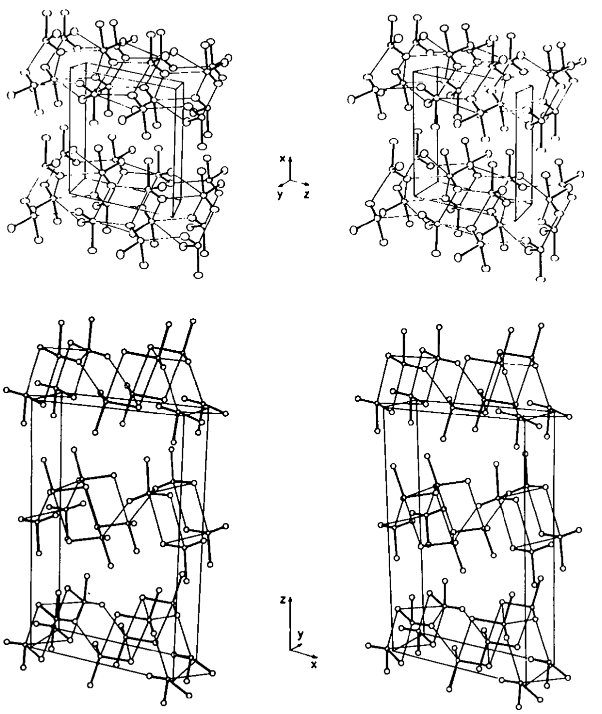

Fig. 1. Stereoplots of the crystal structures with short intermolecular contacts. Above: $SOCl_2$, below: $SOBr_2$.

an octagonal prism; $T_{max}/T_{min} = 4{\cdot}67$. Direct methods; full-matrix least-squares refinement based on F magnitudes, observed reflections only. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Isotropic extinction parameter $F_c' = F_c/(1 + 0{\cdot}002gF_c^2/\sin 2\theta)^{1/4}$ with $g = 0{\cdot}0013$ (2) for $SOBr_2$. Calculations with the program systems *XTLE* (Syntex, 1976) for $SOCl_2$, *SHELXTL* (Sheldrick, 1983) for $SOBr_2$ on Eclipse S/200 and S/140 minicomputers and *ORTEP*II (Johnson, 1976) on a TR 445 computer (Telefunken).

Additional experimental details are given in Table 1, the atomic parameters in Table 2, and interatomic distances and relevant angles in Table 3.* Fig. 1 shows the stereo plots of the crystal structures.

Related literature. Molecular geometries from microwave spectroscopy (Suzuki, Yamaguchi, Onda, Sakaizumi, Ohashi & Yamaguchi, 1981; Mata & Carballo, 1983) and electron diffraction (Gregory, Hargittai & Kolonits, 1976; Brunvoll, Hargittai & Rozsondai, 1982), crystal structure of SOF_2 (Mootz & Korte, 1984).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44703 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Brodalla, D., Mootz, D., Boese, R. & Osswald, W. (1985). *J. Appl. Cryst.* **18**, 316–319.

Brunvoll, J., Hargittai, I. & Rozsondai, B. (1982). *J. Mol. Struct.* **84**, 153–155.

Gregory, D., Hargittai, I. & Kolonits, M. (1976). *J. Mol. Struct.* **31**, 261–267.

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

Johnson, C. K. (1976). *ORTEP*II. ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Mata, F. & Carballo, N. (1983). *J. Mol. Struct.* **101**, 233–238.

Mootz, D. & Korte, L. (1984). *Z. Naturforsch. Teil B*, **39**, 1295–1299.

Pauling, L. (1973). *Die Natur der chemischen Bindung.* Weinheim: Verlag Chemie.

Sheldrick, G. M. (1983). *SHELXTL Users Manual.* Revision 4. Nicolet XRD Corporation, Madison, Wisconsin, USA.

Suzuki, S., Yamaguchi, M., Onda, M., Sakaizumi, T., Ohashi, O. & Yamaguchi, I. (1981). *J. Mol. Struct.* **73**, 41–47.

Syntex (1976). *XTL/XTLE Structure Determination System.* Syntex Analytical Instruments, Cupertino, California, USA.

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Structure of Bis(methanol)(*meso*-tetraphenylporphinato)manganese(III) Hexachloroantimonate Bis(tetrachloroethane) Solvate

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Abstract. $[Mn(C_{44}H_{30}N_4)(CH_3OH)_2][SbCl_6].2C_2H_2Cl_4$, $M_r = 1401{\cdot}93$, triclinic, $P\bar{1}$, $a = 11{\cdot}104$ (3), $b = 12{\cdot}086$ (6), $c = 12{\cdot}619$ (4) Å, $\alpha = 115{\cdot}33$ (3), $\beta = 104{\cdot}75$ (2), $\gamma = 91{\cdot}75$ (3)°, $V = 1461{\cdot}3$ Å^3, $Z = 1$, $D_m = 1{\cdot}58$, $D_x = 1{\cdot}59$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 1{\cdot}359$ mm^{-1}, $F(000) = 698$, $T =$

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Table 1. *Fractional coordinates with e.s.d.'s in parentheses*

$B_{eq} = [V^2\det(\beta_{ij})]^{1/3}$.

	x	y	z	B_{eq}(Å²)
Sb	0·0000	0·0000	0·0000	4·05
Mn	0·0000	0·5000	0·0000	3·00
Cl(1)	−0·14226 (23)	0·05974 (24)	0·11590 (24)	6·21
Cl(2)	0·11453 (21)	0·20400 (20)	0·11047 (23)	6·08
Cl(3)	−0·12533 (22)	0·05570 (23)	−0·14524 (22)	5·90
O	−0·0905 (5)	0·3822 (5)	0·0690 (5)	5·02
N(1)	0·0391 (4)	0·3493 (4)	−0·1277 (4)	3·16
N(2)	0·1703 (5)	0·5211 (5)	0·1157 (4)	3·46
C(1)	−0·0374 (6)	0·2744 (6)	−0·2441 (6)	3·64
C(2)	0·0248 (6)	0·1770 (7)	−0·3073 (6)	4·23
C(3)	0·1401 (6)	0·1932 (6)	−0·2317 (6)	4·05
C(4)	0·1510 (5)	0·3016 (6)	−0·1193 (6)	3·32
C(5)	0·2582 (5)	0·3497 (6)	−0·0170 (6)	3·20
C(6)	0·2649 (5)	0·4511 (6)	0·0918 (6)	3·48
C(7)	0·3717 (6)	0·4993 (7)	0·1987 (6)	3·98
C(8)	0·3419 (6)	0·5963 (7)	0·2868 (6)	4·35
C(9)	0·2185 (6)	0·6120 (6)	0·2355 (6)	3·61
C(10)	0·1570 (6)	0·7078 (6)	0·2957 (6)	3·65
C(11)	0·3744 (6)	0·2919 (6)	−0·0266 (6)	3·57
C(12)	0·4483 (7)	0·3132 (8)	−0·0902 (8)	5·19
C(13)	0·5584 (8)	0·2631 (10)	−0·0952 (9)	6·20
C(14)	0·5962 (7)	0·1923 (8)	−0·0366 (9)	5·15
C(15)	0·5238 (8)	0·1686 (7)	0·0245 (8)	5·07
C(16)	0·4116 (7)	0·2186 (7)	0·0312 (7)	4·65
C(17)	0·2268 (7)	0·7964 (8)	0·4277 (7)	4·39
C(18)	0·2984 (10)	0·9062 (9)	0·4594 (9)	6·76
C(19)	0·3641 (12)	0·9819 (11)	0·5796 (12)	7·47
C(20)	0·3615 (12)	0·9542 (14)	0·6696 (12)	7·26
C(21)	0·2931 (13)	0·8521 (17)	0·6427 (9)	7·72
C(22)	0·2193 (9)	0·7646 (11)	0·5187 (8)	6·52
C(23)	−0·1306 (12)	0·4268 (11)	0·1777 (9)	7·85
C(24)	0·2290 (22)	0·2725 (18)	0·4255 (14)	12·7
C(25)	0·2689 (19)	0·3820 (21)	0·4270 (14)	12·6
Cl(5)	0·0771 (5)	0·3826 (8)	0·4433 (6)	17·4
Cl(6)	−0·3510 (6)	0·5046 (5)	0·4333 (5)	15·7
Cl(7)	0·4023 (4)	0·2385 (6)	0·3930 (4)	13·3
Cl(8)	0·2423 (12)	0·2461 (7)	−0·4495 (7)	19·9

293 K, $R = 0·079$ for 4822 unique observed reflections. The manganese ion is coordinated to two methanol oxygens, Mn—O = 2·283 (5) Å, and four porphinato nitrogen atoms, average Mn—N = 2·002 (5) Å.

Experimental. Crystals of the title compound were obtained in the course of other studies on manganese porphyrinates. A deep purple crystal 0·83 × 0·20 × 0·17 mm was mounted in a glass capillary with the long axis along [010]. Calculated and experimental (by flotation in aqueous zinc iodide solution) densities 1·59 and 1·58 Mg m^{-3}. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using θ–2θ scans at a constant rate of 4° min^{-1} in 2θ to a maximum value of 55°. No significant secular variation of four intensity standards measured every hour. Empirical absorption correction ranged from 0·797 to 1·00. 6939 reflections measured (h 0→14, k ±15, l ±16), 6620 unique, 4822 with $I > 3\sigma(I)$ considered observed. Merging R (intensity) 0·011 for 554 duplicates. Solved by Patterson and Fourier methods. Full-matrix least squares minimized $\sum w(\Delta F)^2$, $w^{-1} = \sigma^2(F) + 0·04\,I$, with a total of 328 variables. The H atoms were positioned according to idealized geometry (C—H = 0·95 Å) and isotropic B 1·3 times B of attached atom. All nonhydrogen atoms refined anisotropically; the 1,1,2,2-tetrachloroethane solvent molecule is disordered. $R = 0·079$, $wR = 0·094$, $S = 2·98$. The final R value, though relatively high, is about average for this class of compounds. Final difference peak 1·39 e Å^{-3} near solvent molecule. Final

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Sb—Cl(1)	2·345 (2)	C(7)—C(8)	1·347 (9)
Sb—Cl(2)	2·368 (2)	C(8)—C(9)	1·415 (8)
Sb—Cl(3)	2·359 (2)	C(9)—C(10)	1·394 (8)
Mn—N(1)	1·999 (5)	C(10)—C(17)	1·513 (9)
Mn—N(2)	2·005 (5)	C(11)—C(12)	1·371 (10)
Mn—O	2·283 (5)	C(11)—C(16)	1·382 (10)
N(1)—C(1)	1·371 (8)	C(12)—C(13)	1·385 (10)
N(1)—C(4)	1·385 (7)	C(13)—C(14)	1·363 (13)
N(2)—C(6)	1·381 (7)	C(14)—C(15)	1·346 (12)
N(2)—C(9)	1·382 (8)	C(15)—C(16)	1·407 (10)
C(1)—C(2)	1·423 (8)	C(17)—C(18)	1·370 (13)
C(1)—C(10^{i})	1·389 (8)	C(17)—C(22)	1·377 (13)
C(2)—C(3)	1·338 (9)	C(18)—C(19)	1·371 (14)
C(3)—C(4)	1·434 (9)	C(19)—C(20)	1·320 (20)
C(4)—C(5)	1·398 (8)	C(20)—C(21)	1·291 (20)
C(5)—C(6)	1·377 (9)	C(21)—C(22)	1·455 (15)
C(5)—C(11)	1·493 (7)	C(23)—O	1·437 (10)
C(6)—C(7)	1·426 (9)		
Cl(1)SbCl(2)	89·85 (9)	C(6)C(7)C(8)	107·5 (5)
Cl(1)SbCl(3)	90·07 (9)	C(7)C(8)C(9)	107·5 (6)
Cl(2)SbCl(3)	89·66 (9)	N(2)C(9)C(8)	109·8 (5)
N(1)MnN(2)	89·53 (20)	N(2)C(9)C(10)	125·3 (5)
N(1)MnO	91·19 (20)	C(8)C(9)C(10)	124·9 (6)
N(2)MnO	91·91 (21)	C(1)C(10)C(9)	124·9 (6)
MnN(1)C(1)	127·2 (4)	C(1)C(10)C(17)	118·7 (6)
MnN(1)C(4)	127·4 (4)	C(9)C(10)C(17)	116·4 (5)
C(1)N(1)C(4)	105·4 (5)	C(5)C(11)C(12)	120·8 (6)
MnN(2)C(6)	127·5 (4)	C(5)C(11)C(16)	120·6 (6)
MnN(2)C(9)	126·7 (4)	C(12)C(11)C(16)	118·6 (6)
C(6)N(2)C(9)	105·8 (5)	C(11)C(12)C(13)	120·5 (7)
N(1)C(1)C(2)	110·3 (5)	C(12)C(13)C(14)	120·9 (8)
N(1)C(1)C(10^{i})	125·5 (6)	C(13)C(14)C(15)	119·4 (7)
C(2)C(1)C(10^{i})	124·2 (6)	C(14)C(15)C(16)	120·8 (8)
C(1)C(2)C(3)	107·6 (6)	C(11)C(16)C(15)	119·7 (7)
C(2)C(3)C(4)	107·2 (5)	C(10)C(17)C(18)	122·1 (8)
N(1)C(4)C(3)	109·5 (5)	C(10)C(17)C(22)	118·9 (8)
N(1)C(4)C(5)	125·8 (5)	C(18)C(17)C(22)	119·1 (8)
C(3)C(4)C(5)	124·7 (5)	C(17)C(18)C(19)	120·4 (11)
C(4)C(5)C(6)	123·7 (5)	C(18)C(19)C(20)	122·7 (13)
C(4)C(5)C(11)	118·7 (5)	C(19)C(20)C(21)	118·3 (11)
C(6)C(5)C(11)	117·5 (5)	C(20)C(21)C(22)	124·0 (12)
N(2)C(6)C(5)	126·0 (5)	C(17)C(22)C(21)	115·6 (11)
N(2)C(6)C(7)	109·3 (5)	MnOC(23)	126·6 (5)
C(5)C(6)C(7)	124·7 (5)		

Symmetry code: (i) $-x$, $1-y$, $-z$.

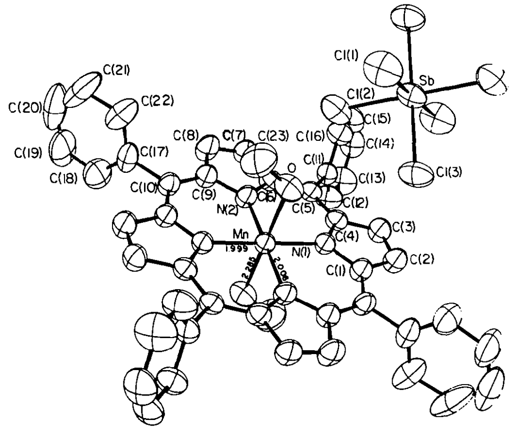

Fig. 1. *ORTEP* diagram (Johnson, 1976) and numbering scheme for all heavy atoms. 50% probability surfaces.

$(\Delta/\sigma)_{\max} = 0{\cdot}05$. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Data reduction carried out using Enraf–Nonius (1983) *SDP* program, a locally modified version of *ALLS* (Lapp & Jacobson, 1979) used for structure refinement and *ORFFE* (Busing, Martin & Levy, 1964) for structural parameters with e.s.d.'s. Table 1 gives atomic coordinates and Table 2 bond distances and angles.* Fig. 1 gives the atom numbering and perspective drawing of the molecule. Mn is in the plane with an average Mn—N_p bond distance of 2·002 (4) Å and an axial Mn—O distance of 2·283 (5) Å. These distances are consistent with a high-spin six-coordinate manganese(III) state (Scheidt & Gouterman, 1983).

Related literature. The structures of a number of six-coordinate Mn^{III} porphyrinates have been reported (Kirner & Scheidt, 1975; Hatano, Anzaik & Iitaka, 1983; Hill & Williamson, 1985*a*,*b*). Most of these have axial oxygen donors. As expected for high-spin six-coordinate manganese(III) species, all have long axial bonds, Mn–O $\simeq$ 2·2–2·3 Å.

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References

Busing, W. R., Martin, K. O. & Levy, H. A. (1964). *ORFFE. A Fortran Crystallographic Function and Error Program.* Report ORNL-TM-306. Oak Ridge National Laboratory, Tennessee, USA.

Enraf–Nonius (1983). *Structure Determination Package.* Enraf–Nonius, Delft, The Netherlands.

Hatano, K., Anzaik, K. & Iitaka, Y. (1983). *Bull. Chem. Soc. Jpn*, **56**, 422–427.

Hill, C. L. & Williamson, M. M. (1985*a*). *Inorg. Chem.* **24**, 2836–2841.

Hill, C. L. & Williamson, M. M. (1985*b*). *Inorg. Chem.* **24**, 3024–3030.

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

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Kirner, J. F. & Scheidt, W. R. (1975). *Inorg. Chem.* **14**, 2081–2086.

Lapp, R. L. & Jacobson, R. A. (1979). *ALLS. A Generalized Crystallographic Least-Squares Program.* National Technical Information Services IS-4708 UC-4, Springfield, Virginia, USA.

Scheidt, W. R. & Gouterman, M. (1983). *Iron Porphyrins, Part One*, edited by A. B. P. Lever & H. B. Gray, pp. 89–139. Reading, MA: Addison-Wesley.

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[*] Lists of structure factors, anisotropic thermal parameters, and fixed hydrogen positions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44681 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Di-μ-acetato-(*O*,*O*′)-bis[*N*-(α-phenylbenzylidene)cyclohexylamine-*N*,*C*]-dipalladium(II)

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Abstract. $[Pd_2(C_2H_3O_2)_2(C_{19}H_{20}N)_2]$, $M_r = 855{\cdot}65$, orthorhombic, *Pbcn*, $a = 19{\cdot}419\ (4)$, $b = 10{\cdot}739\ (4)$, $c = 18{\cdot}224\ (4)$ Å, $V = 3800{\cdot}4$ Å^3, $Z = 4$, $D_x = 1{\cdot}495$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 9{\cdot}750$ cm^{-1}, $F(000) = 1744$, $T = 293$ K. The final *R* value is 0·029 for 2494 significant $[I > 3\sigma(I)]$ reflections. In the dinuclear complex of site symmetry 2 each Pd atom is bonded to an N atom, an *ortho* C atom of the benzyl group and to two O atoms of the bridging acetato groups.

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