Table 1. *Positional parameters and equivalent isotropic thermal parameters for* PdS

	Site symmetry	x	y	z	B_{eq}(Å^2)
Pd(1)	$\bar{4}$	0	0	$\frac{1}{4}$	0·35 (1)
Pd(2)	$2/m$	$\frac{1}{2}$	0	0	0·33 (1)
Pd(3)	m	0·257132 (59)	0·467792 (55)	0	0·33 (1)
S(1)	1	0·30754 (13)	0·19361 (14)	0·22904 (13)	0·37 (1)

Gaskell (1937) obtained: Pd(3) 0·25 (1), 0·48 (1), 0; S(1) 0·32 (1), 0·19 (1), 0·23 (1), for x, y, z respectively.

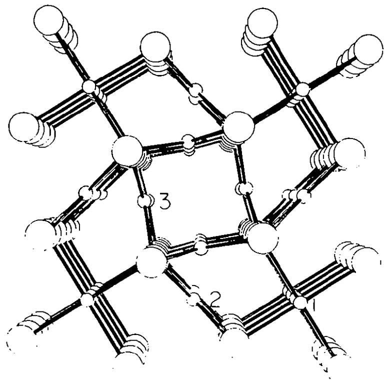

Fig. 1. Perspective view of PdS along [001]. Pd atoms are small open circles; S atoms are large open circles.

Table 2. *Selected bond distances* (Å) *and angles* (°) *for* PdS

Pd(1)–4S(1)	2·341 (1)	S(1)–Pd(1)–S(1)	173·21 (4)
Pd(2)–4S(1)	2·318 (1)	S(1)–Pd(1)–S(1)	90·20 (1)
Pd(3)–2S(1)	2·337 (1)	S(1)–Pd(2)–S(1)	81·57 (5)
Pd(3)–2S(1)	2·346 (1)	S(1)–Pd(3)–S(1)	89·65 (3)
Pd(3)–Pd(3)	3·150 (1)	S(1)–Pd(3)–S(1)	100·08 (5)
Pd(1)–2Pd(1)	3·306 (1)	S(1)–Pd(3)–S(1)	169·46 (4)
Pd(1)–4Pd(2)	3·615 (1)	Pd(2)–S(1)–Pd(3)	93·19 (4)
Pd(1)–4Pd(3)	3·809 (1)	Pd(2)–S(1)–Pd(1)	101·77 (3)
Pd(2)–2Pd(3)	3·389 (1)	Pd(2)–S(1)–Pd(3)	105·34 (4)
Pd(2)–4Pd(3)	3·702 (1)	Pd(1)–S(1)–Pd(3)	108·73 (3)
Pd(3)–4Pd(3)	3·986 (1)	Pd(3)–S(1)–Pd(3)	116·66 (4)
Pd(1)–4Pd(3)	4·144 (1)	Pd(3)–S(1)–Pd(1)	124·73 (4)
S(1)–S(1)	3·029 (2)		
S(1)–2S(1)	3·302 (2)		
S(1)–2S(1)	3·316 (2)		
S(1)–S(1)	3·510 (2)		

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Structure of a Methoxo-Bridged Dichromium Complex

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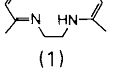
Abstract. Di-μ-methoxo-bis[(7-amino-4-methyl-5-azahept-3-en-2-onato-*O,N,N'*)aquachromium(III)] diperchlorate, $[Cr_2(CH_3O)_2(C_7H_{13}N_2O)_2(H_2O)_2]$-$(ClO_4)_2$, $M_r = 683{\cdot}4$, $P\bar{1}$, $a = 8{\cdot}087$ (2), $b = 8{\cdot}950$ (2), $c = 11{\cdot}018$ (3) Å, $\alpha = 68{\cdot}92$ (2), $\beta = 73{\cdot}59$ (2), $\gamma = 83{\cdot}61$ (2)°, $V = 713{\cdot}75$ Å^3, $Z = 1$, $D_x = 1{\cdot}590$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}00$ mm^{-1}, $F(000) = 354$, $T = 291$ K, $R = 0{\cdot}045$ for 1533 unique observed reflections. The cation has crystallographic $\bar{1}$ symmetry, with a strictly planar central Cr_2O_2 ring and rather unsymmetrical bridging geometry [Cr—O = 1·976 (3) and 1·940 (3) Å, Cr—O—Cr = 100·8 (1)°]. Coordination of Cr is essentially octahedral. The six-membered chelate ring is almost planar [Cr 0·329 (1) Å from the mean plane of the other atoms, r.m.s. $\Delta = 0{\cdot}015$ (6) Å]; the five-membered chelate ring is strongly non-planar to allow for staggering of the H atom substituents.

Experimental. Compound obtained by reaction of $CrCl_2$ with the macrocyclic base 5,7,12,14-tetramethyl-1,4,8,11-tetraazacyclotetradeca-4,6,11,13-tetraene (1) in aqueous methanol, followed by air oxidation and perchlorate ion-exchange (Ramasami, 1984). The macrocyclic ligand is hydrolyzed under the reaction conditions.



(1)

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$)

$U_{eq} = \frac{1}{3}$(trace of the orthogonalized U_{ij} matrix).

	x	y	z	U_{eq}
Cr	5900·3 (8)	6109·4 (8)	3620·7 (6)	33·5 (3)
O(1)	3602 (3)	5724 (3)	4919 (3)	36 (1)
C(1)	1997 (6)	6005 (7)	4582 (5)	64 (3)
O(2)	4999 (4)	7930 (4)	2220 (3)	49 (1)
N(1)	5539 (5)	4515 (5)	2746 (4)	45 (2)
C(2)	7266 (7)	3840 (6)	2317 (5)	60 (3)
C(3)	8545 (6)	5178 (6)	1575 (5)	55 (2)
N(2)	8236 (5)	6300 (5)	2334 (3)	41 (2)
C(4)	9326 (6)	7443 (6)	1994 (4)	46 (2)
C(5)	10915 (7)	7659 (8)	822 (6)	73 (3)
C(6)	9083 (6)	8546 (6)	2685 (5)	50 (2)
C(7)	7789 (6)	8554 (6)	3796 (4)	45 (2)
C(8)	7821 (8)	9680 (6)	4512 (6)	66 (3)
O(3)	6476 (4)	7597 (3)	4339 (3)	43 (1)
Cl	6427 (2)	7928 (1)	−1301 (1)	54 (1)
O(4)	5424 (5)	8910 (5)	−2183 (4)	79 (2)
O(5)	7738 (9)	7245 (10)	−1962 (5)	205 (5)
O(6)	6952 (8)	8796 (6)	−642 (5)	121 (3)
O(7)	5386 (8)	6731 (7)	−277 (6)	153 (4)

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

Cr—O(1)	1·976 (3)	Cr—O(2)	2·032 (3)
Cr—N(1)	2·072 (5)	Cr—N(2)	1·998 (3)
Cr—O(3)	1·930 (4)	Cr—O(1′)	1·940 (3)
O(1)—C(1)	1·421 (6)	N(1)—C(2)	1·478 (6)
C(2)—C(3)	1·506 (7)	C(3)—N(2)	1·483 (8)
N(2)—C(4)	1·304 (7)	C(4)—C(5)	1·515 (6)
C(4)—C(6)	1·416 (9)	C(6)—C(7)	1·369 (6)
C(7)—C(8)	1·493 (9)	C(7)—O(3)	1·301 (6)
Cl—O(4)	1·430 (4)	Cl—O(5)	1·330 (8)
Cl—O(6)	1·401 (7)	Cl—O(7)	1·387 (5)

O(1)—Cr—O(2)	92·5 (1)	O(1)—Cr—N(1)	93·7 (1)
O(2)—Cr—N(1)	90·1 (2)	O(1)—Cr—N(2)	175·1 (2)
O(2)—Cr—N(2)	90·6 (1)	N(1)—Cr—N(2)	82·6 (2)
O(1)—Cr—O(3)	91·9 (1)	O(2)—Cr—O(3)	90·8 (1)
N(1)—Cr—O(3)	174·3 (1)	N(2)—Cr—O(3)	91·8 (2)
O(1)—Cr—O(1′)	79·2 (1)	O(2)—Cr—O(1′)	171·3 (1)
N(1)—Cr—O(1′)	87·7 (1)	N(2)—Cr—O(1′)	97·5 (1)
O(3)—Cr—O(1′)	92·2 (1)	Cr—O(1)—C(1)	125·7 (3)
Cr—O(1)—Cr′	100·8 (1)	C(1)—O(1)—Cr′	124·3 (3)
Cr—N(1)—C(2)	105·5 (4)	N(1)—C(2)—C(3)	109·4 (4)
C(2)—C(3)—N(2)	108·5 (4)	Cr—N(2)—C(3)	113·8 (3)
Cr—N(2)—C(4)	125·3 (4)	C(3)—N(2)—C(4)	120·1 (4)
N(2)—C(4)—C(5)	121·1 (5)	N(2)—C(4)—C(6)	122·2 (4)
C(5)—C(4)—C(6)	116·7 (5)	C(4)—C(6)—C(7)	127·2 (5)
C(6)—C(7)—C(8)	120·9 (5)	C(6)—C(7)—O(3)	124·8 (5)
C(8)—C(7)—O(3)	114·3 (4)	Cr—O(3)—C(7)	126·1 (3)
O(4)—Cl—O(5)	110·7 (3)	O(4)—Cl—O(6)	111·1 (3)
O(5)—Cl—O(6)	112·8 (5)	O(4)—Cl—O(7)	108·7 (3)
O(5)—Cl—O(7)	108·5 (4)	O(6)—Cl—O(7)	104·8 (4)

The prime denotes a symmetry-related atom at $1-x$, $1-y$, $1-z$.

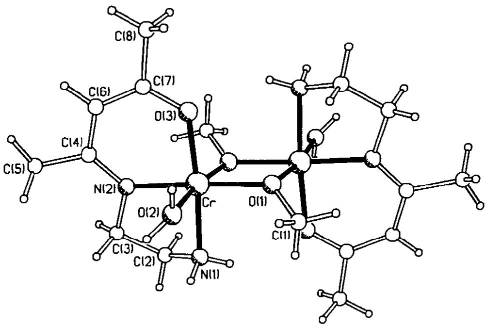


Fig. 1. The cation, showing the numbering scheme.

Crystal size 0·05 × 0·20 × 0·25 mm [dominant faces ±(001)], Stoe–Siemens AED diffractometer, cell parameters from 2θ values of 30 reflections ($20 < 2\theta < 25°$), 1869 reflection intensities measured by ω/θ scan, on-line profile fitting (Clegg, 1981), 2θ 7→45°, h 0→8, k −9→9, l −11→11, no absorption or extinction corrections, no significant intensity variation for three standard reflections. 1869 unique reflections, 336 with $F < 4\sigma(F)$ ignored as 'unobserved'.

Structure solved by automatic multisolution direct methods, refined by blocked-cascade least squares to minimize $\sum w\Delta^2$, $w^{-1} = \sigma^2(F) + 0{\cdot}00045F^2$. Anisotropic thermal parameters for all non-H atoms, H atoms in calculated positions with C—H = 0·96 Å, O—H = N—H = 0·87 Å, H—X—H = 109·5°, rigid methyl groups with freely refined orientation, $U(\mathrm{H}) = 1{\cdot}2\ U_{eq}(X)$. $R = 0{\cdot}045$, $wR = 0{\cdot}055$, slope of normal probability plot = 1·52, $(\Delta/\sigma)_{\max} = 0{\cdot}015$, $(\Delta/\sigma)_{\mathrm{mean}} = 0{\cdot}003$, $(\Delta\rho)_{\max} = 0{\cdot}49$ e Å^{-3} close to $\mathrm{ClO_4^-}$ anion showing high thermal motion (possibly disordered), $(\Delta\rho)_{\min} = -0{\cdot}32$ e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). Programs used: *SHELXTL* (Sheldrick, 1978). Table 1* gives the atom parameters and Table 2 bond lengths and angles. Fig. 1 shows the cation and numbering scheme.

Related literature. For a description of the preparation and use of the macrocyclic ligand, see Truex & Holm (1972), and for the structure of one of its complexes, see Kim & Everett (1981).

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[*] Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42433 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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