Related literature. Crystal structures have been reported for the related products bis(8-methoxy-1-naphthyl)methylplatinum(IV) iodide (Wehman, van Koten, Knaap, Ossor, Pfeffer & Spek, 1988) and a-chloro-b-dichloromethylstannio[dc,ef-bis(8-dimethylamino-1-naphthyl)]platinum(IV) (Smeets, Spek, van Beek & van Koten, 1992). Details of the chemistry will be published elsewhere (van Beek, Wehman-Ooyevaar, Grove, Smeets, Spek & van Koten, 1992).

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Bis[2-(2-hydroxyethyl)piperazinium] Hexachloroosmate(IV) Dichloride

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Abstract. $[C_6H_{15}N_2O]_2[Os^{IV}Cl_6]Cl_2$, $M_r = 738.2$, monoclinic, $P2_1/a$, $a = 12.252 (3)$, $b = 9.176 (2)$, $c = 11.974 (8)$ Å, $\beta = 115.06 (3)°$, $V = 1219 (1)$ Å^3, $Z = 2$, $D_x = 2.01$ Mg m^{-3}, $\lambda(Mo\ K\alpha) = 0.71069$ Å, $\mu = 6.14$ mm^{-1}, $F(000) = 720$, $T = 298$ K, $R = 0.0679$ for 2159 observed reflections. The formula unit consists of the hexachloroosmate(IV) anion, two 2-(2-hydroxyethyl)piperazinium(2+) cations and two chloride ions. The complex is centrosymmetric with the Os atom at the origin. Weak intermolecular hydrogen bonds link the cations with the chloride ions.

Experimental. The complex was obtained under vigorous stirring and heating (363 K) from 1 g K$_2$-(Os^{IV}Cl$_6$) dissolved in 50 cm^3 6 M HCl and the corresponding amount of the ligand in 25 cm^3 of a 1:1 mixture of ethanol and 6 M HCl. The stirring and heating continued until the solution reached one-third of its initial volume and then was left to form bright prismatic crystals at room temperature. Intensity data were collected from a crystal approximately 0.05 × 0.05 × 0.30 mm. The cell parameters were determined by least squares from 25 automatically centred reflections in the range $10 < 2\theta < 25°$. 3063 independent reflections were measured with ω scans in the range $4 < 2\theta < 56°$ on a Philips PW 1100

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diffractometer with graphite-monochromated radiation. Three standard reflections (600, 060, 006) measured after every 80, showed only random deviations from the mean intensity. Lorentz and polarization corrections were applied. From 2928 unique reflections in the index range $h -15 \rightarrow 14$, $k\,0 \rightarrow 12$, $l\,0 \rightarrow 15$ only 2159 $[I > 2.5\sigma(I)]$ were considered observed and included in the refinement. The structure was solved with $MULTAN80$ (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and refined by least squares with $SHELX76$ (Sheldrick, 1976). The function $\Sigma(\Delta F)^2$ was minimized with unit weights. At convergence with isotropic displacement parameters an absorption correction was applied empirically with the program $DIFABS$ (Walker & Stuart, 1983). Anisotropic displacement parameters were then used for non-H atoms. The H atoms bonded to C and N atoms were introduced in calculated atomic positions and refined with an overall isotropic displacement parameter. Final values are $R = 0.0679$, $wR = 0.0679$ and $S = 7.05$ for 125 parameters. Atomic scattering factors were taken from International Tables for X-ray Crystallography (1974, Vol. IV) and the anomalous-scattering factors from Cromer & Liberman (1970). The max. and min. electron densities in the final $\Delta\rho$ map are 3.16 and -5.11 e Å^{-3}, both in the vicinity of the Os atom, max. $\Delta/\sigma = 0.032$ for non-H atoms. Table 1* gives the final atomic parameters with their B_{eq} values (Willis & Pryor, 1975). Fig. 1 (Keller, 1988) shows a view of the molecule with the atom numbering. Molecular geometry calculations were performed using $PARST$ (Nardelli, 1983). Fig. 2 shows a stereoscopic view of the molecular packing.

The infrared spectra of the pure ligand and the complex were obtained in Nujol mulls with a 657 Perkin–Elmer IR spectrophotometer in the 4000–250 cm^{-1} range.

Related literature. In this paper we report the structure of the new Os^{IV} complex salt $(LH_2)_2(OsCl_6)Cl_2$, where L = 2-piperazinylethanol. This complex was prepared as part of a programme directed towards the synthesis of new $(Os^{IV}Cl_6)(LH_2)_n$ $(n = 1,2)$ complex salts (where L = acridine, quinoline, piperazine and pyrazolone derivatives) (Craciunescu, Doadrio & Ghirvu, 1975, 1976) in order to assess their antitumor and antiparasitic properties (Doadrio,

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* List of structure factors, anisotropic displacement parameters, calculated H-atom coordinates, intermolecular distances, bond lengths and angles of the piperazinium cations, torsion angles, least-squares planes and IR spectral data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54902 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0247]

Table 1. *Atomic coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses and B_{eq} values* (Å^2) (*defined according to Willis & Pryor*, 1975)

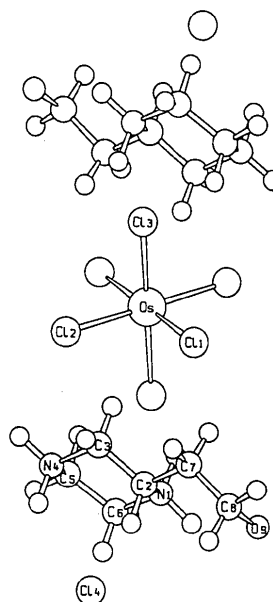|	x	y	z	B_{eq}
Os	0	0	0	1.26
Cl(1)	1023 (4)	2169 (5)	108 (5)	2.25
Cl(2)	−1557 (4)	1269 (6)	210 (5)	2.37
Cl(3)	−1002 (4)	213 (7)	−2151 (4)	2.61
N(1)	1694 (16)	3643 (22)	3274 (18)	2.73
C(2)	1077 (13)	4995 (24)	2616 (12)	1.18
C(3)	−221 (18)	4672 (21)	1756 (19)	2.06
N(4)	−857 (19)	4021 (24)	2506 (20)	3.42
C(5)	−256 (17)	2650 (21)	3164 (17)	1.82
C(6)	1062 (18)	2968 (27)	3974 (18)	2.43
C(7)	1691 (19)	5652 (23)	1882 (18)	2.26
C(8)	2863 (18)	6400 (24)	2660 (19)	2.53
O(9)	3797 (13)	5464 (17)	3452 (15)	3.07
Cl(4)	819 (4)	7038 (6)	4546 (4)	2.35

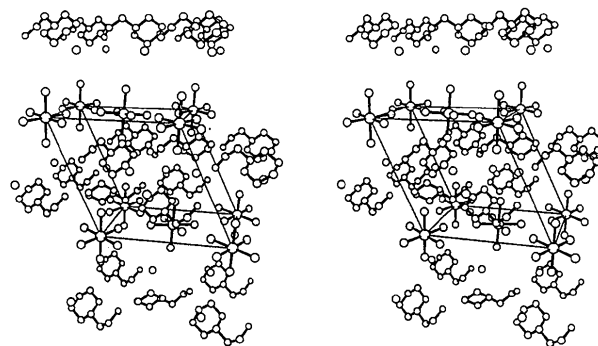Fig. 1. View of the molecule drawn with $SCHAKAL$ (Keller, 1988).

Fig. 2. Stereoscopic view of the molecular packing drawn with $SCHAKAL$ (Keller, 1988).

Table 2. *Selected bond lengths (Å) and angles (°) with e.s.d.'s in parentheses*

Cl(1)—Os	2.326 (4)	Cl(2)—Os—Cl(1)	90.7 (2)
Cl(2)—Os	2.338 (5)	Cl(3)—Os—Cl(1)	90.5 (2)
Cl(3)—Os	2.346 (4)	Cl(3)—Os—Cl(2)	90.3 (2)
C(2)—N(1)	1.49 (3)	C(6)—N(1)—C(2)	112 (2)
C(6)—N(1)	1.50 (3)	C(3)—C(2)—N(1)	110 (2)
C(3)—C(2)	1.51 (3)	C(7)—C(2)—N(1)	112 (2)
C(7)—C(2)	1.50 (3)	C(7)—C(2)—C(3)	109 (2)
N(4)—C(3)	1.54 (3)	N(4)—C(3)—C(2)	109 (2)
C(5)—N(4)	1.50 (3)	C(5)—N(4)—C(3)	112 (2)
C(6)—C(5)	1.52 (3)	C(6)—C(5)—N(4)	109 (2)
C(8)—C(7)	1.51 (3)	C(5)—C(6)—N(1)	113 (2)
O(9)—C(8)	1.42 (3)	C(8)—C(7)—C(2)	114 (2)
		O(9)—C(8)—C(7)	115 (2)

Craciunescu & Ghirvu, 1980; Anadon, Craciunescu, Larrañaga, Sanz & Doadrio, 1977; Ruiz Perez, 1983, and references therein).

The bond lengths and angles given in Table 2 show that the $(Os^{IV}Cl_6)^{2-}$ anion is nearly octahedral. The distances Os^{IV}—Cl in the range 2.326–2.346 (5) Å are shorter than previously reported for Os^{III}—Cl (Blake, Heath, Smith, Yellowlees & Sharp, 1988) and Os^{II}—Cl bonds (Chakravarty, Cotton & Tocher, 1985; Mukhopadhyay & Ray, 1987), but almost identical to those reported by Robinson, Hinckley, Matusz & Kibala (1988) for the same anion. The shortest Os—Os $(\frac{1}{2} + x, \frac{1}{2} - y, z)$ distance is 7.654 (1) Å.

The deposited distances, angles and torsion angles of the piperazinium cation are similar to those reported by Sangin & Brisse (1984). Intermolecular distances and the deposited IR data support the existence of a weak intermolecular hydrogen bond between the cation and the chloride ions (Pimentel & McClellan, 1960; Harlow, Wells, Watt & Simonsen, 1974; Willet, Haugen, Lebsack & Morrey, 1974).

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Redetermination of the Structure of (Acetonitrile)chlorocopper(I) [(H₃CCN)CuCl] at 178 K

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Abstract. [Cu(C₂H₃N)Cl], $M_r = 140.0$, orthorhombic, *Pnma*, $a = 8.545$ (3), $b = 3.8691$ (10), $c = 12.692$ (4) Å, $V = 419.6$ (3) Å³, $Z = 4$, $D_x = 2.217$ Mg m⁻³, $\lambda(Mo\ K\alpha) = 0.71069$ Å, $\mu = 5.6$ mm⁻¹, $F(000) = 272$, $T = 178$ K, $R = 0.022$ for 491 reflections. All non-H atoms lie in the mirror planes at $y = 0.25$. Although the Cu atom displays slightly larger displacement parameters than the Cl atom [U_{eq} 0.0317 (2) and 0.0225 (2) Å² for Cu and Cl], there seems no cogent reason to prefer the non-centrosymmetric space group *Pna*2₁ with the Cu atom displaced from the mirror plane. Zigzag chains