

The serendipitous discovery of the crystal structure of seven-member chelate of hydrated tris-(*cis*-vinyl-1,2 – bis (diphenylphosphineoxide))manganese(ii)perchlorate complex, $\text{Mn}(\text{cis-Ph}_2\text{P}(\text{O})\text{CH}=\text{CHP}(\text{O})\text{Ph}_2)_3(\text{ClO}_4)_2 \cdot (\text{H}_2\text{O})$ and the subsequent synthesis and characterization of related complexes

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ABSTRACT

A hydrated tris-(*cis*-vinyl-1,2 –bis(diphenylphosphineoxide))manganese(II)perchlorate, $[\text{Mn}(\text{cis-Ph}_2\text{P}(\text{O})\text{CH}=\text{CHP}(\text{O})\text{Ph}_2)_3](\text{ClO}_4)_2 \cdot (\text{H}_2\text{O})$ crystal structure has been determined and characterized. The complex was serendipitously discovered while attempting to obtain crystal of its *bis*-ligand analogue. In its structure the the monoclinic crystal is unique in coordination chemistry in that it contains a rare strained seven-member chelate ring system. Six or five coordinate complex are common but not seven-membered ring systems. The complex was then synthesized which shows to be a white air stable complex.

Key words: Coordinate bond, Seven-member chelate, Hydrate octahedral, Crystal data.

INTRODUCTION

Our interest in the coordination chemistry of manganese(II)perchlorate with alkylphosphine oxides¹⁻⁵ has led into crystallographically determining the spatial orientation of the hydrated tris-(vinyl-1,2–bis(diphenylphosphineoxide))manganese(II)perchlorate, $[\text{Mn}(\text{cis-Ph}_2\text{P}(\text{O})\text{CH}=\text{CHP}(\text{O})\text{Ph}_2)_3](\text{ClO}_4)_2 \cdot (\text{H}_2\text{O})$ complex. The complex itself was not target complex in our study and was thus serendipitously discovered. Efforts were spent attempting to crystallize its *bis*-complex derivative, $[\text{Mn}(\text{cis-Ph}_2\text{P}(\text{O})\text{CH}=\text{CHP}(\text{O})\text{Ph}_2)_2(\text{H}_2\text{O})_2](\text{ClO}_4)_2$, a white complex melting at 192-194°C. The *bis*-complex is a 2:1 electrolyte in nitrobenzene and is thus ionic. In fact two water molecules appear to be coordinated to the metal centre giving a pseudo-octahedral environment about the manganese(II).

The *bis*-complex did not crystallize as expected but the *tris*-complex, $[\text{Mn}(\text{cis-Ph}_2\text{P}(\text{O})\text{CH}=\text{CHP}(\text{O})\text{Ph}_2)_3](\text{ClO}_4)_2 \cdot (\text{H}_2\text{O})$, emerged. This drew our attention and interest to synthesize and characterize the new complex. The *tris*-complex is in many ways not similar in structure to the *bis*-complex. The two water molecules coordinated to the metal centre in the *bis*-complex forming part of the inner-sphere metal complex are absent and replaced by a third vinyl-1,2 –bis(diphenylphosphineoxide) ligand. A lone water molecule is found in the outer-sphere. Affinity for water is characteristic for perchlorate complexes. The main difference lies in the position of the water molecule in relation to the metal centre. The *tris*-complex is not only saturated but there is steric hindrance to direct coordination arising from the ligand moieties.

RESULTS AND DISCUSSION

The $[\text{Mn}(\text{cis-Ph}_2\text{P}(\text{O})\text{CH}=\text{CHP}(\text{O})\text{Ph}_2)_3](\text{ClO}_4)_2 \cdot (\text{H}_2\text{O})$ was re-crystallized in 1:3 chloroform to methanol mixture at room temperature to yield crystals suitable for single crystal x-ray analysis. The crystal, Fig 1, is a monomer with three vinyl-1,2-bis(diphenylphosphineoxide) ligands exhibiting chelate mode of coordination. The monoclinic crystal structure of hydrated tris-(vinyl-1,2-bis(diphenylphosphineoxide)) manganese(II) perchlorate, $[\text{Mn}(\text{cis-Ph}_2\text{P}(\text{O})\text{CH}=\text{CHP}(\text{O})\text{Ph}_2)_3](\text{ClO}_4)_2 \cdot (\text{H}_2\text{O})$, complex was been determined by single-crystal x-ray diffraction. The compound crystallizes with space group $P_{21/n}$ and unit cell dimensions: $a = 13.88(2)\text{\AA}$, $b = 26.515(6)\text{\AA}$, $c = 20.961(6)\text{\AA}$; and $\alpha = 90^\circ$, $\beta = 89.97(2)^\circ$, $\gamma = 90^\circ$, Table 1.

The complex shows three *cis*- $\text{Ph}_2\text{P}(\text{O})\text{CH}=\text{CHP}(\text{O})\text{Ph}_2$ ligand moiety coordinate to the metal centre in a chelate fashion forming a perfect octahedral bond to the manganese(II). The molecule has a monomer structure with a two-fold axis. Ligands with *cis*-CH=CH- vinyl backbone⁶ tends to chelate in their coordination. Such coordination results from steric restriction to movement about the C-C double bond.

The $[\text{Mn}(\text{cis-Ph}_2\text{P}(\text{O})\text{CH}=\text{CHP}(\text{O})\text{Ph}_2)_3](\text{ClO}_4)_2 \cdot (\text{H}_2\text{O})$ crystal, Fig. 1, of dimensions

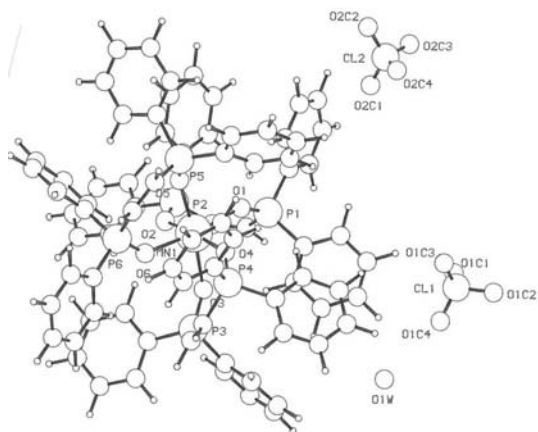


Fig. 1: The Crystal Structure of $[\text{Mn}(\text{cis-Ph}_2\text{P}(\text{O})\text{CH}=\text{CHP}(\text{O})\text{Ph}_2)_3](\text{ClO}_4)_2 \cdot (\text{H}_2\text{O})$

0.40x0.40x0.44mm was sealed in a Lindemann tube for X-ray diffraction. The X-ray diffraction data^{7,8} was collected and processed on a Nicolet R3m/V diffractometers at 293K using graphite monochromated Mo-K α radiation ($\lambda = 0.71073\text{\AA}$). And ω -2 θ scan mode with ω scan width 0.6 below $K\alpha_1$, and 0.6 above $K\alpha_1$. The unit cell constants were determined from the setting angles of 25 accurately centred reflections ($4.0 \leq \theta \leq 14.0^\circ$). The structure was determined using the standard heavy atom technique and refined by full matrix least square procedures with all non-hydrogen atoms refined anisotropically and all hydrogen atoms constrained to chemically reasonable positions with shared

Table 1: Crystal details and details of the structure refinements for $[\text{Mn}(\text{cis-Ph}_2\text{P}(\text{O})\text{CH}=\text{CHP}(\text{O})\text{Ph}_2)_3](\text{ClO}_4)_2 \cdot (\text{H}_2\text{O})$

Parameter	Crystal Details
Empirical Formula	$\text{MnCl}_2\text{P}_6\text{C}_{78}\text{H}_{68}\text{O}_{15}$
Space Group	$P_{21/n}$
Description	Monoclinic
$a / \text{\AA}$	13.88(2)
$b / \text{\AA}$	26.515(6)
$c / \text{\AA}$	20.961(6)
$\alpha / ^\circ$	90
$\beta / ^\circ$	89.97(2)
$\gamma / ^\circ$	90
$V / \text{\AA}^3$	7715.5
Z	4
h	-13 to 12
k	0 to 30
l	0 to 24
$\theta / ^\circ$	0 to 24
Total Reflections	11152
R_{int}	0.037
Unique Reflections	8268
Observed Reflections	4431, $[F \geq 5\sigma(F)]$
Final R	0.078
R_w	0.083
w	$2.077 / \sigma^2(F) + 0.00060F^2$
Max. Fluctuations	
In Fourier Map (ΔF) / $e \text{\AA}^{-3}$	-0.6 to 0.5e
Maximum Δ / σ	0.84

σ = standard deviation; Δ = shift

Table 2: Crystal data for [Mn(*cis*-Ph₂P(O)CH=CHP(O)Ph₂)₃](ClO₄)₂·(H₂O) Fractional Atomic Coordinates and Vibrational Parameters (Å²) for non-Hydrogen Atoms

Atom	x	y	z	Ueq
Mn(1)	0.7722(1)	0.08722(6)	0.23991(7)	0.0424(4)
P(1)	0.5796(2)	0.0157(1)	0.2991(1)	0.0545(9)
O(1)	0.6732(5)	0.0431(3)	0.2981(3)	0.055(2)
C(1)	0.5617(8)	-0.0230(4)	0.2284(5)	0.074(4)
C(101)	0.5626(9)	-0.0289(4)	0.3636(5)	0.056(3)
C(102)	0.6398(10)	-0.0266(5)	0.4120(6)	0.081(4)
C(103)	0.6401(11)	-0.0611(6)	0.4608(7)	0.110(5)
C(104)	0.5724(13)	-0.0981(7)	0.4601(7)	0.115(6)
C(105)	0.5027(9)	-0.0659(5)	0.4139(8)	0.110(5)
C(106)	0.5011(9)	-0.0659(5)	0.3661(7)	0.084(4)
C(107)	0.4769(8)	0.0575(4)	0.3041(6)	0.053(3)
C(108)	0.4633(9)	0.0932(6)	0.2580(6)	0.083(5)
C(109)	0.3879(12)	0.1261(6)	0.2618(8)	0.097(5)
C(110)	0.3263(10)	0.1228(6)	0.3128(8)	0.092(5)
C(111)	0.3396(11)	0.0899(7)	0.3593(7)	0.104(5)
C(112)	0.4137(10)	0.0553(5)	0.3552(6)	0.087(4)
P(2)	0.7557(2)	-0.0355(1)	0.1928(1)	0.0589(9)
O(2)	0.7919(5)	0.0175(2)	0.1879(3)	0.053(2)
C(2)	0.6245(8)	-0.0416(4)	0.1894(5)	0.070(4)
C(201)	0.7978(10)	-0.0679(4)	0.2624(5)	0.068(4)
C(202)	0.8857(13)	-0.0576(5)	0.2863(7)	0.113(6)
C(203)	0.9219(14)	-0.0830(8)	0.3372(9)	0.166(8)
C(204)	0.8725(17)	-0.1179(7)	0.3701(8)	0.127(7)
C(205)	0.7815(16)	-0.1273(5)	0.3482(7)	0.106(7)
C(206)	0.7440(10)	-0.1039(5)	0.2954(7)	0.083(4)
C(207)	0.7979(9)	-0.0716(5)	0.1249(5)	0.064(4)
C(208)	0.8083(14)	-0.1230(6)	0.1256(8)	0.153(9)
C(209)	0.8453(16)	-0.1477(7)	0.0721(8)	0.165(10)
C(210)	0.8733(10)	-0.1222(7)	0.0223(8)	0.097(5)
C(211)	0.8674(13)	-0.0724(7)	0.0212(7)	0.125(5)
C(212)	0.8283(12)	-0.0470(5)	0.0723(7)	0.119(7)
P(3)	0.6527(2)	0.1492(1)	0.1192(2)	0.068(1)
O(3)	0.6666(5)	0.1092(3)	0.1695(3)	0.068(2)
C(3)	0.6968(8)	0.2126(4)	0.1383(6)	0.075(4)
C(301)	0.7143(8)	0.1324(5)	0.0448(6)	0.062(4)
C(302)	0.7811(13)	0.0942(5)	0.0452(6)	0.71(4)
C(303)	0.8327(10)	0.0844(6)	-0.0079(7)	0.099(5)
C(304)	0.8196(12)	0.1127(6)	-0.0628(7)	0.103(6)
C(305)	0.7522(12)	0.1501(6)	-0.0643(7)	0.099(6)
C(306)	0.6983(9)	0.1617(5)	-0.0098(6)	0.077(4)
C(307)	0.5286(10)	0.1613(6)	0.1004(6)	0.082(5)
C(308)	0.4825(14)	0.1271(9)	0.0649(9)	0.158(9)
C(309)	0.3832(18)	0.1303(12)	0.0507(12)	0.190(13)
C(310)	0.3401(19)	0.1717(11)	0.0740(11)	0.164(13)
C(311)	0.3813(17)	0.2114(9)	0.1125(11)	0.167(10)
C(312)	0.4767(14)	0.2034(7)	0.1224(8)	0.132(7)
P(4)	0.7243(2)	0.2124(1)	0.2706(2)	0.0623(9)
O(4)	0.7352(5)	0.1571(3)	0.2835(3)	0.069(2)
C(4)	0.7227(9)	0.2324(5)	0.1912(5)	0.088(4)

Table 2, Cont...

C(401)	0.6116(9)	0.2348(5)	0.3037(5)	0.066(4)
C(402)	0.5522(10)	0.2040(5)	0.3457(6)	0.079(4)
C(403)	0.4746(12)	0.2226(8)	0.3702(7)	0.112(6)
C(404)	0.4383(14)	0.2677(11)	0.3505(11)	0.145(10)
C(405)	0.4882(16)	0.2959(9)	0.3078(10)	0.149(8)
C(406)	0.5726(11)	0.2802(6)	0.2831(7)	0.100(5)
C(407)	0.8189(9)	0.2481(4)	0.3063(8)	0.071(5)
C(408)	0.8283(11)	0.2467(6)	0.3707(9)	0.107(5)
C(409)	0.9071(16)	0.2709(8)	0.4008(9)	0.148(8)
C(410)	0.9693(19)	0.2988(8)	0.3628(16)	0.182(12)
C(411)	0.9596(18)	0.3000(9)	0.2982(13)	0.157(10)
C(412)	0.8867(13)	0.2755(5)	0.2701(9)	0.107(7)
P(5)	0.9506(2)	0.0940(1)	0.3534(1)	0.0500(8)
O(5)	0.8893(4)	0.0716(2)	0.3035(3)	0.049(2)
C(5)	1.0143(7)	0.1489(4)	0.3265(5)	0.053(3)
C(501)	0.8827(9)	0.1145(4)	0.4205(5)	0.055(3)
C(502)	0.7861(9)	0.1026(4)	0.4238(5)	0.057(3)
C(503)	0.7304(10)	0.1162(5)	0.4752(7)	0.080(4)
C(504)	0.7714(12)	0.1419(5)	0.5252(8)	0.086(5)
C(505)	0.8683(15)	0.1539(5)	0.5235(7)	0.102(6)
C(506)	0.9245(9)	0.1405(5)	0.4711(6)	0.076(4)
C(507)	1.0432(8)	0.0514(4)	0.3784(6)	0.054(3)
C(508)	1.0962(10)	0.0282(5)	0.3312(6)	0.082(4)
C(509)	1.1645(10)	-0.0072(5)	0.3462(8)	0.095(5)
C(510)	1.1876(10)	-0.0176(5)	0.4058(9)	0.093(5)
C(511)	1.1376(12)	0.0035(6)	0.4537(7)	0.102(5)
C(512)	1.0638(9)	0.0390(5)	0.4395(6)	0.080(4)
P(6)	0.9852(2)	0.1420(1)	0.1927(1)	0.0480(8)
O(6)	0.8824(4)	0.1262(2)	0.1863(3)	0.048(2)
C(6)	1.0250(7)	0.1657(4)	0.2676(5)	0.051(3)
C(601)	1.0128(8)	0.1911(4)	0.1367(6)	0.053(3)
C(602)	0.9523(9)	0.1988(4)	0.0846(6)	0.0634
C(603)	0.9733(9)	0.233956)	0.0391(6)	0.071(4)
C(604)	1.0551(12)	0.2616(5)	0.0430(7)	0.092(5)
C(605)	1.1155(11)	0.2551(5)	0.0921(8)	0.102(5)
C(606)	1.0948(9)	0.2203(5)	0.1394(6)	0.085(4)
C(607)	1.0611(8)	0.0901(4)	0.1758(4)	0.042(3)
C(608)	1.1582(11)	0.0924(5)	0.1820(6)	0.088(5)
C(609)	1.2167(10)	0.0517(7)	0.1707(7)	0.094(5)
C(610)	1.1771(13)	0.0050(6)	0.1555(6)	0.091(5)
C(611)	1.0780(13)	0.0029(5)	0.1478(6)	0.090(5)
C(612)	1.0205(8)	0.0449(5)	0.1584(5)	0.064(4)
O(1W)	0.2206(11)	0.2375(6)	0.2745(7)	0.241(7)
Cl(1)	0.1962(3)	0.1971(1)	0.4705(2)	0.080(1)
O(1C1)	0.1790(7)	0.1455(3)	0.4595(5)	0.118(4)
O(1C2)	0.1228(8)	0.2162(4)	0.5097(6)	0.167(5)
O(1C3)	0.2780(9)	0.2059(5)	0.5048(7)	0.205(6)
O(1C4)	0.1933(12)	0.2230(5)	0.4156(5)	0.205(7)
Cl(2)	0.7199(5)	0.1266(3)	0.7409(3)	0.153(2)
O(2C1)	0.7021(14)	0.1288(7)	0.6809(7)	0.286(10)
O(2C2)	0.8054(10)	0.1162(5)	0.7679(7)	0.199(6)
O(2C3)	0.6557(14)	0.0900(11)	0.7623(13)	0.385(15)
O(2C4)	0.7133(19)	0.1716(8)	0.7586(15)	0.431(17)

isotropic parameters, to a final $R = 0.078$, $R_w = 0.083$. Tables 1-4 give the crystal details.

The Synthesis of the $[\text{Mn}(\text{cis-Ph}_2\text{P}(\text{O})\text{CH}=\text{CHP}(\text{O})\text{Ph}_2)_3](\text{ClO}_4)_2 \cdot (\text{H}_2\text{O})$ complex

The complex was prepared by mixing hydrated manganese(II) perchlorate (ca. 1.05 mol) with slightly excess of three mole (ca. 3.10 mol) of *cis*-vinyl-1,2-bis(diphenylphosphineoxide) ligand in freshly distilled toluene under argon atmosphere. The mixture was stirred for at least 14 days upon which a white solid was isolated by the standard Schlenk method.

Anal. Calc. for $[\text{Mn}(\text{cis-Ph}_2\text{P}(\text{O})\text{CH}=\text{CHP}(\text{O})\text{Ph}_2)_3](\text{ClO}_4)_2 \cdot (\text{H}_2\text{O})$: C, 60.9; H, 4.7; Mn, 3.6; Found: C, 59.9; H, 4.3; Mn, 3.4%. The complex is air stable though its preparation requires strict adherence to inert and dry conditions. The hydrated manganese(II) perchlorate used for this preparation was achieved by using 50g finely ground hexahydrated manganese(II) perchlorate which was heated *in vacuo* at 45°C for 4 hours and then at 60°C for 5 hours. The resulting solid cake was ground to a fine powder in a dry box under argon

environment, and further heated *in vacuo* at 100°C for 2 hours. Extra caution has to be taken as perchlorates readily exploded. The temperature was gradually raised to 80°C and held there for a further 4 hours. Typical analyses of the 'dry' $(\text{MnClO}_4)_2 \cdot (\text{H}_2\text{O})_2$ suitable for this reaction showed 0.04% w/w H_2O (by Karl Fischer method)⁹. Curiosity has made us synthesize the related chloro and bromo complexes, Table 4.

The $[\text{Mn}(\text{cis-Ph}_2\text{P}(\text{O})\text{CH}=\text{CHP}(\text{O})\text{Ph}_2)_3](\text{ClO}_4)_2 \cdot (\text{H}_2\text{O})$ complex melts at 196-198°C and has characteristic $\nu(\text{PO})$ nujol mull infrared frequencies split at 1190 and 1170 cm^{-1} . Free ligand has $\nu(\text{PO})$ at 1185 cm^{-1} a frequency split upon coordination to form complex. The complex has a room temperature magnetic moment of $5.87\mu_B$ a value consistent with the spin-only magnetic moment value of $5.92\mu_B$. The complex further shows an X-band solid state ESR spectra with a single absorption centred at ca. $g=2$ at room temperature while at -150°C in frozen chloroform glass the complex yields a spectra containing six-lines of equal intensity with a mean hyperfine constant of ca. 170 Gauss indicative of a high spin d^5 manganese(II) ion.

Table 3: Selected Bond Length(Å) between non-hydrogen atoms in $[\text{Mn}(\text{cis-Ph}_2\text{P}(\text{O})\text{CH}=\text{CHP}(\text{O})\text{Ph}_2)_3](\text{ClO}_4)_2 \cdot (\text{H}_2\text{O})$

Bonding Atoms	Bond Length / Å
Mn-O(1)	2.176(6)
Mn-O(2)	2.163(6)
Mn-O(3)	2.161(7)
Mn-O(4)	2.129(7)
Mn-O(5)	2.144(6)
Mn-O(6)	2.186(6)
O(1)-P(1)	1.489(7)
O(2)-P(2)	1.496(7)
O(3)-P(3)	1.508(8)
O(4)-P(4)	1.500(7)
O(5)-P(5)	1.474(7)
O(6)-P(6)	1.480(7)
Chelate Ring Carbons	
C(1)-C(2)	1.29(2)
C(3)-C(4)	1.30(2)
C(5)-C(6)	1.32(2)

Table 4: Selected Bond Angles(°) ring atoms in $[\text{Mn}(\text{cis-Ph}_2\text{P}(\text{O})\text{CH}=\text{CHP}(\text{O})\text{Ph}_2)_3](\text{ClO}_4)_2 \cdot (\text{H}_2\text{O})$

Bonding Atoms	Bond Angle / °
O(1)-Mn-O(2)	84.4(2)
O(3)-Mn-O(4)	84.0(0)
O(5)-Mn-O(6)	83.4(2)
Mn-O(1)-P(1)	145.2(4)
Mn-O(2)-P(2)	136.6(4)
Mn-O(3)-P(3)	139.0(4)
Mn-O(4)-P(4)	143.0(4)
Mn-O(5)-P(5)	143.3(4)
Mn-O(6)-P(6)	138.8(4)
O(1)-P(1)-C(1)	112.5(5)
O(2)-P(2)-C(2)	114.5(5)
O(3)-P(3)-C(3)	116.7(5)
O(4)-P(4)-C(4)	119.5(5)
O(5)-P(5)-C(5)	112.9(4)
O(6)-P(6)-C(6)	117.8(4)

Table 5: Physical and Chemical Characteristics of $[\text{Mn}(\text{ligand})_3]\text{X}_2$, where ligand is *cis*- $\text{Ph}_2\text{P}(\text{O})\text{CH}=\text{CHP}(\text{O})\text{Ph}_2$; $\text{X}=\text{Br}$, I and ClO_4^-

X	Colour	m.p/°C	$\mu_{\text{eff}}/\mu_{\text{B}}$	$\nu(\text{PO})/\text{i.r.}$ cm^{-1}	Elemental analyses(%)			
					C	H	P	Mn
Br	White	210-214	5.61	1188, 1170	62.6(62.4)*	4.5(4.4)	11.9(12.4)	3.5(3.7)
I	Beige	230-232	5.63	1188, 1168	59.2(58.8)	4.2(4.1)	10.8(11.1)	3.1(3.5)
ClO_4^-	White	196-198	5.87	1190(m), 1170(s)	59.9(60.9)	4.3(4.7)	-	3.4(3.6)

*calcd. Values; ClO_4^- complex is a hydrate; Free ligand has $\nu(\text{PO})$ at 1185 cm^{-1}

The $[\text{Mn}(\textit{cis}\text{-Ph}_2\text{P}(\text{O})\text{CH}=\text{CHP}(\text{O})\text{Ph}_2)_3](\text{ClO}_4)_2 \cdot (\text{H}_2\text{O})$ complex shows conductivity measurement of $57.4\text{ Scm}^2\text{mol}^{-1}$ in 10^{-3}M nitrobenzene indicating that it is ionic and is a 2:1 electrolyte. The chloro and bromo complex are 2:1 electrolyte and show similar chemical environments as in perchlorate complex.

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