unlikely, since no Friedel pair differences were observed, and the parameter probably corresponds to a real vibration in a direction approximately bisecting the large Cl(1)—Hg—Cl(3) angle of 141°. The overall geometry and dimensions of the anion are similar to those found previously for $Hg_2X_6^{2-}$ species (Beurskens, Bosman & Cras, 1972; Brotherton, Epstein, White & Willis, 1974; von Brodersen, Pezzei & Thiele, 1983).

Distances between the $[Ph_3Te]_2[Hg_2Cl_6]$ units (Figs. 1 and 2) correspond to normal van der Waals interactions.

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Structure of [*N,N'*-Bis(β-carbamoylethyl)-2-hydroxytrimethylenediamine]copper(II) Perchlorate,* $[Cu(C_9H_{20}N_4O_3)](ClO_4)_2$†

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Abstract. $M_r = 494{\cdot}73$, monoclinic, $P2_1/c$, $Z = 4$, $a = 13{\cdot}997$ (7), $b = 10{\cdot}656$ (9), $c = 14{\cdot}254$ (12) Å, $V = 1783{\cdot}2$ (3) Å^3, $D_x = 1{\cdot}85$, $D_m = 1{\cdot}81$ (3) Mg m^{-3} (by flotation in CCl_4–hexane), Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 2{\cdot}98$ mm^{-1}, $F(000) = 1012$, $T = 296$ (4) K, $R(F) = 0{\cdot}076$ for 2195 observed reflections. The coordination geometry around the Cu atom is a tetragonally distorted octahedron comprising two O atoms from the two amide groups and two N atoms from the two amine groups in an equatorial plane, and the two O atoms of the perchlorate ions above and below this plane at distances of 2·448 (11) and 2·641 (11) Å. This complex has the *RS* configuration for the chiral amine N centres. The two perchlorate ions are situated on opposite sides of the equatorial plane; they form intra- and intermolecular hydrogen bonds with the multidentate ligand and neighbouring molecules.

Introduction. In a previous paper we have reported the crystal structure of a copper(II) complex with *N,N'*-bis(β-carbamoylethyl)ethylenediamine, $[Cu(NO_3)(bcen)(H_2O)]NO_3.H_2O$ (Lee, Lu, Liu, Chung & Lee, 1984). As an extension, the crystal and molecular structure of the title compound, $[Cu(bchtn)](ClO_4)_2$ (Chen, 1982), are described here.

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* (6-Hydroxy-4,8-diazaundecanediamide)copper(II) perchlorate.

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Table 1. *Experimental data and structure refinement parameters*

Crystal size (mm)	0·3 × 0·35 × 0·4
Diffractometer and data collection technique	ω–2θ scan, four-circle diffractometer (Syntex $P\bar{1}$), graphite monochromator
Scan width (°)	2·0 + 0·7 tanθ; starting 1° below $K\alpha_1$ and 1° above $K\alpha_2$
Number and θ range used for measuring lattice parameters	15 reflections with $12° < 2\theta < 25°$
Absorption correction applied (North, Phillips & Mathews, 1968)	Experimental absorption correction based on ψ scan
Transmission factors	0·304 to 0·409
Number of reflections measured	6095
Number of unique reflections	2195 $[I > 3\sigma(I)]$
Method used to solve the structure	Patterson and Fourier methods
Parameters refined (coordinates and anisotropic temperature factors of nonhydrogen atoms)	243
Quantity minimized	$\sum w(\Delta F)^2$; $w = 1/\sigma_F^2$
Extinction correction	Not applied
Range of *h*, *k*, *l*	0, 0, 16 to 16, 14, $\bar{16}$
Atomic scattering factors f' and f''	*International Tables for X-ray Crystallography* (1974)
Number of reflections per parameter	9
Maximum value of $(\sin\theta)/\lambda$ reached in intensity measurement (Å^{-1})	0·6691
R (R_w)	0·076 (0·078)
$R_{\rm int}$	0·039
S	1·75
Final residual electron densities (e Å^{-3}) for max. peak	0·37
Average, max. Δ/σ	0·2, 0·7
Programs used	Tsing Hua crystal package* and *ORTEP*

* The program package and user's guide can be obtained from Professor Tseng-Yuh Lee, Computer Center, National Tsing Hua University, Taiwan.

Table 2. *Atomic positional parameters and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$B_{\rm eq} = \frac{4}{3}\sum_i\sum_j \mathbf{a}_i.\mathbf{a}_j B_{ij}$, where $\mathbf{a}_i$'s are the length vectors in direct space.

	x	*y*	*z*	$B_{\rm eq}$(Å^2)
Cu	0·2144 (1)	−0·2594 (1)	−0·0079 (1)	2·8
Cl(1)	0·5130 (3)	−0·2811 (3)	0·0701 (3)	4·0
Cl(2)	−0·0792 (2)	−0·2606 (2)	−0·0850 (2)	3·2
O(11)	0·3935 (7)	−0·2820 (8)	0·0015 (7)	5·7
O(12)	0·5490 (11)	−0·3955 (14)	0·1382 (13)	10·9
O(13)	0·5636 (8)	−0·2827 (8)	0·0102 (8)	5·1
O(14)	0·5508 (12)	−0·1883 (17)	0·1452 (14)	12·5
O(21)	0·0142 (7)	−0·1801 (8)	−0·0547 (8)	8·1
O(22)	−0·1641 (9)	−0·2393 (10)	−0·1945 (7)	8·0
O(23)	−0·1235 (9)	−0·2263 (8)	−0·0194 (8)	6·6
O(24)	−0·0450 (10)	−0·3860 (8)	−0·0680 (11)	9·8
O(1)	0·2038 (6)	−0·0980 (5)	−0·0797 (5)	2·7
O(2)	0·1375 (7)	−0·3176 (6)	−0·1640 (5)	3·2
O(10)	0·2124 (8)	−0·3770 (7)	0·2293 (7)	5·7
N(1)	0·2631 (9)	0·0876 (8)	−0·0965 (8)	4·5
N(2)	0·2523 (8)	−0·1784 (7)	0·1348 (7)	3·5
N(3)	0·2291 (7)	−0·4335 (7)	0·0526 (6)	2·9
N(4)	0·0262 (8)	−0·4346 (8)	−0·3148 (7)	4·1
C(1)	0·2627 (10)	−0·0018 (8)	−0·0320 (10)	3·8
C(2)	0·3262 (10)	0·0140 (10)	0·0920 (9)	3·9
C(3)	0·2554 (11)	−0·0370 (9)	0·1352 (10)	4·2
C(4)	0·3511 (11)	−0·2304 (10)	0·2396 (9)	4·2
C(5)	0·3237 (12)	−0·3680 (10)	0·2489 (10)	4·3
C(6)	0·3249 (11)	−0·4559 (9)	0·1651 (9)	4·0
C(7)	0·2346 (11)	−0·5280 (9)	−0·0253 (9)	4·2
C(8)	0·1222 (10)	−0·5363 (8)	−0·1339 (9)	4·5
C(9)	0·0963 (10)	−0·4237 (8)	−0·2050 (9)	2·8

Experimental. Preparation of samples: bchtn (3·13 g) was added to an aqueous solution of copper(II) perchlorate (5·01 g) and heated until it completely dissolved; the solution was filtered immediately while hot. After evaporation of the filtrate, blue crystals of $[Cu(bchtn)](ClO_4)_2$ were formed. Single crystals were obtained by recrystallization in a mixture of methanol and ethanol, after slow evaporation of the solvent at room temperature. The crystals obtained are deep blue and rod-like.

Experimental data and structure-solution parameters together with the standard refinement procedures are listed in Table 1. The H atoms of the amine, amide and OH groups were located on difference Fourier maps. Positions of other H atoms were calculated theoretically (tetrahedral geometry assumed). H-atom positions were not refined. The *B* values of the H atoms were initially assigned as 5·0 Å^2 and refined in the last cycle of least-squares calculation.

Discussion. The atomic coordinates and temperature factors are listed in Table 2.* A perspective view of the molecule with the numbering scheme and deviations of atoms from the least-squares plane through Cu—O(1)—N(2)—N(3)—O(2) are shown in Fig. 1.

The four donor atoms of bchtn and Cu$^{\rm II}$ lie approximately in a plane. N(3) and O(1) are above this plane, while N(2) and O(2) are below, so that the square-planar coordination geometry of Cu$^{\rm II}$ undergoes a slight tetrahedral distortion with a dihedral angle of 14·3 (5)° between planes O(1)—Cu—N(2) and O(2)—Cu—N(3). Two perchlorate O atoms are coordinated axially. Like $[Cu(NO_3)(bcen)(H_2O)]NO_3.H_2O$ (Lee, Lu, Liu, Chung & Lee, 1984), the axial Cu$^{\rm II}$—O bond lengths of the title compound are dramatically longer than the equatorial Cu$^{\rm II}$—O and Cu$^{\rm II}$—N bond distances, so that the coordination geometry of the Cu$^{\rm II}$ undergoes a significant tetragonal distortion, and is of the '4+2' type (Pavcovic & Brown, 1982), as would be predicted on the basis of the Jahn–Teller effect.

The amine H atoms H(13) and H(15) form intramolecular hydrogen bonds with O(21) and O(24) of perchlorate ion (2), respectively. On the other hand, the amide H atoms H(17), H(18), H(19) and H(20) form intermolecular hydrogen bonds with neighbouring perchlorate ions. These intra- and intermolecular hydrogen bonds contribute significant stabilizing force in holding the molecule in a stable crystalline state (Fig. 2).

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* List of structure factors, anisotropic thermal parameters, H-atom parameters, and bond lengths and bond angles for the perchlorate ions have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39534 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Bond distances* (Å) *in the coordination sphere*

The superscripts (i) and (ii) indicate the distances between Cu to O atoms, O(11) and O(21), of the two perchlorate ions; superscripts ′ and ″ indicate the distances between Cu to O atoms of water and NO_3^- respectively.

	$[Cu(bchtn)](ClO_4)_2$	$[Cu(NO_3)(bcen)(H_2O)]NO_3.H_2O$*
Cu–O(1)	1·966 (7)	1·957 (5)
Cu–O(2)	1·970 (7)	1·974 (6)
Cu–N(2)	1·998 (10)	1·983 (7)
Cu–N(3)	2·008 (7)	2·002 (6)
Cu–O [short axis]	2·448 (11)^{i} [Cu–O(11)]	2·399 (9)′ [Cu–O*W*(1)]
Cu–O [long axis]	2·641 (11)^{ii} [Cu–O(21)]	2·584 (9)″ [Cu–O(11)]
Bite angles (°) around Cu^{II} atom		
O(1)–Cu–N(2)	93·1 (3)	95·3 (2)
N(2)–Cu–N(3)	93·1 (4)	95·5 (3)
N(3)–Cu–O(2)	93·4 (3)	95·0 (2)
O(1)–Cu–O(2)	81·2 (3)	86·0 (2)

* Lee, Lu, Liu, Chung & Lee (1984).

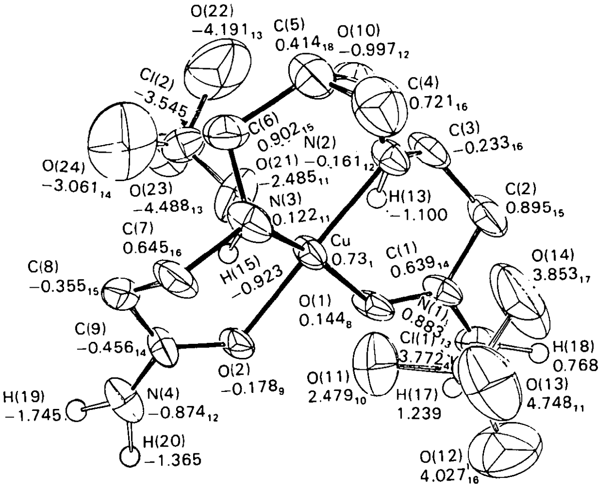

Fig. 1. Perspective view of the complex, showing deviations (in Å) from the major coordination plane (*ORTEP*, Johnson, 1976). E.s.d.'s of the deviation are indicated with subscripts for nonhydrogen atoms; those for H atoms are estimated to be about 0·05 Å. Equation of least-squares plane is $13 \cdot 584x - 0 \cdot 797y - 4 \cdot 791z = 3 \cdot 084$.

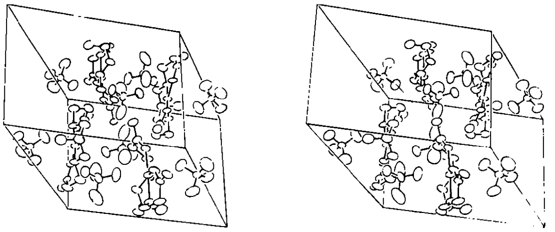

Fig. 2. Stereoscopic drawing of the crystal packing in the unit cell, showing clearly how the perchlorate ions are situated between the ligands and link them together. Origin: lower left corner; *a* axis to the right, *b* axis up, *c* axis towards the reader.

Table 4. *Bond distances* (Å) *and bond angles* (°)

(*a*) Covalent bond lengths			
C(1)=O(1)	1·257 (11)	C(7)–N(3)	1·532 (16)
C(9)=O(2)	1·259 (10)	C(1)–C(2)	1·494 (17)
C(1)–N(1)	1·327 (17)	C(2)–C(3)	1·523 (24)
C(9)–N(4)	1·324 (13)	C(4)–C(5)	1·540 (16)
C(3)–N(2)	1·506 (13)	C(5)–C(6)	1·525 (20)
C(4)–N(2)	1·484 (12)	C(7)–C(8)	1·492 (13)
C(6)–N(3)	1·444 (11)	C(8)–C(9)	1·482 (14)

(*b*) Bond angles around the macrocyclic ring			
Cu–O(1)–C(1)	126·2 (6)	C(6)–N(3)–Cu	116·5 (6)
O(1)–C(1)–C(2)	121·6 (11)	Cu–N(3)–C(7)	109·3 (7)
C(1)–C(2)–C(3)	109·9 (10)	N(3)–C(7)–C(8)	110·5 (10)
C(2)–C(3)–N(2)	112·3 (11)	C(7)–C(8)–C(9)	112·9 (9)
N(2)–C(4)–C(5)	107·6 (8)	C(8)–C(9)–O(4)	122·0 (9)
C(4)–C(5)–C(6)	114·1 (13)	C(9)–O(2)–Cu	129·6 (7)
C(5)–C(6)–N(3)	111·7 (10)		

The title complex has the *RS* configuration for the chiral N centres, which is different from that of $[Cu(NO_3)(bcen)(H_2O)]NO_3.H_2O$ which has the *RR* or the *SS* configuration. In addition, the bite angles around Cu^{II} of the title complex are significantly different from those of $[Cu(NO_3)(bcen)(H_2O)]NO_3.H_2O$, as shown in Table 3. These different bite angles are mainly attributed to the sizes of the different middle chelate rings of these two complexes. $[Cu(bchtn)](ClO_4)_2$ contains a middle six-membered chelate ring, while $[Cu(NO_3)(bcen)(H_2O)]NO_3.H_2O$ contains a middle five-membered chelate ring. On the other hand, the coordination bonds Cu–O and Cu–N in the equatorial plane are consistent between these two complexes within standard deviations. Other bond distances and angles are given in Table 4.

In the trimethylenediamine chelate ring, the distance between Cu and the O atom of the hydroxyl group is longer than 3·5 Å; hence there is no coordination bond between them. The OH group is immediately over C(5), with bond angle 'Cu–C(5)–O(10)' 90·2°.

Both perchlorate ions have distorted tetrahedral geometry and vibrate violently as usual. This can be seen from the higher temperature factors of the O atoms and the fact that the bond lengths between Cl and O atoms in these ions are not equal. However, the average Cl–O bond length and the average O–Cl–O bond angle are close to the ideal values for tetrahedral geometry.

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