Chlorobis(1,10-phenanthroline)(trichloro-acetato)manganese(II)

Liang Chen, Xian-Wen Wang,* Fu-Ping Chen, Ying Chen and Jing-Zhong Chen

Faculty of Materials Science and Chemical Engineering, China University of Geoscience, Wuhan 430074, People's Republic of China

Correspondence e-mail: wangxw10108092@163.com

Key indicators

Single-crystal X-ray study
$T = 298$ K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.030
wR factor = 0.084
Data-to-parameter ratio = 18.7

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

In the title compound, [Mn(C$_2$Cl$_3$O$_2$)Cl(C$_{12}$H$_8$N$_2$)$_2$], the Mn^{II} ion exhibits a distorted octahedral geometry, with one O atom from a trichloroacetate anion and three N atoms from two 1,10-phenanthroline ligands occupying the equatorial plane, and one Cl$^-$ anion and one pyridyl N atom occupying the axial positions. In the crystal structure, complex molecules are linked by C—H···O and C—H···Cl intermolecular hydrogen bonds into layers. In addition, π–π interactions are observed.

Comment

Manganese complexes with carboxylate ligands have received extensive attention due to the fact that carboxylates are good candidates for the investigation of exchange-coupling inter-actions between adjacent metal ions (Soler *et al.*, 2004). Trichloroacetic acid, a type of carboxylate organic ligand, and its coordination chemistry, have received a great deal of attention (Dell'Amico *et al.*, 2000; Ng, 2004; Gomez-Segura *et al.*, 2005; Turta, Prodius *et al.*, 2004; Turta, Shova *et al.*, 2004; Dey *et al.*, 2004). However, there are only a few reports on transition metal complexes with trichloroacetate and rigid chelating organic ligands such as 1,10-phenanthroline or 2,2-bipyridine (de Boer *et al.*, 2005; Yin *et al.*, 2005). We report here the crystal structure of the title compound, (I).

(I)
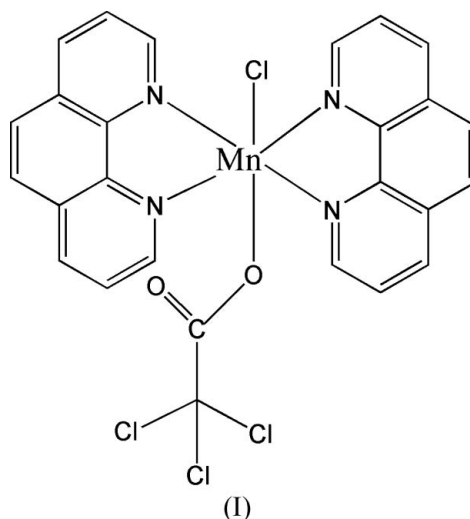
The Mn atom in compound (I) is six-coordinated, by one Cl$^-$ anion, one O atom from a trichloroacetate anion and four N atoms from two 1,10-phenanthroline ligands (Fig. 1). The three *trans* angles at Mn1 (Table 1) indicates a distorted octahedral geometry of the Mn atom.

In the crystal structure, the molecules are linked by C—H···O and C—H···Cl intermolecular hydrogen-bonding
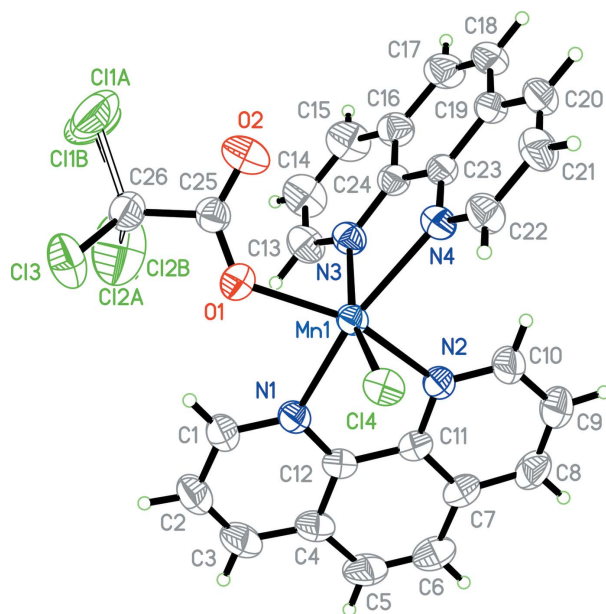
Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering scheme. Both components of the disordered Cl atoms are shown.

interactions (Table 2) into a two-dimensional network, as shown in Fig. 2. The molecular packing is further stabilized by π–π interactions between the 1,10-phenanthroline ring systems of screw-related molecules; the distance between the centroids of the N1-pyridine ring at (x, y, z) and the N2-pyridine ring at the symmetry position $(1 - x, -\frac{1}{2} + y, \frac{1}{2} - z)$ is 3.695 (1) Å.

Experimental

MnCl$_2$·4H$_2$O (0.20 g, 1.0 mmol) and Cl$_3$CCOOH (0.32 g, 2.0 mmol) were dissolved in a water–ethanol mixture (1:1 v/v; 20 ml). The mixture was stirred for *ca* 30 min at 333 K and then 1,10-phenanthroline monohydrate (0.198 g, 1.0 mmol) was added. The mixture was further stirred for another 1 h at 333 K, then filtered, and the resultant filtrate was left to stand for slow evaporation at room temperature. Yellow single crystals of (I) were obtained after a period of 7 d (yield 85%). Analysis, calculated for C$_{26}$H$_{16}$Cl$_4$MnN$_4$O$_2$: C 50.93, H 2.63, N 9.14%; found: C 51.01, H 2.66, N 9.07%.

Crystal data

[Mn(C$_2$Cl$_3$O$_2$)Cl(C$_{12}$H$_8$N$_2$)$_2$]	$Z = 4$
$M_r = 613.17$	$D_x = 1.560$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 18.1550$ (5) Å	$\mu = 0.95$ mm^{-1}
$b = 10.6381$ (3) Å	$T = 298$ (2) K
$c = 14.6858$ (4) Å	Block, yellow
$\beta = 112.992$ (2)°	$0.30 \times 0.25 \times 0.20$ mm
$V = 2611.02$ (13) Å^3	

Data collection

Bruker SMART CCD APEX-II area-detector diffractometer	24639 measured reflections
	6585 independent reflections
ω scans	5462 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.019$
$T_{min} = 0.735$, $T_{max} = 0.827$	$\theta_{max} = 28.5°$
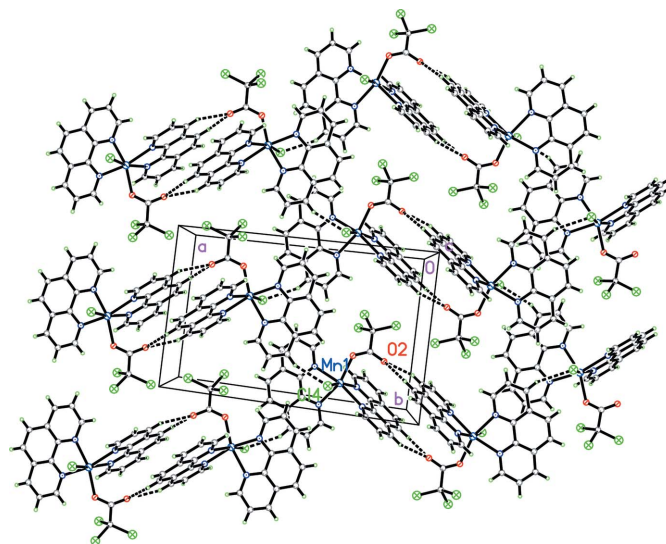
Figure 2
A packing diagram for (I), viewed along the c axis. Hydrogen bonds are shown as dashed lines. Only one disorder component is shown.

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0398P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.030$	$+ 0.8491P]$
$wR(F^2) = 0.084$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
6585 reflections	$\Delta\rho_{max} = 0.31$ e Å^{-3}
353 parameters	$\Delta\rho_{min} = -0.31$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Mn1—O1	2.1378 (12)	Mn1—N2	2.3109 (14)
Mn1—N3	2.2876 (14)	Mn1—N1	2.3131 (13)
Mn1—N4	2.3033 (13)	Mn1—Cl4	2.4374 (5)
O1—Mn1—N3	84.94 (5)	N4—Mn1—N1	161.37 (5)
O1—Mn1—N4	110.18 (5)	N2—Mn1—N1	71.54 (5)
N3—Mn1—N4	72.10 (5)	O1—Mn1—Cl4	100.71 (4)
O1—Mn1—N2	152.53 (5)	N3—Mn1—Cl4	163.26 (4)
N3—Mn1—N2	86.46 (5)	N4—Mn1—Cl4	91.18 (4)
N4—Mn1—N2	91.77 (5)	N2—Mn1—Cl4	94.96 (4)
O1—Mn1—N1	83.89 (5)	N1—Mn1—Cl4	98.28 (4)
N3—Mn1—N1	97.98 (5)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···Cl4[i]	0.93	2.71	3.565 (2)	154
C18—H18···O2[ii]	0.93	2.54	3.334 (3)	144
C20—H20···O2[ii]	0.93	2.43	3.255 (3)	148

Symmetry codes: (i) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x, -y + 2, -z$.

Atoms Cl1 and Cl2 are disordered and were modelled with split positions having site-occupancy factors of 0.604 (17) and 0.396 (17). The C—Cl distances involving the disordered atoms were restrained to be equal. H atoms were positioned geometrically and treated as riding, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXL97*.

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