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Dichlorobis[phosphonic tris(dimethylamide)]-manganese(II)

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(N–C) = 0.004 Å
R factor = 0.032
wR factor = 0.087
Data-to-parameter ratio = 18.0

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

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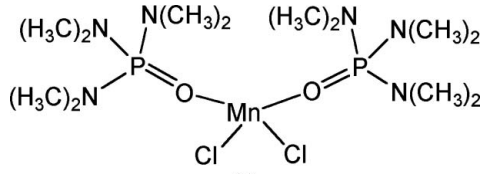
In the title compound, $[MnCl_2(C_6H_{18}N_3OP)]$, the Mn^{II} atom, on a twofold rotation axis, is bonded to two O atoms from two symmetry-related hexamethylphosphophosphoramide (HMPA) ligands and two Cl atoms in a distorted tetrahedral configuration.

Comment

There are numerous examples of hexamethylphosphoramide coordination complexes (Sinha *et al.*, 1982; Herrmann *et al.*, 1996; Bombieri *et al.*, 2001; Süss-Fink *et al.*, 2004). We present here the structure of the title compound, (I).



(I)

The asymmetric unit of (I) contains one-half of the title complex, with the other half generated by crystallographic twofold symmetry; the Mn1 atom lies on the twofold axis. The Mn^{II} atom is bonded to two O atoms from two symmetry-related HMPA ligands and two Cl atoms (Fig. 1). The coordination around Mn^{II} is distorted tetrahedral, with angles subtended at the Mn^{II} atom in the range 106.19 (4)–121.43 (4)° (Table 1). The molecular structure is similar to bis(HMPA)-dibromomagnesium (Allan *et al.*, 1998) and bis-(HMPA)-dichlorocadmium (Hiltunen *et al.*, 1982). Except for intramolecular C—H···O and C—H···N interactions (Table 2), no hydrogen bonds are observed in the crystal structure.

Experimental

$MnCl_2{\cdot}4H_2O$ and hexamethylphosphoramide in a molar ratio of 1:2 were mixed and dissolved in sufficient ethanol by heating to 373 K, at which temperature a clear solution resulted. After the reaction system was cooled slowly to room temperature, pink crystals of (I) were obtained by slow evaporation of the solvent.

Crystal data

$[MnCl_2(C_6H_{18}N_3OP)]$
M_r = 484.25
Monoclinic, $C2/c$
a = 23.400 (3) Å
b = 8.1922 (10) Å
c = 15.7447 (19) Å
β = 127.465 (2)°
V = 2395.6 (5) Å^3
Z = 4
D_x = 1.343 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1098 reflections
θ = 2.4–23.3°
μ = 0.93 mm^{-1}
T = 273 (2) K
Prism, pink
0.43 × 0.29 × 0.23 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.732, T_{max} = 0.808
6167 measured reflections
2163 independent reflections
2030 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 25.3°
$h = -28 \rightarrow 26$
$k = -9 \rightarrow 9$
$l = -18 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.087
S = 1.04
2163 reflections
120 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0494P)^2 + 1.6611P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Mn1—O1	2.0474 (13)	P1—N1	1.6256 (17)
Mn1—Cl1	2.3434 (7)	N1—C1	1.456 (3)
P1—O1	1.4885 (13)	N1—C2	1.462 (3)
O1^{i}—Mn1—O1	106.63 (8)	O1—P1—N2	115.09 (9)
O1—Mn1—Cl1^{i}	107.80 (4)	N1—P1—N2	104.48 (9)
O1—Mn1—Cl1	106.19 (4)	N3—P1—N2	108.19 (10)
Cl1^{i}—Mn1—Cl1	121.43 (4)	P1—O1—Mn1	146.84 (9)
O1—P1—N1	110.06 (9)	C1—N1—C2	112.46 (19)
O1—P1—N3	108.18 (8)	C1—N1—P1	120.20 (15)
N1—P1—N3	110.80 (10)	C2—N1—P1	126.05 (16)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1*B*···O1	0.96	2.54	3.015 (3)	111
C2—H2*B*···N2	0.96	2.51	3.005 (4)	112
C4—H4*B*···N3	0.96	2.53	2.995 (4)	110
C6—H6*B*···O1	0.96	2.50	2.980 (4)	111

The methyl H atoms were placed in calculated positions (C—H = 0.96 Å) and allowed to ride on their parent C atoms, with U_{iso}(H) = 1.5U_{eq}(C).

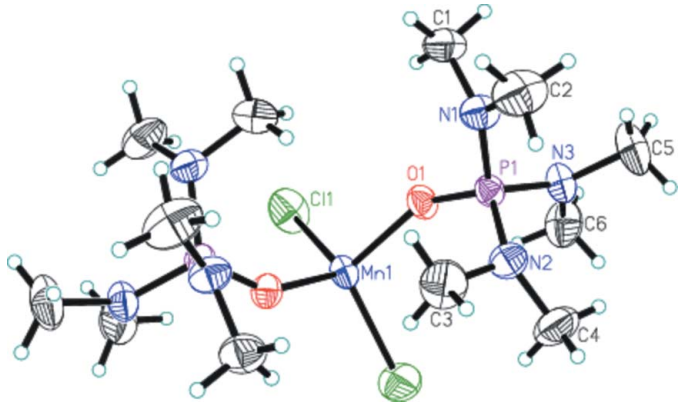


Figure 1
The structure of (I), showing 35% probability displacement ellipsoids. Unlabeled atoms are related by the symmetry code ($-x$, y, 1/2 − z).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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