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

Ag1—O1	2.314 (5)	Ag2—O1$^{\rm v}$	2.619 (5)
Ag1—O4$^{\rm i}$	2.349 (5)	Ag2···Ag2$^{\rm iv}$	3.2606 (11)
Ag1—O4$^{\rm ii}$	2.388 (5)	O1—C1	1.254 (8)
Ag1—C3$^{\rm iii}$	2.452 (7)	O2—C1	1.252 (8)
Ag1—C2$^{\rm iii}$	2.539 (6)	O3—C4	1.242 (8)
Ag1···Ag2	3.0032 (8)	O4—C4	1.256 (8)
Ag2—O3$^{\rm ii}$	2.185 (5)	C1—C2	1.508 (10)
Ag2—O2	2.230 (4)	C2—C3	1.345 (10)
Ag2—O2$^{\rm iv}$	2.585 (5)	C3—C4	1.498 (10)
O1—Ag1—O4$^{\rm i}$	108.1 (2)	O2$^{\rm iv}$—Ag2···Ag1	162.63 (11)
O1—Ag1—O4$^{\rm ii}$	108.6 (2)	O3$^{\rm ii}$—Ag2···Ag2$^{\rm iv}$	141.02 (14)
O4$^{\rm i}$—Ag1—O4$^{\rm ii}$	81.4 (2)	O2—Ag2···Ag2$^{\rm iv}$	52.16 (12)
O1—Ag1···Ag2	72.88 (13)	O2$^{\rm iv}$—Ag2···Ag2$^{\rm iv}$	42.94 (10)
O4$^{\rm i}$—Ag1···Ag2	149.55 (13)	Ag1···Ag2···Ag2$^{\rm iv}$	126.50 (3)
O4$^{\rm ii}$—Ag1···Ag2	70.00 (12)	Ag2—O2—Ag2$^{\rm iv}$	84.90 (15)
O3$^{\rm ii}$—Ag2—O2	162.0 (2)	Ag1$^{\rm vi}$—O4—Ag1$^{\rm vii}$	98.6 (2)
O3$^{\rm ii}$—Ag2—O2$^{\rm iv}$	99.2 (2)	C3—C2—C1	127.0 (6)
O2—Ag2—O2$^{\rm iv}$	95.10 (15)	C3—C2—Ag1$^{\rm viii}$	70.8 (4)
O3$^{\rm ii}$—Ag2···Ag1	86.94 (13)	C2—C3—C4	124.8 (6)
O2—Ag2···Ag1	76.31 (12)	C2—C3—Ag1$^{\rm viii}$	78.0 (4)

Symmetry codes: (i) $\frac{3}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $x - \frac{1}{2}, -\frac{1}{2} - y, \frac{1}{2} + z$; (iii) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (iv) $-x, -y, 1 - z$; (v) $x - 1, y, z$; (vi) $\frac{3}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (vii) $\frac{1}{2} + x, -\frac{1}{2} - y, z - \frac{1}{2}$; (viii) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$.

Data collection: *DIF*4 (Stoe & Cie, 1991). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1991). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *EMACS* (Free Software Foundation Inc., 1993).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1232). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.

Chen, X.-M. & Mak, T. C. W. (1991*a*). *Polyhedron*, **10**, 1723–1726.

Chen, X.-M. & Mak, T. C. W. (1991*b*). *J. Chem. Soc. Dalton Trans.* pp. 1219–1222.

Free Software Foundation Inc. (1993). *EMACS*. 675 Massachusetts Ave., Cambridge, MA 02139, USA.

Jansen, M. (1987). *Angew. Chem. Int. Ed. Engl.* **26**, 1098–1110.

Mak, T. C. W., Yip, W.-H., Kennard, C. H. L., Smith, G. & O'Reilly, E. J. (1986). *Aust. J. Chem.* **39**, 541–546.

Michaelides, A., Kiritsis, V., Skoulika, S. & Aubry, A. (1993). *Angew. Chem. Int. Ed. Engl.* **32**, 1495–1497.

Munakata, M., Kitagawa, S., Kosome, S. & Asahara K. (1986). *Inorg. Chem.* **25**, 2622–2627.

Sagatys, D. S., Smith, G., Bott, R. C., Lynch, D. E. & Kennard, C. H. L. (1993). *Polyhedron*, **12**, 709–713.

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

Sheldrick, G. M. (1991). *SHELXTL-Plus*. Revision 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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

Stoe & Cie (1991). *DIF4. Diffractometer Control Program*. Version 7.08. Stoe & Cie, Darmstadt, Germany.

Stoe & Cie (1991). *REDU4. Data Reduction Program*. Version 7.08. Stoe & Cie, Darmstadt, Germany.

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(Ammine)(aqua)[*N*-(2,6-dimethylphenyl)-*N*-(carboxymethyl)glycinato-*N*,*O*,*O*″]copper(II)

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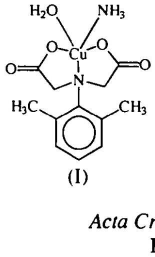
Abstract

The structure of the title compound [(ammine)(aqua)(2,6-dimethylphenyliminodiacetato-*N*,*O*,*O*″)copper(II)] consists of discrete neutral [$Cu(C_{12}H_{13}NO_4)(H_2O)(NH_3)$] molecules. The Cu atom is five-coordinate, with four short bonds formed with the N atom and two O atoms of the doubly deprotonated ligand and the N atom of the ammonia molecule. The water molecule is coordinated in an axial position at a longer distance. The coordination polyhedron can be described as a square-based pyramid with a small trigonal-bipyramidal distortion.

Comment

The crystal structure of the title compound, (I), consists of discrete neutral [Cu*L*(H_2O)(NH_3)] molecules, where H_2L is *N*-(2,6-dimethylphenyl)-*N*-(carboxymethyl)glycine.



(I)

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The Cu atom involves a five-coordinate CuN_2O_3 chromophore. Four short bonds of about 2.0 Å are formed with the N and two O atoms from the doubly deprotonated ligand L^{2-}, and the N atom of the ammonia molecule, while a water molecule is coordinated in an axial position at a longer distance [2.345 (7) Å]. Bond distances and angles are comparable with those reported for μ-pyrazine-bis[aqua(2,6-xylyliminodiacetato)copper(II)] (Suárez-Varela *et al.*, 1994), [*N*-(*o*-tolyl)iminodiacetato-*N*,*O*,*O*]copper(II) (Chen *et al.*, 1992), diaqua[*N*-(3-methoxyphenyl)iminodiacetato-*N*,*O*,*O*]copper(II) (Liu *et al.*, 1991) and diaqua[(*N*-*p*-methylphenyl)iminodiacetato-*O*,*O*,*N*]copper(II) (Wang, Tian, Miao, Liu & Chen, 1992).

The angles of the coordination polyhedron are not dissimilar to those of a square-based pyramidal geometry. Distortion can be measured by using the index $\tau = (\beta-\alpha)/60$, where β is the largest angle and α the second (Addison, Rao, Reedijk, Rijin & Verschoor, 1984). This index is unity for perfect trigonal-bipyramidal geometry and zero for square-pyramidal geometry. The calculated value for $[CuL(H_2O)(NH_3)]$ is $(172.4-169.9)/60 = 0.042$, indicating that the coordination polyhedron could be well described as a square-based pyramid with a small trigonal-bipyramidal distortion. In this square-pyramidal geometry, the basal plane is defined by O(2), N(1), O(3) and N(2) and the apical position is occupied by O(1*W*) from the water molecule, in accordance with the relatively large Cu—O(1*W*) distance. Deviations from planarity up to 0.068 (6) Å are observed for the equatorial donor atoms. As usual, the Cu atom is displaced by 0.071 (1) Å from the basal plane towards the O(1*W*) axial donor.

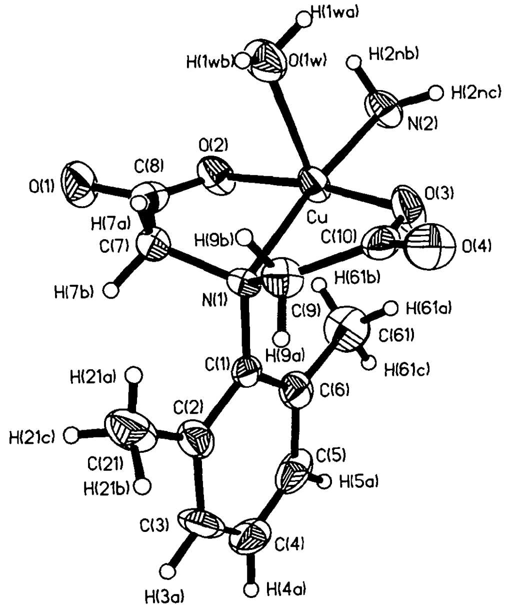

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the title complex. Ellipsoids are drawn at the 50% probability level.

The tridentate behaviour of the ligand results in the formation of two five-membered rings which have in common the Cu—N(1) bond. The dihedral angle between the mean least-squares planes of these two rings is 21.0 (2)°. The benzene ring is essentially planar, as expected. The dihedral angle between the benzene plane and the mean equatorial plane around the Cu atom is 87.5 (3)°.

Short O···O contacts (<3.0 Å) involving water molecules and O atoms of the carboxylato groups suggest intermolecular hydrogen bonding: $O(1W)\cdots O(3^{i}) = 2.958(8)$, $O(1W)\cdots O(1^{ii}) = 2.836(9)$ Å [symmetry codes: (i) $-x, -y, -z$; (ii) $\frac{1}{2}-x, -\frac{1}{2}+y, \frac{1}{2}-z$].

Experimental

0.5 g of the complex $[CuL(H_2O)_3]$ (Salinas, Guiraúm & Avila-Rosón, 1982) were added over concentrated ammonia (50 ml) and the resulting solution boiled for 5 min and then filtered. From the filtrate, blue crystals of $[CuL(H_2O)(NH_3)]$ were obtained. The crystal density D_m was measured by flotation.

Crystal data

$[Cu(C_{12}H_{13}NO_4)(H_2O)(NH_3)]$
$M_r = 333.8$
Monoclinic
$P2_1/n$
$a = 12.384$ (2) Å
$b = 7.684$ (2) Å
$c = 15.739$ (3) Å
$\beta = 111.65$ (3)°
$V = 1392.0$ (5) Å^3
$Z = 4$
$D_x = 1.593$ Mg m^{-3}
$D_m = 1.596$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 12.5$–17.5°
$\mu = 1.589$ mm^{-1}
$T = 294$ K
Short prism
0.23 × 0.23 × 0.19 mm
Deep blue

Data collection

Stoe Siemens AED-2 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{\min} = 0.645$, $T_{\max} = 0.731$
7394 measured reflections
6987 independent reflections

1380 observed reflections $[F > 6\sigma(F)]$
$R_{\rm int} = 0.0683$
$\theta_{\max} = 32.5$°
$h = 0 \rightarrow 20$
$k = 0 \rightarrow 12$
$l = -26 \rightarrow 24$
4 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F
$R = 0.0387$
$wR = 0.0459$
$S = 1.9165$
1380 reflections
205 parameters
Only H-atom U's refined
Unit weights applied
$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.35$ e Å^{-3}
$\Delta\rho_{\min} = -0.43$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 55, 99 & 149)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	U_{eq}
Cu	0.0243 (1)	0.1307 (1)	0.1693 (1)	0.031 (1)
N(1)	0.0361 (5)	−0.0698 (7)	0.2639 (3)	0.024 (2)
O(2)	0.1434 (5)	0.2435 (6)	0.2710 (3)	0.038 (2)
C(1)	−0.0331 (6)	−0.0641 (9)	0.3222 (4)	0.027 (2)
N(2)	−0.0050 (7)	0.3322 (8)	0.0832 (4)	0.037 (3)
O(1*W*)	0.1697 (5)	0.0325 (8)	0.1189 (4)	0.046 (3)
O(3)	−0.0926 (5)	−0.0155 (7)	0.0826 (3)	0.046 (2)
C(2)	−0.0204 (7)	−0.2024 (10)	0.3851 (5)	0.038 (3)
C(6)	−0.1111 (6)	0.0718 (10)	0.3166 (4)	0.035 (3)
C(5)	−0.1734 (7)	0.0689 (12)	0.3754 (5)	0.046 (3)
C(3)	−0.0900 (8)	−0.1951 (12)	0.4400 (5)	0.049 (4)
C(21)	0.0601 (8)	−0.3565 (12)	0.4005 (5)	0.053 (4)
C(4)	−0.1644 (7)	−0.0621 (13)	0.4357 (5)	0.050 (4)
C(61)	−0.1309 (8)	0.2243 (11)	0.2527 (6)	0.053 (4)
C(8)	0.2051 (6)	0.1342 (12)	0.3293 (4)	0.034 (2)
O(1)	0.2977 (5)	0.1689 (8)	0.3915 (3)	0.054 (2)
O(4)	−0.1225 (5)	−0.2963 (7)	0.0504 (3)	0.049 (2)
C(7)	0.1642 (6)	−0.0540 (9)	0.3209 (4)	0.031 (3)
C(10)	−0.0778 (7)	−0.1765 (11)	0.1040 (5)	0.036 (3)
C(9)	0.0029 (7)	−0.2242 (9)	0.2002 (4)	0.032 (3)

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

Cu—N(1)	2.109 (5)	O(3)—C(10)	1.278 (10)
Cu—O(2)	1.936 (4)	C(2)—C(3)	1.428 (14)
Cu—N(2)	2.000 (6)	C(2)—C(21)	1.509 (12)
Cu—O(1*W*)	2.345 (7)	C(6)—C(5)	1.405 (13)
Cu—O(3)	1.940 (5)	C(6)—C(61)	1.505 (11)
N(1)—C(1)	1.470 (11)	C(5)—C(4)	1.360 (13)
N(1)—C(7)	1.512 (8)	C(3)—C(4)	1.362 (14)
N(1)—C(9)	1.509 (9)	C(8)—O(1)	1.232 (7)
O(2)—C(8)	1.270 (8)	C(8)—C(7)	1.521 (11)
C(1)—C(2)	1.421 (10)	O(4)—C(10)	1.233 (9)
C(1)—C(6)	1.403 (11)	C(10)—C(9)	1.520 (9)
N(1)—Cu—O(2)	84.9 (2)	C(2)—C(1)—C(6)	119.8 (8)
N(1)—Cu—N(2)	172.4 (3)	Cu—O(3)—C(10)	112.2 (4)
O(2)—Cu—N(2)	96.1 (2)	C(1)—C(2)—C(3)	117.1 (7)
N(1)—Cu—O(1*W*)	98.1 (2)	C(1)—C(2)—C(21)	126.5 (8)
O(2)—Cu—O(1*W*)	88.7 (2)	C(3)—C(2)—C(21)	116.4 (7)
N(2)—Cu—O(1*W*)	89.4 (3)	C(1)—C(6)—C(5)	118.6 (7)
N(1)—Cu—O(3)	85.0 (2)	C(1)—C(6)—C(61)	124.1 (8)
O(2)—Cu—O(3)	169.9 (2)	C(5)—C(6)—C(61)	117.3 (8)
N(2)—Cu—O(3)	94.0 (2)	C(6)—C(5)—C(4)	123.2 (8)
O(1*W*)—Cu—O(3)	92.4 (2)	C(2)—C(3)—C(4)	123.2 (8)
Cu—N(1)—C(1)	120.9 (4)	C(5)—C(4)—C(3)	118.0 (9)
Cu—N(1)—C(7)	98.3 (4)	O(2)—C(8)—O(1)	124.5 (8)
C(1)—N(1)—C(7)	110.6 (5)	O(2)—C(8)—C(7)	118.3 (5)
Cu—N(1)—C(9)	100.1 (4)	O(1)—C(8)—C(7)	117.2 (6)
C(1)—N(1)—C(9)	111.1 (6)	N(1)—C(7)—C(8)	111.7 (5)
C(7)—N(1)—C(9)	115.2 (6)	O(3)—C(10)—O(4)	123.9 (6)
Cu—O(2)—C(8)	111.9 (5)	O(3)—C(10)—C(9)	118.3 (6)
N(1)—C(1)—C(2)	118.2 (6)	O(4)—C(10)—C(9)	117.8 (7)
N(1)—C(1)—C(6)	122.0 (6)	N(1)—C(9)—C(10)	113.0 (6)

Data collection: *DIF*4 (Stoe & Cie, 1988*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1987). Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*; *ORTEP* (Johnson, 1965).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HR1039). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Addison, A. W., Rao, T. N., Reedijk, J., Rijn, J. & Verschoor, G. C. (1984). *J. Chem. Soc. Dalton Trans.* pp. 1349–1356.

Chen, Y.-T., Liu, H.-Q., Liu, J.-J., Bu, X.-H., Wang, J.-L., Zhang, L.-J. & Miao, F.-M. (1992). *J. Coord. Chem.* **25**, 43–51.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Liu, X.-L., Zhang, L.-J., Miao, F.-M., Liu, H.-Q., Rong, Y.-M-, Jin, C.-Y. & Chen, Y.-T. (1991). *Chin. Chem. Lett.* **2**, 463–464.

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

Salinas, F., Guiraúm, A. & Avila-Rosón, J. C. (1982). *An Quím.* **78B**, 232.

Sheldrick, G. M. (1987). *SHELXTL-Plus*. Release 3.4 for Nicolet *R*3*m*/*V* crystallographic system. Nicolet Instrument Corporation, Madison, Wisconsin, USA.

Stoe & Cie (1988*a*). *DIF*4. *Diffractometer Control Program*. Version 7.0. Stoe & Cie, Darmstadt, Germany.

Stoe & Cie (1988*b*). *REDU*4. *Data Reduction Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.

Suárez-Varela, J., Colacio, E., Romerosa, A., Avila-Rosón, J. C., Hidalgo, M. A. & Romero, J. (1994). *Inorg. Chim. Acta*, **217**, 39–44.

Wang, J.-L., Tian, G.-H., Miao, F.-M., Liu, H.-Q. & Chen, R.-T. (1992). *Jiegou Huaxue* (*J. Struct. Chem.*), **11**, 384–387.

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Diaqua[*N*-(2-ethylphenyl)-*N*-(carboxymethyl)glycinato-*N*,*O*,*O*″]copper(II)

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Abstract

The (4+1) coordination environment around Cu^{II} in the title compound, [$Cu(C_{12}H_{13}NO_4)(H_2O)_2$] [diaqua-

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