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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1386). Services for accessing these data are described at the back of the journal.

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Tetrakis[1-(1-phenylcyclohexyl)piperidinium] Tetrachloromanganate(II) Dichloride

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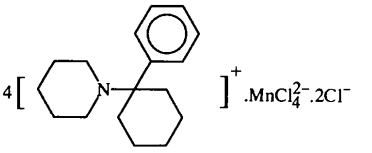
Abstract

The crystal structure of the title compound, $(C_{17}H_{26}N)_4[MnCl_4]Cl_2$, has been determined. The cation is 1-(1-phenylcyclohexyl)piperidinium. The metal atom is located on a $\bar{4}$ rotation axis with four Cl atoms coordinated to form a flattened tetrahedron.

† Deceased.

Comment

The present structural investigation was carried out because of our interest in the geometry and environment of the tetrachlorometallate(II) anion (Harlow *et al.*, 1974, 1975; Nelson & Simonsen, 1981). The structure of the title compound, (I), is isostructural with the Cu



(I)

and Ni salts. The four Cl atoms around each metal atom form a flattened tetrahedron with two equal large angles and four equal smaller angles: 121.10 (3) and 103.99 (1)° for Mn, 132.6 (1) and 99.3 (1)° for Cu, and 122.4 (1) and 103.4 (1)° for Ni (Nelson & Simonsen, 1981). Each Cl^- ion is hydrogen bonded to the N atoms of two symmetry-related 1-(1-phenylcyclohexyl)-piperidinium (PCP) cations [N···Cl 3.183 (2) Å].

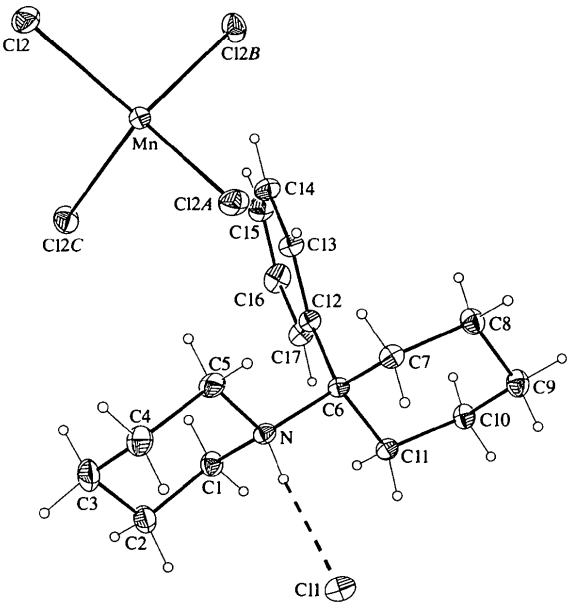


Fig. 1. A view of the title compound showing the atom-labeling scheme. Displacement ellipsoids are scaled to the 30% probability level. H atoms are drawn to an arbitrary scale.

Experimental

Crystals of the title compound were provided by Dr William J. Wells III.

Crystal data

$(C_{17}H_{26}N)_4[MnCl_4]Cl_2$
M_r = 1245.20

Mo $K\alpha$ radiation
λ = 0.71073 Å

Tetragonal
$P\bar{4}2_1c$
a = 15.747 (1) Å
c = 13.148 (1) Å
V = 3260.3 (4) Å^3
Z = 2
D_x = 1.268 Mg m^{-3}
D_m not measured

Cell parameters from 35 reflections
θ = 24.7–25.0°
μ = 0.492 mm^{-1}
T = 173 (2) K
Prism
0.80 × 0.70 × 0.46 mm
Colorless

Data collection

Siemens diffractometer
ω scan
Absorption correction: none
12 559 measured reflections
3237 independent reflections
2777 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

θ_{max} = 32.50°
$h = -23 \rightarrow 23$
$k = -23 \rightarrow 23$
$l = 0 \rightarrow 19$
4 standard reflections
every 96 reflections
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.089
S = 1.06
3237 reflections
181 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2 + 0.5476P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.04$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0125 (10)
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = 0.00 (3)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\sum_i\sum_j U^{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Mn	0	0	0	0.02046 (12)
Cl2	−0.11804 (3)	−0.05617 (4)	0.08839 (5)	0.03157 (13)
C1	0.34898 (15)	−0.09177 (14)	−0.2155 (2)	0.0273 (4)
C2	0.3723 (2)	−0.17418 (15)	−0.1616 (2)	0.0353 (5)
C3	0.3048 (2)	−0.1993 (2)	−0.0846 (2)	0.0415 (6)
C4	0.2916 (2)	−0.1269 (2)	−0.0101 (2)	0.0366 (5)
C5	0.26981 (14)	−0.04437 (15)	−0.0645 (2)	0.0293 (5)
C6	0.32578 (12)	0.06796 (13)	−0.19102 (14)	0.0193 (3)
C7	0.31490 (13)	0.13391 (13)	−0.10540 (15)	0.0233 (4)
C8	0.31498 (15)	0.22439 (14)	−0.1483 (2)	0.0290 (4)
C9	0.3960 (2)	0.24432 (15)	−0.2057 (2)	0.0314 (5)
C10	0.41190 (15)	0.17850 (14)	−0.2887 (2)	0.0266 (4)
C11	0.40945 (13)	0.08783 (14)	−0.2461 (2)	0.0221 (3)
C12	0.24903 (13)	0.06421 (13)	−0.26199 (14)	0.0220 (4)
C13	0.16673 (13)	0.07913 (15)	−0.2259 (2)	0.0271 (4)
C14	0.0971 (2)	0.0767 (2)	−0.2902 (2)	0.0342 (5)
C15	0.1082 (2)	0.0584 (2)	−0.3923 (2)	0.0383 (6)
C16	0.1888 (2)	0.0417 (2)	−0.4294 (2)	0.0384 (6)
C17	0.25884 (15)	0.0451 (2)	−0.3658 (2)	0.0294 (4)
Cl1	0	−1/2	−0.00298 (5)	0.02837 (14)
N	0.33815 (10)	−0.02079 (10)	−0.13987 (13)	0.0213 (3)

The labels of the x and y axes were chosen to minimize the absolute Flack parameter (Sheldrick *et al.*, 1985).

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXL*93 (Sheldrick, 1993). Program(s) used to refine structure: *SHELXL*93. Molecular graphics: *SHELXL*93. Software used to prepare material for publication: *SHELXL*93.

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Diiodotetrakis[tris(dimethylamino)-phosphine oxide-*O*]bismuth(III) Pentaiodide, $[BiI_2\{OP(NMe_2)_3\}_4][I_5]$

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Abstract

The title compound, $[BiI_2(C_6H_{18}N_3OP)_4](I_5)$, contains the pseudo-octahedral Bi^{III} cation $[BiI_2\{OP(NMe_2)_3\}_4]^+$, which has crystallographic $\bar{4}$ symmetry, and *trans* iodide ligands. The central I atom of the disordered $[I_5]^-$ anion is also situated on a site of $\bar{4}$ symmetry and the atoms of this anion form an infinite three-dimensional network, resulting in a 'cage'-type structure. The cations are situated within the 'cage' cavities.