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Aquabis(2,4-dichloro-6-formylphenolato-κ^2O,O')(*N*,*N*′-dimethylformamide-κO)-manganese(II)

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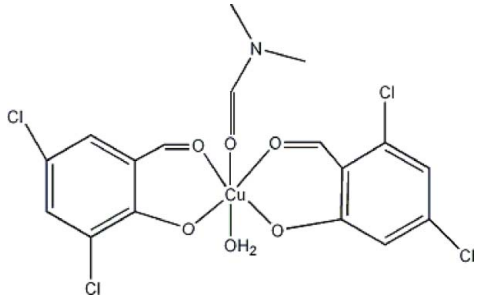
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.013 Å; R factor = 0.077; wR factor = 0.229; data-to-parameter ratio = 14.0.

In the title compound, $[Mn(C_7H_3Cl_2O_2)_2(C_3H_7NO)(H_2O)]$, the Mn^{II} atom is octahedrally coordinated by four O atoms from two bidentate 2,4-dichloro-6-formylphenolate ligands forming the equatorial plane, and by one O atom from a water molecule and one O atom from *N*,*N*-dimethylformamide *trans* in axial positions. A pseudo-dimer is constructed through O—H···O, O—H···Cl and slipped π–π stacking (with a centroid-to-centroid distance of 3.692 Å and interplanar distance of 3.47 Å, giving an offset angle of 20°) interactions. Short Cl···Cl interactions may help in stabilizing the packing.

Related literature

For related literature, see: Cohen *et al.* (1964); Desiraju (1989); Mathews *et al.* (1991); Willey *et al.* (1994); Zaman *et al.* (2004); Zhang *et al.* (2007); Zordan *et al.* (2005).



Experimental

Crystal data

$[Mn(C_7H_3Cl_2O_2)_2(C_3H_7NO)(H_2O)]$
M_r = 526.04
Monoclinic, $P2_1/c$
a = 10.479 (2) Å
b = 8.9988 (18) Å
c = 22.561 (5) Å
β = 92.51 (3)°
V = 2125.4 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.16 mm^{-1}
T = 293 (2) K
0.47 × 0.34 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.612, T_{max} = 0.761
10253 measured reflections
3770 independent reflections
2353 reflections with $I > 2\sigma(I)$
R_{int} = 0.062

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.077
$wR(F^2)$ = 0.229
S = 1.08
3770 reflections
270 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6···O1^{i}	0.85 (2)	2.19 (6)	2.874 (8)	138 (8)
O6—H6A···O3^{i}	0.85 (6)	2.02 (6)	2.748 (7)	143 (9)
O6—H6···Cl1^{i}	0.85 (2)	2.67 (5)	3.426 (7)	149 (8)
O6—H6A···Cl3^{i}	0.85 (6)	2.73 (6)	3.442 (6)	142 (8)

Symmetry code: (i) $-x, -y, -z+1$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2214).

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supplementary materials

Acta Cryst. (2007). E**63**, m2107 [**doi:10.1107/S1600536807033417**]

Aquabis(2,4-dichloro-6-formylphenolato-κ^2O,O')(*N*,*N*'-dimethylformamide-κO)manganese(II)

G.-Z. Li, S.-H. Zhang, Z. Liu and L.-X. Jin

Comment

Interest in packing arrangements of halogenated compounds goes back many years to what Schmidt called the 'chloro effect', where the presence of chloro substituents on aromatic compounds frequently results in stacking arrangements with a short (*ca* 4 Å) crystallographic axis (Cohen *et al.*, 1964; Zordan *et al.*, 2005; Desiraju, 1989; Zaman *et al.*, 2004; Zhang *et al.*, 2007). The title compound, (I), contains the dichloride ligand 3,5-Dichloro-2-hydroxy-benzaldehyde, with two Cl atoms accessible at the periphery of each ligand.

In compound (I), the Mn^{II} atom is octahedrally coordinated by four O atoms from two bidentate 3,5-Dichloro-2-hydroxy-benzaldehyde ligands forming the equatorial plane, one O atom from H_2O and one O atom from *N*,*N*-Dimethyl-formamide *trans* on axial positions, forming a slightly distorted octahedral geometry (Fig. 1). The O—H···O hydrogen bonds result in the formation of pseudo dimers through inversion centres (Fig. 2). Within the dimer, there are slippest π-π stacking between the C1—C6 and C8—C13 rings with centroid-to-centroid distance of 3.692 and interplanar distance of 3.47 giving an offset angle of 20°. Some O—H···Cl interactions are also present in these dimers (Table 1). The shortest Cl–Cl contacts, 3.573 (8) Å, (Mathews & Manohar, 1991; Willey *et al.*, 1994) observed between Cl2 and $Cl3^{i}$ [symmetry codes: (i) *x*, 1/2 + *y*, −1/2 + *z*] might help in stabilizing the crystal packing.

Experimental

A ethanol solution (30 ml) containing 3,5-Dichloro-2-hydroxy-benzaldehyde (0.382 g, 2 mmol) was dropwise added to an aqueous solution containing amino-methanesulfonic acid(0.222 g, 2 mmol) and sodium hydroxide (0.080 g, 2 mmol) with stirred during 10 min. After stirring for 1 h, an aqueous solution of manganese chloride (0.396 g, 2 mmol) was added to the resulting solution and stirred for 2 h. The yellow-green solid compound was separated out and dissolved by *N*,*N*-Dimethyl-formamide, then the yellow-green solution was filtrated. After 15 days, colorless crystals were produced from the filtrate (yield: 48.6%, based on Mn).

Figures

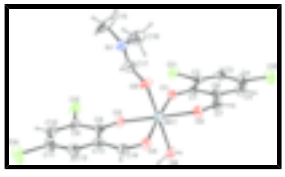

Fig. 1. Molecular view of (I) with the atom labelling scheme. Ellipsoids are drawn at the 30% probability level. H atoms are represented as small sphere of arbitrary radii.

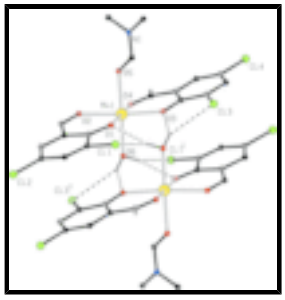

Fig. 2. View of the pseudo dimer formed by O–H···O and O–H···Cl hydrogen bonding interaction. H atoms not involved in H bonds have been omitted for clarity. [Symmetry code: (i) −*x*, −*y*, 1 − *z*].

Aquabis(2,4-dichloro-6-formylphenolato-κ^2*O*,*O*')(*N*,*N*'-dimethylformamide-\ κ*O*)manganese(II)

Crystal data

$[Mn(C_7H_3Cl_2O_2)_2(C_3H_7NO)(H_2O)]$	F_{000} = 1060
M_r = 526.04	D_x = 1.644 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 264 reflections
a = 10.479 (2) Å	θ = 1.8–25.1°
b = 8.9988 (18) Å	μ = 1.16 mm^{-1}
c = 22.561 (5) Å	T = 293 (2) K
β = 92.51 (3)°	Block, colorless
V = 2125.4 (8) Å^3	0.47 × 0.34 × 0.25 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3770 independent reflections
Radiation source: fine-focus sealed tube	2353 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.062
T = 293(2) K	θ_{max} = 25.1°
φ and ω scans	θ_{min} = 1.8°
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −12→12
T_{min} = 0.612, T_{max} = 0.761	k = −10→10
10253 measured reflections	l = −26→17

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.077	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.229	$w = 1/[\sigma^2(F_o^2) + (0.0588P)^2 + 19.7821P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.08	$(\Delta/\sigma)_{max}$ = 0.001
3770 reflections	$\Delta\rho_{max}$ = 1.38 e Å^{-3}
270 parameters	$\Delta\rho_{min}$ = −0.42 e Å^{-3}
3 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.0004 (7)	0.0754 (9)	0.3582 (4)	0.0342 (19)
C2	−0.1247 (7)	0.1196 (9)	0.3346 (4)	0.0367 (19)
C3	−0.1756 (8)	0.0685 (11)	0.2826 (4)	0.045 (2)
H3	−0.2553	0.1028	0.2689	0.054*
C4	−0.1109 (8)	−0.0350 (12)	0.2488 (4)	0.049 (2)
C5	0.0103 (9)	−0.0785 (11)	0.2674 (4)	0.052 (2)
H5	0.0562	−0.1437	0.2444	0.062*
C6	0.0644 (7)	−0.0244 (11)	0.3210 (4)	0.039 (2)
C7	0.1920 (9)	−0.0777 (11)	0.3359 (4)	0.049 (2)
H7	0.2285	−0.1370	0.3074	0.058*
C8	0.2317 (8)	0.1811 (10)	0.5888 (4)	0.0371 (19)
C9	0.1969 (8)	0.2856 (10)	0.6319 (4)	0.041 (2)
C10	0.2705 (9)	0.3182 (11)	0.6815 (4)	0.052 (2)
H10	0.2419	0.3870	0.7086	0.063*
C11	0.3880 (9)	0.2491 (13)	0.6916 (4)	0.056 (3)
C12	0.4278 (8)	0.1482 (11)	0.6519 (4)	0.049 (2)
H12	0.5065	0.1021	0.6588	0.058*
C13	0.3533 (7)	0.1108 (10)	0.6003 (4)	0.0366 (19)
C14	0.4044 (7)	0.0009 (10)	0.5623 (4)	0.039 (2)
H14	0.4804	−0.0433	0.5759	0.047*
C15	0.3455 (15)	0.6253 (13)	0.4451 (8)	0.113 (6)
H15A	0.3114	0.6656	0.4083	0.169*
H15B	0.4196	0.6810	0.4581	0.169*
H15C	0.2821	0.6314	0.4744	0.169*
C16	0.4926 (12)	0.4435 (15)	0.4035 (6)	0.085 (4)
H16A	0.5162	0.3412	0.4090	0.127*

H16B	0.5617	0.5061	0.4175	0.127*
H16C	0.4743	0.4623	0.3621	0.127*
C17	0.3068 (9)	0.3661 (12)	0.4519 (4)	0.053 (3)
H17	0.2331	0.3898	0.4713	0.064*
Cl1	−0.2081 (2)	0.2480 (3)	0.37414 (12)	0.0573 (7)
Cl2	−0.1816 (3)	−0.1083 (4)	0.18426 (12)	0.0761 (9)
Cl3	0.0525 (2)	0.3769 (3)	0.61989 (13)	0.0652 (8)
Cl4	0.4803 (3)	0.2921 (5)	0.75486 (14)	0.0919 (12)
Mn1	0.19920 (11)	0.04952 (15)	0.46251 (6)	0.0371 (4)
N1	0.3796 (8)	0.4755 (9)	0.4368 (4)	0.057 (2)
O1	0.0414 (5)	0.1221 (7)	0.4086 (2)	0.0382 (14)
O2	0.2581 (5)	−0.0552 (7)	0.3810 (3)	0.0472 (16)
O3	0.1589 (5)	0.1547 (7)	0.5430 (2)	0.0389 (14)
O4	0.3610 (5)	−0.0425 (7)	0.5142 (3)	0.0417 (14)
O5	0.3276 (6)	0.2326 (7)	0.4424 (3)	0.0521 (17)
O6	0.0987 (5)	−0.1542 (7)	0.4855 (3)	0.0408 (14)
H6	0.095 (8)	−0.165 (11)	0.5228 (7)	0.061*
H6A	0.031 (5)	−0.187 (11)	0.468 (3)	0.061*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.031 (4)	0.035 (5)	0.037 (5)	0.005 (4)	0.004 (3)	0.003 (4)
C2	0.031 (4)	0.031 (5)	0.049 (5)	0.001 (4)	0.007 (4)	0.011 (4)
C3	0.035 (4)	0.049 (6)	0.051 (6)	0.002 (4)	−0.006 (4)	0.013 (5)
C4	0.041 (5)	0.065 (7)	0.040 (5)	−0.005 (5)	0.001 (4)	0.000 (5)
C5	0.052 (5)	0.052 (6)	0.052 (6)	0.004 (5)	0.002 (4)	−0.008 (5)
C6	0.029 (4)	0.054 (6)	0.035 (5)	0.003 (4)	0.005 (3)	−0.001 (4)
C7	0.048 (5)	0.055 (7)	0.044 (5)	0.014 (5)	0.007 (4)	−0.008 (5)
C8	0.035 (4)	0.037 (5)	0.039 (5)	−0.007 (4)	0.006 (4)	0.000 (4)
C9	0.039 (5)	0.032 (5)	0.052 (5)	−0.005 (4)	0.004 (4)	−0.007 (4)
C10	0.056 (6)	0.044 (6)	0.058 (6)	−0.008 (5)	0.009 (5)	−0.012 (5)
C11	0.046 (5)	0.080 (8)	0.041 (5)	−0.025 (5)	0.004 (4)	−0.007 (5)
C12	0.037 (5)	0.057 (7)	0.052 (6)	−0.008 (4)	0.002 (4)	0.002 (5)
C13	0.029 (4)	0.043 (5)	0.038 (5)	−0.004 (4)	0.004 (3)	0.005 (4)
C14	0.025 (4)	0.038 (5)	0.054 (6)	0.004 (4)	0.002 (4)	0.005 (4)
C15	0.113 (12)	0.025 (7)	0.200 (17)	0.012 (7)	0.011 (11)	−0.009 (8)
C16	0.076 (8)	0.066 (9)	0.113 (10)	−0.004 (7)	0.028 (7)	0.010 (7)
C17	0.044 (5)	0.059 (8)	0.057 (6)	0.002 (5)	0.000 (4)	0.001 (5)
Cl1	0.0431 (12)	0.0522 (16)	0.0768 (17)	0.0185 (11)	0.0043 (11)	0.0017 (13)
Cl2	0.0656 (17)	0.103 (3)	0.0579 (17)	−0.0133 (16)	−0.0124 (13)	−0.0144 (16)
Cl3	0.0547 (15)	0.0574 (17)	0.0835 (19)	0.0128 (13)	0.0027 (13)	−0.0221 (14)
Cl4	0.0704 (19)	0.142 (3)	0.0612 (18)	−0.034 (2)	−0.0145 (14)	−0.0208 (19)
Mn1	0.0270 (6)	0.0398 (8)	0.0445 (8)	0.0008 (6)	0.0025 (5)	−0.0040 (6)
N1	0.052 (5)	0.037 (5)	0.083 (6)	0.001 (4)	0.005 (4)	0.000 (4)
O1	0.033 (3)	0.040 (4)	0.042 (3)	0.009 (3)	0.005 (3)	0.000 (3)
O2	0.038 (3)	0.054 (4)	0.049 (4)	0.015 (3)	0.001 (3)	−0.009 (3)
O3	0.027 (3)	0.044 (4)	0.046 (3)	0.002 (3)	0.000 (2)	−0.008 (3)

O4	0.029 (3)	0.043 (4)	0.054 (4)	0.008 (3)	0.001 (3)	−0.006 (3)
O5	0.047 (4)	0.033 (4)	0.077 (5)	−0.008 (3)	0.011 (3)	0.002 (3)
O6	0.025 (3)	0.043 (4)	0.055 (4)	−0.006 (3)	0.005 (3)	−0.005 (3)

Geometric parameters (Å, °)

C1—O1	1.271 (9)	C12—H12	0.9300
C1—C6	1.423 (11)	C13—C14	1.429 (12)
C1—C2	1.443 (11)	C14—O4	1.222 (10)
C2—C3	1.348 (12)	C14—H14	0.9300
C2—Cl1	1.722 (9)	C15—N1	1.409 (14)
C3—C4	1.398 (13)	C15—H15A	0.9600
C3—H3	0.9300	C15—H15B	0.9600
C4—C5	1.377 (13)	C15—H15C	0.9600
C4—Cl2	1.735 (9)	C16—N1	1.457 (14)
C5—C6	1.399 (12)	C16—H16A	0.9600
C5—H5	0.9300	C16—H16B	0.9600
C6—C7	1.446 (12)	C16—H16C	0.9600
C7—O2	1.222 (10)	C17—O5	1.241 (12)
C7—H7	0.9300	C17—N1	1.300 (13)
C8—O3	1.279 (9)	C17—H17	0.9300
C8—C9	1.411 (12)	Mn1—O3	2.107 (6)
C8—C13	1.435 (11)	Mn1—O1	2.114 (5)
C9—C10	1.363 (12)	Mn1—O4	2.179 (6)
C9—Cl3	1.733 (9)	Mn1—O2	2.180 (6)
C10—C11	1.390 (14)	Mn1—O5	2.187 (6)
C10—H10	0.9300	Mn1—O6	2.188 (6)
C11—C12	1.353 (14)	O6—H6	0.85 (2)
C11—Cl4	1.733 (9)	O6—H6A	0.85 (6)
C12—C13	1.414 (12)		
O1—C1—C6	125.7 (7)	N1—C15—H15A	109.5
O1—C1—C2	120.6 (7)	N1—C15—H15B	109.5
C6—C1—C2	113.7 (7)	H15A—C15—H15B	109.5
C3—C2—C1	123.0 (8)	N1—C15—H15C	109.5
C3—C2—Cl1	119.3 (6)	H15A—C15—H15C	109.5
C1—C2—Cl1	117.6 (7)	H15B—C15—H15C	109.5
C2—C3—C4	121.3 (8)	N1—C16—H16A	109.5
C2—C3—H3	119.4	N1—C16—H16B	109.5
C4—C3—H3	119.4	H16A—C16—H16B	109.5
C5—C4—C3	119.1 (8)	N1—C16—H16C	109.5
C5—C4—Cl2	120.1 (8)	H16A—C16—H16C	109.5
C3—C4—Cl2	120.8 (7)	H16B—C16—H16C	109.5
C4—C5—C6	119.8 (9)	O5—C17—N1	125.4 (10)
C4—C5—H5	120.1	O5—C17—H17	117.3
C6—C5—H5	120.1	N1—C17—H17	117.3
C5—C6—C1	123.0 (8)	O3—Mn1—O1	100.2 (2)
C5—C6—C7	114.9 (8)	O3—Mn1—O4	83.7 (2)
C1—C6—C7	122.1 (8)	O1—Mn1—O4	175.3 (2)
O2—C7—C6	128.6 (8)	O3—Mn1—O2	175.1 (2)

O2—C7—H7	115.7	O1—Mn1—O2	83.7 (2)
C6—C7—H7	115.7	O4—Mn1—O2	92.6 (2)
O3—C8—C9	121.1 (8)	O3—Mn1—O5	89.3 (2)
O3—C8—C13	123.8 (8)	O1—Mn1—O5	96.9 (2)
C9—C8—C13	115.1 (7)	O4—Mn1—O5	85.7 (2)
C10—C9—C8	123.7 (8)	O2—Mn1—O5	87.2 (3)
C10—C9—Cl3	119.0 (7)	O3—Mn1—O6	93.2 (2)
C8—C9—Cl3	117.3 (7)	O1—Mn1—O6	91.3 (2)
C9—C10—C11	120.2 (9)	O4—Mn1—O6	85.8 (2)
C9—C10—H10	119.9	O2—Mn1—O6	89.7 (2)
C11—C10—H10	119.9	O5—Mn1—O6	170.8 (2)
C12—C11—C10	119.2 (9)	C17—N1—C15	122.3 (10)
C12—C11—Cl4	121.2 (8)	C17—N1—C16	119.0 (9)
C10—C11—Cl4	119.6 (8)	C15—N1—C16	118.3 (11)
C11—C12—C13	122.0 (9)	C1—O1—Mn1	130.0 (5)
C11—C12—H12	119.0	C7—O2—Mn1	127.0 (6)
C13—C12—H12	119.0	C8—O3—Mn1	130.2 (5)
C12—C13—C14	116.8 (8)	C14—O4—Mn1	127.2 (5)
C12—C13—C8	119.8 (8)	C17—O5—Mn1	125.4 (6)
C14—C13—C8	123.4 (8)	Mn1—O6—H6	112 (6)
O4—C14—C13	128.2 (8)	Mn1—O6—H6A	126 (6)
O4—C14—H14	115.9	H6—O6—H6A	110 (7)
C13—C14—H14	115.9		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O6—H6···O1^{i}	0.85 (2)	2.19 (6)	2.874 (8)	138 (8)
O6—H6A···O3^{i}	0.85 (6)	2.02 (6)	2.748 (7)	143 (9)
O6—H6···Cl1^{i}	0.85 (2)	2.67 (5)	3.426 (7)	149 (8)
O6—H6A···Cl3^{i}	0.85 (6)	2.73 (6)	3.442 (6)	142 (8)

Symmetry codes: (i) −*x*, −*y*, −*z*+1.

Fig. 1

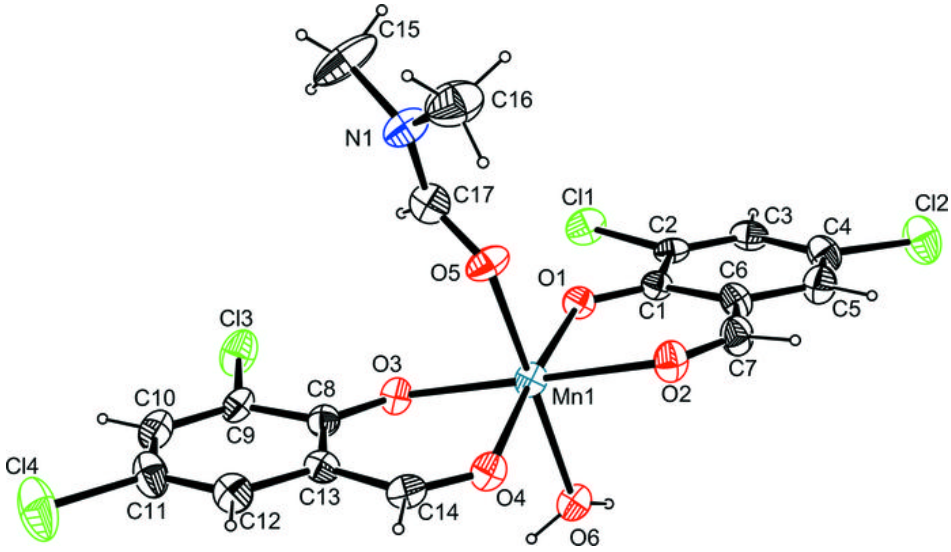
C15
C16
N1
C17
O5
Cl1
C2
C3
C4
Cl2
O1
C1
C6
C5
Cl3
O3
C8
Mn1
O2
C7
C10
C9
Cl4
C11
C12
C13
C14
O4
O6

Fig. 2