

the hexakis(imidazole)Cu<sup>II</sup> complex shows a marked tetragonal distortion (McFadden, McPhail, Garner & Mabbs, 1975). The complexes containing Mn<sup>II</sup>, Fe<sup>II</sup>, Co<sup>II</sup> and Ni<sup>II</sup> are high spin. In Fig. 3, the metal–ligand bond lengths in [M(ImH)<sub>6</sub>]<sup>2+</sup> and [M(H<sub>2</sub>O)<sub>6</sub>]<sup>2+</sup> (M = Mn to Zn) are compared. The parallelism of the two curves suggests that coordination of the imidazole rings is not significantly affected by steric hindrance.

Bond lengths in the coordinated imidazole rings are not significantly different from those in free imidazole as determined by neutron diffraction (Craven, McMullan, Bell & Freeman, 1977; McMullan, Epstein, Ruble & Craven, 1979; cf. also Martinez-Carrera, 1966).

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### *trans*-Diaquatetrakis(imidazole)manganese(II) Dichloride, [Mn(C<sub>3</sub>H<sub>4</sub>N<sub>2</sub>)<sub>4</sub>(H<sub>2</sub>O)<sub>2</sub>]Cl<sub>2</sub>

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**Abstract.**  $M_r = 434.19$ , monoclinic,  $C2/c$ ,  $a = 12.488$  (2),  $b = 11.121$  (1),  $c = 14.526$  (2) Å,  $\beta = 107.7$  (1)°,  $V = 1921.5$  (7) Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.51$  (1),  $D_x = 1.501$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu(\text{Mo } K\alpha) = 9.63$  cm<sup>-1</sup>,  $F(000) = 892$ ,  $T = 291$  K,  $R = 0.030$  for 2096 unique reflections. The metal atom is octahedrally coordinated [Mn–N(imidazole) = 2.282 (2), 2.213 (6) Å; Mn–O(water) = 2.230 (2) Å]. The Mn–O(water) bonds are longer and two Mn–N(imidazole) bonds are shorter than the corresponding bonds in [Mn(H<sub>2</sub>O)<sub>6</sub>]<sup>2+</sup> and [Mn(imidazole)<sub>6</sub>]<sup>2+</sup> ions. The two remaining Mn–N(imidazole) bonds are similar in length to those in the [Mn(imidazole)<sub>6</sub>]<sup>2+</sup> ion.

**Introduction.** More than 25 manganese-containing metalloproteins and manganese-activated enzymes are now known (McEuen, 1981). Structural data for the types of bonds to be expected in manganese–protein interactions are, however, still limited. The present structure is a Mn<sup>II</sup> complex in which the ligands are water and imidazole (ImH = C<sub>3</sub>H<sub>4</sub>N<sub>2</sub>, the functional group of the amino acid histidine).

**Experimental.** Crystals of [Mn(H<sub>2</sub>O)<sub>2</sub>(ImH)<sub>4</sub>]Cl<sub>2</sub> appeared during attempts to crystallize [Mn(ImH)<sub>6</sub>]Cl<sub>2</sub>·4H<sub>2</sub>O (Garrett, Guss & Freeman, 1983), when a warm ethanolic solution of MnCl<sub>2</sub> and imidazole in a molecular ratio of 1:4 was slowly cooled. Density by

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flotation in chloroform/bromoform, diffraction data from specimen coated with aerosol lacquer to inhibit decomposition, crystal bounded by faces {100}, {010} and {001} [separations 0.08, 0.27 and 0.22 mm, respectively], Enraf-Nonius CAD-4/F diffractometer, graphite monochromator, unit-cell dimensions by least-squares refinement of  $2\theta$  values for 24 reflections [ $28 < 2\theta < 40^\circ$ ]. Profile analysis of a representative reflection indicated that  $\omega/2\theta$  scan would optimize conditions for measurement of integrated intensities. Scan speeds determined by required precision  $\sigma(I) < 0.01I$ , subject to maximum scan time of 60 s. Extreme one-sixths of scans used to derive backgrounds, no significant decomposition or changes in orientation revealed by three reference reflections after every 7000 s of X-ray exposure and by orientation checks after every 100 reflections. 3028 reflections measured with  $(\sin\theta/\lambda) < 0.64 \text{ \AA}^{-1}$ , including all reflections with  $0 \leq h \leq 15$ ,  $0 \leq k \leq 14$ ,  $-18 \leq l \leq 18$  plus additional  $hkl$  reflections for use in subsequent error analysis; Lorentz, polarization and absorption corrections (Busing & Levy, 1957; Coppens, Leiserowitz & Rabinovich, 1956), grid for absorption calculations  $4 \times 8 \times 10$  [ $1.081 < A^* < 1.237$ ], 2096 unique reflections, 534 below threshold [ $I < 3\sigma_{\text{stat}}(I)$ ],  $R_{\text{merge}} = 0.018$  where  $R_{\text{merge}} = \{\sum_{hkl} \sum_i k_i^{-2} [|\bar{F}(hkl)| - k_i |F_i(hkl)|]^2 / \sum_{hkl} \sum_i |F_i(hkl)|^2\}^{1/2}$ ,  $k_i$  is the relative scale factor for the  $i$ th value,  $F_i$ . Error analysis (Freeman & Guss, 1972) showed that the systematic variance,  $V_s$ , was dependent only on  $|F|$ ,  $\sigma^2(F)$  for each reflection recalculated as the sum of the statistical variance and  $V_s$ , where  $V_s = 2.125 - 0.0612|F| + 0.00067|F|^2$ . Atomic positions of all non-hydrogen atoms from Patterson and Fourier methods, full-matrix least-squares refinement, isotropic,  $R = 0.102$ ,  $R_w = 0.112$ ,  $w = 1/\sigma^2(F)$ , only observed reflections used, atomic scattering factors for Mn<sup>2+</sup>, Cl<sup>-</sup>, O, N, C from Cromer & Mann (1968) and for H from Stewart, Davidson & Simpson (1965), anomalous-dispersion corrections for Mn and Cl applied (*International Tables for X-ray Crystallography*, 1974), program used for structure analysis and refinement: XRAY76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). After two cycles of anisotropic refinement, difference Fourier map showed all hydrogen atoms, which were included in the refinement with thermal parameters fixed at 1.25 times  $\langle U_{ii} \rangle$  for the attached C, N and O atoms.  $R = 0.030$ ,  $R_w = 0.031$ ,  $S = 1.148$ ; in the final cycle the maximum and average ratios of shift to error were  $1 \times 10^{-4}$  and  $6 \times 10^{-6}$ , respectively, in a difference Fourier map the maximum and minimum heights were 0.4 and  $-0.4 \text{ e \AA}^{-3}$ , respectively. †

† Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving hydrogen atoms, hydrogen bonds and least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38569 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

**Discussion.** Final atomic parameters are given in Table 1. Bond lengths and angles are given in Table 2.

The complex cation is centrosymmetric. The coordination of the Mn<sup>II</sup> atom is approximately octahedral (Fig. 1). Owing to symmetry, pairs of *trans* imidazole rings are parallel. One pair of rings is approximately perpendicular [ $82(1)^\circ$ ] to the plane containing the four coordinated N(imidazole) atoms,

Table 1. Atomic positional parameters ( $\times 10^4$  for non-hydrogen atoms,  $\times 10^3$  for hydrogen atoms) and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^3$ ;  $U_{\text{iso}}$  for H)

E.s.d.'s of positional parameters in parentheses. E.s.d.'s of  $U_{\text{eq}}$ 's and  $U_{\text{iso}}$ 's are 2 in the least significant digit for Cl, 1 in the least significant digit for other atoms.

$$U_{\text{eq}} = (U_{11}U_{22}U_{33})^{1/3}.$$

	x	y	z	$U_{\text{eq}}/U_{\text{iso}}$
Mn	2500	2500	5000	26.5
Cl	4090.6 (5)	3505.2 (5)	746.3 (5)	46.4
O	1120 (2)	1194 (2)	4921 (1)	43
N(1)	2411 (2)	2023 (2)	3451 (1)	34
N(3)	2628 (2)	2179 (2)	2013 (2)	47
N(6)	1219 (2)	3902 (2)	4405 (1)	33
N(8)	371 (2)	5638 (2)	4026 (2)	43
C(2)	2908 (2)	2617 (2)	2910 (2)	42
C(4)	1916 (2)	1248 (3)	1967 (2)	44
C(5)	1783 (2)	1159 (2)	2846 (2)	39
C(7)	1301 (2)	5069 (2)	4536 (2)	40
C(9)	-355 (2)	4792 (3)	3532 (2)	47
C(10)	165 (2)	3732 (2)	3762 (2)	39
H(2)	340 (2)	329 (3)	310 (2)	54
H(3)	286 (3)	243 (3)	161 (2)	57
H(4)	166 (2)	85 (3)	144 (2)	57
H(5)	137 (2)	64 (2)	307 (2)	48
H(7)	191 (2)	553 (2)	493 (2)	53
H(8)	26 (3)	637 (3)	394 (2)	57
H(9)	-98 (2)	496 (3)	313 (2)	59
H(10)	-7 (2)	299 (3)	354 (2)	49
H(11)	108 (3)	57 (3)	470 (2)	54
H(12)	65 (3)	133 (3)	515 (2)	54

Table 2. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ) with e.s.d.'s in parentheses

Atomic labels refer to ImH(1); for ImH(2) add 5 to the numeral in each atomic label.

	ImH(1)	ImH(2)
Mn—O	2.230 (2)	
Mn—N(1)	2.282 (2)	2.213 (6)
N(1)—C(2)	1.317 (5)	1.310 (3)
C(2)—N(3)	1.334 (5)	1.331 (7)
N(3)—C(4)	1.354 (4)	1.351 (6)
C(4)—C(5)	1.340 (4)	1.339 (4)
C(5)—N(1)	1.374 (6)	1.375 (9)
N(1)—Mn—N(6)	88.06 (7)	
O—Mn—N(1)	89.57 (8)	
O—Mn—N(6)	89.06 (7)	
Mn—N(1)—C(2)	125.5 (2)	128.7 (2)
Mn—N(1)—C(5)	129.8 (2)	126.6 (2)
C(5)—N(1)—C(2)	104.5 (2)	104.6 (2)
N(1)—C(2)—N(3)	111.6 (2)	111.9 (2)
C(2)—N(3)—C(4)	107.4 (2)	107.2 (2)
N(3)—C(4)—C(5)	106.2 (2)	106.5 (2)
C(4)—C(5)—N(1)	110.2 (2)	109.8 (2)

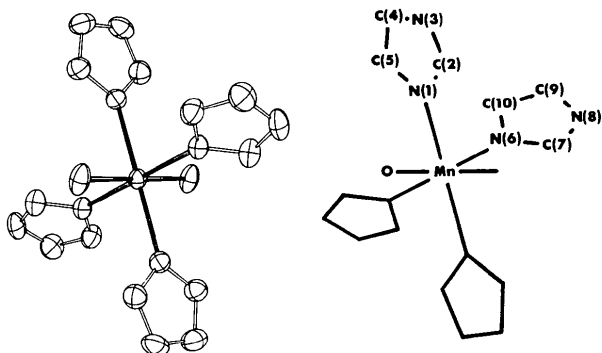


Fig. 1. The  $[\text{Mn}(\text{H}_2\text{O})_2(\text{ImH})_4]^{2+}$  cation, showing (left) thermal ellipsoids (Johnson, 1976) at the 50% probability level and (right) atomic labels.

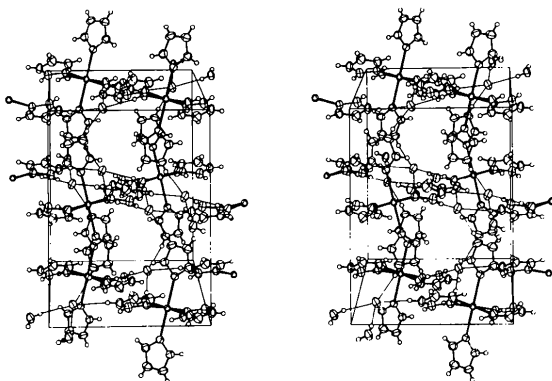


Fig. 2. Stereoview (Johnson, 1976) showing the packing and hydrogen bonding in a crystal of  $[\text{Mn}(\text{H}_2\text{O})_2(\text{ImH})_4]\text{Cl}_2$ . Origin at the top left front corner,  $x$  axis into the paper,  $y$  axis left to right.

while the other pair is at an angle of  $59.4(1)^\circ$  to that plane. The Mn–N(imidazole) bond length in the first pair (2.282 Å) is close to the average value in  $[\text{Mn}(\text{ImH})_6]^{2+}$  (2.273 Å) (Garrett *et al.*, 1983). The second pair of Mn–N(imidazole) bonds are significantly shorter (2.213 Å). The contraction in the latter bonds is balanced by a lengthening of the Mn–O(water) bonds (2.230 Å) when compared with the average value in  $[\text{Mn}(\text{H}_2\text{O})_6]^{2+}$  (2.177 Å) (Carrell & Glusker, 1973).

The complexes are linked parallel to the  $xy$  plane by hydrogen bonds between coordinated  $\text{H}_2\text{O}$  molecules and interstitial  $\text{Cl}^-$  ions (Fig. 2). Each  $\text{Cl}^-$  ion is also hydrogen bonded to two imidazole rings, one belonging to a complex in the same layer and the other belonging to a complex in an adjacent layer.

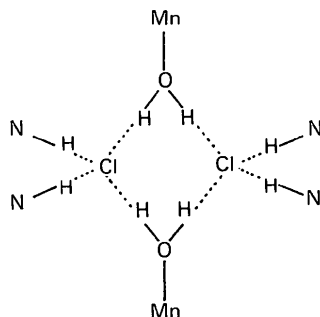


Table 3. Mn<sup>II</sup>–N(imidazole) and Mn<sup>II</sup>–N(imidazolate) bond lengths (Å) as functions of coordination geometry

Bond lengths for bridging imidazolate ( $\text{Im}^-$ ) are indicated by \*.  $\text{MeImH}$  = 2-methylimidazole.

Complex	Mn–N bond length	Geometry	Reference
$[\text{Mn}(\text{H}_2\text{O})_2(\text{ImH})_4]^{2+}$	2.282 (2)	Octahedral	Present work
	2.213 (6)		
$[\text{Mn}(\text{ImH})_6]^{2+}$	2.276 (2)	Octahedral	Garrett <i>et al.</i> (1983)
	2.276 (2)		
	2.266 (1)		
$[\text{Mn}_3(\text{ImH})_2(\text{Im})_6]_\infty$	2.31 (1)	Octahedral	Lehnert & Seel (1980)
	2.25 (1)*		
	2.26 (1)*		
$[\text{Mn}(\text{MeImH})_3\text{Cl}_2]$	2.249 (4)	Distorted trigonal bipyramidal	Phillips, Shreeve & Skapski (1976)
	2.196 (3)		
	2.194 (4)		
$[\text{Mn}_3(\text{ImH})_2(\text{Im})_6]_\infty$	2.12 (1)*	Tetrahedral	Lehnert & Seel (1980)
	2.11 (1)*		
	2.10 (1)*		
	2.08 (1)*		

In the  $z$  direction, the  $\text{ImH}(2)$  rings of adjacent complexes form stacks in which the distances between imidazole ring planes are alternately 3.33 (1) and 3.50 Å.

Within the limits of precision, the dimensions of the coordinated imidazole rings in the present complex are the same as in free imidazole as determined by neutron diffraction (Craven, McMullan, Bell & Freeman, 1977; McMullan, Epstein, Ruble & Craven, 1979). Manganese–imidazole complexes in which the Mn<sup>II</sup> atoms have coordination numbers 6, 5 and 4, respectively, are listed in Table 3. As noted previously in the cases of Zn<sup>II</sup>– and Co<sup>II</sup>–imidazole complexes (Bear, Duggan & Freeman, 1975), a significant decrease in the metal–ligand bond lengths accompanies a decrease in coordination number. An additional and similar effect results from the substitution of imidazolate for imidazole (Table 3). The ‘short’ Mn–N(imidazole) bonds in the present complex are outliers in Table 3 but, as already stated, the shortening of these bonds is compensated by a lengthening of two Mn–O bonds.

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**Dibromo[ $\alpha$ -(*tert*-butyl-1 aziridinyl-2)benzylidèneamine]zinc(II), [ZnBr<sub>2</sub>(C<sub>13</sub>H<sub>18</sub>N<sub>2</sub>)] (1), et  
Dibromo[ $\alpha$ -(*tert*-butyl-1 aziridinyl-2)benzylamine]zinc(II), [ZnBr<sub>2</sub>(C<sub>13</sub>H<sub>20</sub>N<sub>2</sub>)] (2)**

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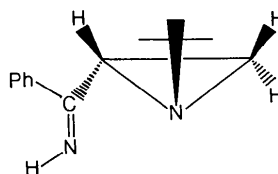
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(Reçu le 16 septembre 1982, accepté le 29 avril 1983)

**Abstract.** (1)  $M_r = 427.5$ , monoclinic,  $P2_1/c$ ,  $a = 7.750$  (2),  $b = 23.044$  (4),  $c = 11.852$  (3) Å,  $\beta = 130.42$  (2)°,  $V = 1611$  (2) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.76$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $\mu = 6.8$  mm<sup>-1</sup>,  $F(000) = 840$ ,  $T = 295$  K,  $R = 0.037$  for 1422 unique reflections. (2)  $M_r = 429.5$ , triclinic,  $P\bar{1}$ ,  $a = 7.787$  (4),  $b = 9.855$  (1),  $c = 10.823$  (4) Å,  $\alpha = 101.91$  (2),  $\beta = 100.87$  (3),  $\gamma = 88.61$  (2)°,  $V = 798$  (2) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.79$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $\mu = 6.9$  mm<sup>-1</sup>,  $F(000) = 424$ ,  $T = 295$  K,  $R = 0.058$  for 2380 unique reflections. (1) and (2) are chelate compounds, the Zn atoms in each being tetrahedrally surrounded by the two Br atoms and the two N atoms of the ligand involved. The formation of (1) would explain the stereospecificity of the reduction of the chelating imine by Zn(BH<sub>4</sub>)<sub>2</sub> or (ZnBr<sub>2</sub> + 2 NaBH<sub>4</sub>) into the sole *erythro* isomer of the corresponding amine, the configuration of which is precisely characterized by means of (2).

**Introduction.** La structure de (1) et (2) a été établie en vue d'expliquer la stéréospécificité de la réduction en milieu protique (CH<sub>3</sub>OH) par Zn(BH<sub>4</sub>)<sub>2</sub> ou par (ZnBr<sub>2</sub> + 2 NaBH<sub>4</sub>) de l'imine (aziridinoimine):



En effet, avec ces deux réducteurs, l'isomère *érythro* de l'amine correspondante est seul obtenu. Les modes opératoires ainsi que la synthèse directe de (1) et (2) ont été décrits par ailleurs (Bartnik, Laurent & Lesniak, 1982). En l'absence de zinc, la réduction donne un mélange d'isomères *érythro* et *thréo*. (1) et (2) peuvent être obtenus par addition de ZnBr<sub>2</sub> à une solution dans le méthanol de l'imine pour (1) et de l'amine pour (2). La réduction stéréospécifique de l'imine peut s'effectuer aussi bien à partir de (1) mis en suspension dans le méthanol. En outre, la formation de (2) est un moyen de déterminer la configuration de l'isomère *érythro* de l'amine obtenue après réduction de l'imine.

**Partie expérimentale.** Pour le chélate (1): cristallisation dans le méthanol; parallélépipède taillé 0,15 × 0,20 × 0,40 mm; diffractomètre Nonius CAD-4; paramètres