-aminoethyl)oxamide] in the title compound, μ-[N,N'-bis(2-aminoethyl)oxamido(2−)-N,N',O:N'',N''',O']-bis[aqua(quinoxaline-N^1)copper(II)] diperchlorate, $[Cu_2(C_6H_{12}N_4O_2)(C_8H_6N_2)_2(H_2O)_2](ClO_4)_2$, is bis-tridentate in a *trans* conformation bridging two copper(II) ions to form a centrosymmetric binuclear molecule. The symmetry-equivalent central copper(II) ions are located in a distorted square-based pyramid, with a water molecule bonded at the axial site.

Comment

As extending bridging ligands transmitting a strong magnetic exchange interaction, oxamidates have played a key role in the design of polymetallic systems (Ojima & Nonoyama, 1988). One of the most important factors for the versatility of these ligands is their easy *cis*–*trans* conformational change affording symmetric or asymmetric oxamidato bridges. In contrast with symmetric oxamidato-bridged copper(II) complexes in the *cis* conformation (Journaux, Sletten & Kahn, 1985), few copper(II) complexes bridged by oxamidato groups displaying the *trans* conformation have been synthesized and characterized structurally by X-ray diffraction (Lloret, Julve, Real, Faus, Ruiz, Mollar, Castro & Bois, 1992). We report here the crystal structure of a dinuclear copper(II) complex with a bridging N,N'-bis(2-aminoethyl)oxamide (oxen) ligand in the *trans* conformation, (I).



(I)

The title compound, (I), forms centrosymmetric binuclear molecules, with oxen acting as a bis-tridentate ligand, in which the Cu^{II} ions are bridged by a *trans*-oxamidato group with an inversion center in the middle of the C5—$C5^{i}$ bond [Fig. 1; symmetry code: (i) $-x$, $-y$, $-z$]. The unique Cu^{II} ion is in a distorted square-pyramidal geometry with three atoms from the oxen ligand (N1, N4 and $O51^{i}$) and one atom from quinoxaline (N11) in the basal plane and the apical site occupied by an O atom (O1) of the water molecule [Cu—O1 2.139 (2) Å]. The largest deviation from the least-squares plane through N1, N4, $O51^{i}$ and N11 is 0.072 (2) Å out of this plane towards the apical O1 atom. The shorter bond distance for Cu—N4 [1.916 (2) Å] compared with Cu—$O51^{i}$ and Cu—N1 [2.098 (2) and 2.128 (2) Å, respectively] is similar to other oxamidato-bridged copper(II) complexes (Lloret, Julve, Faus, Journaux, Philoche-Levisalles & Jeannin, 1989). The significant shortening of the Cu—N4 bond length agrees with the strong basicity of the deprotonated amide N atom.

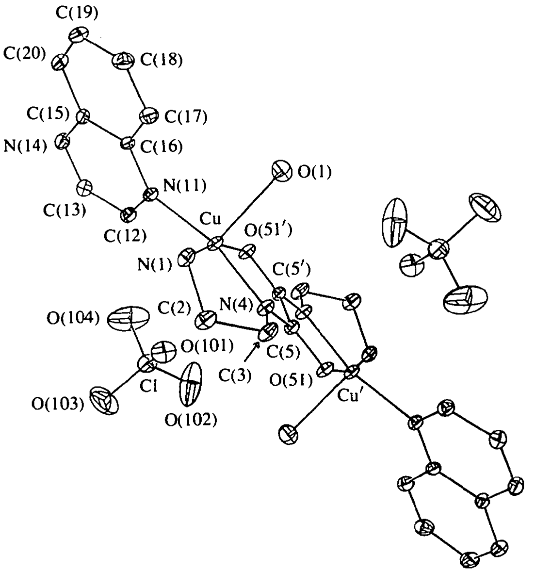


Fig. 1. The molecular structure of the title complex showing the atom-labelling scheme. Displacement ellipsoids are plotted at the 40% probability level.

The bis-tridentate character of the oxen ligand with a *trans* conformation produces two five-membered chelate metal cycles on each Cu^{II} ion. The three atoms (Cu, C3 and C5) around N4 lie in a plane, with bond angles of 116.9 (1), 114.9 (2) and 128.2 (1)° for Cu—N4—C3, Cu—N4—C5 and C3—N4—C5, respectively. This, together with the bond lengths and planarity of the oxamidato ligand, reveals that N4 and its symmetry-related $N4^{i}$ atom are sp^2 hybridized and that the π electrons of C5═O51 and $C5^{i}$═$O51^{i}$ are delocalized to form a conjugated system. The oxen and quinoxaline ligands, each showing no significant deviation from planarity, form a dihedral of 44.5° with respect to one another. The copper–copper separation through the oxen ligand is 5.335 (1) Å.

Experimental

The H_2oxen ligand and its mononuclear compound, Cu(oxen).-$2H_2O$, were obtained according to the literature method of Ojima & Yamada (1968). The dinuclear complex was prepared by adding an aqueous solution of copper perchlorate (1 mmol, 5 ml) to a heated aqueous solution of Cu(oxen).$2H_2O$ (1 mmol, 40 ml) with stirring. An aqueous solution of quinoxaline (2 mmol, 5 ml) was then added to the resulting blue solution. After standing at room temperature for several days, block-shaped green–blue single crystals were obtained by slow evaporation.

Crystal data

$[Cu_2(C_6H_{12}N_4O_2)(C_8H_6N_2)_2$-$(H_2O)_2](ClO_4)_2$
$M_r = 794.51$
Triclinic
$P\overline{1}$
$a = 6.936\ (2)$ Å
$b = 9.747\ (3)$ Å
$c = 12.057\ (1)$ Å
$\alpha = 101.81\ (1)°$
$\beta = 102.65\ (2)°$
$\gamma = 112.03\ (1)°$
$V = 699.2\ (3)$ Å^3
$Z = 1$
$D_x = 1.892$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10.2$–$13.4°$
$\mu = 1.795$ mm^{-1}
$T = 293\ (1)$ K
Block
0.3 × 0.2 × 0.1 mm
Blue–green

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (North, Phillips & Mathews, 1968)
$T_{min} = 0.78$, $T_{max} = 0.84$
2740 measured reflections
2468 independent reflections

2345 observed reflections $[I > 3\sigma(I)]$
$R_{int} = 0.011$
$\theta_{max} = 25°$
$h = -8 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = 0 \rightarrow 14$
3 standard reflections
frequency: 60 min
intensity decay: 0.4%

Refinement

Refinement on F
$R = 0.026$
$wR = 0.028$
$S = 0.66$
2345 reflections
251 parameters
All H-atom parameters refined
Unit weights applied

$(\Delta/\sigma)_{max} = 0.19$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = 0.12$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$B_{eq} = (1/3)\Sigma_i\Sigma_jB_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
Cu	−0.14962 (5)	0.10383 (3)	−0.17221 (2)	1.989 (6)
O1	0.1176 (4)	0.3298 (3)	−0.1253 (2)	4.76 (6)
N11	−0.2035 (3)	0.0290 (2)	−0.3485 (2)	2.09 (4)
N1	−0.3559 (3)	0.2187 (2)	−0.1643 (2)	2.48 (4)
C2	−0.4344 (4)	0.2054 (3)	−0.0605 (2)	3.07 (6)
C3	−0.2584 (4)	0.2092 (3)	0.0410 (2)	2.27 (5)
N4	−0.1453 (3)	0.1186 (2)	−0.0107 (2)	2.21 (4)
C5	−0.0501 (3)	0.0405 (3)	0.0402 (2)	1.94 (5)
O51	−0.0317 (3)	0.0236 (2)	0.1436 (1)	2.30 (3)
O103	0.2834 (6)	0.4552 (3)	0.1229 (3)	10.7 (1)
Cl	0.4084 (1)	0.41754 (7)	0.21281 (6)	3.40 (2)
O101	0.3973 (3)	0.2678 (2)	0.1740 (2)	4.34 (5)
O102	0.3451 (6)	0.4395 (4)	0.3150 (3)	10.3 (1)
O104	0.6141 (5)	0.5105 (4)	0.2425 (3)	8.9 (1)
N14	−0.2970 (3)	−0.1115 (2)	−0.5905 (2)	2.72 (5)
C12	−0.2476 (4)	−0.1144 (3)	−0.3890 (2)	2.39 (5)
C13	−0.2972 (4)	−0.1843 (3)	−0.5107 (2)	2.67 (6)
C15	−0.2475 (4)	0.0379 (3)	−0.5505 (2)	2.24 (5)
C16	−0.2038 (3)	0.1106 (3)	−0.4293 (2)	2.01 (5)
C17	−0.1623 (4)	0.2643 (3)	−0.3937 (2)	2.64 (6)
C18	−0.1655 (4)	0.3410 (3)	−0.4769 (2)	3.21 (6)
C19	−0.2023 (5)	0.2702 (3)	−0.5961 (2)	3.40 (6)
C20	−0.2423 (4)	0.1220 (3)	−0.6331 (2)	3.07 (6)

Table 2. *Selected geometric parameters (Å, °)*

Cu—O1	2.139 (2)	Cu—O51$^{\mathrm{i}}$	2.098 (2)
Cu—N4	1.916 (2)	Cu—N1	2.128 (2)
Cu—N11	2.001 (2)		
O1—Cu—N11	99.88 (8)	O1—Cu—N1	87.01 (1)
O1—Cu—N4	94.75 (9)	O1—Cu—O51$^{\mathrm{i}}$	98.88 (10)
N11—Cu—N1	101.88 (9)	N11—Cu—N4	164.89 (9)
N11—Cu—O51$^{\mathrm{i}}$	90.03 (8)	N1—Cu—N4	82.54 (9)
N1—Cu—O51$^{\mathrm{i}}$	165.68 (8)	N4—Cu—O51$^{\mathrm{i}}$	83.96 (8)

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

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Program(s) used to solve structure: *MULTAN*11/84 (Main, Germain & Woolfson, 1984). Program(s) used to refine structure: *SDP-Plus* (Frenz, 1985). Molecular graphics: *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SZ1007). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Frenz, B. A. (1985). *Enraf–Nonius SDP-Plus Structure Determination Package*. Version 3.0. Enraf–Nonius, Delft, The Netherlands.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Journaux, Y., Sletten, J. & Kahn, O. (1985). *Inorg. Chem.* **24**, 4063–4069.

Lloret, F., Julve, M., Faus, J., Journaux, Y., Philoche-Levisalles, M. & Jeannin, Y. (1989). *Inorg. Chem.* **28**, 3702–3706.

Lloret, F., Julve, M., Real, J. A., Faus, J., Ruiz, R., Mollar, M., Castro, I. & Bois, C. (1992). *Inorg. Chem.* **31**, 2956–2961.

Main, P., Germain, G. & Woolfson, M. M. (1984). *MULTAN*11/84. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Universities of York, England, and Louvain, Belgium.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Ojima, H. & Nonoyama, K. (1988). *Coord. Chem. Rev.* **82**, 85–111.

Ojima, H. & Yamada, K. (1968). *Nippon Kagaku Zasshi*, **89**, 490–494.

Acta Cryst. (1996). C**52**, 2754–2757

Triple-Decker $[Cp^*Fe(C_8H_6)Fe(C_8H_7)]$

ANGEL ALVAREZ-LARENA,[a] JOSÉ L. BRIANSÓ,[a] JUAN F. PINIELLA,[a] JOAN FARRAN,[a] JUAN M. MANRÍQUEZ,[b] IVONNE CHÁVEZ,[c] BEATRIZ OELCKERS,[d] ELIES MOLINS[e] AND CARLES MIRAVITLLES[e]

[a]*Unidad de Cristalografía, Universidad Autónoma de Barcelona, 08193 Bellaterra, Spain,* [b]*Centro de Investigación Minera y Metalúrgica, Av. Parque A. Rabat 6500, Santiago, Chile,* [c]*Departamento de Ciencias de los Materiales, Facultad de Química y Biología, Universidad de Santiago de Chile, Casilla 307, Santiago, Chile,* [d]*Instituto de Química, Universidad Católica de Valparaíso, Av. Brasil 2950, Valparaíso, Chile, and* [e]*Institut de Ciència de Materials (CSIC), Campus Universidad Autónoma de Barcelona, 08193 Bellaterra, Spain. E-mail: angel@santiuste.uab.es*

(*Received 23 February 1996; accepted 17 June 1996*)

Abstract

The title compound, [2(1,2,3,3a,6a-η)-1,4-dihydropentalenyl][μ-(1,2,3,3a,6a-η:3a,4,5,6,6a-η)pentalene]-[1(η^5)-pentamethylcyclopentadienyl]diiron, $[Fe_2(C_8H_7)(C_{10}H_{15})(C_8H_6)]$, has a metallocenic structure with the two pentalene ligands adopting an eclipsed conformation. In the central pentalene ligand, all the peripheral bonds are of similar length whereas the bridge bond is longer; there are two significantly different Fe—C distances, *i.e.* a mean value of 2.121 (3) Å for the Fe—C_{bridge} distances and a mean value of 2.028 (3) Å for the distances from the Fe atoms to all other C atoms. In the terminal pentalene ligand, the C—C and C—Fe distances in the ring bonded to the Fe atom are all similar.

Comment

The detailed physical and structural characterization of binuclear complexes of the type $[Cp^*–M–\text{pentalene}–M–Cp^*]^{n+}$ (M = Fe, Co, Ni; n = 0, 1, 2; Cp* = η^5-pentamethylcyclopentadienyl) has been reported recently (Manríquez *et al.*, 1995). In these complexes, the mixed-valence iron derivative was found to be fully delocalized as demonstrated by ^{57}Fe Mössbauer spectroscopy. The salt exhibited a single intermediate-

Acta Crystallographica Section C
ISSN 0108-2701 ©1996