

Table 3. Comparison of Co—O(OH<sub>2</sub>) and Co—N(py) distances (Å) in cobaloximes H<sub>2</sub>OCo(DH)<sub>2</sub>X and pyCo(DH)<sub>2</sub>X [ $\alpha$  bendings (°) in pyridine complexes are also reported]

X	Co—O	X	Co—N	$\alpha^*$
py	1.916 (3)	H <sub>2</sub> O	1.926 (3)	8.1
Br	1.96 (1)	py	1.946	—
NO <sub>2</sub>	1.980 (5)	N <sub>3</sub>	1.973 (1)	2.9
CN	1.992 (4)	O <sub>2</sub> CMe <sub>2</sub> Ph	1.994 (3)	—
Me	2.058 (3)	Me	2.068 (3)	3.2

All data are from Bresciani-Pahor, Forcolin, Marzilli, Randaccio, Summers & Toscano (1985) except those of the present work.

\* Values of  $\alpha$  indicate that the bending of the equatorial ligand is towards the group X.

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## The Aqua and the Perchlorato Copper(II) Complexes of *N,N'*-Bis( $\beta$ -carbamoylethyl)-*N*-methylethylenediamine\*

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**Abstract.** Aqua(4-methyl-4,7-diazadecanediamide)-copper(II) (4-methyl-4,7-diazadecanediamide)perchloratocopper(II) triperchlorate monohydrate, [Cu(C<sub>9</sub>H<sub>20</sub>N<sub>4</sub>O<sub>2</sub>)(H<sub>2</sub>O)][Cu(C<sub>9</sub>H<sub>20</sub>N<sub>4</sub>O<sub>2</sub>)(ClO<sub>4</sub>)](ClO<sub>4</sub>)<sub>3</sub>.H<sub>2</sub>O, *M<sub>r</sub>* = 993.5, triclinic, *P*1̄, *a* = 12.626 (3), *b* = 12.891 (2), *c* = 13.387 (3) Å,  $\alpha$  = 114.19 (2),  $\beta$  = 82.79 (2),  $\gamma$  = 107.07 (3)°, *V* = 1900 (1) Å<sup>3</sup>, *Z* = 2, *D<sub>m</sub>* = 1.731 (7), *D<sub>x</sub>* = 1.737 Mg m<sup>-3</sup>,  $\lambda$ (Mo *K* $\alpha$ ) = 0.7093 Å,  $\mu$  = 1.49 mm<sup>-1</sup>, *F*(000) = 1022.6, *T* = 297 (3) K, *R* = 0.076, *wR* = 0.068 for 4042 observed reflections. There are two independent complex cations: an aqua and a perchlorato complex. The coordination geometry about Cu<sup>II</sup> for both of these complexes is a distorted square pyramid with the diaminodiamide (*N*-Mebcen) equatorial and the O atom of H<sub>2</sub>O or ClO<sub>4</sub><sup>-</sup> axial. The two chiral amine N centres have the *RR* or *SS* configuration. In [Cu(ClO<sub>4</sub>)(*N*-Mebcen)]<sup>+</sup>, the conformation of the quadridentate ligand, *N*-Mebcen, is

the unstable form, with both terminal six-membered chelate rings in a twist form and with the central five-membered ring in a *gauche* form. On the other hand, in [Cu(H<sub>2</sub>O)(*N*-Mebcen)]<sup>2+</sup>, one of the two six-membered rings adopts a twist form, and the other six- and five-membered chelate rings are of chair form and *gauche* form, respectively.

**Introduction.** Recently, we have initiated systematic studies on transition-metal complexes of diaminodiamides. Previously, we have reported the crystal structures of the copper(II) complexes of *N,N'*-bis( $\beta$ -carbamoylethyl)ethylenediamine, bceen (Lee, Lu, Liu, Chung & Lee, 1984), *N,N'*-bis( $\beta$ -carbamoylethyl)-*N,N'*-dimethylethylenediamine, *N*-Me<sub>2</sub>bceen (Lu, Shan, Chao & Chung, 1987), *N,N'*-bis( $\beta$ -carbamoylethyl)-2-hydroxytrimethylenediamine, bchtn (Lee, Hong, Liu, Chung & Lee, 1984), *N,N'*-bis( $\beta$ -carbamoylethyl)trimethylenediamine, bctn (Hong, Lee, Lee, Chao & Chung, 1987). We now report the crystal structures of the aqua and the perchlorato copper(II) complexes of *N,N'*-bis( $\beta$ -carbamoylethyl)-*N*-methylethylenediamine, *N*-

\* 4-Methyl-4,7-diazadecanediamine.

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Mebcen,  $\{[\text{Cu}(\text{H}_2\text{O})(N\text{-Mebcen})](\text{ClO}_4)_2\} \{[\text{Cu}(\text{ClO}_4)(N\text{-Mebcen})](\text{ClO}_4)(\text{H}_2\text{O})\}$ .

**Experimental.** The ligand, *N*-Mebcen, was prepared by using the following procedure: 12.4 ml (0.2 mol) of *N*-methylethylenediamine and 28.4 g (0.4 mol) of acrylamide in 80 ml acetonitrile were refluxed for 2 h. The solution was cooled in a refrigerator for 1.5 months and the product filtered off, recrystallized from acetonitrile, and dried in air, m.p. 361–362 K (found: C, 50.8; H, 9.32; N, 26.5%; calc. for  $\text{C}_9\text{H}_{20}\text{N}_4\text{O}_2$ : C, 50.0; H, 9.30; N, 25.9%). *N*-Mebcen was reacted with  $\text{Cu}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$  in water. A deep blue crystal  $0.1 \times 0.3 \times 0.5$  mm grown from 95% alcohol was used for the X-ray structure determination.  $D_m$  by flotation. CAD-4 diffractometer, setting angles of 25 reflections ( $20 \leq 2\theta \leq 28^\circ$ ) used to determine unit-cell constants and crystal orientation.  $[(\sin\theta)/\lambda]_{\max} = 0.594 \text{ \AA}^{-1}$  ( $-14 \leq h \leq 14$ ,  $0 \leq k \leq 15$ ,  $-15 \leq l \leq 14$ ),  $\omega$ - $2\theta$  scan, width =  $2(1.0 + 0.35 \tan\theta)^\circ$ ,  $2\theta$  scan speed =  $20/3$  to  $20/10^\circ \text{ min}^{-1}$ , graphite-monochromated Mo  $K\alpha$  radiation. Three standard reflections monitored every 2 h, varied within  $2\sigma(I)$ . 6652 reflections measured, 4042 observed with  $I > 2.5\sigma(I)$ . Empirical absorption correction based on azimuthal rotation from three reflections: 313, 526, 738 (North, Phillips & Mathews, 1968). The minimum, maximum and average normalized correction factors are 0.8615, 0.9995 and 0.9277, respectively. The positions of two Cu atoms were determined by direct methods and checked by Patterson synthesis. Other atoms were located by successive difference synthesis and full-matrix least-squares fit. The function minimized was  $\sum w(|F_o| - |F_c|)^2$ , where  $w = 1/\sigma^2(F_o)$ . Final values of  $R$  and  $wR$  are 0.076 and 0.068, respectively, based on 4042 independent reflections. 487 parameters with anisotropic temperature factors for non-H atoms.  $S = 4.92$ ,  $(\Delta/\sigma)_{\max} = 0.4$ ,  $(\Delta\rho)_{\max} = 0.95(8)$  (around perchlorate ion),  $(\Delta\rho)_{\min} = -0.66(8) \text{ e \AA}^{-3}$ . Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

**Discussion.** The atomic parameters are listed in Table 1. Bond lengths and bond angles are listed in Table 2.\* Fig. 1 shows the displacements of atoms from the best plane through the coordinated N and O atoms of *N*-Mebcen. The coordination geometry about the Cu ion in each of these two complexes is a distorted square pyramid, in which the four donor atoms of *N*-Mebcen form a slightly distorted plane and an O atom of  $\text{H}_2\text{O}$  or  $\text{ClO}_4^-$  occupies the axial position. The equatorial

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a stereoview of the crystal structure have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43900 (33 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic parameters  $x$ ,  $y$ ,  $z$  and equivalent isotropic thermal parameters ( $\text{Å}^2$ )

	$x$	$y$	$z$	$B_{\text{eq}}^*$
Cu(1)	0.09655 (14)	0.77035 (14)	0.33985 (14)	3.21
Cu(2)	0.33906 (16)	0.22045 (14)	1.15403 (14)	3.68
Cl(1)	0.0478 (3)	0.2844 (3)	0.1997 (3)	4.63
Cl(2)	0.2664 (3)	0.6105 (3)	0.1133 (3)	4.69
Cl(3)	0.6151 (3)	0.2684 (3)	0.2853 (3)	4.44
Cl(4)	0.6674 (4)	0.7796 (4)	0.3318 (3)	6.0
O(11)	0.1377 (9)	0.2632 (10)	0.1327 (9)	7.6
O(12)	0.0775 (10)	0.3775 (11)	0.2989 (9)	9.8
O(13)	-0.0084 (12)	0.1836 (13)	0.2157 (13)	13.4
O(14)	-0.0246 (9)	0.3055 (11)	0.1404 (10)	8.8
O(21)	0.2901 (10)	0.5090 (10)	0.0293 (9)	8.5
O(22)	0.3593 (10)	0.6954 (12)	0.1624 (11)	12.4
O(23)	0.2075 (13)	0.5877 (13)	0.1952 (11)	15.7
O(24)	0.2067 (18)	0.6406 (14)	0.0600 (14)	20.9
O(31)	0.7185 (12)	0.2920 (13)	0.2484 (15)	18.9
O(32)	0.6081 (14)	0.1894 (14)	0.3270 (15)	18.3
O(33)	0.6054 (13)	0.3735 (12)	0.3623 (11)	14.6
O(34)	0.5369 (10)	0.2187 (10)	0.2035 (9)	8.3
O(41)	0.5830 (25)	0.6870 (23)	0.324 (3)	53.2
O(42)	0.6215 (13)	0.8477 (13)	0.3109 (11)	12.8
O(43)	0.7171 (12)	0.8214 (11)	0.4247 (10)	11.3
O(44)	0.738 (3)	0.749 (3)	0.2500 (16)	47.2
O(1)	0.2120 (7)	0.8994 (7)	0.3175 (8)	4.2
O(2)	0.0191 (7)	0.7597 (8)	0.2182 (7)	4.2
O(3)	0.2717 (8)	0.0571 (7)	1.1350 (7)	4.4
O(4)	0.3173 (7)	0.2646 (7)	1.3102 (6)	3.5
N(1)	0.1807 (8)	0.7855 (8)	0.4646 (8)	3.2
N(2)	0.0034 (8)	0.6144 (9)	0.3365 (8)	3.4
N(3)	-0.0883 (11)	0.6813 (13)	0.0717 (10)	7.3
N(4)	0.3710 (10)	1.0297 (11)	0.3214 (10)	5.8
N(5)	0.3726 (9)	0.1727 (9)	0.9949 (8)	3.5
N(6)	0.3880 (10)	0.3872 (9)	1.1699 (9)	4.7
N(7)	0.3299 (10)	0.3766 (10)	1.4871 (9)	5.3
N(8)	0.1957 (13)	-0.1331 (10)	1.0587 (10)	7.9
C(11)	0.3084 (10)	0.9435 (11)	0.3482 (11)	3.7
C(12)	0.3594 (10)	0.9133 (12)	0.4269 (11)	4.0
C(13)	0.3001 (11)	0.7979 (11)	0.4366 (10)	3.5
C(14)	0.1678 (11)	0.8807 (12)	0.5689 (11)	4.6
C(15)	0.1292 (13)	0.6680 (12)	0.4773 (11)	5.0
C(16)	0.0096 (12)	0.6235 (12)	0.4457 (11)	4.7
C(17)	-0.1128 (12)	0.5794 (12)	0.2948 (12)	4.9
C(18)	-0.1081 (12)	0.5656 (12)	0.1762 (12)	4.9
C(19)	-0.0544 (11)	0.6774 (12)	0.1589 (10)	3.9
C(21)	0.2396 (12)	-0.0306 (11)	1.0516 (10)	4.6
C(22)	0.2487 (14)	-0.0312 (13)	0.9409 (11)	6.5
C(23)	0.2731 (12)	0.0886 (13)	0.9328 (11)	5.2
C(24)	0.4666 (13)	0.1258 (13)	0.9598 (12)	5.5
C(25)	0.3945 (13)	0.2834 (12)	0.9730 (11)	5.3
C(26)	0.4575 (12)	0.3863 (12)	1.0732 (13)	5.4
C(27)	0.4256 (13)	0.4745 (12)	1.2695 (12)	5.6
C(28)	0.3579 (12)	0.4735 (11)	1.3653 (12)	4.7
C(29)	0.3325 (11)	0.3620 (11)	1.3862 (10)	3.6
O(W1)	0.9710 (9)	0.8715 (9)	0.4448 (9)	6.5
O(W2)	0.0756 (24)	0.067 (3)	0.323 (3)	58.5

\*  $B_{\text{eq}}$  is the mean of the principal axes of the thermal ellipsoid.

Cu—N distances span a very narrow range, 1.979 (10)–1.996 (10) Å, which are typical for copolymer(II) polyamine complexes. The equatorial Cu—N and Cu—O bond lengths of  $[\text{Cu}(\text{ClO}_4)(N\text{-Mebcen})]^+$  (*B*) are shorter by ca 0.009 Å than those of  $[\text{Cu}(\text{H}_2\text{O})(N\text{-Mebcen})]^{2+}$  (*A*). This is the result of the shorter axial Cu—O bond length (Dunitz, 1979). The two asymmetric N atoms in each of these two complex cations are of the same *R* or *S* configuration; the *N*-methyl group of *N*-Mebcen and the  $\text{H}_2\text{O}$  in *A* or  $\text{ClO}_4^-$  in *B*, are on the same side of the  $\text{CuN}_2\text{O}_2$  plane. In *B*, the conformation of *N*-Mebcen is the unstable form, with both six-membered chelate rings having a twist form and the five-membered ring having a *gauche* form. On

the other hand, one of the two six-membered rings, that including O(1)—Cu(1)—N(1), adopts a chair form in *A*. The rather high *R* value of 0.076 is due to poor crystallinity of the specimen and large thermal motion of ClO<sub>4</sub><sup>-</sup>.

The structures of the copper(II) complexes with several diaminodiamides have been reported. A comparison of the selected structural data of these complexes with those in this work is given in Table 3. The

Table 2. Bond lengths (Å), bond angles (°) and hydrogen-bond lengths (Å)

Cu(1)—O(1)	1.964 (8)	Cu(1)—O(2)	1.940 (8)
Cu(1)—N(1)	2.003 (10)	Cu(1)—N(2)	1.994 (10)
Cu(2)—O(3)	1.946 (8)	Cu(2)—O(4)	1.934 (8)
Cu(2)—N(5)	1.988 (10)	Cu(2)—N(6)	1.981 (10)
Cl(1)—O(11)	1.376 (11)	Cl(1)—O(12)	1.378 (12)
Cl(1)—O(13)	1.374 (13)	Cl(1)—O(14)	1.429 (11)
Cl(2)—O(21)	1.416 (11)	Cl(2)—O(22)	1.362 (12)
Cl(2)—O(23)	1.331 (13)	Cl(2)—O(24)	1.327 (15)
Cl(3)—O(31)	1.331 (14)	Cl(3)—O(32)	1.325 (14)
Cl(3)—O(33)	1.354 (12)	Cl(3)—O(34)	1.381 (11)
Cl(4)—O(41)	1.323 (19)	Cl(4)—O(42)	1.314 (13)
Cl(4)—O(43)	1.295 (12)	Cl(4)—O(44)	1.326 (20)
O(1)—C(11)	1.255 (15)	O(2)—C(19)	1.239 (16)
O(3)—C(21)	1.225 (15)	O(4)—C(29)	1.227 (15)
N(1)—C(13)	1.484 (17)	N(1)—C(14)	1.462 (16)
N(1)—C(15)	1.534 (16)	N(2)—C(16)	1.429 (17)
N(2)—C(17)	1.510 (18)	N(3)—C(19)	1.316 (18)
N(4)—C(11)	1.315 (16)	N(5)—C(23)	1.472 (18)
N(5)—C(24)	1.428 (18)	N(5)—C(25)	1.513 (16)
N(6)—C(26)	1.468 (18)	N(6)—C(27)	1.372 (18)
N(7)—C(29)	1.284 (16)	N(8)—C(21)	1.311 (17)
C(11)—C(12)	1.526 (18)	C(12)—C(13)	1.507 (19)
C(15)—C(16)	1.496 (21)	C(17)—C(18)	1.519 (20)
C(18)—C(19)	1.501 (19)	C(21)—C(22)	1.468 (20)
C(22)—C(23)	1.530 (21)	C(25)—C(26)	1.543 (22)
C(27)—C(28)	1.452 (21)	C(28)—C(29)	1.511 (18)
Cu(1)—O(W1)	2.311 (2)	Cu(2)—O(34)	2.672 (4)

O(1)—Cu(1)—O(2)	87.0 (4)	O(1)—Cu(1)—N(1)	91.1 (4)
O(1)—Cu(1)—N(2)	165.4 (4)	O(2)—Cu(1)—N(1)	178.1 (4)
O(2)—Cu(1)—N(2)	93.3 (4)	N(1)—Cu(1)—N(2)	88.5 (4)
O(3)—Cu(2)—O(4)	88.1 (3)	O(3)—Cu(2)—N(5)	91.5 (4)
O(3)—Cu(2)—N(6)	172.5 (5)	O(4)—Cu(2)—N(5)	175.5 (4)
O(4)—Cu(2)—N(6)	92.5 (4)	N(5)—Cu(2)—N(6)	88.5 (4)
O(11)—Cl(1)—O(12)	112.5 (8)	O(11)—Cl(1)—O(13)	108.8 (9)
O(11)—Cl(1)—O(14)	106.7 (7)	O(12)—Cl(1)—O(13)	110.2 (9)
O(12)—Cl(1)—O(14)	111.7 (8)	O(13)—Cl(1)—O(14)	106.8 (8)
O(21)—Cl(2)—O(22)	112.9 (8)	O(21)—Cl(2)—O(23)	112.9 (9)
O(21)—Cl(2)—O(24)	101.6 (9)	O(22)—Cl(2)—O(23)	104.2 (9)
O(22)—Cl(2)—O(24)	115.3 (11)	O(23)—Cl(2)—O(24)	110.3 (13)
O(31)—Cl(3)—O(32)	105.8 (12)	O(31)—Cl(3)—O(33)	104.2 (10)
O(31)—Cl(3)—O(34)	113.1 (10)	O(32)—Cl(3)—O(33)	112.8 (12)
O(32)—Cl(3)—O(34)	107.5 (8)	O(33)—Cl(3)—O(34)	113.3 (8)
O(41)—Cl(4)—O(42)	102.8 (14)	O(41)—Cl(4)—O(43)	110.4 (14)
O(41)—Cl(4)—O(44)	109.7 (27)	O(42)—Cl(4)—O(43)	118.3 (9)
O(42)—Cl(4)—O(44)	105.2 (13)	O(43)—Cl(4)—O(44)	109.9 (13)
Cu(1)—O(1)—C(11)	131.0 (8)	Cu(1)—O(2)—C(19)	129.9 (8)
Cu(2)—O(3)—C(21)	129.9 (8)	Cu(2)—O(4)—C(29)	131.1 (8)
Cu(1)—N(1)—C(13)	110.8 (7)	Cu(1)—N(1)—C(14)	114.7 (8)
Cu(1)—N(1)—C(15)	103.5 (7)	C(13)—N(1)—C(14)	109.9 (9)
C(13)—N(1)—C(15)	109.7 (10)	C(14)—N(1)—C(15)	107.9 (10)
Cu(1)—N(2)—C(16)	103.9 (8)	Cu(1)—N(2)—C(17)	114.7 (7)
C(16)—N(2)—C(17)	114.7 (10)	Cu(2)—N(5)—C(23)	108.9 (8)
Cu(2)—N(5)—C(24)	114.5 (9)	Cu(2)—N(5)—C(25)	105.7 (7)
C(23)—N(5)—C(24)	111.0 (11)	C(23)—N(5)—C(25)	107.3 (10)
C(24)—N(5)—C(25)	109.0 (10)	Cu(2)—N(6)—C(26)	105.1 (8)
Cu(2)—N(6)—C(27)	119.4 (9)	C(26)—N(6)—C(27)	117.7 (12)
O(1)—C(11)—N(4)	119.8 (12)	O(1)—C(11)—C(12)	123.8 (11)
N(4)—C(11)—C(12)	116.1 (11)	C(11)—C(12)—C(13)	115.9 (10)

N(2)—H(12)···O(23)	2.1965	N(3)—H(18)···O(14)	2.4122
N(3)—H(18)···O(21)	2.5790	N(3)—H(17)···O(44)	2.1553
N(4)—H(19)···O(32)	2.1471	N(4)—H(20)···O(3)	2.1280
N(7)—H(37)···O(33)	2.1061	N(7)—H(38)···O(43)	2.2258
N(8)—H(40)···O(24)	2.6750	O(W1)—H(W1)···O(43)	2.1538
O(W2)—H(W22)···O(1)	2.1419		

coordination numbers of these diaminodiamide complexes are five or six. The coordination geometry about copper(II) for the five-coordinate complex is distorted square-pyramidal; that for the six-coordinate complex is tetragonally distorted octahedral. The equatorial Cu—N bond lengths are in the range 1.98–2.01 Å; the equatorial Cu—O bond lengths are in the range 1.93–1.99 Å. The chelate angles for the five-membered rings are in the range 86–89°; the chelate angles for the six-membered rings are in the range 92–95°.

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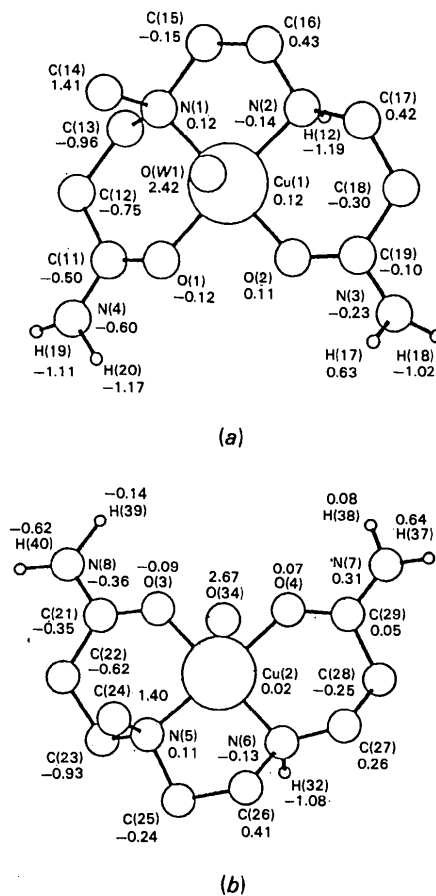


Fig. 1. (a) Molecular structure, showing the displacements (Å) of atoms from the best plane through the coordinated N and O atoms of [Cu(H<sub>2</sub>O)(*N*-Mebcen)]<sup>2+</sup> (*A*) (the atom positions below the plane are indicated by negative signs). (b) The same as (a) but for the molecule of [Cu(ClO<sub>4</sub>)(*N*-Mebcen)]<sup>+</sup> (*B*).

Table 3. Selected structural data for copper(II) complexes of diaminodiamides

	[Cu(NO <sub>3</sub> )(bcen)- (H <sub>2</sub> O)]NO <sub>3</sub> ·H <sub>2</sub> O	[Cu(H <sub>2</sub> O)(N-Mebcen)] (ClO <sub>4</sub> ) <sub>2</sub> (A)	[Cu(ClO <sub>4</sub> )(N-Mebcen)] (ClO <sub>4</sub> ) <sub>2</sub> ·H <sub>2</sub> O (B)	{[Cu(N-Me <sub>2</sub> bcen)] <sub>2</sub> (ClO <sub>4</sub> )}(ClO <sub>4</sub> ) <sub>3</sub>	[Cu(bcen)(H <sub>2</sub> O)- (ClO <sub>4</sub> )](ClO <sub>4</sub> ) <sub>2</sub> ·H <sub>2</sub> O	[Cu(bchtn)](ClO <sub>4</sub> ) <sub>2</sub>
Cu—N (Å)	1.983 (7) 2.002 (6)	2.007 (10) 2.005 (9)	1.979 (10) 1.990 (10)	1.977 (6) 2.010 (6)	2.006 (5) 2.009 (4)	1.998 (10) 2.008 (7)
Equatorial Cu—O (Å)	1.957 (5) 1.974 (6)	1.938 (8) 1.961 (8)	1.926 (8) 1.949 (8)	1.945 (5) 1.952 (5)	1.972 (4) 1.987 (4)	1.966 (7) 1.970 (7)
Axial Cu—O (Å)	2.548 (9) 2.399 (9)*	2.312 (20)*	2.673 (20)	2.483 (13)	2.500 (4) 2.702 (5)*	2.448 (11) 2.641 (11)
Chelate angle for 5-membered ring (°)	85.6 (3)	88.6 (4)	88.2 (4)	87.3 (3)	—	—
Chelate angle for 6-membered ring (°)	95.3 (2) 95.0 (2)	93.4 (4) 90.6 (4)	92.0 (4) 92.6 (4)	91.8 (2) 92.9 (2)	93.3 (2) 94.1 (2) 93.6 (2)	93.1 (3) 93.1 (4) 93.4 (3)
Coordination geometry about Cu <sup>II</sup>	Tetragonally distorted octahedral	Distorted square-pyramidal	Distorted square-pyramidal	Distorted square-pyramidal	Tetragonally distorted octahedral	Tetragonally distorted octahedral
Conformation of chelate rings	chair <i>gauche</i> chair	chair <i>gauche</i> twist	chair <i>gauche</i> chair	chair <i>gauche</i> chair	chair <i>gauche</i> chair	chair <i>gauche</i> chair
Coordination number	6	5	5	5 (binuclear)	6	6
Configuration for the two chiral N centres	<i>SS</i> or <i>RR</i>	<i>SS</i> or <i>RR</i>	<i>SS</i> or <i>RR</i>	<i>SS</i> or <i>RR</i>	<i>RS</i>	<i>RS</i>
Crystal system	Monoclinic	Triclinic	Triclinic	Orthorhombic	Triclinic	Monoclinic
Reference	Lee, Lu, Liu, Chung & Lee (1984)	Present work	Present work	Lu <i>et al.</i> (1987)	Hong <i>et al.</i> (1987)	Lee, Hong, Liu, Chung & Lee (1984)

\* Cu—OH<sub>2</sub> bond distance.

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## Structure of Poly-diaqua-bis( $\mu$ -4,4'-bi-1,2,4-triazole- $N^1, N^{1'}$ )-manganese(II) Dinitrate Dihydrate

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**Abstract.** [Mn(C<sub>4</sub>H<sub>4</sub>N<sub>6</sub>)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>](NO<sub>3</sub>)<sub>2</sub>·2H<sub>2</sub>O,  $M_r = 523.24$ , orthorhombic, *Pbca*,  $a = 13.648$  (2),  $b = 21.312$  (3),  $c = 13.979$  (3) Å,  $V = 4066$  Å<sup>3</sup>,  $Z = 8$ ,  $D_m = 1.69$  (2),  $D_x = 1.71$  Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.54184$  Å,  $\mu = 61.5$  mm<sup>-1</sup>,  $F(000) = 2083$ ,  $T = 293$  K,  $R = 0.063$  for 1583 independent reflections. The Mn atoms are octahedrally surrounded by two *trans* oriented water molecules and four ligand N atoms. Each ligand coordinates through its N1 and N1'

atoms constituting a bridge between adjacent Mn atoms. In this way, two-dimensional arrays of Mn atoms are formed. The layers are connected to each other by means of hydrogen bridges through the water molecules. The nitrate anions are not coordinated and are fixed in the lattice by hydrogen bridging with the water ligands and lattice water.

**Introduction.** In order to be able to study magnetic interactions between first-row transition-metal ions, magnetochemists need ligands which can create a

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