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Acta Cryst. (1992). **C48**, 364–366

Diaqua-*N,N'*-trimethylenebis(5'-chlorosalicylideneaminato)manganese(III) Perchlorate Monohydrate: a Schiff-Base Complex Linked into Infinite Spirals by Hydrogen and π Bonds

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(Received 2 July 1991; accepted 15 August 1991)

Abstract. [Mn(C₁₇H₁₈Cl₂N₂O₄)]ClO₄·H₂O, $M_r = 557.65$, orthorhombic, $P2_12_12_1$, $a = 13.650$ (1), $b = 20.784$ (9), $c = 7.773$ (2) Å, $V = 2205$ (2) Å³, $Z = 4$, $D_x = 1.679$ Mg m⁻³, $F(000) = 1136$, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.992$ mm⁻¹, $T = 293$ K, $R = 0.057$, 1565 unique reflexions [$I \geq 2\sigma(I)$]. The role of manganese in photosynthesis has focused attention on how individual complex molecules are able to associate to form multinuclear structures. In the title molecule the Schiff base forms a square-planar belt around manganese with Mn—N distances of 2.029 (8), 2.044 (8) Å and Mn—O distances of 1.894 (6), 1.866 (6) Å. Two capping water molecules at 2.230 (6) and 2.206 (7) Å complete a distorted octahedral arrangement. Infinite spirals of the complex, generated by a screw axis along *c*, are linked by hydrogen bonds from coordinated water to phenoxy oxygen and π bonds between overlapping aromatic rings. The hydrogen-bonding scheme is completed by lattice water, which, in addition to bridging alternate members of the spiral *via* coordinated water, forms two links to perchlorate anions.

Experimental. The compound was prepared following the method of Boucher (Boucher & Coe, 1975). To a solution of the ligand (2.85 mmol) in ethanol Mn(ClO₄)₂·6H₂O (2.85 mmol) in methanol was added. The mixture was stirred in air for five days before filtering. On standing the filtrate at room temperature, small dark-green crystals were deposited. These were separated by filtration and washed

with ethanol and diethyl ether before being dried in air.

Crystal dimensions 0.4 × 0.3 × 0.1 mm, Rigaku AFC-6S diffractometer, graphite-monochromated Mo *K*α radiation, unit-cell dimensions from setting angles of 25 accurately centered reflexions ($21.0 \leq 2\theta \leq 30.8^\circ$), ω - 2θ scan mode, ω scan width ($0.94 + 0.30\tan\theta$)° and scan speed of 4° min⁻¹ with up to 2 additional scans of weak reflexions [$I < 10\sigma(I)$], $0 \leq h \leq 16$, $0 \leq k \leq 24$, $0 \leq l \leq 9$, $0 \leq \theta \leq 25^\circ$, 2264 reflexions measured, 2252 unique 1565 observed [$I \geq 2\sigma(I)$], intensity standards (25 $\bar{1}$, 15 $\bar{1}$, 1 $\bar{8}0$) measured every 150 reflexions showed 24% decline, Lp, decomposition and absorption corrections applied (ψ -scan method, min. and max. transmission 0.91 and 1.00 respectively), *MITHRIL* (Gilmore, 1984) used to solve the phase problem, all non-H atoms found in Fourier map, H atoms placed in chemically reasonable positions except for those attached to water, which were located from a ΔF map and refined, full-matrix least-squares refinement based on *F* using *TEXSAN* crystallographic software (Molecular Structure Corporation, 1985), final $R = 0.057$ ($wR = 0.058$, $w = 1/[\sigma^2(F_o) + (0.03F_o)^2]$); a parallel refinement of the inverted structure gave a final $R = 0.058$ and $wR = 0.059$, indicating that the correct enantiomer had been chosen, anisotropic thermal parameters for heavier atoms, isotropic for hydrogen. Maximum fluctuation in final ΔF map in range -0.47 to 0.46 e Å⁻³, maximum Δ/σ 0.08. Scattering factors from Cromer & Waber (1974), computation

Table 1. Positional parameters and B_{eq} (\AA^2) for non-H atoms
$$B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq}
Mn(1)	0.2014 (1)	0.42446 (7)	0.0248 (2)	2.25 (6)
Cl(1)	0.7013 (3)	0.3833 (2)	0.3124 (5)	5.6 (2)
Cl(2)	-0.2209 (2)	0.6019 (2)	0.3131 (5)	5.8 (2)
Cl(3)	-0.0677 (2)	0.2917 (2)	-0.5285 (5)	4.4 (2)
O(1)	0.3248 (4)	0.4660 (3)	0.023 (1)	2.7 (3)
O(2)	0.1491 (5)	0.5074 (3)	0.025 (1)	3.0 (3)
O(3)	0.2005 (6)	0.4168 (4)	-0.2614 (8)	3.2 (4)
O(4)	0.1932 (6)	0.4218 (4)	0.3081 (8)	4.0 (4)
O(5)	-0.0978 (8)	0.2684 (7)	-0.690 (1)	8.8 (8)
O(6)	-0.1383 (8)	0.2734 (5)	-0.404 (1)	7.8 (7)
O(7)	-0.0545 (8)	0.3567 (4)	-0.541 (2)	9.9 (8)
O(8)	0.0230 (6)	0.2592 (4)	-0.486 (2)	6.2 (5)
O(9)	0.2109 (7)	0.3139 (4)	-0.483 (1)	4.5 (5)
N(2)	0.2704 (6)	0.3381 (4)	0.038 (1)	2.7 (4)
N(6)	0.0632 (6)	0.3866 (4)	0.023 (1)	2.5 (4)
C(1)	0.3567 (8)	0.3323 (5)	0.096 (1)	2.8 (5)
C(3)	0.2202 (9)	0.2776 (4)	-0.011 (2)	4.0 (6)
C(4)	0.116 (1)	0.2733 (5)	0.052 (2)	3.9 (6)
C(5)	0.0469 (8)	0.3189 (5)	-0.032 (2)	4.1 (6)
C(7)	-0.0112 (7)	0.4178 (5)	0.077 (1)	2.9 (5)
C(8)	0.4255 (7)	0.3824 (5)	0.135 (1)	2.3 (2)
C(9)	0.4105 (8)	0.4458 (5)	0.091 (1)	2.6 (5)
C(10)	0.4864 (8)	0.4906 (5)	0.110 (2)	3.0 (6)
C(11)	0.5757 (9)	0.4708 (7)	0.176 (2)	4.0 (7)
C(12)	0.589 (1)	0.4082 (6)	0.228 (1)	3.5 (6)
C(13)	0.517 (1)	0.3639 (5)	0.205 (2)	3.5 (6)
C(14)	-0.0128 (8)	0.4851 (5)	0.123 (1)	3.1 (6)
C(15)	0.0655 (8)	0.5268 (5)	0.091 (1)	2.6 (5)
C(16)	0.0506 (8)	0.5924 (6)	0.128 (1)	3.4 (6)
C(17)	-0.036 (1)	0.6151 (6)	0.194 (2)	3.7 (6)
C(18)	-0.1100 (9)	0.5719 (7)	0.229 (1)	3.9 (7)
C(19)	-0.100 (1)	0.5087 (6)	0.193 (2)	3.9 (6)

carried out on a Digital VAX station 3520. Literature survey performed *via* the Cambridge Structural Database 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 title molecule, including atomic labelling, is displayed in Fig. 1 and the hydrogen-bonding scheme in Fig. 2.

Related literature. The extended hydrogen- and π -bonded spirals seen in the title compound may be contrasted with dimers formed by $[\text{Mn}_2(\text{salpd})_2(\text{EtOH})_3(\text{H}_2\text{O})](\text{BPh}_4)_2 \cdot \text{EtOH}$, where *salpd* = propane-1,3-diylbis(salicylideneaminato) (Ashmawy, Beagley, McAuliffe, Parish & Pritchard, 1990). It is probable that the bulkier EtOH ligands prevent efficient stacking of the molecules; however, at 5.21 (1) \AA the Mn...Mn distance is comparable to that in the present case [5.17 (1) \AA].

We thank the Xunta de Galicia for a grant.

* Lists of structure factors, anisotropic thermal parameters, bond lengths and bond angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54600 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL0491]

Table 2. Selected bond lengths (\AA), angles ($^\circ$) and hydrogen-bond geometry (\AA , $^\circ$)

Mn(1)—O(1)	1.894 (6)	Mn(1)—O(4)	2.206 (7)	
Mn(1)—O(2)	1.866 (6)	Mn(1)—N(2)	2.029 (8)	
Mn(1)—O(3)	2.230 (6)	Mn(1)—N(6)	2.044 (8)	
O(1)—Mn(1)—O(2)	85.4 (3)	O(1)—Mn(1)—N(2)	89.4 (3)	
O(1)—Mn(1)—O(3)	91.8 (3)	O(2)—Mn(1)—N(6)	90.2 (3)	
O(1)—Mn(1)—O(4)	93.6 (3)			
<i>A</i> —H... <i>B</i>	<i>A</i> —H	H... <i>B</i>	<i>A</i> ... <i>B</i>	<i>A</i> —H... <i>B</i>
O(3)—H(3C)...O(9)	0.951	1.935	2.75 (1)	142
O(3)—H(3D)...O(2) ⁱ	0.942	2.163	3.08 (1)	163
O(9)—H(9B)...O(8)	0.7 (1)	2.1 (1)	2.81 (1)	165 (15)
O(4)—H(4C)...O(9) ⁱⁱ	0.944	2.329	2.78 (1)	109
O(4)—H(4D)...O(1) ⁱⁱⁱ	0.959	1.972	2.88 (1)	157
O(9)—H(9A)...O(6) ^{iv}	0.7 (1)	2.3 (1)	2.88 (2)	136 (13)

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

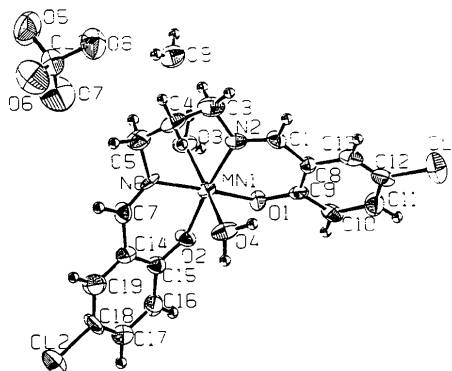


Fig. 1. The title molecule drawn using ORTEPII (Johnson, 1976).

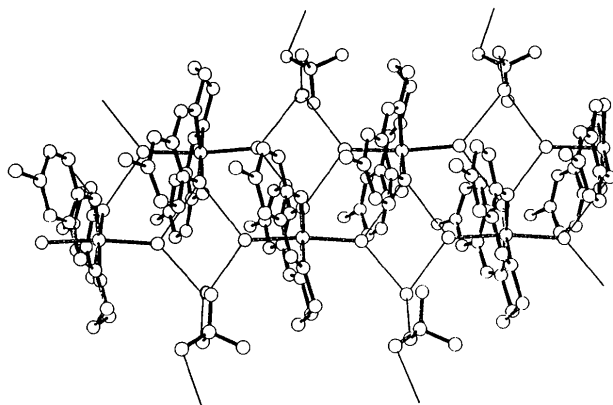


Fig. 2. The hydrogen-bonding scheme.

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Acta Cryst. (1992). **C48**, 366–368

2-(Chlorodimethylstannyl)-4-methylbenzenesulfonyl Pyrrolidide

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(Received 25 June 1991; accepted 19 August 1991)

Abstract. [Sn(C₁₃H₂₀ClNO₂S)], *M_r* = 408.51, orthorhombic, *Pbca*, *a* = 7.596 (5), *b* = 18.654 (13), *c* = 23.447 (19) Å, *V* = 3322 (4) Å³, *Z* = 8, *D_x* = 1.633 Mg m⁻³, λ(Mo *K*α) = 0.71073 Å, μ = 1.82 mm⁻¹, *F*(000) = 1632, *T* = 291 (1) K, final *R* = 0.047 for 1652 unique observed [*F* ≥ 4.0σ(*F*)] diffractometer data. This is the first case of complexation of a triorganotin halide by an SO₂ group forming an intramolecular pentacoordination at the Sn atom. The five atoms bound to Sn form a distorted trigonal bipyramid with Cl and O in the apical positions [Sn—Cl 2.438 (3), Sn—O 2.529 (6) Å, Cl—Sn—O 171.4 (2)°] and the C atoms in the equatorial plane [Sn—C 2.113 (8), 2.13 (1), 2.171 (8) Å, C—Sn—C 107.4 (3), 124.1 (3), 122.2 (4)°]. The O—Sn—C and Cl—Sn—C angles are in the range from 75.3 (2) (ring angle) to 99.1 (3)° and the bond angles at S are

in the range from 105.5 (4) to 118.5 (4)°. Short intermolecular contacts around the free O at S indicate weak hydrogen bonds.

Experimental. The crystals were obtained from hexane/dichloromethane, 3:1, m.p. 419 K. A well developed crystal platelet of size ~0.06 × 0.13 × 0.35 mm was used. Its quality was checked with optical polarizing microscopy. The crystal was mounted on a glass fibre. *D_m* was not determined. Intensity data were collected with ω/2θ scans, constant scan speed, scan width 0.8° + dispersion (0.02° steps, 1 s per step). A modified Hilger & Watts diffractometer (Lange & Burzlaff, 1991) with graphite-monochromated Mo *K*α radiation was used. The lattice parameters were determined from a least-squares fit of 20 reflections with 2θ_{max} = 17.7°. Three standard reflections (200, 060, 0,0,10) were recorded every 100 reflections and an intensity loss of up to 7% was detected during data collection; 13 110

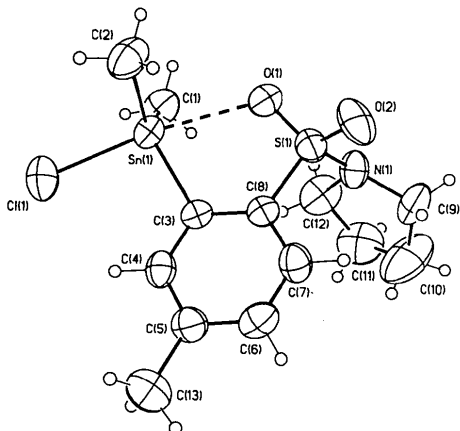


Fig. 1. General view (*SHELXTL-Plus* graphic) of the asymmetric unit, showing the atom-numbering scheme. Anisotropic ellipsoids represent 50% probability boundaries.

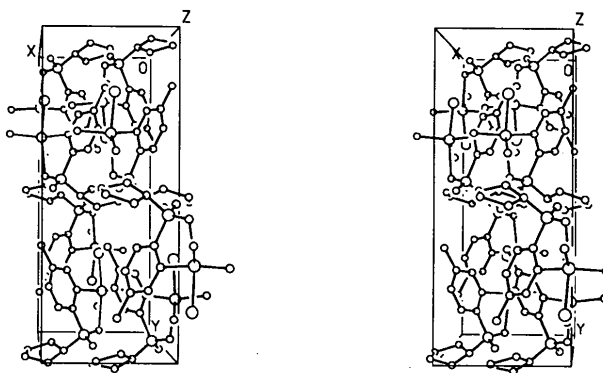


Fig. 2. Stereoscopic view (*SHELXTL-Plus* graphic) of the unit cell. H atoms are omitted for clarity.