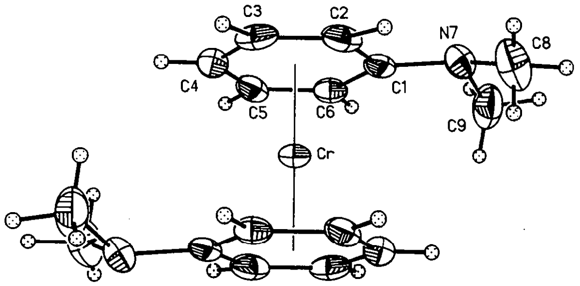

Fig. 1. View of the complex (1) showing the atomic labeling scheme. The non-H atoms are scaled to the 50% probability level while the H atoms are drawn to an arbitrary size. The Cr atom lies on an inversion center. The pyramidal geometry at the N atom is apparent.

atoms are listed in Table 2.* The atomic labeling scheme is shown in Fig. 1. The least-squares planes program was supplied by Cordes (1983). Other computer programs used in this work are listed in reference 11 of Gadol & Davis (1982).

Related literature. The Cr—C bond lengths in (1) are similar to those found in bis(η^6-benzene)-chromium(0) [av. 2·142 (2) Å] (Keulen & Jellinek, 1966).

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* Tables of anisotropic thermal parameters, H-atom positional parameters, bond distances and angles involving the H atoms, least-squares planes, structure factor amplitudes and a unit-cell packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52582 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of (2,3,7,8,12,13,17,18-Octaethylporphinato)dioxoosmium(VI)

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Abstract. $[Os(C_{36}H_{44}N_4)O_2].C_6H_5Cl$, $M_r = 867·53$, triclinic, $P\bar{1}$, $a = 8·177$ (1), $b = 10·668$ (1), $c = 11·791$ (1) Å, $\alpha = 73·91$ (1), $\beta = 83·18$ (1), $\gamma = 75·07$ (1)°, $V = 953·6$ Å^3, $Z = 1$, $D_x = 1·51$ g cm^{-3}, $D_m = 1·50$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·71073$ Å, $\mu = 34·5$ cm^{-1}, $F(000) = 438$, $T = 293$ K, $R = 0·048$ for 5721 unique observed reflections. The Os atom is centered in the porphinato plane with an average Os—N = 2·052 (6) Å and Os—O = 1·745 (5) Å. The 24-atom porphyrin core is effectively planar with the largest deviation from the mean plane less than 0·02 Å.

Experimental. The title compound was synthesized following the literature procedure (Buchler & Smith, 1974; Che, Poon, Chung & Gray, 1985) and crystals of this complex were obtained by slow diffusion of pentane into a chlorobenzene solution. D_m measured by flotation. A purple, air-stable crystal of $Os(O)_2(oep).C_6H_5Cl$ (oep = octaethylporphinato) with approximate dimensions 0·32 × 0·21 × 0·12 mm was mounted on the end of a glass fiber. All measurements were performed with graphite-monochromated Mo $K\alpha$ radiation on an Enraf–Nonius CAD-4 diffractometer. Intensity data were measured by θ–2θ scans with a constant scan rate of 3° min^{-1} (in θ). Data were collected to a maximum 2θ of 63·7° and θ–2θ CAD-4 moving-crystal moving-counter background measurements were used for backgrounds (total background counting time 1/2 peak counting time). 25 reflections used for measuring lattice parameters $7 < \theta < 10°$. Range of *hkl*: 0–11,

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Table 1. *Fractional coordinates and equivalent isotropic thermal parameters*

$$B_{eq} = (4/3)[a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + ab(\cos\gamma)\beta(1,2) + ac(\cos\beta)\beta(1,3) + bc(\cos\alpha)\beta(2,3)].$$

	x	y	z	B_{eq}(Å^2)
Os	1·0000	1·0000	1·0000	2·07
Cl	0·1272 (12)	0·2355 (11)	0·3913 (10)	8·66
O	0·8653 (7)	1·0678 (5)	1·1055 (5)	2·94
N(1)	1·1311 (7)	1·1469 (6)	0·9726 (5)	2·50
N(2)	1·1623 (7)	0·8800 (6)	1·1282 (5)	2·69
C(*a*1)	1·0955 (9)	1·2699 (7)	0·8912 (6)	2·66
C(*a*2)	1·2704 (9)	1·1409 (7)	1·0298 (7)	2·74
C(*a*3)	1·2977 (9)	0·9099 (8)	1·1647 (7)	2·82
C(*a*4)	1·1564 (9)	0·7543 (7)	1·1930 (6)	2·69
C(*b*1)	1·2205 (10)	1·3435 (7)	0·8972 (7)	2·95
C(*b*2)	1·3278 (9)	1·2632 (8)	0·9832 (7)	2·89
C(*b*3)	1·3799 (9)	0·8001 (9)	1·2563 (7)	3·13
C(*b*4)	1·2950 (10)	0·7003 (8)	1·2745 (7)	3·10
C(*m*1)	0·9634 (10)	1·3151 (8)	0·8155 (7)	3·09
C(*m*2)	1·3479 (9)	1·0292 (7)	1·1167 (7)	2·86
C(11)	1·2261 (11)	1·4829 (9)	0·8217 (7)	3·47
C(12)	1·1337 (15)	1·5937 (10)	0·8783 (11)	4·91
C(21)	1·4723 (11)	1·2938 (9)	1·0275 (8)	3·57
C(21)	1·4275 (15)	1·3302 (13)	1·1460 (11)	4·72
C(31)	1·5280 (11)	0·7980 (10)	1·3216 (8)	3·72
C(32)	1·4745 (18)	0·8596 (22)	1·4254 (15)	6·24
C(41)	1·3269 (12)	0·5649 (9)	1·3628 (8)	3·95
C(42)	1·2129 (17)	0·5618 (13)	1·4729 (10)	5·53
C(1)	0·0572 (20)	0·1139 (21)	0·4492 (17)	7·55
C(2)	−0·0011 (22)	0·0534 (24)	0·3757 (16)	7·14
C(3)	0·0563 (23)	0·0637 (20)	0·5717 (15)	6·9

Table 2. *Bond lengths* (Å) *and angles* (°) *in the* Os(O)$_2$(oep) *molecule*

Os—O	1·745 (5)	C(*a*4)—C(*m*1)′	1·399 (11)
Os—N(1)	2·052 (6)	C(*b*1)—C(*b*2)	1·370 (11)
Os—N(2)	2·052 (6)	C(*b*1)—C(11)	1·515 (11)
N(1)—C(*a*1)	1·378 (9)	C(*b*2)—C(21)	1·489 (10)
N(1)—C(*a*2)	1·371 (9)	C(*b*3)—C(*b*4)	1·371 (12)
N(2)—C(*a*3)	1·372 (8)	C(*b*3)—C(31)	1·502 (10)
N(2)—C(*b*4)	1·357 (10)	C(*b*4)—C(41)	1·508 (12)
C(*a*1)—C(*b*1)	1·458 (10)	C(11)—C(12)	1·507 (14)
C(*a*1)—C(*m*1)	1·385 (10)	C(21)—C(22)	1·530 (14)
C(*a*2)—C(*b*2)	1·442 (10)	C(31)—C(32)	1·514 (15)
C(*a*2)—C(*m*2)	1·400 (10)	C(41)—C(42)	1·504 (16)
C(*a*3)—C(*b*3)	1·434 (11)	C(1)—Cl	1·508 (25)
C(*a*3)—C(*m*2)	1·387 (11)	C(1)—C(2)	1·401 (26)
C(*a*4)—C(*b*4)	1·468 (9)	C(1)—C(3)	1·395 (25)

N(1)—Os—N(2)	89·7 (2)	C(*b*2)—C(*b*1)—C(*a*1)	106·9 (6)
N(1)—Os—O	90·3 (2)	C(*a*1)—C(*b*1)—C(11)	125·4 (7)
N(2)—Os—O	90·6 (2)	C(*b*2)—C(*b*1)—C(11)	127·7 (7)
Os—N(1)—C(*a*1)	125·8 (4)	C(*b*1)—C(*b*2)—C(*a*2)	106·9 (6)
Os—N(1)—C(*a*2)	126·9 (5)	C(*b*1)—C(*b*2)—C(21)	128·3 (7)
Os—N(2)—C(*a*3)	127·2 (6)	C(*a*2)—C(*b*2)—C(21)	124·7 (7)
Os—N(2)—C(*a*4)	126·2 (5)	C(*b*4)—C(*b*3)—C(*a*3)	107·1 (6)
C(*a*1)—N(1)—C(*a*2)	107·3 (6)	C(*b*4)—C(*b*3)—C(31)	127·0 (8)
C(*a*3)—N(2)—C(*a*4)	106·6 (6)	C(*a*3)—C(*b*3)—C(31)	125·9 (8)
N(1)—C(*a*1)—C(*b*1)	108·9 (6)	C(*b*3)—C(*b*4)—C(*a*4)	105·7 (7)
N(1)—C(*a*1)—C(*m*1)	125·3 (6)	C(*b*3)—C(*b*4)—C(41)	128·8 (7)
C(*m*1)—C(*a*1)—C(*b*1)	125·8 (7)	C(*a*4)—C(*b*4)—C(41)	125·5 (7)
N(1)—C(*a*2)—C(*b*2)	109·9 (6)	C(*a*1)—C(*m*1)—C(*a*4)	126·9 (7)
N(1)—C(*a*2)—C(*m*2)	124·1 (6)	C(*a*3)—C(*m*2)—C(*a*2)	128·1 (6)
C(*m*2)—C(*a*2)—C(*b*2)	125·9 (6)	C(12)—C(11)—C(*b*1)	113·8 (7)
N(2)—C(*a*3)—C(*b*3)	110·5 (7)	C(22)—C(21)—C(*b*2)	112·4 (7)
N(2)—C(*a*3)—C(*m*2)	123·9 (7)	C(32)—C(31)—C(*b*3)	112·6 (8)
C(*m*2)—C(*a*3)—C(*b*3)	125·6 (6)	C(42)—C(41)—C(*b*4)	113·3 (8)
N(2)—C(*a*4)—C(*b*4)	110·2 (6)	Cl—C(1)—C(2)	118 (2)
N(2)—C(*a*4)—C(*m*1)	125·5 (6)	Cl—C(1)—C(3)	118 (2)
C(*m*1)′—C(*a*4)—C(*b*4)	124·3 (6)	C(2)—C(1)—C(3)	122 (2)

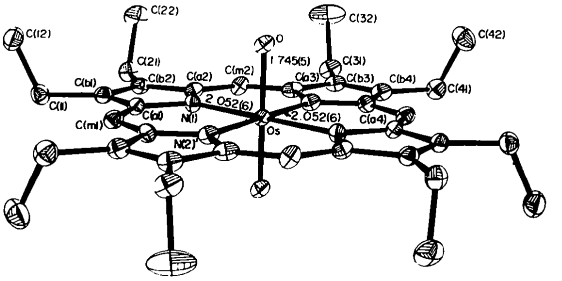

Fig. 1. *ORTEP* (Johnson, 1976) diagram and numbering scheme for all heavy atoms. 30% probability surfaces.

−15–15, −17–17, 6496 reflections measured, 5833 unique. 5721 with $I > 3\sigma(I)$ considered observed. Merging R 0·040 for 403 duplicates. Four standard reflections, 20% intensity decrease. The data were corrected for Lorentz, polarization, crystal decay, and absorption effects using the Blessing (1987) suite for data reduction. The empirical absorption was based on azimuthal (ψ) scans of eight reflections. Solved by direct methods: *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Full-matrix least squares minimized $w(\Delta F)^2$ with a total of 232 variables. The H atoms were positioned according to idealized geometry (C—H = 0·95 Å) and given isotropic B's of 1·3 times B of attached atom. All non-H atoms refined anisotropically. R = 0·048, wR = 0·049, S = 1·71. Weighting scheme $w = 1/[\sigma(F_o)]^2$. Final difference peak 3·9 e Å^{-3} near the Os atom. Final $(\Delta/\sigma)_{max}$ = 0·29. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). 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 atomic numbering of the independent half of the molecule and a perspective drawing of this molecule.

Related literature. Not many Os–porphyrin structures are reported in the literature. During our investigation a similar structure of a related compound has been reported (Che, Chung & Lai, 1988) using *meso*-tetra-*p*-tolylporphyrin (ttp). The measured osmyl Os—O distance with (oep) and (ttp) porphyrins are very close, 1·745 (5) and 1·743 (3) Å, respectively. The average Os—N distance is 2·052 (6) with the (oep) and 2·066 (4) Å with the (ttp) por-

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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 52571 (48 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Values of M—O distances* (Å) *in some metalloporphyrin complexes*

Complex	*M*—O	References
$Os^{VI}(O)_2(oep)$	1·745 (5)	This work
$Os^{VI}(O)_2(ttp)$	1·743 (3)	Che, Chung & Lai (1988)
$Mo^{IV}(O)(ttp)$	1·656 (6)	Diebold, Chevrier & Weiss (1979)
$Mo^{V}(Cl)(O)(tpp)^{a}$	1·714 (3)	Ledon & Mentzen (1978)
$[Mo^{VI}(O)(tpp)]_2O$	1·707 (3)	Johnson & Scheidt (1977)
$Ti^{IV}(O)(oepMe2)^{b}$	1·619 (4)	Dwyer, Puppe, Buchler & Scheidt (1975)
$Ti^{IV}(O)(oep)$	1·613 (5)	Guilard, Latour, Lecomte, Marchon, Protas & Ripoll (1978)
$Cr^{IV}(O)(ttp)$	1·572 (6)	Groves, Kruper, Haushalter & Butler (1982)
$Cr^{IV}(O)(tpp)$	1·62 (2)	Budge, Gatehouse, Nesbit & West (1981)
$V^{IV}(O)(oep)$	1·620 (2)	Molinaro & Ibers (1976)
$V^{IV}(O)(ttp)$	1·625 (16)	Drew, Mitchell & Scott (1984)
$V^{IV}(O)(etp)^{c}$	1·599 (6)	Drew, Mitchell & Scott (1984)
$V^{IV}(O)(dpep)^{d}$	1·582 (3)	Miller, Hambley & Taylor (1984)
$Nb^{V}(F)(O)(oep)$	1·749 (3)	Lecomte, Protas, Richard, Barbe & Guilard (1982)

(*a*) oepMe2 = α,γ-dimethyl-α,γ-dihydrooctaethylporphyrin; (*b*) tpp = tetraphenylporphyrin; (*c*) etp = etioporphyrin; (*d*) dpep = deoxophylloerythroetioporphyrin.

phyrin. In Table 3 are reported the *M*—O distances for several metalloporphyrin complexes; five-coordinate complexes have *M*—O $\simeq$ 1·6 Å and six-coordinate species have *M*—O $\simeq$ 1·7 Å.

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Structure of *catena*-Poly[bis(ethylenediamine)zinc(II)-μ-cyano-dicyanonickel(II)-μ-cyano]

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Abstract. $[NiZn(C_2H_8N_2)_2(CN)_4]$, M_r = 348·35, monoclinic, $P2_1/n$, a = 7·173 (3), b = 10·606 (4), c = 10·091 (6) Å, β = 115·91 (4)°, V = 690·53 (6) Å^3, Z = 2, D_m = 1·66 (1), D_x = 1·675 Mg m^{-3}, $F(000)$ = 356, λ(Mo $K\alpha$) = 0·71069 Å, μ = 3·1 mm^{-1}, T = 295 K, R = 0·058 for 1048 reflexions. The structure is built up of infinite electroneutral $[\text{—Zn(en)}_2\text{—NC—Ni(CN)}_2\text{—CN—}]_\infty$ chains parallel to the c axis (en = $H_2NCH_2CH_2NH_2$). The chains are significantly bent at the N(1) atom, the angle C(1)—N(1)—Zn

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