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References

AZEVEDO, C. G., CALHORDA, M. J., CARRONDO, M. A. A. F. DE C. T., DIAS, A. R., FÉLIX, V. & ROMÃO, C. C. (1990). *J. Organomet. Chem.* **391**, 345–360.

CALHORDA, M. J., CARRONDO, M. A. A. F. DE C. T., DIAS, A. R., DOMINGOS, A. M. T., DUARTE, M. T. L. S., GARCIA, M. H. & ROMÃO, C. C. (1987). *J. Organomet. Chem.* **320**, 63–81.

CARRIEDO, G. A., JEFFREY, J. C. & STONE, F. G. A. (1984). *J. Chem. Soc. Dalton Trans.* pp. 1597–1603.

CARRONDO, M. A. A. F. DE C. T., DIAS, A. R., GARCIA, M. H., MATIAS, P. M., ROBALO, M. P., GREEN, M. L. H. HIGGINS, J. Y. & YANG, Y. Y. (1990). *Organomet. Chem.* **395**, 279–292.

COWIE, M. & GAUTHIER, M. D. (1980). *Inorg. Chem.* **19**, 3142–3148.

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

NORRESTAM, R., MERTZ, S. & CROSSLAND, I. (1983). *Acta Cryst.* C**39**, 1554–1556.

NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1991). C**47**, 2453–2454

Bis(pyridine)[bis(salicylidene)-1,3-diaminopropanato]manganese(III) Perchlorate*

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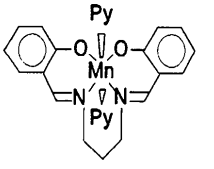
Abstract. $[Mn(C_{17}H_{16}N_2O_2)(C_{10}H_{10}N_2)][ClO_4]$, M_r = 592·9, orthorhombic, $P2_12_12_1$, a = 12·596 (6), b = 13·836 (7), c = 15·848 (8) Å, V = 2761·9 Å^3, Z = 4, D_x = 1·426 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 0·061 mm^{-1}, $F(000)$ = 1224, T = 293 K, R = 0·057 for 1440 unique reflexions [$I \geq 2\sigma(I)$]. The manganese environment is approximately octahedral, comprising a square-planar arrangement of oxygen and nitrogen from the Schiff base, capped by pyridine. Non-bonded interactions are minimized by staggering the pyridine rings relative to the manganese—Schiff-base bonds, O—Mn—N—C −42·9 (7), −63·2 (6)° and by the long pyridine Mn—N bonds of 2·307 (7) and 2·388 (7) Å.

Experimental. The complex aqua[*N*,*N*′-(1,3-propanediyl)bis(salicylideneaminato)]manganese(III) perchlorate dihydrate was prepared as previously described (Ashmawy, McAuliffe, Parish & Tames, 1985). To a solution of this complex (0·5 g) in methanol (200 ml) 2·0 g pyridine was added with stirring. On standing in the refrigerator for 5 d small black crystals of the perchlorate salt of (1) were deposited. These were separated by filtration, washed with cold methanol (3 × 25 ml) and dried over P_2O_5.



(1)

Crystal size 0·4 × 0·2 × 0·2 mm, Nicolet *R*3*m*/*V* diffractometer, graphite-monochromated Mo $K\alpha$ radiation, unit-cell dimensions from setting angles of 25 accurately centred reflexions (8·4 ≤ θ ≤ 14·2°), ω–2θ scan mode, ω-scan width 0·6° below $K\alpha_1$ and 0·6° above $K\alpha_2$ and scan speed ranging from 2 to 5° min^{-1} according to the intensity gathered in a pre-scan, 0 ≤ h ≤ 10, 0 ≤ k ≤ 15, 0 ≤ l ≤ 17, 0 ≤ θ ≤ 25°. 2240 unique reflexions measured, 1440 observed [$I \geq 2\sigma(I)$], intensity standards ($\bar{1}2\bar{3}$, $\bar{4}\bar{2}\bar{2}$, $30\bar{6}$) measured every 200 reflexions, no systematic drift,

* Alternative name: [*N*,*N*′-(1,3-propanediyl)bis(salicylideneaminato)]bis(pyridine)manganese(III) perchlorate.

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Table 1. *Fractional atomic coordinates and vibrational parameters* (Å^2) *for non-H atoms*

$B_{eq} = (1/3)\sum_i\sum_j B_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
Mn(1)	0·45820 (10)	0·60167 (9)	0·59398 (8)	4·45 (6)
O(1)	0·4264 (4)	0·7320 (3)	0·6100 (3)	4·4 (3)
O(2)	0·4917 (4)	0·6320 (4)	0·4824 (3)	5·6 (3)
N(2)	0·4192 (5)	0·5755 (5)	0·7177 (4)	4·7 (4)
N(6)	0·4930 (5)	0·4619 (5)	0·5703 (5)	4·9 (4)
N(20)	0·6332 (5)	0·6197 (5)	0·6340 (5)	4·9 (4)
N(26)	0·2800 (5)	0·5693 (5)	0·5512 (4)	5·1 (4)
C(1)	0·3596 (7)	0·6338 (7)	0·7592 (5)	5·3 (5)
C(3)	0·4613 (8)	0·4917 (6)	0·7622 (6)	6·7 (5)
C(4)	0·4551 (7)	0·3976 (6)	0·7123 (6)	6·2 (5)
C(5)	0·5289 (7)	0·3961 (6)	0·6377 (7)	6·8 (5)
C(7)	0·4882 (6)	0·4258 (6)	0·4952 (7)	5·6 (5)
C(8)	0·3179 (7)	0·7237 (6)	0·7325 (6)	4·7 (5)
C(9)	0·3541 (7)	0·7707 (6)	0·6577 (5)	4·6 (4)
C(10)	0·3104 (7)	0·8612 (6)	0·6395 (6)	5·2 (5)
C(11)	0·2354 (8)	0·9035 (7)	0·6913 (7)	6·3 (5)
C(12)	0·2035 (8)	0·8598 (8)	0·7629 (7)	7·2 (6)
C(13)	0·2440 (7)	0·7702 (7)	0·7854 (6)	5·8 (5)
C(14)	0·4600 (7)	0·4761 (6)	0·4201 (5)	5·1 (4)
C(15)	0·4648 (6)	0·5760 (7)	0·4147 (6)	5·7 (5)
C(16)	0·4424 (8)	0·6223 (7)	0·3384 (6)	7·3 (6)
C(17)	0·4174 (9)	0·5684 (10)	0·2687 (7)	9·0 (7)
C(18)	0·4126 (8)	0·4683 (10)	0·2745 (7)	8·7 (7)
C(19)	0·4359 (7)	0·4228 (7)	0·3461 (7)	6·8 (5)
C(21)	0·7087 (8)	0·5840 (7)	0·5858 (6)	5·9 (5)
C(22)	0·8144 (8)	0·5885 (8)	0·6073 (8)	7·3 (6)
C(23)	0·8432 (7)	0·6283 (8)	0·6818 (8)	8·1 (7)
C(24)	0·7686 (7)	0·6655 (8)	0·7299 (7)	7·4 (6)
C(25)	0·6636 (8)	0·6618 (6)	0·7079 (7)	6·1 (5)
C(27)	0·2240 (7)	0·6427 (7)	0·5197 (6)	5·6 (5)
C(28)	0·1218 (9)	0·6320 (8)	0·4914 (6)	7·1 (6)
C(29)	0·0757 (7)	0·5428 (9)	0·4941 (6)	6·9 (6)
C(30)	0·1343 (8)	0·4657 (8)	0·5270 (6)	6·5 (5)
C(31)	0·2350 (8)	0·4821 (7)	0·5557 (6)	5·5 (5)
Cl(1)	0·6545 (3)	0·6693 (2)	0·9646 (2)	8·2 (2)
O(3)	0·6922 (8)	0·7511 (6)	0·9373 (8)	16·7 (8)
O(4)	0·5741 (12)	0·6505 (12)	0·9031 (6)	22 (1)
O(5)	0·7069 (11)	0·5910 (7)	0·9629 (8)	20 (1)
O(6)	0·5959 (7)	0·6797 (6)	1·0389 (5)	11·9 (6)

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

Mn(1)—O(1)	1·864 (5)	Mn(1)—N(6)	2·019 (7)
Mn(1)—O(2)	1·866 (5)	Mn(1)—N(20)	2·307 (7)
Mn(1)—N(2)	2·053 (7)	Mn(1)—N(26)	2·388 (7)
O(1)—Mn(1)—O(2)	87·7 (2)	O(2)—Mn(1)—N(26)	89·2 (2)
O(1)—Mn(1)—N(2)	89·4 (2)	N(2)—Mn(1)—N(6)	93·5 (3)
O(1)—Mn(1)—N(20)	93·6 (2)	N(2)—Mn(1)—N(20)	89·2 (3)
O(1)—Mn(1)—N(26)	91·0 (2)	N(2)—Mn(1)—N(26)	90·8 (3)
O(2)—Mn(1)—N(6)	89·4 (3)	N(6)—Mn(1)—N(20)	87·0 (2)
O(2)—Mn(1)—N(20)	91·1 (3)	N(6)—Mn(1)—N(26)	88·4 (3)

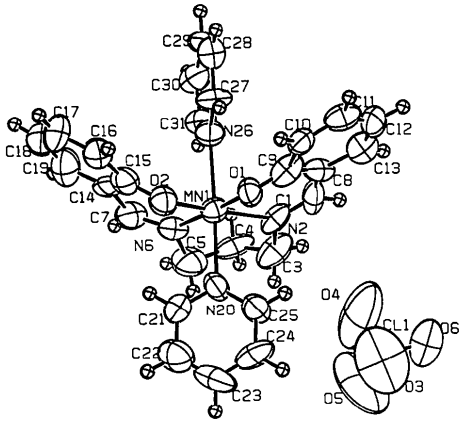

Fig. 1. The title compound, including atomic labelling scheme, drawn using *ORTEP*II (Johnson, 1976).

Lp corrections applied, absorption ignored. Heavier atoms positioned using Patterson techniques (*SHELXTL-Plus*88; Sheldrick, 1988), H atoms placed in chemically reasonable positions, full-matrix least squares based on F using *TEXSAN* (Molecular Structure Corporation, 1985). Final $R = 0{\cdot}057$, $wR = 0{\cdot}051$, $w = 1/[\sigma^2(F) + 0.03F^2]$, anisotropic thermal parameters for heavier atoms, fixed isotropic for H atoms. The absolute configuration was confirmed when a $\Delta F''$ multiplier refined to a value of 0·9 (2) (Rogers, 1981). Min. and max. fluctuations in final ΔF map $-0{\cdot}28$ and $0{\cdot}42$ e Å^{-3}, max. Δ/σ 0·013. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV), computation carried out on MicroVAX computers. Literature surveyed *via* the Cambridge Structural Database (Allen, Kennard & Taylor, 1983) using the Crystal Structure Search and Retrieval Interactive System (CSSR, 1984). Fractional atomic coordinates and vibrational parameters for non-H atoms are presented in Table 1* and selected bond lengths and angles in Table 2. The molecule including atomic labelling is displayed in Fig. 1.

Related literature. The tetragonal bipyrimidal manganese environment is very similar to that of manganese in polymeric 1,4-di(1-imidazolyl)butane-[*N*,*N*′-ethylenebis(salicylideneaminato)]manganese-(III) perchlorate (Matsumoto, Takemoto, Ohyosi & Okawa, 1988).

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* Lists of structure factors, anisotropic thermal parameters, complete bond lengths and angles involving H, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54088 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H., Kennard, O. & Taylor, R. (1983). *Acc. Chem. Res.* **16**, 146–153.

Ashmawy, F. M., McAuliffe, C. A., Parish, R. V. & Tames, J. (1985). *J. Chem. Soc. Dalton Trans.* pp. 1391–1398.

CSSR (1984). SERC Daresbury Laboratory, Warrington, England.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Matsumoto, N., Takemoto, N., Ohyosi, A. & Okawa, H. (1988). *Bull. Chem. Soc. Jpn*, **61**, 2984–2986.

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200A Research Forest Drive, The Woodlands, TX 77381, USA.

Rogers, D. (1981). *Acta Cryst.* A**37**, 734–741.

Sheldrick, G. M. (1988). *SHELXTL-Plus* 88 *Structure Determination Software Programs*. Nicolet Instrument Corporation, Madison, Wisconsin, USA.