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Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
 Mean $\sigma(\text{C}-\text{C}) = 0.006\text{ \AA}$
 R factor = 0.047
 wR factor = 0.117
 Data-to-parameter ratio = 13.4

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Dichlorobis(1,10-phenanthroline)manganese(II)–salicylaldoxime (1/1)

The structure of the title compound, $[\text{MnCl}_2(\text{C}_{12}\text{H}_8\text{N}_2)_2] \cdot \text{C}_7\text{H}_7\text{NO}_2$ or $[\text{MnCl}_2(\text{phen})_2] \cdot \text{saox}$ (saox is salicylaldoxime and phen is 1,10-phenanthroline), consists of a discrete saox molecule and a neutral $[\text{MnCl}_2(\text{phen})_2]$ molecule. The Mn^{II} atom is coordinated by two chloride ions and four N atoms of two phen ligands, forming a distorted octahedral coordination geometry, with four Mn–N distances ranging from 2.275 (3) to 2.315 (2) Å, and Mn–Cl bond lengths of 2.4619 (9) and 2.4285 (9) Å.

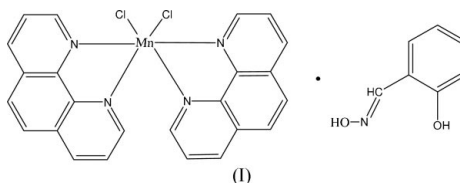
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Comment

One of the interesting aspects of manganese complexes is that manganese plays an important role in biological systems (Lawrence & Sawyer, 1978; Ruttinger & Dismukes, 1997). We chose phen and saox (saox is salicylaldoxime and phen is 1,10-phenanthroline) as ligands to react with Mn^{II} salts, because of the biological importance of α -diimines, which chelate with some ions of 3d transition metals and 2-hydroxyoximes (Keeney *et al.*, 1984). We report here the synthesis and crystal structure of the title compound, $[\text{MnCl}_2(\text{phen})_2] \cdot \text{saox}$, (I).



The X-ray structure analysis reveals that (I) contains a neutral $[\text{MnCl}_2(\text{phen})_2]$ molecule and a discrete saox molecule, as shown in Fig. 1. In the $[\text{MnCl}_2(\text{phen})_2]$ molecule, the Mn^{II} atom is coordinated by two chloride anions and four N atoms of two phen ligands in a distorted octahedral coordination geometry; the Mn–N bond lengths range from 2.275 (3) to 2.315 (2) Å, and the Mn–Cl bond lengths are 2.4619 (9) and 2.4285 (9) Å. A packing diagram of (I) is presented in Fig. 2 and it shows the existence of an $\text{O2} \cdots \text{H2} \cdots \text{N5}$ hydrogen bond in the uncoordinated saox molecule, and an $\text{O1} - \text{H1} \cdots \text{Cl1}(2 - x, -y, -z)$ hydrogen bond between the uncoordinated saox molecule and the inversion-related $[\text{MnCl}_2(\text{phen})_2]$ molecule.

Experimental

$\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$, salicylaldoxime and 1,10-phenanthroline (molar ratio 1:1:1) were dissolved in ethanol (20 ml) and refluxed for 3 h. The resulting solution was allowed to stand at room temperature for a week and brown crystals of (I) were obtained.

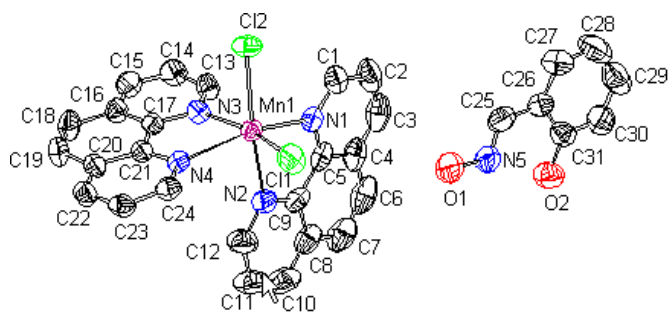


Figure 1
A view of the asymmetric unit of (I), showing the atom-numbering scheme and displacement ellipsoids at the 30% probability level. H atoms have been omitted for clarity.

Crystal data

[MnCl₂(C₁₂H₈N₂)₂] \cdot C₇H₇NO₂
 $M_r = 623.38$
 Triclinic, $P\bar{1}$
 $a = 10.7688$ (6) Å
 $b = 10.8408$ (6) Å
 $c = 13.3473$ (7) Å
 $\alpha = 89.032$ (1) $^\circ$
 $\beta = 66.477$ (1) $^\circ$
 $\gamma = 88.276$ (1) $^\circ$
 $V = 1428.04$ (13) Å³

$Z = 2$
 $D_x = 1.450$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 4106 reflections
 $\theta = 1.7$ – 25.1 $^\circ$
 $\mu = 0.69$ mm⁻¹
 $T = 293$ (2) K
 Prism, brown
 $0.56 \times 0.50 \times 0.40$ mm

Data collection

Siemens SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.807$, $T_{\max} = 1.000$
 7520 measured reflections

4964 independent reflections
 4019 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$
 $\theta_{\max} = 25.1$ $^\circ$
 $h = -11 \rightarrow 12$
 $k = -12 \rightarrow 12$
 $l = -15 \rightarrow 15$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.117$
 $S = 1.08$
 4964 reflections
 370 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0427P)^2 + 1.3089P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.29$ e Å⁻³
 $\Delta\rho_{\min} = -0.39$ e Å⁻³

Table 1

Selected geometric parameters (Å, $^\circ$).

Mn1–N1	2.275 (3)	Mn1–N3	2.315 (2)
Mn1–N4	2.304 (2)	Mn1–Cl2	2.4285 (9)
Mn1–N2	2.310 (3)	Mn1–Cl1	2.4619 (9)
N1–Mn1–N4	152.68 (9)	N2–Mn1–Cl1	92.02 (8)
N1–Mn1–N2	72.65 (10)	N3–Mn1–Cl1	165.16 (7)
N4–Mn1–N2	85.61 (9)	Cl2–Mn1–Cl1	96.86 (4)
N1–Mn1–N3	90.71 (9)	C13–N3–Mn1	125.8 (2)
N4–Mn1–N3	71.78 (8)	C17–N3–Mn1	115.87 (18)
N2–Mn1–N3	88.02 (10)	C24–N4–Mn1	126.1 (2)
N1–Mn1–Cl2	94.17 (7)	C21–N4–Mn1	116.45 (18)
N4–Mn1–Cl2	105.09 (6)	C12–N2–Mn1	126.8 (2)
N2–Mn1–Cl2	165.58 (8)	C9–N2–Mn1	115.2 (2)
N3–Mn1–Cl2	86.28 (7)	C1–N1–Mn1	125.4 (2)
N1–Mn1–Cl1	103.48 (7)	C5–N1–Mn1	115.6 (2)
N4–Mn1–Cl1	93.42 (6)		

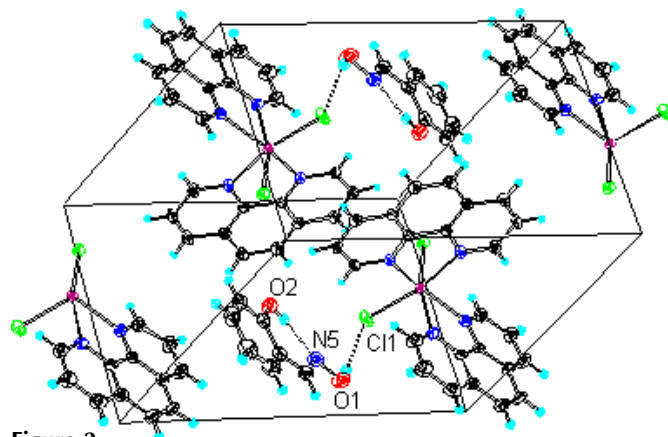


Figure 2
A packing diagram of the title compound. Hydrogen bonds are indicated by dotted lines.

Table 2

Hydrogen-bonding geometry (Å, $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2–H2 \cdots N5	0.82	1.90	2.613 (4)	145
O1–H1 \cdots Cl1'	0.82	2.34	3.055 (3)	147

Symmetry code: (i) $2 - x, -y, -z$.

H atoms were placed in calculated positions, with O–H distances of 0.82 Å and C–H distances of 0.93 Å. The $U_{\text{iso}}(\text{H})$ values were constrained to be $1.5U_{\text{eq}}$ of the carrier atom for hydroxy H atoms and $1.2U_{\text{eq}}$ for all other H atoms. A rotating-group refinement was used for the hydroxy groups.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART* and *SAINT* (Siemens, 1994); data reduction: *SAINT* and *XPREP* in *SHELXTL* (Siemens, 1994); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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References

- Keeney, M. E., Osseo-Asare, K. & Woode, K. A. (1984). *Coord. Chem. Rev.* **59**, 141–201.
 Lawrence, G. D. & Sawyer, D. T. (1978). *Coord. Chem. Rev.* **27**, 173–193.
 Ruttigter, W. & Dismukes, G. C. (1997). *Chem. Rev.* **79**, 1–24.
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
 Siemens (1994). *SAINT* and *SHELXTL*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Siemens (1996). *SMART*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.