observed and used for all calculations (program system *SHELX*76; Sheldrick, 1976).

The structure was solved by routine direct methods. In the least-squares refinement $|F|$ magnitudes were used to refine an overall scale factor, atomic coordinates and anisotropic temperature factors. Final $R = 0{\cdot}035$, $wR = 0{\cdot}037$, $w^{-1} = \sigma^2(F) + 0{\cdot}0075|F|^2$ and $(\Delta/\sigma)_{\max} < 0{\cdot}001$ for 14 variables. Max. features in the final $\Delta\rho$ map (± 2 e Å^{-3}) near the heavy atoms, obviously resulting from insufficient absorption correction by ψ scans. Atomic scattering factors and f', f'' values were taken from *International Tables for X-ray Crystallography* (1974). Bond distances and angles were calculated using the program *SADIAN* (Baur & Wenninger, 1969). Final atomic coordinates and equivalent isotropic temperature factors are given in Table 1, derived bond distances in Table 2.* $ErVO_4$-II crystallizes with the scheelite ($CaWO_4$) structure (Dickinson, 1920; Zalkin & Templeton, 1964). The structure comprises VO_4 tetrahedra [V—O = 1·722 (3) Å] and ErO_8 dodecahedra [Er—O = 2·333 (3) (4×), 2·368 (3) Å (4×)].

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Er	0	$\frac{1}{4}$	$\frac{5}{8}$	0·0030 (1)
V	0	$\frac{1}{4}$	$\frac{1}{8}$	0·0035 (5)
O	0·1460 (6)	0·5050 (6)	0·2054 (3)	0·0051 (9)

Origin at centre ($\bar{1}$).

Table 2. *Interatomic distances* (Å)

VO_4 tetrahedron			ErO_8 dodecahedron		
V—O	1·722 (3)	4×	Er—O	2·333 (3)	4×
O—O	2·745 (5)	4×		2·368 (3)	4×
	2·940 (4)	2×	O—O	2·692 (4)	4×
				2·843 (4)	4×
				2·854 (4)	2×
				2·881 (5)	2×
				2·882 (5)	2×
				3·534 (4)	4×

Related literature. The zircon-type structure of the normal-pressure-phase $ErVO_4$-I has been refined by Patscheke, Fuess & Will (1968). The *P*,*T*-phase diagrams for rare-earth orthovanadates obtained by Stubican & Roy (1963*a*,*b*) using an opposed-anvil high-pressure apparatus are definitely wrong, most probably due to kinetic problems at the rather low temperatures investigated (Range & Meister, 1990).

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* Lists of structure factors, anisotropic temperature factors and bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52562 (7 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 Bis(η^6-*N*,*N*-dimethylaniline)chromium(0)

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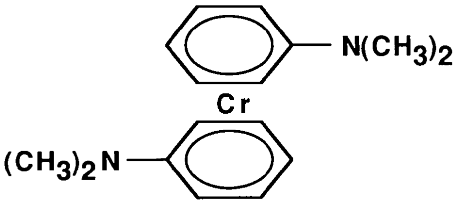
Abstract. $[Cr(C_8H_{11}N)_2]$, $M_r = 294{\cdot}36$, orthorhombic, *Pbca*, $a = 16{\cdot}871$ (8), $b = 10{\cdot}194$ (2), $c = 8{\cdot}185$ (3) Å, $V = 1407{\cdot}7$ (9) Å^3, $Z = 4$, $D_x = 1{\cdot}39$ g cm^{-3}, $\mu = 7{\cdot}806$ cm^{-1}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}7107$ Å, $F(000) = 624$, $T = 163$ K, $R = 0{\cdot}0546$ for 831 reflections [$F_o \geq 4\sigma(F_o)$]. The Cr atom lies on an inversion center.

0108-2701/90/061094-03$03.00

In the complex, the aromatic rings are parallel and the dimethyl amine groups are *anti* by symmetry. The Cr is 1·622 (2) Å from the ring centroid with Cr—C bond lengths ranging from 2·129 (6) to 2·193 (5) Å which is normal for this type of compound. The longest Cr—C bond is to the C atom bearing the dimethyl amine group. The geometry at the N atom is pyramidal and the N is 0·323 (4) Å from the plane through the three ligating C atoms.

Experimental. (1) was synthesized by the co-condensation of *N*,*N*-dimethylaniline with Cr vapor at 77 K (Graves & Lagowski, 1976). Crystals were obtained *in vacuo* by thermal gradient sublimation at 343 K. Crystals of (1) are air sensitive and were mounted in a glove bag that was purged with dry, oxygen-free nitrogen. Crystals were mounted in capillaries which were then flame sealed. The data crystal was a red plate of approximate dimensions 0·08 × 0·26 × 0·47 mm. The data were collected on a Syntex $P2_1$ diffractometer using a graphite monochromator and a Syntex LT-1 low-temperature delivery system.



(1)

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

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

	x	y	z	U_{eq}(Å^2)
Cr	0·0	0·0	0·0	0·0228 (3)
C1	−0·0620 (3)	−0·1117 (5)	0·1900 (6)	0·027 (2)
C2	−0·0043 (5)	−0·0242 (5)	0·2602 (5)	0·035 (2)
C3	0·0741 (4)	−0·0240 (5)	0·2084 (6)	0·033 (2)
C4	0·0995 (4)	−0·1099 (6)	0·0852 (7)	0·032 (2)
C5	0·0454 (4)	−0·1946 (6)	0·0143 (8)	0·030 (2)
C6	−0·0343 (4)	−0·1969 (6)	0·0640 (7)	0·026 (2)
N7	−0·1389 (3)	−0·1184 (5)	0·2505 (5)	0·036 (2)
C8	−0·1690 (5)	0·0006 (12)	0·3290 (10)	0·060 (3)
C9	−0·1967 (4)	−0·1798 (8)	0·1468 (11)	0·052 (3)

Table 2. *Bond lengths* (Å) *and angles* (°)

1	2	3	1—2	1—2—3
C1	Cr		2·193 (5)	
C2	Cr		2·145 (4)	
C3	Cr		2·129 (6)	
C4	Cr		2·136 (6)	
C5	Cr		2·130 (6)	
C6	Cr		2·154 (6)	
C2	C1	C6	1·440 (8)	116·4 (5)
C6	C1	N7	1·427 (8)	122·2 (5)
N7	C1	C2	1·391 (7)	121·2 (5)
C3	C2	C1	1·389 (10)	121·5 (5)
C4	C3	C2	1·403 (8)	120·6 (5)
C5	C4	C3	1·384 (9)	119·3 (6)
C6	C5	C4	1·405 (9)	121·4 (6)
C1	C6	C5		120·8 (5)
C8	N7	C9	1·463 (12)	112·7 (6)
C8	N7	C1		116·1 (5)
C9	N7	C1	1·437 (9)	116·4 (5)

Lattice parameters were obtained from the least-squares refinement of 15 reflections with $19·3 < 2\theta < 24·1°$. The data were collected using the ω-scan technique (2314 reflections, of which 1616 were unique, $R_{\text{int}} = 0·0276$), with a 2θ range from 4·0–55·0°, with a 1·0° ω scan at 3–6° min^{-1} ($h = -21 \rightarrow 6$, $k = 0 \rightarrow 13$, $l = 0 \rightarrow 10$). A problem with the ω drive caused the abortion of the data collection before two full equivalent sets of data could be obtained. Two reflections (020; $\bar{3}11$) were remeasured every 96 reflections to monitor instrument and crystal stability (maximum correction on I was < 2%; Henslee & Davis, 1975). The data were also corrected for Lp effects and absorption (based on crystal shape; transmission factor range 0·8045–0·9412). The data reduction procedure is described in Riley and Davis (1976). Reflections having $F_o < 4\sigma(F_o)$ were considered unobserved (785 reflections). The structure was solved by the heavy-atom method (Sheldrick, 1976) and refined by full-matrix least squares (Sheldrick, 1976) with the positional parameters of the Cr fixed by symmetry. In all, 132 parameters were refined. The non-H atoms were refined with anisotropic thermal parameters. All H-atom positions were obtained from a ΔF map and refined with isotropic thermal parameters. The function $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 1/[\sigma(F_o)]^2$ and $\sigma(F_o) = 0·5I^{-1/2}\{[\sigma(I)]^2 + (0·02I)^2\}^{1/2}$. The intensity, I, is given by $(I_{\text{peak}} - I_{\text{background}}) \times$ (scan rate); 0·02 is a factor to downweight intense reflections and to account for instrument instability and k is the correction due to Lp effects, absorption and decay. $\sigma(I)$ was estimated from counting statistics; $\sigma(I) = [(I_{\text{peak}} + I_{\text{background}})^{1/2} \times \text{(scan rate)}]$. The final $R = 0·0546$ for 831 reflections, with $wR = 0·0390$ ($R_{\text{all}} = 0·126$, $wR_{\text{all}} = 0·0438$) and goodness of fit = 1·468. The maximum $|\Delta/\sigma| = 0·14$ in the final refinement cycle and the minimum and maximum peaks in the final ΔF map were −0·93 (near Cr) and 0·41 e Å^{-3}, respectively. The scattering factors for the non-H atoms were taken from Cromer & Mann (1968), with the anomalous-dispersion corrections taken from Cromer & Liberman (1970). The scattering factors for the H atoms were obtained from Stewart, Davidson & Simpson (1965). Values used to calculate the linear absorption coefficient are from *International Tables for X-ray Crystallography* (1974). Figures were generated using *SHELXTL-PLUS* (Sheldrick, 1987). The positional and thermal parameters for non-H atoms are listed in Table 1, while the bond lengths and angles for the non-H

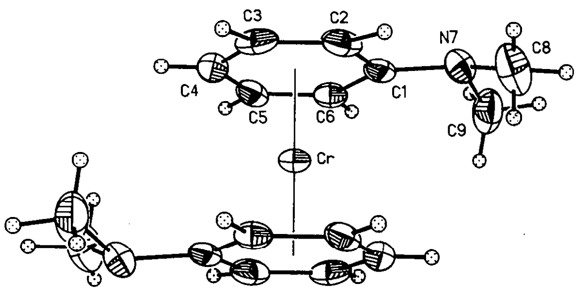


Fig. 1. View of the complex (1) showing the atomic labeling scheme. The non-H atoms are scaled to the 50% probability level while the H atoms are drawn to an arbitrary size. The Cr atom lies on an inversion center. The pyramidal geometry at the N atom is apparent.

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. $[Os(C_{36}H_{44}N_4)O_2].C_6H_5Cl$, $M_r = 867{\cdot}53$, triclinic, $P\bar{1}$, $a = 8{\cdot}177\ (1)$, $b = 10{\cdot}668\ (1)$, $c = 11{\cdot}791\ (1)$ Å, $\alpha = 73{\cdot}91\ (1)$, $\beta = 83{\cdot}18\ (1)$, $\gamma = 75{\cdot}07\ (1)°$, $V = 953{\cdot}6$ Å^3, $Z = 1$, $D_x = 1{\cdot}51$ g cm^{-3}, $D_m = 1{\cdot}50$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 34{\cdot}5$ cm^{-1}, $F(000) = 438$, $T = 293$ K, $R = 0{\cdot}048$ 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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