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

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

	x	y	z	U_{eq}
P1	0.17587 (8)	0.34179 (6)	0.40911 (5)	0.0485 (6)
Al1	0.42955 (9)	0.33478 (6)	0.54316 (6)	0.0633 (6)
O1	0.2853 (2)	0.3511 (2)	0.4771 (2)	0.0754 (16)
O2	0.0922 (2)	0.2685 (2)	0.4164 (1)	0.0627 (13)
C1	0.5546 (5)	0.3257 (4)	0.4883 (3)	0.096 (3)
C2	0.4474 (5)	0.4243 (3)	0.6215 (3)	0.095 (3)
C11	0.2249 (3)	0.3257 (3)	0.3234 (2)	0.066 (2)
C12	0.2986 (4)	0.3866 (4)	0.3017 (3)	0.100 (3)
C13	0.3381 (6)	0.3734 (7)	0.2362 (5)	0.138 (5)
C14	0.3039 (6)	0.3037 (8)	0.1925 (4)	0.153 (6)
C15	0.2306 (6)	0.2385 (5)	0.2119 (3)	0.126 (4)
C16	0.1891 (4)	0.2517 (3)	0.2777 (3)	0.088 (3)
C21	0.0951 (4)	0.4410 (2)	0.3998 (2)	0.072 (2)
C22	0.1412 (6)	0.5123 (4)	0.4453 (4)	0.137 (4)
C23	0.0830 (7)	0.5900 (4)	0.4342 (6)	0.179 (6)
C24	−0.0270 (7)	0.5976 (4)	0.3841 (5)	0.149 (5)
C25	−0.0773 (6)	0.5278 (5)	0.3408 (4)	0.143 (4)
C26	−0.0171 (5)	0.4503 (3)	0.3498 (4)	0.111 (3)

Table 2. *Selected geometric parameters* (Å, °)

Al1—O1	1.781 (3)	P1—O1	1.512 (3)
Al1—O2^{i}	1.794 (3)	P1—O2	1.505 (2)
Al1—C1	1.931 (4)	P1—C11	1.782 (4)
Al1—C2	1.945 (4)	P1—C21	1.771 (4)
O1—Al1—O2^{i}	101.3 (2)	C21—P1—O1	107.1 (2)
C1—Al1—O1	110.1 (2)	C21—P1—O2	109.5 (2)
C2—Al1—O1	106.8 (2)	C21—P1—C11	107.9 (2)
C1—Al1—O2^{i}	109.7 (2)	Al1—O1—P1	162.4 (2)
C2—Al1—O2^{i}	109.8 (2)	Al1^{i}—O2—P1	149.1 (2)
C1—Al1—C2	117.8 (2)	C12—C11—P1	119.6 (4)
O1—P1—O2	114.2 (1)	C16—C11—P1	120.7 (3)
C11—P1—O1	109.7 (2)	C22—C21—P1	121.3 (4)
C11—P1—O2	108.3 (2)	C26—C21—P1	121.8 (3)

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

The large U_{ij} terms observed for some phenyl C atoms may indicate disorder. H atoms were placed in calculated positions with a common refined displacement parameter. Nine reflections were omitted as likely to be suffering from extinction.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1033). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(Ethanol)(2,3,7,8,12,13,17,18-octaethylporphinato)manganese(III) Perchlorate

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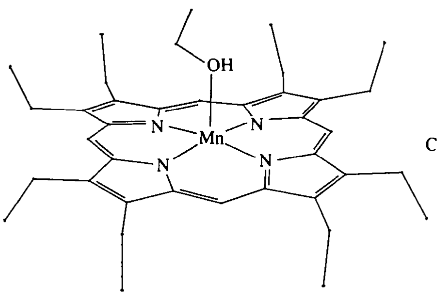
Abstract

The crystal structure of $[Mn(C_{36}H_{44}N_4)(C_2H_6O)]ClO_4$ has been determined. The average Mn—N$_p$ bond distance is 1.997 (5) Å and the axial Mn—O bond length is 2.145 (2) Å. The manganese(III) ion has a displacement of 0.17 Å from the mean plane of the 24-atom porphinato core.

Comment

The title compound, $[Mn(OEP)(EtOH)]ClO_4$ (EtOH is ethanol and OEP is the dianion of 2,3,7,8,12,13,17,18-octaethylporphyrin), (I), was obtained in our attempted preparation of $[Mn(OEP)]_2O$ from $CHCl_3$ and hexanes. The source of the ethanol ligand is believed to be the stabilizer in $CHCl_3$.



(I)

An *ORTEP*II diagram (Johnson, 1976) and labeling scheme for the molecule are shown in Fig. 1. The average values for the unique chemical classes of bond distances and angles of the 24-atom porphinato core and the perpendicular displacements of each atom from the mean plane of the core are given in Fig. 2. The agreement between chemically equivalent bond distances and angles is quite satisfactory.

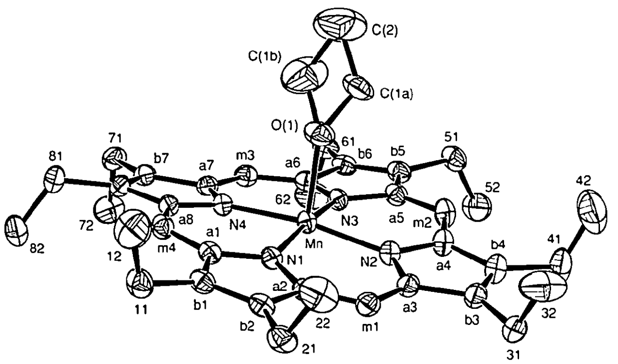


Fig. 1. *ORTEP*II (Johnson, 1976) diagram of [Mn(OEP)(EtOH)] with the labeling scheme. For clarity, the C atom labels of the porphyrin C(51), C(*a*7), *etc.* are shown only as 51, *a*7, *etc.* Displacement ellipsoids are drawn at the 30% probability level. Porphyrin H atoms are omitted for clarity. The disorder of the ligated ethanol is also shown.

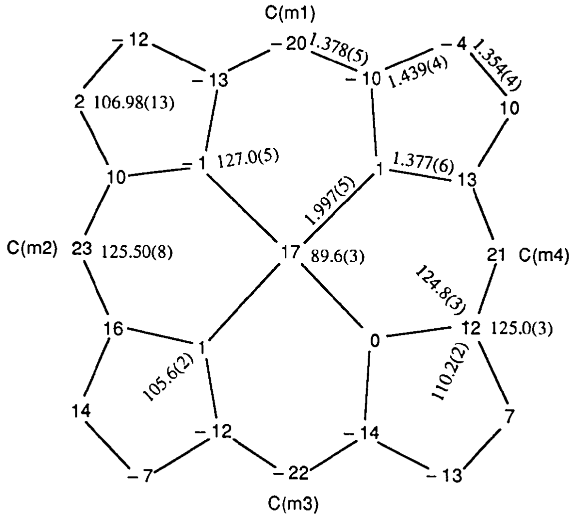


Fig. 2. Formal diagram of the porphinato core of [Mn(OEP)(EtOH)]-ClO_4, displaying the average values of the bond parameters. The numbers in parentheses are the estimated standard deviations calculated on the assumption that the average values were all drawn from the same population. Also displayed are the perpendicular displacements, in units of 0.01 Å, of each atom from the 24-atom mean plane of the core.

The average Mn—N_p (N_p = pyrrole N atom) bond length of 1.997 (5) Å and the Mn^{III} displacement of 0.17 Å are comparable with those of previously reported five-coordinate manganese(III) porphyrinates with O-atom donor axial ligands, such as $[Mn(TPP)(H_2O)]$-CF_3SO_3 (Williamson & Hill, 1986), $[Mn(TPP)(H_2O)]$-$SbF_6.2C_6H_6$ (Williamson & Hill, 1987), [Mn(OEP)-$(H_2O)]ClO_4$ (Cheng, Cukiernik, Fries, Marchon & Scheidt, 1995) and $[Mn(OEP)(OClO_3)]$ (Cheng & Scheidt, 1995). The axial Mn—O bond distance of 2.145 (2) Å is also within the range 2.105 (4)–2.183 (2) Å observed for these molecules. The large variation of the observed axial Mn—O bond lengths is due to the differences in π–π interactions between the adjacent porphyrin macrocycles and/or hydrogen-bonding effects of the aqua ligand. As in the other five-coordinate octaethylporphyrinates, this complex forms π–π dimers in the solid state. The mean planes of the two porphinato cores with close 'face-to-face' contact have an interplanar distance of 3.53 Å and a lateral shift of 3.40 Å, consistent with the four-up/four-down peripheral ethyl-group orientation (Scheidt & Lee, 1987).

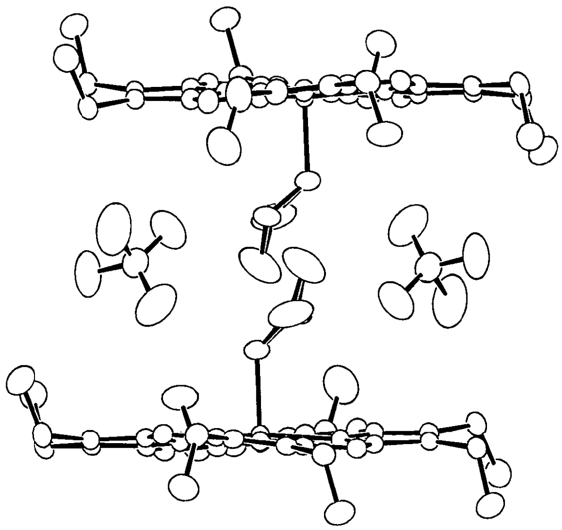


Fig. 3. *ORTEP*II (Johnson, 1976) edgeview showing the arrangement of the porphyrin rings and perchlorate anions in the lattice. Only one orientation (*a*) of the perchlorate is shown; the other orientation (*b*) is omitted for clarity.

Experimental

The title compound was crystallized by diffusion of hexanes into chloroform.

Crystal data

$[Mn(C_{36}H_{44}N_4)(C_2H_6O)]$-$ClO_4$
$M_r = 733.23$
Triclinic
$P\bar{1}$
$a = 12.8859\ (4)$ Å
$b = 12.9276\ (10)$ Å
$c = 13.5328\ (7)$ Å
$\alpha = 64.067\ (9)°$
$\beta = 87.820\ (6)°$
$\gamma = 67.207\ (6)°$
$V = 1845.4\ (2)$ Å^3
$Z = 2$
$D_x = 1.319$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 250 reflections
$\theta = 5.1$–$20.8°$
$\mu = 0.461$ mm^{-1}
$T = 293\ (2)$ K
Rectangular prism
0.55 × 0.25 × 0.25 mm
Black

Data collection

Enraf–Nonius FAST area-detector diffractometer
Ellipsoid-mask fitting scans
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{min} = 0.549$, $T_{max} = 0.748$

18 191 measured reflections
8973 independent reflections
7059 observed reflections $[I > 2\sigma(I)]$
$R_{int} = 0.0587$
$\theta_{max} = 29.85°$
$h = -17 \rightarrow 17$
$k = -16 \rightarrow 17$
$l = -15 \rightarrow 18$

Refinement

Refinement on F^2
$R(F) = 0.0672$
$wR(F^2) = 0.1767$
$S = 1.106$
8972 reflections
482 parameters
Idealized with riding model
$w = 1/[\sigma^2(F_o^2) + (0.1017P)^2 + 1.0542P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.033$
$\Delta\rho_{max} = 0.718$ e Å^{-3}
$\Delta\rho_{min} = -0.540$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	U_{eq}
Mn	0.87902 (3)	0.18852 (3)	0.03474 (3)	0.03443 (13)
N(1)	0.9964 (2)	0.0345 (2)	0.1569 (2)	0.0379 (4)
N(2)	0.9946 (2)	0.1920 (2)	−0.0678 (2)	0.0415 (5)
N(3)	0.7572 (2)	0.3248 (2)	−0.0950 (2)	0.0375 (4)
N(4)	0.7590 (2)	0.1653 (2)	0.1289 (2)	0.0370 (4)
C(*a*1)	0.9831 (2)	−0.0272 (2)	0.2662 (2)	0.0388 (5)
C(*a*2)	1.1096 (2)	−0.0250 (2)	0.1528 (2)	0.0400 (5)
C(*a*3)	1.1076 (2)	0.1111 (3)	−0.0431 (2)	0.0412 (6)
C(*a*4)	0.9788 (3)	0.2840 (3)	−0.1757 (2)	0.0493 (7)
C(*a*5)	0.7736 (2)	0.4020 (3)	−0.1979 (2)	0.0420 (6)
C(*a*6)	0.6415 (2)	0.3707 (2)	−0.0970 (2)	0.0384 (5)
C(*a*7)	0.6429 (2)	0.2308 (2)	0.0978 (2)	0.0373 (5)
C(*a*8)	0.7757 (2)	0.0873 (2)	0.2423 (2)	0.0373 (5)
C(*b*1)	1.0912 (2)	−0.1264 (2)	0.3313 (2)	0.0414 (6)
C(*b*2)	1.1690 (2)	−0.1251 (2)	0.2608 (2)	0.0423 (6)
C(*b*3)	1.1641 (2)	0.1519 (3)	−0.1376 (2)	0.0463 (6)
C(*b*4)	1.0845 (3)	0.2581 (3)	−0.2192 (3)	0.0543 (7)
C(*b*5)	0.6656 (2)	0.4986 (2)	−0.2646 (2)	0.0439 (6)
C(*b*6)	0.5840 (2)	0.4771 (2)	−0.2036 (2)	0.0419 (6)
C(*b*7)	0.5864 (2)	0.1922 (2)	0.1921 (2)	0.0395 (5)
C(*b*8)	0.6686 (2)	0.1058 (3)	0.2818 (2)	0.0402 (5)
C(*m*1)	1.1606 (2)	0.0083 (3)	0.0592 (2)	0.0439 (6)
C(*m*2)	0.8771 (3)	0.3836 (3)	−0.2339 (2)	0.0513 (7)
C(*m*3)	0.5883 (2)	0.3245 (2)	−0.0075 (2)	0.0402 (5)
C(*m*4)	0.8807 (2)	−0.0001 (3)	0.3058 (2)	0.0409 (5)
C11	1.1106 (3)	−0.2108 (3)	0.4543 (2)	0.0549 (7)
C21	1.2943 (3)	−0.2052 (3)	0.2878 (3)	0.0541 (7)
C31	1.2887 (3)	0.0886 (3)	−0.1397 (3)	0.0540 (7)
C41	1.0988 (4)	0.3376 (4)	−0.3357 (3)	0.0803 (13)
C51	0.6519 (3)	0.6025 (3)	−0.3793 (2)	0.0555 (7)
C61	0.4573 (3)	0.5459 (3)	−0.2377 (3)	0.0527 (7)
C71	0.4594 (2)	0.2371 (3)	0.1867 (3)	0.0492 (7)
C81	0.6518 (3)	0.0394 (3)	0.4000 (2)	0.0501 (7)
C12	1.1515 (5)	−0.1662 (5)	0.5228 (4)	0.0944 (15)
C22	1.3646 (3)	−0.1398 (4)	0.2992 (4)	0.0829 (13)
C32	1.3584 (4)	0.1410 (5)	−0.1095 (6)	0.107 (2)
C42	1.1120 (6)	0.4474 (6)	−0.3494 (5)	0.123 (2)
C52	0.6428 (5)	0.5693 (4)	−0.4706 (3)	0.0820 (12)
C62	0.4072 (3)	0.4800 (4)	−0.2775 (4)	0.0836 (13)
C72	0.4126 (3)	0.1761 (4)	0.1368 (4)	0.0707 (10)
C82	0.6201 (4)	−0.0688 (4)	0.4234 (3)	0.0761 (11)
O(1)	0.9061 (2)	0.3127 (2)	0.0858 (2)	0.0613 (6)
C(1*a*)†	0.8775 (9)	0.4432 (7)	0.0216 (7)	0.074 (3)
C(1*b*)†	0.8160 (13)	0.4233 (19)	0.0920 (18)	0.140 (7)
C(2)	0.8391 (9)	0.5257 (7)	0.0711 (9)	0.183 (4)
Cl	1.13468 (11)	0.23226 (12)	0.30314 (9)	0.0855 (3)
O(2*a*)†	1.2501 (6)	0.1351 (7)	0.3331 (4)	0.142 (3)
O(3*a*)†	1.1361 (9)	0.3459 (7)	0.2349 (7)	0.155 (4)
O(4*a*)†	1.1138 (6)	0.2383 (10)	0.4067 (6)	0.172 (4)
O(5*a*)†	1.0575 (14)	0.2005 (13)	0.2771 (11)	0.266 (9)
O(2*b*)†	1.022 (2)	0.313 (3)	0.288 (2)	0.216 (11)
O(3*b*)†	1.213 (2)	0.276 (3)	0.282 (2)	0.180 (8)
O(4*b*)†	1.1165 (8)	0.2325 (14)	0.1921 (9)	0.095 (4)
O(5*b*)†	1.1199 (17)	0.132 (2)	0.3792 (18)	0.168 (7)

† Partial occupancy (see below).

Table 2. *Selected geometric parameters (Å, °)*

Mn—O(1)	2.145 (2)	C(*b*5)—C(*b*6)	1.350 (4)
Mn—N(1)	1.991 (2)	C(*b*7)—C(*b*8)	1.356 (4)
Mn—N(2)	1.997 (2)	C(*b*1)—C11	1.501 (4)
Mn—N(3)	2.003 (2)	C(*b*2)—C21	1.494 (4)
Mn—N(4)	1.996 (2)	C(*b*3)—C31	1.499 (4)
N(1)—C(*a*1)	1.381 (3)	C(*b*4)—C41	1.511 (5)
N(1)—C(*a*2)	1.374 (3)	C(*b*5)—C51	1.503 (4)
N(2)—C(*a*3)	1.371 (3)	C(*b*6)—C61	1.498 (4)
N(2)—C(*a*4)	1.379 (4)	C(*b*7)—C71	1.502 (4)
N(3)—C(*a*5)	1.380 (3)	C(*b*8)—C81	1.504 (4)
N(3)—C(*a*6)	1.371 (3)	C11—C12	1.487 (6)
N(4)—C(*a*7)	1.373 (3)	C21—C22	1.510 (5)
N(4)—C(*a*8)	1.387 (3)	C31—C32	1.475 (5)
C(*a*1)—C(*b*1)	1.443 (3)	C41—C42	1.426 (8)
C(*a*2)—C(*b*2)	1.436 (4)	C51—C52	1.495 (5)
C(*a*3)—C(*b*3)	1.442 (4)	C61—C62	1.513 (5)
C(*a*4)—C(*b*4)	1.440 (4)	C71—C72	1.518 (5)
C(*a*5)—C(*b*5)	1.440 (4)	C81—C82	1.506 (5)
C(*a*6)—C(*b*6)	1.441 (3)	O(1)—C(1*a*)	1.414 (7)
C(*a*7)—C(*b*7)	1.436 (4)	O(1)—C(1*b*)	1.49 (2)
C(*a*8)—C(*b*8)	1.431 (4)	C(1*a*)—C(2)	1.421 (11)
C(*a*1)—C(*m*4)	1.379 (4)	C(1*b*)—C(2)	1.38 (2)
C(*a*2)—C(*m*1)	1.378 (4)	Cl—O(2*a*)	1.452 (6)
C(*a*3)—C(*m*1)	1.381 (4)	Cl—O(3*a*)	1.358 (6)
C(*a*4)—C(*m*2)	1.371 (4)	Cl—O(4*a*)	1.447 (7)
C(*a*5)—C(*m*2)	1.371 (4)	Cl—O(5*a*)	1.323 (9)
C(*a*6)—C(*m*3)	1.384 (4)	Cl—O(2*b*)	1.38 (3)
C(*a*7)—C(*m*3)	1.379 (4)	Cl—O(3*b*)	1.30 (2)
C(*a*8)—C(*m*4)	1.379 (4)	Cl—O(4*b*)	1.528 (9)
C(*b*1)—C(*b*2)	1.358 (4)	Cl—O(5*b*)	1.33 (2)
C(*b*3)—C(*b*4)	1.351 (4)		

N(1)—Mn—N(2)	89.99 (9)	C(*b*3)—C(*b*4)—C(*a*4)	107.2 (3)
N(1)—Mn—N(3)	171.04 (9)	C(*b*6)—C(*b*5)—C(*a*5)	106.9 (2)
N(1)—Mn—N(4)	89.55 (9)	C(*b*5)—C(*b*6)—C(*a*6)	107.0 (2)
N(2)—Mn—N(3)	89.32 (9)	C(*b*8)—C(*b*7)—C(*a*7)	107.1 (2)
N(2)—Mn—N(4)	170.11 (9)	C(*b*7)—C(*b*8)—C(*a*8)	106.8 (2)
N(3)—Mn—N(4)	89.60 (9)	C(*a*1)—C(*b*1)—C11	125.1 (3)
N(1)—Mn—O(1)	93.14 (9)	C(*a*2)—C(*b*2)—C21	124.8 (3)
N(2)—Mn—O(1)	95.01 (10)	C(*a*3)—C(*b*3)—C31	125.1 (3)
N(3)—Mn—O(1)	95.82 (9)	C(*a*4)—C(*b*4)—C41	124.6 (3)
N(4)—Mn—O(1)	94.88 (10)	C(*a*5)—C(*b*5)—C51	124.5 (3)
C(*a*1)—N(1)—Mn	126.8 (2)	C(*a*6)—C(*b*6)—C61	124.9 (3)
C(*a*2)—N(1)—Mn	127.2 (2)	C(*a*7)—C(*b*7)—C71	124.6 (2)
C(*a*3)—N(2)—Mn	127.4 (2)	C(*a*8)—C(*b*8)—C81	126.0 (2)
C(*a*4)—N(2)—Mn	126.4 (2)	C(*b*1)—C(*b*2)—C21	128.1 (3)
C(*a*5)—N(3)—Mn	126.4 (2)	C(*b*2)—C(*b*1)—C11	128.0 (3)
C(*a*6)—N(3)—Mn	127.7 (2)	C(*b*3)—C(*b*4)—C41	128.2 (3)
C(*a*7)—N(4)—Mn	127.5 (2)	C(*b*4)—C(*b*3)—C31	128.0 (3)
C(*a*8)—N(4)—Mn	126.9 (2)	C(*b*5)—C(*b*6)—C61	128.1 (3)
C(*a*1)—N(1)—C(*a*2)	105.7 (2)	C(*b*6)—C(*b*5)—C51	128.6 (3)
C(*a*3)—N(2)—C(*a*4)	105.9 (2)	C(*b*7)—C(*b*8)—C81	127.1 (3)
C(*a*5)—N(3)—C(*a*6)	105.5 (2)	C(*b*8)—C(*b*7)—C71	128.2 (3)
C(*a*7)—N(4)—C(*a*8)	105.4 (2)	C(*a*2)—C(*m*1)—C(*a*3)	125.4 (3)
N(1)—C(*a*1)—C(*b*1)	109.9 (2)	C(*a*4)—C(*m*2)—C(*a*5)	125.5 (3)
N(1)—C(*a*2)—C(*b*2)	110.5 (2)	C(*a*6)—C(*m*3)—C(*a*7)	125.5 (2)
N(2)—C(*a*3)—C(*b*3)	110.2 (2)	C(*a*8)—C(*m*4)—C(*a*1)	125.6 (2)
N(2)—C(*a*4)—C(*b*4)	109.8 (3)	C(*b*1)—C11—C12	113.7 (3)
N(3)—C(*a*5)—C(*b*5)	110.2 (2)	C(*b*2)—C21—C22	113.4 (3)
N(3)—C(*a*6)—C(*b*6)	110.3 (2)	C(*b*3)—C31—C32	113.5 (3)
N(4)—C(*a*7)—C(*b*7)	110.3 (2)	C(*b*4)—C41—C42	114.0 (4)
N(4)—C(*a*8)—C(*b*8)	110.3 (2)	C(*b*5)—C51—C52	113.9 (3)
N(1)—C(*a*1)—C(*m*4)	124.8 (2)	C(*b*6)—C61—C62	112.6 (3)
N(1)—C(*a*2)—C(*m*1)	125.1 (2)	C(*b*7)—C71—C72	111.6 (3)
N(2)—C(*a*3)—C(*m*1)	124.7 (2)	C(*b*8)—C81—C82	114.1 (3)
N(2)—C(*a*4)—C(*m*2)	125.2 (3)	C(1*a*)—O(1)—Mn	127.8 (3)
N(3)—C(*a*5)—C(*m*2)	124.7 (2)	C(1*b*)—O(1)—Mn	126.1 (5)
N(3)—C(*a*6)—C(*m*3)	124.5 (2)	O(1)—C(1*a*)—C(2)	120.5 (8)
N(4)—C(*a*7)—C(*m*3)	125.1 (2)	O(1)—C(1*b*)—C(2)	118.5 (12)
N(4)—C(*a*8)—C(*m*4)	124.2 (2)	O(2*a*)—Cl—O(3*a*)	108.9 (5)
C(*m*4)—C(*a*1)—C(*b*1)	125.3 (2)	O(2*a*)—Cl—O(4*a*)	102.0 (4)
C(*m*1)—C(*a*2)—C(*b*2)	124.4 (3)	O(2*a*)—Cl—O(5*a*)	113.7 (9)
C(*m*1)—C(*a*3)—C(*b*3)	125.0 (3)	O(3*a*)—Cl—O(4*a*)	103.5 (6)
C(*m*2)—C(*a*4)—C(*b*4)	124.9 (3)	O(3*a*)—Cl—O(5*a*)	119.7 (6)
C(*m*2)—C(*a*5)—C(*b*5)	125.0 (3)	O(4*a*)—Cl—O(5*a*)	107.0 (9)
C(*m*3)—C(*a*6)—C(*b*6)	125.1 (2)	O(2*b*)—Cl—O(3*b*)	120.0 (17)
C(*m*3)—C(*a*7)—C(*b*7)	124.6 (2)	O(2*b*)—Cl—O(4*b*)	91.0 (12)
C(*m*4)—C(*a*8)—C(*b*8)	125.4 (2)	O(2*b*)—Cl—O(5*b*)	93.2 (15)
C(*b*2)—C(*b*1)—C(*a*1)	106.9 (2)	O(3*b*)—Cl—O(4*b*)	98.2 (11)
C(*b*1)—C(*b*2)—C(*a*2)	107.0 (2)	O(3*b*)—Cl—O(5*b*)	138.8 (14)
C(*b*4)—C(*b*3)—C(*a*3)	106.9 (3)	O(4*b*)—Cl—O(5*b*)	105.3 (11)

Cell determination and intensity data collection were performed on an Enraf–Nonius FAST area-detector diffractometer with an Mo rotating anode source. Our detailed methods and procedures for small-molecule X-ray data collection with the FAST system have been described previously (Scheidt & Turowska-Tyrk, 1994). All reflections were used in least-squares refinement, including negative intensities.

Both the ethanol ligand and the perchlorate anion were found to be disordered. For the ethanol ligand, two positions [C(1*a*) and C(1*b*)] are found for the C atom bonded to the O atom, with a separation of 1.22 Å, and refined occupancies of 0.531 (14) and 0.469 (14), respectively. The terminal C atom of the ethanol ligand is essentially disordered; however, we were unable to resolve it with the room-temperature data. For the perchlorate anion, the Cl atom has full occupancy, while the O atoms are disordered and have been resolved into two fragments (a and *b*) with different orientations. Group occupancies were applied and the refined values are 0.668 (8) and 0.332 (8), respectively. All the H atoms were idealized using riding models.

Data collection: *MADNES* (Messerschmitt & Pflugrath, 1987). Cell refinement: *MADNES*. Data reduction: *MADNES*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1159). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(2-methyl-4-nitroanilinium) Tetrachlorocadmate

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Abstract

The crystal structure of bis(2-methyl-4-nitroanilinium) tetrachlorocadmate, $(C_7H_9N_2O_2)_2[CdCl_4]$, has been determined by X-ray diffraction at room temperature. The