$> |0{\cdot}991|$ e Å^{-3} was found. Calculations were performed on a PDP 11/34 computer with the *SDP* system (B. A. Frenz & Associates, Inc., 1982). Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B).

Fractional coordinates and equivalent isotropic temperature factors for non-H atoms are given in Table 1.* Bond distances and angles are given in Table 2. Fig. 1 shows the perspective drawing of the macrocycle molecule. Fig. 2 shows the packing drawing of the title compound.

Related literature. In recent years, there has been a growing interest in polycyclic polyamines (Whimp, Baily & Curtis, 1970; Hay, Fraser & Ferguson, 1987). Metal complexes of Cu^{2+}, Ni^{2+}, Co^{2+}, Cr^{3+} with macrocyclic tetraaza ligands have been studied extensively (House, Hay & Ali, 1983). The crystal structure of the bridged tetraaza macrocyclic molecule 5,7,7,12,14,14-hexamethyl-1,4,8,11-tetraazatricyclo[9.3.1.1.^{4,8}]hexadecane has previously been determined (Alcock, More & Mok, 1980) as well as the structure of 1,4,8,11-tetraazatricyclo[9.3.1.1^{4,8}]-hexadecane (Gabe, Le Page & Prasad, 1982). The characteristic infinite chains of $[Ag_2(SCN)_4]_n$ are the same as in $Zn[Ag(SCN)_2]$ (Lu, Huang & Huang, 1982).

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, least-squares planes and C—H bond distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54125 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Alcock, N. W., Moore, P. & Mok, K. F. (1980). *J. Chem. Soc. Perkin Trans.* 2, pp. 1186–1190.

B. A. Frenz, & Associates, Inc. (1982). College Station, Texas, USA, and Enraf–Nonius, Delft, Holland.

Gabe, E. J., Le Page, Y. & Prasad, L. (1982). *Acta Cryst.* **B38**, 2752–2754.

Hay, R. W., Fraser, L. & Ferguson, G. (1987). *J. Chem. Soc. Chem. Commun.* **22**, 1715–1716.

House, D. A., Hay, R. W. & Ali, A. M. (1983). *Inorg. Chim. Acta*, **76**(1) 47–49.

Lu, S. F., Huang, M. Y. & Huang, J. L. (1982). *J. Struct. Chem. USSR*, **2**, 71–76.

Whimp, P. O., Baily, M. F. & Curtis, N. F. (1970). *J. Chem. Soc. A*, pp. 1956–1963.

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Structure of the Complex [*N*-(*p*-Chlorophenyl)iminodiacetato]copper(II)

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Abstract. $[Cu(C_{10}H_8ClNO_4)]$, $M_r = 305{\cdot}17$, orthorhombic, *Pbca*, $a = 8{\cdot}783\ (2)$, $b = 9{\cdot}304\ (3)$, $c = 26{\cdot}993\ (5)$ Å, $V = 2206\ (3)$ Å^3, $Z = 8$, $D_x = 1{\cdot}838$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 22{\cdot}28$ cm^{-1}, $F(000) = 1224$, room temperature, $R = 0{\cdot}037$, $wR = 0{\cdot}045$ for 634 observed reflections with $I > 3\sigma(I)$. Cu is coordinated by one amino N and three carboxyl O atoms in an approximately square-planar configuration. Two five-membered rings with an envelope conformation are formed. As the N and two carboxyl O atoms are from one ligand and the third O atom is from another, a chain structure is formed.

Experimental. Crystals of the title compound were obtained by the slow evaporation of an ethanol solution of *p*-chlorophenyliminodiacetic acid and $Cu(ClO_4)_2$. A green single crystal ($0{\cdot}15 \times 0{\cdot}14 \times 0{\cdot}14$ mm) was used for the data collection on an Enraf–Nonius CAD-4 four-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71073$ Å). Unit-cell dimensions were obtained from a least-squares fit of 25 reflections in the range $12{\cdot}8 < 2\theta < 24{\cdot}1°$. Intensities in the range $2 < 2\theta < 25°$ (h: 0→10, k: 0→11, l: 0→32) were measured using the $\omega/2\theta$ scan mode. Three standard reflections monitored after each group of 200 measurements showed no significant change in intensity. Maximum counting time for each reflection was 60 s. Systematic absences $0kl$ for $k = 2n + 1$, $h0l$ for $l = 2n + 1$ and $hk0$ for $h = 2n + 1$ indicated space group *Pbca*. 2263 unique reflections were collected. 634 with $I > 3\sigma(I)$

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Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

$B_{eq} = \frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^3B(3,3) + ac(\cos\beta)B(1,3)]$.

	x	*y*	*z*	B_{eq}(Å^2)
Cu(1)	0·4224 (2)	0·3510 (2)	0·32663 (6)	1·48 (2)
O(1)	0·6067 (9)	0·2932 (8)	0·2933 (3)	1·9 (2)
O(2)	0·8014 (8)	0·3813 (8)	0·2509 (3)	2·0 (2)
O(3)	0·246 (1)	0·4507 (8)	0·3531 (3)	1·4 (2)
O(4)	0·3753 (8)	0·156 (1)	0·3455 (3)	2·1 (2)
N(1)	0·5173 (9)	0·5501 (9)	0·3275 (4)	1·4 (2)
C(1)	0·686 (1)	0·397 (1)	0·2768 (4)	1·3 (2)
C(2)	0·622 (1)	0·544 (1)	0·2843 (5)	1·6 (3)
C(3)	0·241 (2)	0·579 (1)	0·3420 (4)	1·3 (2)
C(4)	0·385 (1)	0·645 (1)	0·3196 (4)	1·5 (2)
C(11)	0·590 (1)	0·567 (1)	0·3753 (4)	1·7 (2)
C(12)	0·516 (2)	0·620 (2)	0·4140 (5)	3·7 (4)
C(13)	0·583 (2)	0·629 (2)	0·4595 (5)	5·2 (4)
C(14)	0·725 (2)	0·582 (2)	0·4675 (5)	3·9 (4)
C(15)	0·814 (2)	0·535 (3)	0·4287 (6)	6·9 (6)
C(16)	0·739 (2)	0·523 (2)	0·3805 (5)	5·3 (4)
Cl(1)	0·8028 (6)	0·5849 (7)	0·5260 (2)	7·4 (2)

Table 2. *Bond lengths* (Å) *and bond angles* (°)

Cu(1)—O(1)	1·928 (4)	Cu(1)—O(3)	1·940 (4)
Cu(1)—O(4^i)	1·928 (5)	Cu(1)—N(1)	2·031 (5)
O(1)—C(1)	1·270 (7)	O(2)—C(1)	1·240 (8)
O(3)—C(3)	1·234 (7)	O(4)—C(3)	1·253 (8)
N(1)—C(2)	1·487 (8)	N(1)—C(4)	1·476 (7)
N(1)—C(11)	1·447 (8)	C(1)—C(2)	1·493 (9)
C(3)—C(4)	1·525 (9)	C(11)—C(12)	1·332 (10)
C(11)—C(16)	1·374 (11)	C(12)—C(13)	1·364 (11)
C(13)—C(14)	1·345 (13)	C(14)—C(15)	1·376 (13)
C(14)—Cl(1)	1·718 (8)	C(15)—C(16)	1·462 (11)
O(1)—Cu(1)—O(3)	167·0 (2)	O(1)—Cu(1)—O(4^i)	92·3 (2)
O(1)—Cu(1)—N(1)	85·2 (2)	O(3)—Cu(1)—O(4^i)	100·5 (2)
O(3)—Cu(1)—N(1)	83·5 (2)	O(4)—Cu(1)—N(1)	160·4 (2)
Cu(1)—O(1)—C(1)	114·3 (4)	Cu(1)—O(3)—C(3)	113·8 (4)
Cu(1)—O(4)—C(3)	133·8 (4)	Cu(1)—N(1)—C(2)	102·1 (4)
Cu(1)—N(1)—C(4)	102·7 (4)	Cu(1)—N(1)—C(11)	106·8 (4)
C(2)—N(1)—C(4)	113·4 (5)	C(2)—N(1)—C(11)	115·4 (4)
C(4)—N(1)—C(11)	114·5 (5)	O(1)—C(1)—O(2)	123·8 (5)
O(1)—C(1)—C(2)	116·3 (6)	O(2)—C(1)—C(2)	119·4 (6)
N(1)—C(2)—C(1)	111·9 (5)	O(3)—C(3)—O(4)	124·3 (6)
O(3)—C(3)—C(4)	117·0 (6)	O(4)—C(3)—C(4)	118·5 (5)
N(1)—C(4)—C(3)	110·8 (5)	N(1)—C(11)—C(12)	121·5 (7)
N(1)—C(11)—C(16)	118·7 (7)	C(12)—C(11)—C(16)	119·8 (7)
C(11)—C(12)—C(13)	121·2 (8)	C(12)—C(13)—C(14)	121·8 (9)
C(13)—C(14)—C(15)	120·5 (8)	C(13)—C(14)—Cl(1)	120·7 (7)
C(15)—C(14)—Cl(1)	118·7 (7)	C(14)—C(15)—C(16)	116·5 (9)
C(11)—C(16)—C(15)	119·9 (9)		

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

were corrected for Lorentz and polarization factors but not for absorption. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B). Calculations were performed on a PDP 11/34 computer using the *SDP* program (B. A. Frenz & Associates, Inc., 1982). The structure was solved by Patterson synthesis and subsequent ΔF maps. All H atoms except H(16) (which was placed in a calculated position) were located. The structure was refined (on *F*) by full-matrix least squares with anisotropic thermal parameters for non-H atoms and isotropic for H atoms. The final discrepancy indices were $R = 0{\cdot}037$ and $wR = 0{\cdot}045$ with unit weights. 179 parameters were refined. Maximum shift/e.s.d. in final cycle was 1·25 and no residual electron density was $> |0{\cdot}602|$ e Å^{-3}.

Fractional coordinates of all non-H atoms with their standard deviations are given in Table 1.* Selected bond lengths and angles are listed in Table 2. The atomic numbering scheme of the molecule is shown in Fig. 1.

Related literature. Iminodiacetic acid derivatives have a strong tendency to form stable metal chelates (Albertson & Oskarsson, 1968; Mootz & Wunderlich, 1980). Normally the ligand forms three bonds to a central atom (Okamoto, Yasui & Hidaka, 1987). In the present structure the iminodiacetic acid also acts as a bridge between Cu atoms, thus forming four bonds to Cu. Other related Cu^{II} structures contain the ligands *p*-methylphenyl iminodiacetate (Chen, Liu, Liu, Bu, Wang, Zhang & Miao, 1991), *n*-*p*-hydroxyliminodiacetate (Abodovskaya, Shkolnikova & Dyamlova, 1984) and iminodiacetate (a dihydrate is formed) (Podder, Dattagupta, Saha & Saenger, 1979).

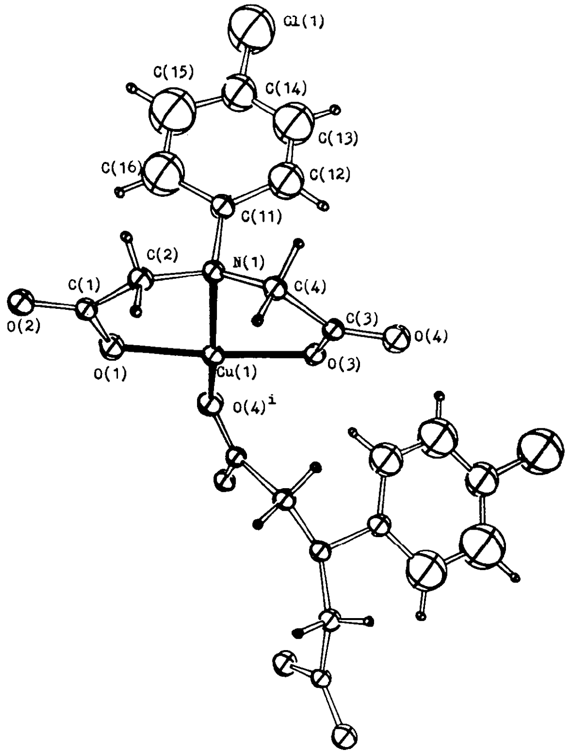

Fig. 1. Molecular structure and atom-numbering scheme. Symmetry code: (i) $\frac{1}{2} - x, \frac{1}{2} + y, z$.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, least-squares planes and C—H bond lengths have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54126 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

ABODOVSKAYA, A. E., SHKOLNIKOVA, L. M. & DYAMLOVA, N. N. (1984). *Koord. Khim.* **10**(2), 161–167.

ALBERTSON, N. J. & OSKARSSON, A. (1968). *Acta. Chem. Scand.* **22**, 1700–1703.

B. A. FRENZ, & ASSOCIATES, INC. (1982). *SDP Structure Determination Package.* College Station, Texas, USA, and Enraf–Nonius, Delft, Holland.

CHEN, Y.-T., LIU, H.-Q., LIU, J.-J., BU, X.-H., WANG, J.-I., ZHANG, L.-J. & MIAO, F. M. (1991). *J. Coord. Chem.* In the press.

MOOTZ, D. & WUNDERLICH, H. (1980). *Acta Cryst.* **B36**, 445–447, 721–724.

OKAMOTO, K., YASUI, T. & HIDAKA, J. (1987). *Chem. Lett.* pp. 551–554.

PODDER, A., DATTAGUPTA, J. K., SAHA, N. N. & SAENGER, W. (1979). *Acta Cryst.* **B35**. 53–56.

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Structure of a Zinc(II) Complex with a Non-Symmetrical Tetradentate Schiff Base

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Abstract. {*N*-[2-(2-Aminoethylamino)ethyl]salicylideneaminato-*O*,*N*,*N*′,*N*″}aquazinc(II) bromide, $[Zn(C_{11}H_{16}N_3O)(H_2O)]Br$, $M_r = 369{\cdot}57$, monoclinic, $P2_1/c$, $a = 9{\cdot}129\,(1)$, $b = 14{\cdot}207\,(1)$, $c = 11{\cdot}003\,(1)$ Å, $\beta = 102{\cdot}36\,(1)^\circ$, $V = 1393{\cdot}9\,(2)$ Å^3, $Z = 4$, $D_m = 1{\cdot}77\,(1)$, $D_x = 1{\cdot}76$ Mg m^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}71073$ Å, $\mu = 4{\cdot}6$ mm^{-1}, $F(000) = 744$, $T = 295$ K, $R = 0{\cdot}030$ for 1995 unique observed reflections. The crystal comprises a cationic Zn^{II} complex which includes the saden ligand (saden = {*N*-[2-(2-aminoethylamino)ethyl]salicylideneaminato-*O*,*N*,*N*′,*N*″}) and a water molecule. The Zn atom is surrounded by three N and two O atoms (one from H_2O) with a distorted trigonal bipyramidal arrangement. Intermolecular hydrogen bridges of the O(water)···O type join the molecules in dimers. Other than this, there is one Br···HO(water) and two Br···HN intermolecular distances, which correspond to hydrogen bonds much weaker than the O···O ones, in agreement with infrared spectra ($\nu_{\mathrm{Br\cdots DO}} = 2381$ cm^{-1}, $\nu_{\mathrm{Br\cdots DN}} = 2347$ and 2326 cm^{-1}).

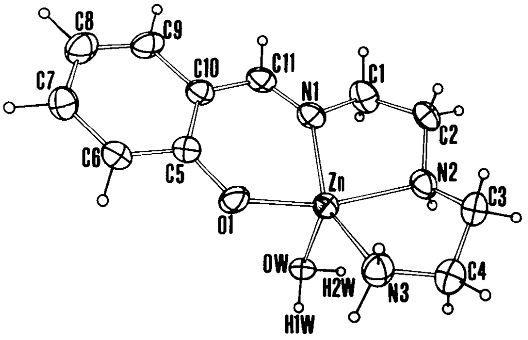

Fig. 1. View of the complex cation with atom numbering. Thermal ellipsoids are scaled to 50% probability.

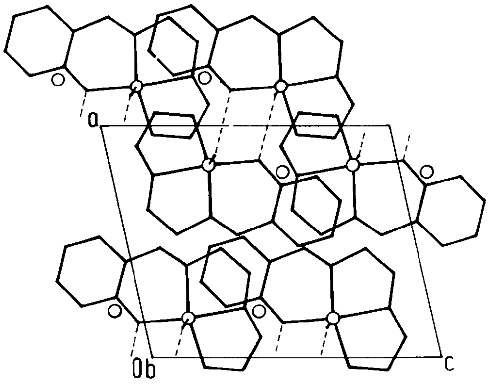

Fig. 2. Packing scheme. The intermolecular O···O hydrogen bonds are indicated by dashed lines.

Experimental. To an aqueous solution of $ZnBr_2$ (2·25 g, 0·01 ml) an equivalent amount of the Schiff base in methanol solution was slowly added. The resulting mixture was neutralized by 0·84 g (0·01 mol) $NaHCO_3$, filtered and allowed to stand in air. Small yellowish crystals were obtained over a few days. The yield was 1·3 g (35%). Density was determined by flotation in a methylene iodide/toluene mixture. Parameters describing the data

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