$S = 1.085$
4180 reflections
132 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0395P)^2 + 0.7536P]$ where $P = (F_o^2 + 2F_c^2)/3$
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = −0.01 (4)

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

Pd1—N1	2.0872 (19)	O2—C1	1.304 (4)
Pd1—Cl1	2.2991 (6)	O3—C3	1.213 (4)
N1—C2	1.484 (3)	O4—C3	1.310 (3)
N1—C4	1.485 (3)	C1—C2	1.523 (4)
N1—C5	1.498 (3)	C3—C4	1.500 (4)
O1—C1	1.190 (4)		
N1—Pd1—Cl1	91.70 (6)	C2—N1—Pd1	105.60 (14)
C2—N1—C4	109.47 (19)	C4—N1—Pd1	112.74 (15)
C2—N1—C5	112.77 (19)	C5—N1—Pd1	105.91 (14)
C4—N1—C5	110.3 (2)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4C···O2W	0.82	1.79	2.602 (5)	170
O1W—H2W1···Cl1	1.03	2.23	3.133 (4)	146
O3W—H2O3···O1	0.86	2.15	2.858 (6)	139
O2—H2C···O1W^{i}	0.82	1.83	2.646 (6)	176
O2W—H2W2···O3W^{ii}	1.09	2.55	3.539 (8)	151
O2W—H1W2···O2W^{iii}	0.86	2.63	3.338 (7)	140

Symmetry codes: (i) $x-1, y, z$; (ii) $\frac{1}{2}-y, \frac{1}{2}+x, \frac{1}{4}+z$; (iii) $1-y, 1-x, \frac{1}{2}-z$.

H atoms on oxygen were located from difference maps, while those on carbon were placed in calculated positions, with C—H distances of 0.97 Å. $U_{\rm iso}$ values for the H atoms on C2 and C4 were assigned as $1.2U_{\rm eq}$ of the C atom, while those on C5 and the O atoms were assigned as $1.5U_{\rm eq}$ of the attached atom. H atoms were not refined.

Data collection: *SMART* (Siemens, 1996*a*). Cell refinement: *SAINT* (Siemens, 1996*b*). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

LZ thanks the National Natural Science Foundation of China (grant Nos. 2987017 and 29823001) and the Natural Science Foundation of Jiangsu Province (grant No. BK97015) for financial support. SSSR thanks Universiti Sains Malaysia for a Visiting Postdoctoral Research Fellowship, and HKF would like to thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 190-9609-2801.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1173). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 1222–1224

{4,4′-Dimethyl-6′-morpholiniomethyl-6-morpholinomethyl-2,2′-[1,2-ethanediylbis(iminomethylene)]diphenolato}copper(II) perchlorate dihydrate

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(*Received 25 January 1999; accepted 26 April 1999*)

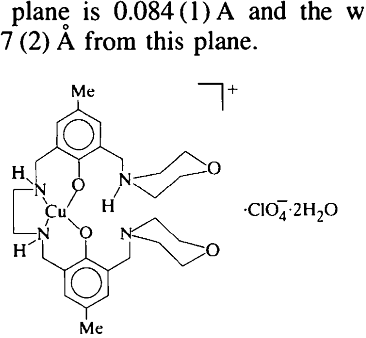
Abstract

In the title compound, $[Cu(C_{28}H_{41}N_4O_4)]ClO_4\cdot 2H_2O$, the copper ion is in a square-pyramidal environment, with the basal plane being defined by the two phenoxy O and the two amino N atoms. Both morpholino rings are in chair conformations. The molecules are packed in the crystal with a three-dimensional hydrogen-bonding network involving the water molecules and the perchlorate ion.

Comment

The study of metal complexes is focused very much towards the field of biomimicry where they can be used as model compounds for biological metallo compounds like proteins and enzymes (Suzuki *et al.*, 1984).

In the title complex, (I), the copper ion is coordinated by two phenoxy O and two amine N atoms in a square-pyramidal arrangement. The fifth coordination site is occupied by a water molecule [Cu*cdots*O2*W* 2.475 (3) Å]. The distance of the Cu atom from the coordination plane is 0.084 (1) Å and the water O2*W* atom is 2.507 (2) Å from this plane.



(I)

The two amine groups are in a '*gauche*' conformation, with an N2—C14—C15—N3 torsion angle of 55.6 (3)° (Nasipuri, 1991). The bond distances around the N atom of the protonated morpholino ring are different from those around the unprotonated morpholino N atom. The perchlorate ion has a tetrahedral geometry, with the average bond length and angle being 1.416 (3) Å and 109.5 (3)°, respectively.

Both morpholino rings are in a chair conformation. It is worth noticing that protonation at the N4 morpholine atom has only a small influence on the pyramidalization of nitrogen, the sum of the bond angles formed by the non-H atoms being 329.1° at N1 and 333.2° at N4. The same can be said for the values of the total puckering amplitudes (Cremer & Pople, 1975), which are 0.563 (4) Å for the N1-morpholine ring and 0.575 (3) Å for the N4-morpholine ring. The values of the dihedral angles formed by the best planes through the morpholine and the attached phenyl rings are 65.2 (1)° for the N1-morpholine ring and 81.0 (1)° for the N4-morpholine ring.

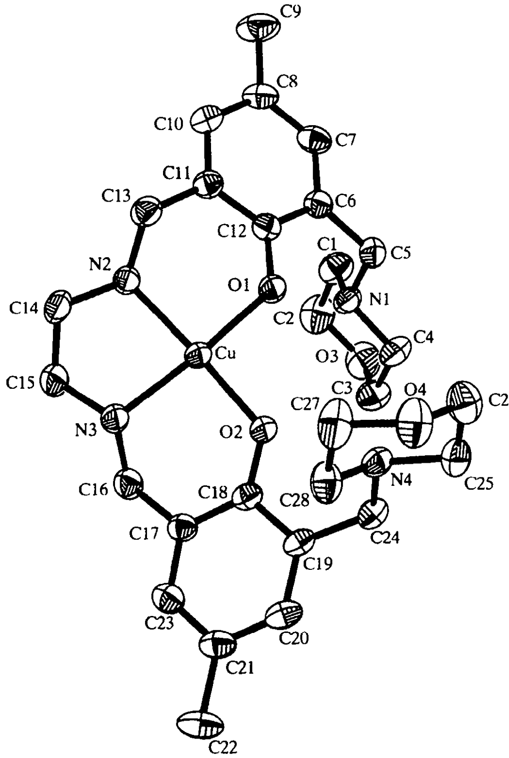


Fig. 1. *ORTEP* plot (Sheldrick, 1997) at 50% probability of the structure of (I) with the atom-numbering scheme. The perchlorate anion, the hydrate molecules and the H atoms have been omitted for clarity.

The water molecules and perchlorate ion stabilize the crystal packing. As can be seen from Table 2, one of the water molecules (O1*W*) forms three hydrogen bonds: two intramolecular (N—H···O and O—H···O) and one intermolecular. The other water molecule (O2*W*) is involved in one intramolecular (O—H···N) hydrogen bond with the complex and one intermolecular (O—H···O) hydrogen bond with the perchlorate ion.

Experimental

The ligand was prepared by the reduction of *N*,*N*′-bis[2-hydroxy-5-methyl-3-(morpholinomethyl)benzyl]ethylenediamine (0.5 g, 1 mmol) (Karunakaran & Kandaswamy, 1995) with sodium borohydride (0.15 g, 4 mmol) in ethanol. The ligand (0.5 g, 1 mmol) was dissolved in methanol and added to a methanolic solution of copper acetate monohydrate (0.2 g, 1 mmol). The resulting solution was refluxed on a water bath for 2 h and filtered while hot. Black crystals formed on cooling the solution to room temperature (273 K). The crystals were filtered and dried in a vacuum.

Crystal data

$[Cu(C_{28}H_{41}N_4O_4)]ClO_4 \cdot 2H_2O$
$M_r = 696.67$
Monoclinic
$P2_1$
$a = 10.546 (1)$ Å
$b = 14.229 (1)$ Å
$c = 10.807 (1)$ Å
$\beta = 99.75 (1)°$
$V = 1598.3 (2)$ Å^3
$Z = 2$
$D_x = 1.448$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 38 reflections
$\theta = 5.44–12.51°$
$\mu = 0.827$ mm^{-1}
$T = 293 (2)$ K
Rectangular block
0.78 × 0.60 × 0.38 mm
Dark green

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical *via* ψ scans (Siemens, 1994)
$T_{min} = 0.573$, $T_{max} = 0.768$
4742 measured reflections
3818 independent reflections (plus 275 Friedel-related reflections)

3821 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 27.5°$
$h = -1 \rightarrow 13$
$k = -1 \rightarrow 18$
$l = -14 \rightarrow 13$
3 standard reflections every 97 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.074$
$S = 1.017$
4093 reflections
428 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.211$ e Å^{-3}
$\Delta\rho_{min} = -0.207$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0020 (9)
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = −0.005 (9)

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

Cu—O1	1.918 (2)	Cl—O8	1.403 (3)
Cu—O2	1.924 (2)	Cl—O7	1.416 (3)
Cu—N3	2.010 (2)	Cl—O5	1.421 (4)
Cu—N2	2.023 (2)	Cl—O6	1.423 (5)
O1—Cu—O2	90.38 (8)	O1—Cu—N2	91.67 (9)
O1—Cu—N3	172.59 (10)	O2—Cu—N2	175.89 (10)
O2—Cu—N3	93.21 (8)	N3—Cu—N2	84.35 (9)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*W*1···O3	0.91 (3)	2.03 (4)	2.885 (4)	156 (4)
O2*W*—H2*W*2···N1	0.88 (3)	2.07 (3)	2.945 (4)	169 (3)
N3—H1N3···O7	0.87 (3)	2.42 (3)	3.203 (5)	150 (3)
N2—H1N2···O4^{i}	0.89 (2)	2.14 (2)	2.958 (3)	154 (3)
O2*W*—H1*W*2···O7^{ii}	0.90 (4)	2.05 (4)	2.927 (5)	165 (4)
N4—H1N4···O1*W*^{iii}	0.88 (3)	1.90 (3)	2.728 (3)	156 (3)
O1*W*—H2*W*1···O1^{iv}	0.87 (4)	1.84 (3)	2.652 (3)	156 (4)

Symmetry codes: (i) $x, y, z-1$; (ii) $1-x, \frac{1}{2}+y, -z$; (iii) $-x, y-\frac{1}{2}, -z$; (iv) $-x, \frac{1}{2}+y, -z$.

The water H atoms and those attached to N atoms were localized in difference maps, while all other H atoms were placed in calculated positions riding on their attached C atoms.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

The authors would like to thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 190-9609-2801. SSSR thanks the Universiti Sains Malaysia for a Visiting Postdoctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1413). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 1224–1226

An *exo*-polyhedral bicyclic 11-vertex *closo*-ruthenaborane: $[(PPh_3)(CH_3COS)_2\text{-}RuB_{10}H_8]$

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(*Received 14 January 1999; accepted 29 March 1999*)

Abstract

The title compound, 1,2:1,3-bis(μ-thioacetato-*S*:*O*)-1-(triphenylphosphine-*P*)-1-ruthena-*closo*-undecaborane, $[Ru(C_4H_{14}B_{10}O_2S_2)(C_{18}H_{15}P)]$, contains an 11-vertex *closo*-type C_{2v} $\{RuB_{10}\}$ cluster and two symmetric *exo*-polyhedral five-membered Ru—S—C—O—B rings. The Ru—S bond lengths are 2.443 (2) and 2.445 (2) Å, and the Ru—B bond lengths are in the range 2.036 (9)–2.393 (9) Å.

Comment

There has been considerable interest in the incidence of cyclic *exo*-polyhedral metal-to-cluster linkages in polyhedral metalloborane compounds (Kennedy, 1984, 1986; Barton & Srivastava, 1995). However, such ruthenaboranes have not been fully explored. Only six complexes representative of two types of rings, to our knowledge, are known to date. It has been reported that the reaction of $[RuCl_2(PPh_3)_3]$ and $[B_{10}H_{10}]^{2-}$ with MeCOOH affords two novel ruthenaboranes with one and two five-membered Ru—O—C(Me)—O—B rings (Fontaine *et al.*, 1987). When the same reaction was carried out in the presence of PhCOOH or *o*-$PhNHC_6H_4$-COOH instead of MeCOOH, three 11-vertex *closo*-type ruthenaborane compounds, all with one or two five-membered Ru—O—C(Ar)—O—B rings (where Ar is