Acta Cryst. (1985). C**41**, 1832–1833

Structure of Dichlorobis(triphenylphosphine oxide)manganese(II)

BY KOYCHI TOMITA

Instituto de Química de Araraquara, Caixa Postal 174, 14800 *Araraquara, SP, Brazil*

(*Received* 13 *June* 1985; *accepted* 15 *July* 1985)

Abstract. $[MnCl_2\{PO(C_6H_5)_3\}_2]$, $M_r = 682\cdot43$, orthorhombic, $Fdd2$, $a = 20\cdot990\ (3)$, $b = 33\cdot028\ (6)$, $c = 9\cdot756\ (8)$ Å, $V = 6763\ (6)$ Å^3, $Z = 8$, $D_m = 1\cdot36\ (1)$, $D_x = 1\cdot340\ (1)$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0\cdot71069$ Å, $\mu = 6\cdot16$ cm^{-1}, $F(000) = 2808$, $T = ca$ 293 K, $R = 0\cdot046$ for 1137 observed reflections. The Mn atom lies on a twofold axis and is tetrahedrally coordinated. The structure displays no unusual features.

Experimental. The title complex was synthesized by Goodgame & Cotton (1961) and characterized by Davolos (1978). D_m by flotation (CH_3OH + $CHBr_3$), pale-yellow plates, $0\cdot4 \times 0\cdot3 \times 0\cdot2$ mm, CAD-4 diffractometer, unit-cell dimensions from 25 reflections ($11 < \theta < 18°$), absorption ignored, $\theta < 23°$, $0 \le h \le 23$, $0 \le k \le 36$, $0 \le l \le 10$, three standard reflections showed no appreciable intensity variation, 1222 independent reflections, 1137 observed reflections [$I > 3\sigma(I)$], Lorentz and polarization corrections. Structure solved by *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and difference Fourier maps. Refinement by least squares using *SHELX*76 (Sheldrick, 1976), $\sum w(|F_o| - |F_c|)^2$ minimized, atomic coordinates, anisotropic thermal parameters and scale factor refined, isotropic thermal parameters for H atoms, final R 0·046, unit weights, maximum Δ/σ in final refinement cycle 0·04, maximum and minimum height in final difference Fourier map 0·3 and $-0\cdot5$ e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). Calculations performed on the VAX 11/780 computer at the Instituto de Física e Química de São Carlos, USP, Brazil. Positional and equivalent isotropic thermal parameters are listed in Table 1.* The atom-numbering scheme, bond lengths and angles are shown in Fig. 1. A view of the molecule is shown in Fig. 2 (Johnson, 1965).

Table 1. *Atomic coordinates* (×10^4 *excepting z coordinates of* C: ×10^3) *and equivalent isotropic thermal parameters* (*Hamilton,* 1959) *with e.s.d.'s in parentheses*

	x	y	z	B_{eq}(Å^2)
Mn	0	0	0	2·83 (5)
Cl	750 (1)	344 (1)	−1251 (4)	5·5 (1)
P	870 (1)	−709 (1)	1862 (3)	3·12 (7)
O	410 (3)	−397 (2)	1383 (7)	4·1 (2)
C(1)	1685 (4)	−552 (3)	168 (1)	3·3 (3)
C(2)	1931 (5)	−512 (3)	38 (1)	4·8 (4)
C(3)	2570 (5)	−386 (3)	23 (1)	5·4 (4)
C(4)	2944 (5)	−305 (3)	136 (1)	5·0 (4)
C(5)	2692 (5)	−340 (3)	266 (2)	5·6 (5)
C(6)	2055 (5)	−471 (3)	285 (1)	5·0 (4)
C(7)	731 (4)	−808 (3)	366 (1)	3·6 (3)
C(8)	391 (8)	−519 (4)	438 (1)	7·6 (6)
C(9)	300 (10)	−559 (5)	578 (2)	10·2 (8)
C(10)	530 (8)	−908 (5)	644 (1)	7·9 (6)
C(11)	859 (8)	−1197 (4)	573 (2)	6·9 (6)
C(12)	965 (6)	−1149 (3)	431 (1)	5·5 (4)
C(13)	792 (4)	−1169 (3)	91 (1)	3·6 (3)
C(14)	1315 (5)	−1440 (3)	76 (1)	5·1 (4)
C(15)	1246 (6)	−1787 (3)	−7 (2)	6·1 (5)
C(16)	670 (7)	−1865 (3)	−76 (1)	6·3 (5)
C(17)	146 (7)	−1590 (4)	−60 (1)	6·5 (5)
C(18)	208 (5)	−1238 (3)	24 (1)	4·5 (4)

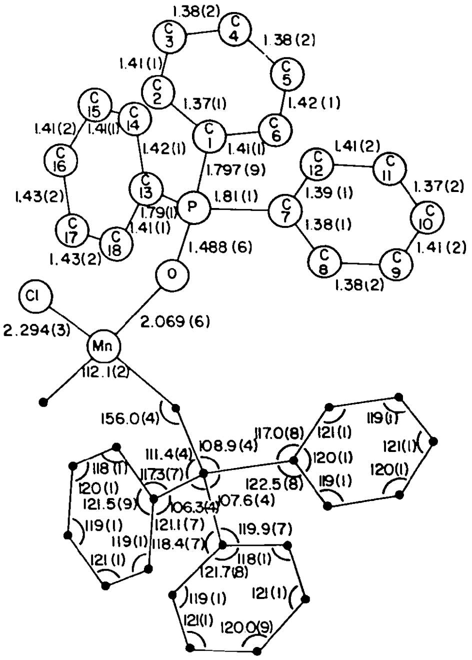

Fig. 1. Numbering scheme and bond lengths (Å) and angles (°) with e.s.d.'s in parentheses.

---

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42372 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/85/121832-02$01.50

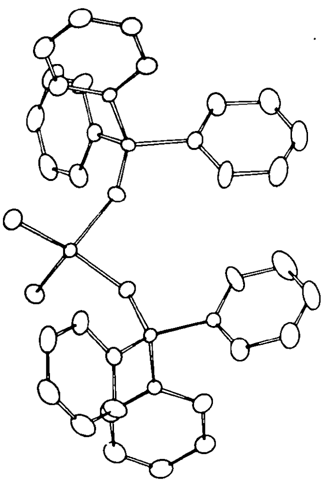

Fig. 2. View of the molecule.

This work was supported by FAPESP, CNPq and FINEP. We thank the Instituto de Física e Química de São Carlos, USP, Brazil, for computing facilities.

References

Davolos, M. R. (1978). Dissertation, Univ. of São Paulo, Brazil.

Goodgame, D. M. L. & Cotton, F. A. (1961). *J. Chem. Soc.* pp. 3735–3741.

Hamilton, W. C. (1959). *Acta Cryst.* **12**, 609–610.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Johnson, C. K. (1965). *ORTEP*. Report ORNL–3794. Oak Ridge National Laboratory, Tennessee.

Main, P., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Sheldrick, G. M. (1976). *SHELX76*. A program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1985). C**41**, 1833–1834

Structure of a Bicyclic Sulfur–Nitrogen–Carbon Heterocyclic Molecule

By A. W. Cordes

Department of Chemistry, University of Arkansas, Fayetteville, AR 72701, *USA*

and R. T. Oakley and René T. Boeré

*Guelph–Waterloo Centre for Graduate Work in Chemistry, Guelph Campus, Department of Chemistry and Biochemistry, University of Guelph, Guelph, Ontario, Canada N*1*G* 2*W*1

(*Received* 26 *June* 1985; *accepted* 30 *July* 1985)

Abstract. 7-Phenyl-1λ^4,3$\lambda^4\delta^2$,5λ^4-trithia-2,4,6,8,9-penta-azabicyclo[3.3.1]nonane, $C_7H_5N_5S_3$, $M_r = 255{\cdot}3$, monoclinic, $P2_1/n$, $a = 5{\cdot}958$ (1), $b = 22{\cdot}954$ (2), $c = 7{\cdot}428$ (1) Å, $\beta = 106{\cdot}25$ (1)°, $V = 975{\cdot}2$ (4) Å^3, $Z = 4$, $D_x = 1{\cdot}74$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 7{\cdot}00$ cm^{-1}, $F(000) = 520$, $T = 293$ K, $R = 0{\cdot}030$ for 1363 unique observed reflections. The planar SNCNS and SNSNS components of the bicyclic molecule make dihedral angles of 137·1 and 118·3° with the SNS bridging unit. The S—N bonds connecting the NSN fragment to the CN_3S_2 ring are much longer (1·728 Å) than those in the remainder of the molecule (1·546–1·630 Å).

Experimental. Compound prepared by reaction of *N,N,N'*-tris(trimethylsilyl)benzamidine with $S_3N_3Cl_3$. Crystals obtained from acetonitrile solutions. Yellow needle data crystal 0·60 × 0·16 × 0·14 mm mounted on a glass fiber. Density not measured. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using variable-speed ω–2θ scans. Unit cell determined from least-squares analysis of angle data for 25 reflections with $20 < 2\theta < 28°$. Analytical absorption correction based on crystal shape varied from 0·95 to 1·00. Data collected to $\sin\theta/\lambda$ of 0·59 Å^{-1}, $-7 \le h \le 7$, $0 \le k \le 27$, $0 \le l \le 8$. Three standard reflections (252, 1,12,0, $\bar{3}73$) varied ±4·1% over 16·9 h of data collection; anisotropic-drift correction applied. 1839 reflections measured, 1708 unique ($R_{\rm int} = 0{\cdot}01$), 345 reflections with $I < 3\sigma(I)$ considered unobserved. Solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier methods. Full-matrix least squares minimized $\sum w\Delta F^2$. H atoms refined isotropically, other

0108-2701/85/121833-02$01.50