C(14)	−0.6923 (9)	−0.4649 (8)	−0.5945 (6)	0.024 (3)
C(15)	−0.790 (1)	−0.4266 (9)	−0.5922 (7)	0.034 (3)
C(16)	−0.883 (1)	−0.4411 (9)	−0.6563 (7)	0.036 (3)
C(17)	−0.8688 (10)	−0.4953 (8)	−0.7164 (7)	0.031 (3)
C(18)	−0.770 (1)	−0.5352 (9)	−0.7160 (8)	0.040 (3)
C(19)	−0.685 (1)	−0.5251 (9)	−0.6530 (8)	0.040 (3)
C(20)	−0.4790 (9)	−0.5216 (7)	−0.5174 (6)	0.025 (3)
C(21)	−0.495 (2)	−0.6047 (7)	−0.498 (1)	0.038 (3)
C(22)	−0.421 (1)	−0.6681 (9)	−0.4907 (8)	0.038 (3)
C(23)	−0.325 (1)	−0.6535 (10)	−0.5162 (8)	0.044 (4)
C(24)	−0.304 (1)	−0.5747 (10)	−0.5412 (8)	0.044 (3)
C(25)	−0.378 (1)	−0.5081 (9)	−0.5429 (7)	0.036 (3)
C(26)	−0.472 (1)	−0.175 (1)	−0.6691 (9)	0.052 (4)
C(27)	−0.677 (1)	−0.2371 (10)	−0.6683 (8)	0.043 (3)
C(28)	−0.516 (1)	−0.3515 (9)	−0.6948 (8)	0.041 (3)
C(29)	−0.333 (1)	−0.1859 (9)	−0.3564 (8)	0.036 (3)
C(30)	−0.539 (1)	−0.1093 (10)	−0.4148 (9)	0.049 (4)
C(31)	−0.370 (1)	−0.0971 (9)	−0.4993 (8)	0.044 (3)

Table 2. *Selected geometric parameters (Å, °)*

Pt(1)—P(1)	2.317 (6)	P(2)—C(14)	1.81 (2)
Pt(1)—P(2)	2.352 (7)	P(2)—C(20)	1.73 (2)
Pt(1)—P(3)	2.331 (7)	P(3)—C(26)	1.84 (3)
Pt(1)—P(4)	2.345 (7)	P(3)—C(27)	1.77 (3)
P(1)—C(1)	1.80 (2)	P(3)—C(28)	1.83 (3)
P(1)—C(7)	1.79 (3)	P(4)—C(29)	1.80 (3)
P(1)—C(13)	1.84 (2)	P(4)—C(30)	1.80 (3)
P(2)—C(13)	1.80 (3)	P(4)—C(31)	1.84 (3)
P(1)—Pt(1)—P(2)	70.3 (2)	C(13)—P(2)—C(20)	105 (1)
P(1)—Pt(1)—P(3)	164.9 (2)	C(14)—P(2)—C(20)	109 (1)
P(1)—Pt(1)—P(4)	94.1 (2)	Pt(1)—P(3)—C(26)	123 (1)
P(2)—Pt(1)—P(3)	96.8 (2)	Pt(1)—P(3)—C(27)	112.9 (10)
P(2)—Pt(1)—P(4)	164.2 (2)	Pt(1)—P(3)—C(28)	111.7 (9)
P(3)—Pt(1)—P(4)	98.4 (3)	C(26)—P(3)—C(27)	101 (1)
Pt(1)—P(1)—C(1)	116.3 (9)	C(26)—P(3)—C(28)	101 (1)
Pt(1)—P(1)—C(7)	121.2 (9)	C(27)—P(3)—C(28)	103 (1)
Pt(1)—P(1)—C(13)	93.2 (8)	Pt(1)—P(4)—C(29)	112.0 (10)
C(1)—P(1)—C(7)	109 (1)	Pt(1)—P(4)—C(30)	112 (1)
C(1)—P(1)—C(13)	107 (1)	Pt(1)—P(4)—C(31)	121.2 (10)
C(7)—P(1)—C(13)	106 (1)	C(29)—P(4)—C(30)	104 (1)
Pt(1)—P(2)—C(13)	93.1 (8)	C(29)—P(4)—C(31)	100 (1)
Pt(1)—P(2)—C(14)	121.2 (8)	C(30)—P(4)—C(31)	103 (1)
Pt(1)—P(2)—C(20)	115.7 (8)	P(1)—C(13)—P(2)	94 (1)
C(13)—P(2)—C(14)	109 (1)		

Data collection used a Rigaku AFC-7*S* four-circle diffractometer equipped with an Oxford Systems low-temperature attachment. The temperature for data collection was 150 K. The Pt-atom position was located by heavy-atom Patterson methods and the remaining non-H atoms were found from difference Fourier syntheses. As there were no identifiable faces, the data were corrected for absorption using *DIFABS* (Walker & Stuart, 1983). This correction was applied to the raw data with the model at isotropic convergence. In the final refinement calculations, anisotropic displacement parameters were adjusted only for Pt, Cl, P and O atoms. Fixed contributions for the scattering of methyl, methylene and phenyl H atoms [C—H = 0.96 Å] were added to the structure factors.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *PATTY* in *DIRDIF*92 (Beurskens *et al.*, 1992). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1219). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Manganese Quinaldinate Complex: *trans*-[Diaquabis(2-quinolinecarboxylato)-manganese(II)]–Water–Ethanol (1/2/2)

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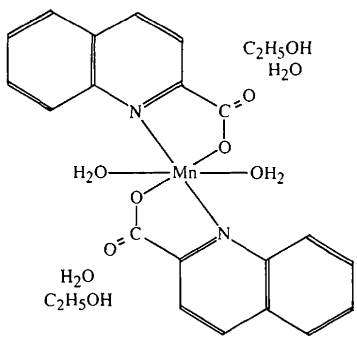
Abstract

The title compound, $[Mn(C_{10}H_6NO_2)_2(H_2O)_2].2C_2H_5\text{-}OH.2H_2O$, has a six-coordinate Mn atom at the center of symmetry, with two bidentate quinaldinate ligands and two water molecules in *trans* configurations. Two uncoordinated water molecules and two uncoordinated ethanol molecules stabilize the complex by participation in an extended hydrogen-bonding network.

Comment

The structure analysis of the title compound, (I), is a continuation of the investigation of metal complexes of aromatic and heterocyclic amino acids (Haendler, 1989, 1993, 1994; Boudreau & Haendler, 1992). The structure of the analogous pentacoordinate copper complex has been reported (Haendler, 1986).



(I)

Manganese(II) complexes are inherently less stable than those of the succeeding elements, but can sometimes be isolated by formation in non-aqueous solvents. Quinaldinic acid can act as a bidentate ligand, forming a five-membered ring system. In the case of Cu, this results in pentacoordination, the fifth position being occupied by a water molecule. The Mn complex is hexacoordinate, with the Mn atom at the center of symmetry (Fig. 1). Two water molecules complete the octahedron. The crystal is stabilized by an extended hydrogen-bonding network (Fig. 2), incorporating two additional water molecules and two ethanol molecules. The coordinating atoms are in their respective *trans* positions.

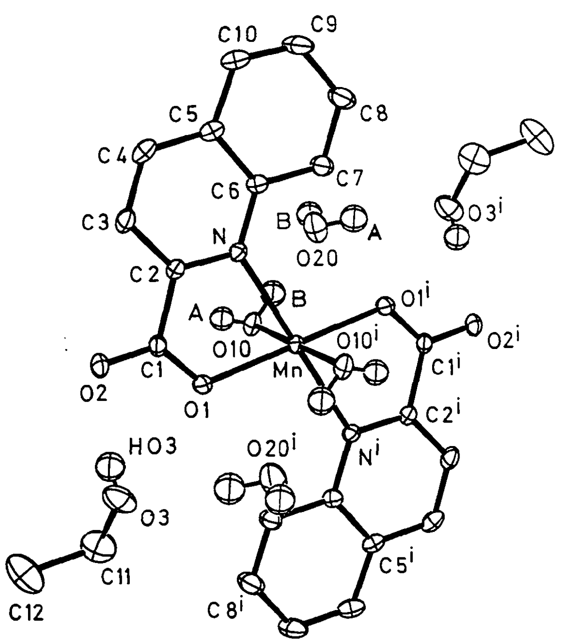


Fig. 1. A plot of the manganese complex showing displacement ellipsoids at the 25% probability level. Ring and chain H atoms are omitted for clarity.

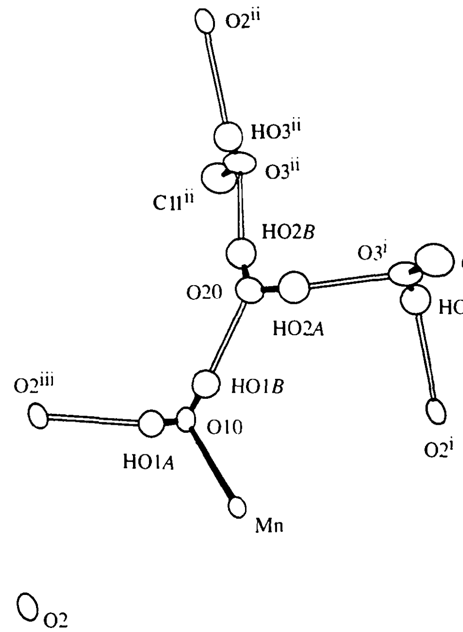


Fig. 2. A representation of the proposed hydrogen-bonding scheme of Table 3. Hydrogen bonds are represented by open bonds.

The Mn atom is smaller than Cu and in the Mn complex the bonds to the ligand are considerably longer, and the bite angle in the five-membered chelate ring is smaller: Mn—O 2.125 (2) Å, Cu—O 1.954 (3), 1.962 (3) Å, Mn—N 2.324 (3) Å, Cu—N 2.014 (3), 2.012 (3) Å, O—Mn—N 74.3 (1)°, O—Cu—N 82.1 (1), 82.5 (1)°. In a six-membered bidentate ring complex reported by McAuliffe *et al.* (1994), the corresponding distances are Mn—O 1.83 (1), 1.85 (1) Å, and Mn—N 1.97 (1), 1.99 (1) Å. The Mn atom is also hexacoordinate, with two ethanol molecules in *trans* positions at Mn—O distances of 2.27 (1) and 2.28 (1) Å, compared with Mn—O of 2.209 (3) Å for the two coordinated water molecules of the title compound.

Experimental

A suitable crystal was formed by slow evaporation of a mixture of ethanol solutions of manganous acetate tetrahydrate and quinaldinic acid in a molar ratio of 1:2. Analysis: calculated for $[Mn(C_{10}H_6NO_2)_2].2C_2H_5OH.2H_2O$ C 51.16, H 5.72, N 4.97%; found C 51.62, H 5.45, N 4.95%.

Crystal data

$[Mn(C_{10}H_6NO_2)_2(H_2O)_2]$.-
$2C_2H_6O.2H_2O$
M_r = 563.3
Triclinic
$P\bar{1}$
a = 7.365 (2) Å
b = 9.102 (3) Å
c = 11.165 (3) Å
α = 77.52 (3)°

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 15 reflections
θ = 10–25°
μ = 0.52 mm^{-1}
T = 293 (1) K
Rectangular parallelepiped
0.70 × 0.55 × 0.18 mm

$\beta = 73.47\ (2)°$
$\gamma = 71.27\ (3)°$
$V = 673.1\ (4)\ Å^3$
$Z = 1$
$D_x = 1.389\ Mg\ m^{-3}$
D_m not measured

Pale yellow

Data collection

Nicolet four-circle diffractometer
ω scans
Absorption correction: ψ scan (North, Phillips & Matthews, 1968)
$T_{min} = 0.786$, $T_{max} = 1.000$
3260 measured reflections
3098 independent reflections
2737 observed reflections $[I > 2.5\sigma(I)]$

$R_{int} = 0.012$
$\theta_{max} = 27.45°$
$h = -8 \rightarrow 9$
$k = 0 \rightarrow 11$
$l = -13 \rightarrow 14$
6 standard reflections monitored every 300 reflections
intensity decay: insignificant

Refinement

Refinement on F
$R = 0.045$
$wR = 0.065$
$S = 1.54$
2737 reflections
217 parameters
Only coordinates of H atoms refined
$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{max} = 0.01$ for non-H atoms, 0.03 for H atoms
$\Delta\rho_{max} = 0.47\ e\ Å^{-3}$
$\Delta\rho_{min} = -0.42\ e\ Å^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters ($Å^2$)*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Mn	0	0	0	2.11 (3)
O1	−0.2058 (4)	0.0293 (3)	−0.1095 (2)	2.81 (11)
O2	−0.4057 (4)	−0.0704 (3)	−0.1614 (3)	3.40 (13)
O10	−0.2371 (4)	0.1086 (4)	0.1534 (3)	3.07 (13)
O20	−0.1212 (6)	−0.0125 (5)	0.3724 (3)	5.2 (2)
N	−0.1072 (4)	−0.2247 (3)	0.0593 (3)	2.19 (11)
C1	−0.2903 (5)	−0.0747 (4)	−0.0975 (3)	2.35 (15)
C2	−0.2426 (5)	−0.2177 (4)	0.0007 (3)	2.27 (14)
C3	−0.3374 (6)	−0.3364 (5)	0.0219 (4)	3.13 (18)
C4	−0.2849 (6)	−0.4663 (5)	0.1063 (4)	3.42 (18)
C5	−0.1407 (6)	−0.4798 (4)	0.1706 (3)	2.78 (15)
C6	−0.0529 (5)	−0.3545 (4)	0.1452 (3)	2.48 (14)
C7	0.0925 (7)	−0.3648 (5)	0.2076 (4)	3.50 (19)
C8	0.1494 (8)	−0.4943 (5)	0.2929 (5)	4.6 (2)
C9	0.0621 (8)	−0.6184 (5)	0.3180 (5)	4.5 (2)
C10	−0.0769 (7)	−0.6118 (5)	0.2594 (4)	3.7 (2)
O3	−0.2840 (6)	0.0860 (5)	−0.3916 (3)	5.16 (19)
C11	−0.3978 (11)	0.2442 (8)	−0.4000 (6)	6.1 (4)
C12	−0.5628 (14)	0.2728 (10)	−0.4601 (9)	7.9 (5)

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

Mn—O1	2.125 (2)	N—C2	1.319 (4)
Mn—O10	2.209 (3)	N—C6	1.379 (4)
Mn—N	2.324 (3)	C1—C2	1.526 (5)
O1—C1	1.256 (4)	C2—C3	1.411 (5)
O2—C1	1.243 (4)	C11—C12	1.477 (11)
O3—C11	1.413 (8)		
O1—Mn—N	74.3 (1)	O2—C1—C2	118.3 (3)
O1—Mn—O10	91.3 (1)	Mn—N—C6	129.5 (2)
N—Mn—O10	89.4 (1)	C2—N—C6	119.0 (3)
Mn—O1—C1	120.4 (2)	C1—C2—C3	119.9 (3)
Mn—N—C2	111.4 (2)	N—C2—C3	123.4 (3)
O1—C1—C2	117.0 (3)	O1—C1—O2	124.7 (3)
N—C2—C1	116.7 (3)	O3—C11—C12	112.6 (6)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
O10—HO1A···O2[i]	0.67 (5)	2.09 (5)	2.737 (4)	163 (5)
O10—HO1B···O20	0.81 (5)	1.92 (5)	2.709 (5)	167 (5)
O20—HO2A···O3[ii]	0.87 (6)	2.05 (6)	2.902 (6)	165 (5)
O20—HO2B···O3[iii]	0.78 (5)	2.01 (5)	2.778 (5)	169 (5)
O3—HO3···O2	0.63 (5)	2.14 (5)	2.713 (4)	151 (7)

Symmetry codes: (i) $1 - x, -y, -z$; (ii) $-x, -y, -z$; (iii) $x, y, 1 + z$.

The structure was solved using a combination of the VAX version of the *DIRDIF* system (Beurskens, *et al.*, 1985) and *NRCVAX* programs (Gabe, Lee & Le Page, 1985). A Patterson map gave the location of the Mn atom and two O atoms, and *DIRDIF* analysis located the remaining non-H atoms. These 25 atoms were refined anisotropically and the positions of the six ring H atoms were calculated. The water and hydroxy H atoms were then located on a difference Fourier map and the positions of the other five H atoms of the ethanol moiety were determined by a combination of Fourier analysis and model superposition on a scaled projection. In the final full-matrix refinement, the positions of these five H atoms were fixed, as were the individual U values of all H atoms. The *NRCVAX* package was used throughout the analysis.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1078). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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