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Structural Researches on Metal Complexes of Polydentate Ligands Containing Carbonyl and α-Diimine Moieties. IV.* Pentacoordination in Dichloro-2-(2′-pyridyl)-3-(*N*-2-picolylimino)-4-oxo-1,2,3,4-tetrahydroquinazolinemanganese(II)

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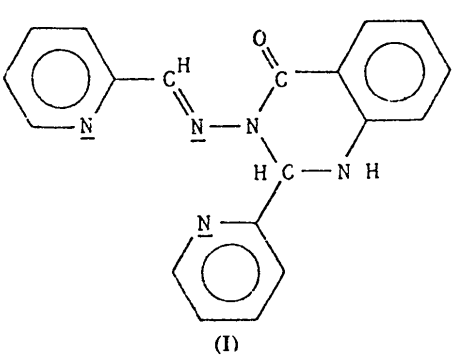
The crystals of the title compound ($C_{19}H_{15}Cl_2MnN_5O$) are triclinic ($P\bar{1}$); the unit-cell dimensions are $a=9\cdot306$ (4), $b=15\cdot493$ (6), $c=10\cdot200$ (4) Å, $\alpha=127\cdot1$ (2), $\beta=123\cdot6$ (2) $\gamma=74\cdot0$ (2)°; $Z=2$. The structure has been determined from diffractometer data by Patterson and Fourier methods and refined by block-diagonal least-squares calculations to $R=3\cdot6$% for 2011 independent reflexions. The manganese atom is five-coordinated through two chlorine atoms (Mn–Cl = 2·338, 2·341 Å), two nitrogen atoms (Mn–N = 2·261, 2·259 Å) and an oxygen atom (Mn–O = 2·203 Å). The organic molecule behaves as a tridentate ligand as has already been observed for the Cu(II) and Co(II) complexes, but now the oxygen atom takes the place of a pyridine nitrogen. Packing is mainly determined by a N–H···Cl hydrogen bond, which gives rise to chains running parallel to **c**.

Introduction

The structure analysis of the title compound was undertaken as a part of a programme of research currently in progress in our laboratory on metal complexes of polydentate ligands containing carbonyl and α-diimine groups. Our work on this subject includes three X-ray structural determinations carried out on the following complexes: $Cu(PPQ)Cl_2$ (Mangia, Nardelli, Pelizzi & Pelizzi, 1974), $[Co(PPQ)(NCS)(SCN)]_2$ (Mangia, Nardelli & Pelizzi, 1974), $Cu_2(DIP)Cl_2.H_2O$ (Mangia, Pelizzi & Pelizzi, 1974) [PPQ and DIP denote 2-(2′-pyridyl)-3-(*N*-2-picolylimino)-4-oxo-1,2,3,4-tetrahydroquinazoline and 2,6-diacetylpyridinebis(picolinoylhydrazone) respectively] and a comprehensive spectroscopic study of the behaviour of PPQ as a ligand (Pelizzi & Pelizzi, 1974).

The crystal analyses of the PPQ complexes revealed that in both cases the ligand acts as tridentate through the underlined nitrogen atoms:



(I)

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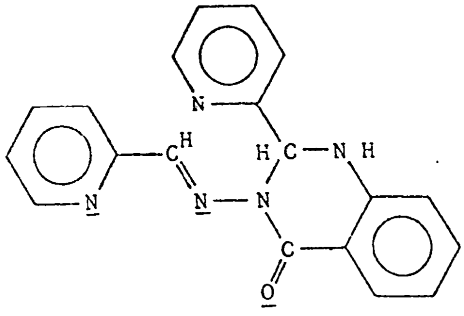
* Part III: *Acta Cryst.* (1974). B**30**, 2146–2150.

From these studies and from the different spectroscopic properties shown by these complexes with respect to others, such as those of Cd, Mn, Zn, it was inferred that the organic molecule can assume two different conformations in coordinating to metal. In order to elucidate this behaviour the crystal structure of Mn(PPQ)Cl_2 was determined, showing that the ligand now assumes the conformation (II), which is derived from conformation (I) by rotation around the N–N bond in order to put oxygen in a suitable situation for coordination.



(II)

Experimental

The compound was prepared as previously described (Pelizzi & Pelizzi, 1974) and occurs in small orange slightly pleochroic crystals. Unit-cell dimensions, preliminarily determined from rotation and Weissenberg photographs, were refined by a least-squares procedure applied to diffractometer measurements for 20 reflexions. These values, together with other pertinent crystal data, are listed in Table 1. Most crystals were twinned, but a thin untwinned platelet, measuring 0·18 × 0·14 × 0·48 mm was selected and mounted on an automated single-crystal Siemens AED diffractometer with its *c* axis aligned along the φ axis of the instrument. 3546 independent reflexions up to a $\theta_{max} = 26°$ were recorded utilizing Mo $K\alpha$ radiation and the ω–2θ scan technique. Of these, 2011 reflexions had intensities greater than $2\sigma(I)$ and were coded as 'observed'.

Table 1. *Crystallographic data*

$C_{19}H_{15}Cl_2MnN_5O$; F.W. 455·2
Triclinic, space group $P\bar{1}$
$a = 9{\cdot}306 \pm 4$, $b = 15{\cdot}493 \pm 6$, $c = 10{\cdot}200 \pm 4$ Å
$\alpha = 127{\cdot}1 \pm 2$, $\beta = 123{\cdot}6 \pm 2$, $\gamma = 74{\cdot}0 \pm 2°$
$U = 973{\cdot}9$ Å^3, $Z = 2$, $D_m = 1{\cdot}53$ g cm^{-3}, $D_x = 1{\cdot}55$ g cm^{-3}
μ(Mo $K\alpha$) = 10·0 cm^{-1}; $F(000) = 462$

The intensity data were corrected for Lorentz and polarization effects, but no correction was made for absorption because the μr value was only 0·08. The absolute scale was determined first by Wilson's method and later by correlating observed and calculated values.

Table 2. *Final atomic fractional coordinates* (× 10^4) *and thermal parameters* (Å^2) *with estimated standard deviations in parentheses for non-hydrogen atoms*

Anisotropic thermal parameters are in the form: $\exp[-\frac{1}{4}(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)]$·

	x/a	y/b	z/c	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Mn	349 (1)	1328 (0)	3917 (1)	7·29 (3)	3·92 (3)	4·95 (2)	1·12 (2)	4·38 (2)	2·81 (2)
Cl(1)	−2407 (1)	503 (1)	2252 (1)	7·23 (6)	4·80 (4)	5·34 (4)	0·93 (4)	4·13 (4)	2·57 (4)
Cl(2)	1754 (1)	2308 (1)	7237 (1)	9·29 (7)	4·60 (4)	5·19 (4)	1·13 (3)	5·02 (5)	2·65 (4)
O	−435 (3)	2604 (2)	3363 (3)	7·8 (1)	5·1 (1)	6·5 (1)	1·7 (1)	5·3 (1)	4·0 (1)
N(1)	1981 (4)	−101 (2)	3423 (4)	6·9 (2)	3·8 (1)	4·7 (1)	0·5 (1)	3·6 (1)	2·6 (1)
N(2)	1550 (4)	1162 (2)	2372 (4)	7·1 (2)	3·1 (1)	4·2 (1)	0·6 (1)	3·7 (1)	2·1 (1)
N(3)	1118 (4)	1887 (2)	1882 (4)	7·2 (2)	3·4 (1)	4·5 (1)	0·9 (1)	4·0 (1)	2·4 (1)
N(4)	3685 (4)	3545 (3)	4698 (4)	4·6 (2)	6·6 (2)	6·4 (2)	0·7 (1)	3·6 (1)	3·5 (1)
N(5)	965 (4)	2504 (2)	214 (4)	10·0 (2)	4·5 (1)	5·2 (1)	2·0 (1)	5·6 (2)	3·2 (1)
C(1)	2197 (5)	−753 (3)	3962 (5)	7·1 (2)	4·9 (2)	6·0 (2)	0·6 (2)	3·8 (2)	3·5 (2)
C(2)	3235 (5)	−1590 (3)	3696 (5)	6·6 (2)	5·0 (2)	6·3 (2)	0·5 (2)	2·8 (2)	4·1 (2)
C(3)	4120 (5)	−1781 (3)	2853 (5)	6·7 (2)	3·4 (1)	5·9 (2)	0·7 (1)	3·1 (2)	2·5 (1)
C(4)	3943 (5)	−1119 (3)	2294 (5)	7·0 (2)	3·9 (1)	5·3 (2)	1·0 (1)	4·0 (2)	2·3 (1)
C(5)	2866 (5)	−294 (2)	2596 (4)	6·6 (2)	3·2 (1)	3·7 (1)	0·4 (1)	3·0 (1)	1·9 (1)
C(6)	2604 (5)	452 (3)	2055 (5)	7·5 (2)	3·8 (1)	4·5 (2)	0·9 (1)	4·2 (2)	2·2 (1)
C(7)	2117 (5)	2081 (3)	1355 (5)	9·7 (2)	4·0 (1)	6·6 (2)	2·6 (1)	6·7 (2)	3·5 (1)
C(8)	3736 (5)	2834 (3)	3107 (6)	6·2 (2)	5·6 (2)	8·7 (2)	3·3 (1)	6·0 (2)	5·5 (2)
C(9)	5173 (7)	2791 (4)	2990 (8)	10·6 (3)	10·5 (3)	14·9 (4)	6·7 (3)	10·4 (3)	10·2 (3)
C(10)	6556 (6)	3550 (5)	4592 (9)	6·8 (3)	14·7 (4)	17·9 (5)	5·0 (3)	8·1 (3)	13·9 (4)
C(11)	6484 (6)	4282 (4)	6159 (8)	5·7 (2)	10·9 (3)	13·0 (4)	1·6 (2)	3·7 (3)	9·5 (3)
C(12)	5048 (5)	4267 (4)	6198 (7)	3·8 (2)	9·2 (3)	8·6 (3)	0·4 (2)	2·7 (2)	5·8 (2)
C(13)	75 (4)	2646 (2)	2515 (4)	6·0 (2)	3·5 (1)	3·9 (1)	0·0 (1)	3·0 (1)	1·9 (1)
C(14)	−430 (4)	3442 (2)	2036 (4)	5·7 (2)	3·3 (1)	3·5 (1)	−0·2 (1)	2·5 (1)	1·7 (1)
C(15)	4 (4)	3321 (2)	824 (4)	5·9 (2)	3·3 (1)	3·3 (1)	−0·6 (1)	2·3 (1)	1·6 (1)
C(16)	−658 (5)	4018 (3)	205 (5)	6·7 (2)	4·1 (1)	4·0 (2)	−0·4 (1)	2·6 (1)	2·3 (1)
C(17)	−1601 (5)	4829 (3)	876 (5)	6·4 (2)	4·7 (2)	5·2 (2)	0·1 (1)	2·5 (2)	3·6 (1)
C(18)	−1983 (5)	4980 (3)	2138 (5)	6·2 (2)	4·8 (2)	5·8 (2)	1·0 (1)	3·4 (2)	3·4 (2)
C(19)	−1408 (5)	4276 (3)	2690 (5)	6·1 (2)	4·2 (1)	4·8 (2)	0·7 (1)	3·2 (2)	2·8 (1)

Structure determination and refinement

The positions of Mn, Cl(1) and Cl(2) were deduced from a three-dimensional Patterson map, assuming the space group $P\bar{1}$. From these, all the other non-hydrogen atoms were located by the heavy-atom technique. The structure was refined by a block-diagonal anisotropic least-squares procedure to a conventional R index $[\sum \Delta F_{\text{meas}}/\sum F_{\text{meas}}]$ of 4·6%. At this stage a three-dimensional difference Fourier synthesis was calculated from which all the hydrogen atoms were located. The contributions of these atoms, refined isotropically, improved the R value to 3·6%.

The quantity minimized was $\sum w(\Delta F_{\text{meas}})^2$, in which unit weights were used at first and the scheme $1/w = A + B|F_{\text{meas}}| + C|F_{\text{meas}}|^2$ was then assumed; the coefficients $A = 0{\cdot}9322$, $B = -0{\cdot}0094$ and $C = 0{\cdot}0010$ were obtained by plotting ΔF_{meas} *vs* $\bar{F}_{\text{meas}}$.

The scattering factors for the heavy atoms were those of Cromer & Mann (1968) and for the hydrogen atoms those of Stewart, Davidson & Simpson (1965). The final atomic and thermal parameters are quoted in Tables 2 and 3.*

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30538 (16 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

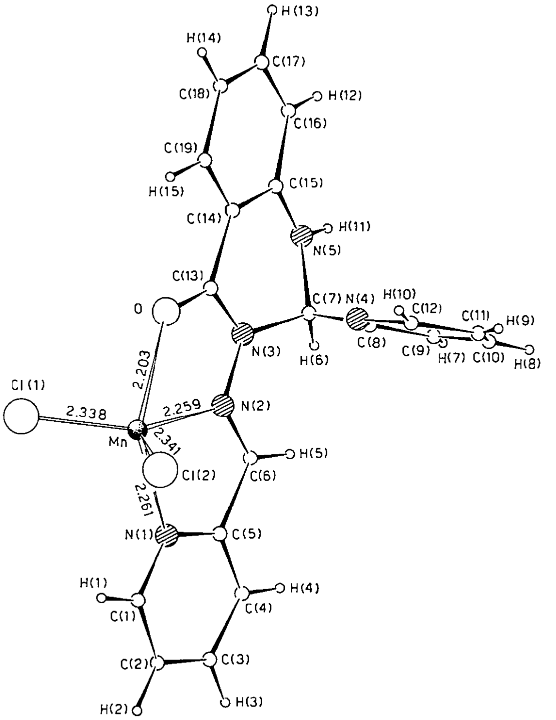

Fig. 1. Clinographic projection of the structure with bond distances in the coordination polyhedron.

Table 3. *Final atomic fractional coordinates* (× 10³) *and isotropic thermal parameters* (Å²) *for hydrogen atoms*

	x/a	*y/b*	*z/c*	*B*
H(1)	155 (4)	−57 (3)	452 (5)	6·0 (0·8)
H(2)	339 (4)	−202 (3)	414 (4)	5·9 (0·8)
H(3)	485 (4)	−234 (3)	263 (5)	6·1 (0·8)
H(4)	457 (4)	−122 (3)	170 (4)	5·8 (0·8)
H(5)	327 (4)	38 (2)	153 (4)	4·9 (0·7)
H(6)	241 (4)	141 (3)	62 (4)	5·9 (0·8)
H(7)	508 (5)	222 (3)	175 (5)	8·9 (1·0)
H(8)	740 (6)	362 (4)	477 (7)	12·8 (1·5)
H(9)	705 (4)	478 (3)	709 (5)	6·3 (0·8)
H(10)	485 (5)	487 (3)	732 (5)	8·4 (1·0)
H(11)	130 (4)	240 (3)	−46 (5)	6·6 (0·9)
H(12)	−38 (4)	386 (2)	−71 (4)	5·6 (0·8)
H(13)	−204 (4)	532 (2)	48 (4)	4·9 (0·7)
H(14)	−263 (5)	556 (3)	264 (5)	7·3 (0·9)
H(15)	−179 (4)	431 (3)	344 (4)	6·2 (0·8)

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

(*a*) In the coordination polyhedron

Mn–Cl(1)	2·338 (9)	Mn–N(1)	2·261 (9)
Mn–Cl(2)	2·341 (8)	Mn–N(2)	2·259 (9)
Mn–O	2·203 (8)		

Cl(1)–Mn–Cl(2)	115·80 (5)	Cl(2)–Mn–N(2)	122·98 (8)
Cl(1)–Mn–N(1)	102·24 (9)	Cl(2)–Mn–O	103·45 (8)
Cl(1)–Mn–N(2)	121·21 (8)	N(1)–Mn–N(2)	70·2 (1)
Cl(1)–Mn–O	98·22 (9)	N(1)–Mn–O	139·9 (1)
Cl(2)–Mn–N(1)	98·12 (8)	N(2)–Mn–O	69·7 (1)

(*b*) In the PPQ ligand

N(1)—C(1)	1·341 (8)	C(13)–N(3)	1·375 (8)
C(1)—C(2)	1·365 (9)	C(13)–O	1·232 (6)
C(2)—C(3)	1·359 (9)	C(7)—C(8)	1·517 (7)
C(3)—C(4)	1·383 (9)	C(8)—C(9)	1·387 (11)
C(4)—C(5)	1·376 (8)	C(9)—C(10)	1·379 (10)
C(5)—N(1)	1·345 (8)	C(10)–C(11)	1·318 (10)
C(5)—C(6)	1·471 (8)	C(11)–C(12)	1·366 (11)
C(6)—N(2)	1·267 (8)	C(12)–N(4)	1·332 (7)
N(2)—N(3)	1·371 (7)	N(4)—C(8)	1·323 (7)
N(3)—C(7)	1·465 (8)	C(15)–C(16)	1·403 (8)
C(7)—N(5)	1·444 (7)	C(16)–C(17)	1·356 (9)
N(5)—C(15)	1·354 (8)	C(17)–C(18)	1·391 (7)
C(15)–C(14)	1·399 (7)	C(18)–C(19)	1·370 (8)
C(14)–C(13)	1·460 (8)	C(19)–C(14)	1·389 (8)

C(1)—N(1)—Mn	125·0 (3)	N(5)—C(15)–C(16)	122·5 (4)
C(5)—N(1)—Mn	118·1 (3)	C(16)–C(15)–C(14)	118·4 (4)
C(1)—N(1)—C(5)	116·8 (4)	C(15)–C(14)–C(19)	120·2 (4)
C(2)—C(1)—N(1)	123·7 (5)	C(15)–C(14)–C(13)	119·2 (4)
C(3)—C(2)—C(1)	119·1 (5)	C(19)–C(14)–C(13)	120·5 (4)
C(4)—C(3)—C(2)	118·9 (5)	C(14)–C(13)–N(3)	116·0 (4)
C(5)—C(4)—C(3)	118·9 (4)	C(14)–C(13)–O	123·7 (4)
C(6)—C(5)—C(4)	122·7 (4)	N(3)—C(13)–O	120·2 (4)
C(6)—C(5)—N(1)	114·7 (4)	C(13)–O——Mn	120·3 (3)
N(1)—C(5)—C(4)	122·6 (4)	C(15)–C(16)–C(17)	120·0 (4)
C(5)—C(6)—N(2)	116·5 (4)	C(16)–C(17)–C(18)	121·9 (5)
C(6)—N(2)—N(3)	123·4 (4)	C(17)–C(18)–C(19)	118·7 (4)
N(3)—N(2)—Mn	116·3 (2)	C(18)–C(19)–C(14)	120·7 (4)
C(6)—N(2)—Mn	120·2 (3)	C(7)—C(8)—C(9)	120·2 (4)
N(2)—N(3)—C(13)	113·4 (3)	C(7)—C(8)—N(4)	117·4 (4)
N(2)—N(3)—C(7)	122·5 (4)	C(9)—C(8)—N(4)	122·4 (4)
C(13)–N(3)—C(7)	120·8 (4)	C(8)—C(9)—C(10)	117·5 (5)
N(3)—C(7)—N(5)	106·7 (4)	C(9)—C(10)–C(11)	120·2 (6)
N(3)—C(7)—C(8)	111·0 (3)	C(10)–C(11)–C(12)	119·6 (6)
N(5)—C(7)—C(8)	113·1 (4)	C(11)–C(12)–N(4)	122·7 (5)
C(7)—N(5)—C(15)	119·5 (3)	C(12)–N(4)—C(8)	117·6 (4)
N(5)—C(15)–C(14)	119·0 (4)		

Results and discussion

A clinographic projection of the structure is represented in Fig. 1 together with the interatomic distances in the coordination polyhedron. All bond distances and valence angles are reported in Table 4.

The metal atom is five-coordinated by two chlorine ions (Mn–Cl = 2·338, 2·341 Å) and by two nitrogen atoms (Mn–N = 2·261, 2·259 Å) and the oxygen atom (Mn–O = 2·203 Å) from the organic molecule, so the bonding geometry in the coordination polyhedron can be described as a distorted trigonal bipyramid with Cl(1), Cl(2) and N(2) in the equatorial plane. Considering bond angles the most relevant deviation from the theoretical values for an ideal trigonal bipyramid concerns the O–Mn–N(1) angle which is 139·9° instead of 180°. Such a situation must be related to the conformation assumed by the organic ligand in coordination. Pentacoordination is rather unusual for Mn(II). Indeed, to our knowledge, only five X-ray structures of pentacoordinated Mn(II)-complexes have been reported, three of which are nitrosylcarbonyl derivatives. Table 5 lists the bond distances in the coordination polyhedra of these compounds. As expected, the Mn–O distances involving water molecules (2·03, 2·06 Å) are relevantly shorter than the same distances involving the carbonyl oxygen; the same can be said for the Mn–N distances which are much shorter when nitrosylic nitrogen is involved. No comparison can be made for the Mn–Cl distances in pentacoordinated complexes; the values observed in the present compound (2·338, 2·341 Å) are relevantly shorter than the values found in octahedral hexacoordinated complexes, *i.e.* 2·508, 2·516, 2·470, 2·506, 2·758, 2·628, 2·577, 2·434 Å in $5MnCl_2.14C_2H_5OH$ (L'Haridon & Le Bihan, 1973); 2·415 Å in *fac*-chloro-[1,3-bis(dimethylarsino)propane]tricarbonylmanganese (Bear & Trotter, 1973).

The PPQ ligand is tridentate with conformation (II). In the clinographic projection of Fig. 2 differences in the coordination between the two pentacoordinated complexes $Mn(PPQ)Cl_2$ and $Cu(PPQ)Cl_2$ are depicted. As a consequence of the different orientation of the systems O–C(13) and N(4)–C(8)–C(7) with respect to the N(2)–N(3) bond, two five-membered chelation rings are present in the Mn-complex, while a five-membered ring and a six-membered ring are formed in the Cu-complex. The two chelation rings in the Mn-complex are planar within experimental errors [max.

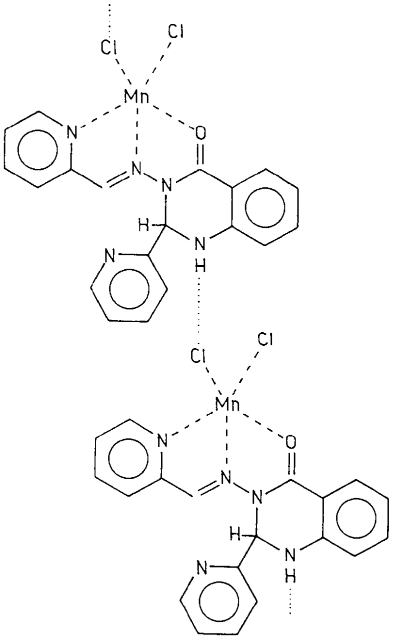

Fig. 3. A chain of complex molecules joined by intermolecular hydrogen bonds (diagrammatic).

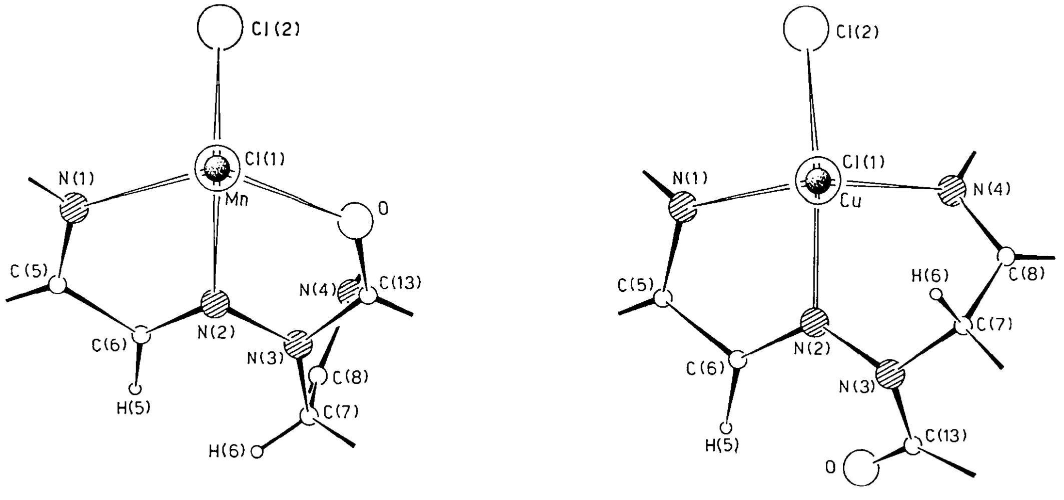

Fig. 2. Clinographic projections of the metal environments in the pentacoordinated complexes $Mn(PPQ)Cl_2$ and $Cu(PPQ)Cl_2$.

Table 5. *Bond distances* (Å) *in the coordination polyhedron for pentacoordinated* Mn(II)-*complexes*

	I	II	III	IV	V	VI
Mn–Br		2·491				
Mn–Cl	2·338 2·341					
Mn–O	2·203		2·28 2·03 2·06			
Mn–N	2·261 2·259	2·19 2·27		1·73	1·76*	1·797
Mn–C				1·78 1·75	1·80* 1·78* 1·83	1·851 1·886
Mn–P				2·278 2·279	2·305	

I. Present work. II. Tris-(2-dimethylaminoethyl)aminemanganese bromide (Di Vaira & Orioli, 1968). III. Zeolite A (Yanagida, Vance & Seff, 1973). IV. Nitrosyldicarbonylbis-(triphenylphosphine)manganese (Enemark & Ibers, 1967). V. Nitrosyltricarbonyltriphenylphosphinemanganese (Enemark & Ibers, 1968). (The starred distances correspond to a disordered situation, each value referring to ½ N and ½ C.) VI. Nitrosyltetracarbonylmanganese (Frenz, Enemark & Ibers, 1969).

deviation 0·04 Å for MnN(1)C(5)C(6)N(2) and 0·02 Å for MnN(2)N(3)C(13)O] and are nearly coplanar, the dihedral angle being 1·3°; the mean least-squares plane through them makes an angle of 2·1° with the adjacent pyridinic ring N(1)···C(5). The second pyridinic ring, N(4)···C(12), is nearly perpendicular to that plane (dihedral angle 82·1°) and to the plane running through the phenyl ring (dihedral angle 87·5°).

The six-membered heteroatomic ring N(3)C(13)C(14)C(15)N(5)C(7) of the quinazoline system shows a rather flattened half-chair conformation as a consequence of the tetrahedral character of C(7) and of the imperfect hybridization of N(3) and N(5). These two atoms are both out of the planes through their three adjacent atoms by 0·15 Å and the sum of the valence angles is 356·7° for N(3) and 354·4° for N(5). N(2) and C(13) are sp^2 hybridized as indicated by their planar surroundings N(3)MnC(6) and N(3)OC(14) respectively.

There is only one crystallographically independent hydrogen bond, $\mathrm{N(5)}\text{–}\mathrm{H(11)}\cdots\mathrm{Cl}(2^{\mathrm{i}}) = 3{\cdot}33$ Å, with $\mathrm{N(5)H(11)Cl}(2^{\mathrm{i}}) = 166°$ ($\mathrm{i} = x, y, z-1$), which joins adjacent molecules in a zigzag pattern parallel to **c** as depicted in the schematic drawing of Fig. 3.

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